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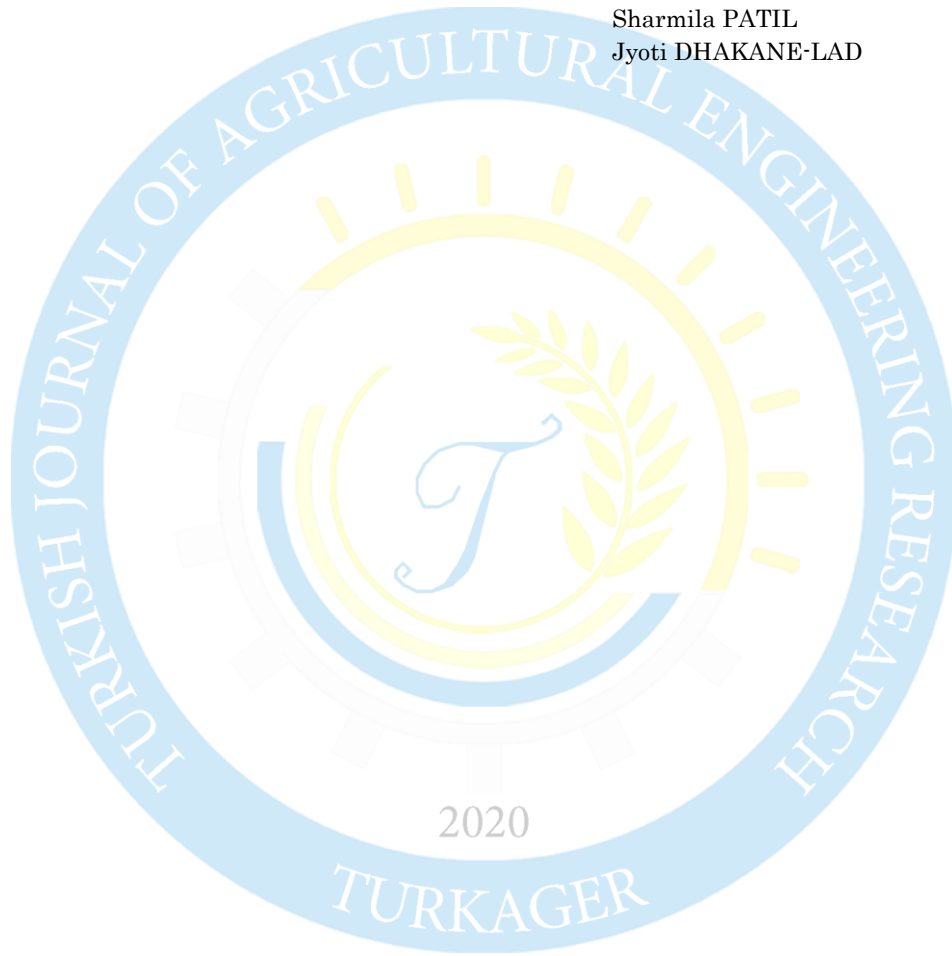
# Turkish Journal of Agricultural Engineering Research (TURKAGER)

Volume 3,  
Issue 1,  
June 30, 2022

No	Articles	Author/s	Pages
1	Kıvırcık Marulda ( <i>Lactuca sativa</i> L. var. <i>crispa</i> ) Borlu Gübre ve Humik Asit Uygulamalarının Bazı Bitki Özelliklerine Etkisi <i>Effect of Boron Fertilizer and Humic Acid Applications on Some Plant Characteristics of Curly Lettuce (Lactuca sativa L. var. crispa)</i>	Özge KURT <b><u>Atnan UĞUR</u></b>	1-14
2	Comparative Investigation of n-Hexane and Ethanol Solvents Used in Eleais guinesis Kernel Oil Extraction and Optimization via Two Computational Modelling	<b><u>Opololaoluwa Oladimarum IJAOLA</u></b>	15-30
3	Optimization of Some Physical and Functional Properties of Extruded Soybean Crud Residue-Base Floating Fish Feed	Saheed Aiola OLAOYE <b><u>Olanrewaju Temitope OWOSENI</u></b> Ayoola Patrick OLALUSI	31-50
4	Farmers' Participation in Watershed Management in Sodo Zuria District of Southern Ethiopia	Daniel BALTA <b><u>Marisennayya SENAPATHY</u></b> Marisennayya PANDIKUMAR Chinaza Godswill AWUCHI	51-63
5	Characterization and Classification of Soil on Varying Lithology in Okigwe Imo Southern Eastern, Nigeria	Patricia Akunna ORIAKU <b><u>Christopher Ikechukwu OBINECHE</u></b>	64-77
6	Adana İlinde Satışa Sunulan Yaş ve Kuru Yolum Yapılmış Tavuk Etlerinin Mikrobiyolojik Yönden Karşılaştırılması <i>Microbiological Comparison of Wet and Dry Plucked Chicken Meat Sold in Adana Province</i>	<b><u>Fatma YENİLMEZ</u></b>	78-85
7	Development of Olive Harvesting Machine by Shaking	<b><u>Mohamed Ibrahim GHONIMY</u></b> Samy Mohamed YOUNIS El-Sayed Youssif GHONIEM	86-102
8	Aljinat Bazlı Yenilebilir Kaplama Uygulamalarının Minimal İşlem Görmüş Kavun Dilimlerinin Fizikokimyasal Kalitesine Etkileri <i>Effects of Alginate Based Edible Coating Applications on Physicochemical Quality of Minimal Processed Melon Slices</i>	Bengü DEMİR <b><u>Aslıhan DEMİRDÖVEN</u></b>	103-118
9	Fuel Properties of Sandbox ( <i>Hura crepitans</i> Linn.) Methyl Ester and its Blends	<b><u>David Nwabueze ONWE</u></b> Adeleke Isaac BAMGBOYE	119-130
10	Evaluation of Aqua-Crop Model using Onion Crop under Deficit Irrigation and Mulch in Semi-arid Nigeria	<b><u>Nura Jafar SHANONO</u></b> Baba Saleh ABBA Nuraddeen Mukhtar NASIDI	131-145
11	Diyabetik Bal Kabağı ( <i>Cucurbita moschata</i> Duch.) Marmelatı Üretimi <i>Production of Diabetic Pumpkin (Cucurbita moschata Duch.) Marmalade</i>	<b><u>Ali CİNGÖZ</u></b> Ashhan DEMİRDÖVEN	146-156
12	Motorbike Powered Trailer for Transporting Poultry Birds	<b><u>Ayoola Olawole JONGBO</u></b> Timothy Oluwatimilehin ADELAJA	157-169



No	Articles	Author/s	Pages
13	Karnabaharın ( <i>Brassica oleracea</i> L.) Model ve Kantitatif Renk Kalite Özelliklerine Kurutma İşlemlerinin Etkisi <i>The Effect of Drying Processes on Model and Quantitative Color Quality Characteristics of Cauliflower (Brassica oleracea L.)</i>	<b>Burcu AKSÜT</b> Hakan POLATCI	170-180
14	Effect of Fermentation on Drying Characteristics of Three Varieties of Trifoliate Yam	Obiageli UGWUANYI NNADI Brendan Ekete EJE <b>Patrick Ejike IDE</b>	181-193
<b>Review Article</b>			
15	Improving the Nutritional and Functional Properties of Pearl Millet Pasta: A Review	Kirti JALGAONKAR <b>Manoj Kumar MAHAWAR</b> Sharmila PATIL Jyoti DHAKANE-LAD	194-203





## Effect of Boron Fertilizer and Humic Acid Applications on Some Plant Characteristics of Curly Lettuce (*Lactuca sativa* L. var. *crispa*)

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### ABSTRACT

This study determined the effects of boron fertilizer and humic acid on yield and plant properties were determined in curly lettuce varieties under unheated plastic greenhouse conditions. The plants were grown in a mixture of peat: perlite prepared at a ratio of 3:1. Pots of 50x18x16 cm were used as planting pots. The study was set up in a randomized plot design with 3 replications and each pot was considered as application replication. In the study, the seedlings of Firtina, Olenka and Campania curly lettuce varieties were used as plant material. Boron fertilizer was tested at doses of 0, 50, 100, 200 and 400 g da<sup>-1</sup>, and humic acid at 0 and 0.2% doses. Yield (g m<sup>-2</sup>), leaf width (cm), leaf length (cm), number of leaves (number/plant) and root length (cm) were determined in lettuce plants. Boron fertilizer at a dose of 50 g da<sup>-1</sup> increased plant yield by approximately 4%. With boron fertilizer applications, while the number of leaves decreased, the root length values increased. While humic acid applications increased plant yield and leaf length values, they had a decreasing effect on leaf number and root length values. The response of cultivars to boron fertilizer applications was different.

#### RESEARCH ARTICLE

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## Kıvırcık Marulda (*Lactuca sativa* L. var. *crispa*) Borlu Gübre ve Humik Asit Uygulamalarının Bazı Bitki Özelliklerine Etkisi

### ÖZET

Bu araştırmada ısıtmasız plastik sera koşullarında kıvırcık marul çeşitlerinde borlu gübre ve humik asidin verim ve bitki özelliklerine etkisi belirlenmiştir. Bitkiler 3:1 oranında hazırlanan torf:perlit karışımında yetiştirilmiştir. Ekim kabı olarak 50x18x16 cm boyutlarındaki saksılar kullanılmıştır. Çalışma tesadüf parselleri deneme deseninde 3 tekerrürlü olarak kurulmuş ve her bir saksı uygulama tekerrürü olarak kabul edilmiştir. Çalışmada bitkisel materyal olarak Fırtına, Olenka ve Campania kıvırcık marul çeşitleri kullanılmıştır. Borlu gübre 0, 50, 100, 200 ve 400 g da<sup>-1</sup> dozlarında, humik asit ise 0 ve %0.2 dozlarında denenmiştir. Marul bitkilerinde verim (g m<sup>-2</sup>), kök uzunluğu (cm) ve yaprak sayısı (adet/bitki) değerleri ile yaprak uzunluğu (cm) ve yaprak eni (cm) değerleri belirlenmiştir. Borlu gübre 50 g da<sup>-1</sup> dozunda uygulandığında yaklaşık %4 oranında bitki veriminde artış sağlamıştır. Borlu gübre uygulamaları ile bitkilerde yaprak sayısı azalırken, kök uzunluğu değeri ise artış göstermiştir. Humik asit uygulamaları bitki verimini ve yaprak uzunluğu değerlerini artırırken, yaprak sayısı ve kök uzunluğu değerlerine azaltıcı yönde etki etmiştir. Borlu gübre uygulamalarına çeşitlerin tepkisi farklı bulunmuştur.

#### ARAŞTIRMA MAKALESİ

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#### Anahtar Kelimeler:

- Kıvırcık marul,
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### GİRİŞ

Sebzecilik, toplayıcı toplumdan günümüze insanoğlunun beslenme amacıyla yaptığı en eski faaliyetlerdendir. Dünya nüfusunun hızlı artışı ve sebzelerin beslenme ve sağlık üzerine olan etkilerinin daha iyi bilinmeye başlaması, sebze üretimini ekonomik açıdan önemli boyutlara taşımıştır. Sebzeler besin değeri, lif, sekonder metabolitler, karotenoidler, flavanoidler, glukosinolatlar, vitamin C ve mineral madde içerikleri açısından sağlıklı gıdalar olarak görülmektedir. Marul kolay yetişmesi, gevrek yapısı, taze görünümü, aroması, tadı, mineral ve fitokimyasal içerikleri ile salata ve garnitürlerin aranan sebzesidir (Ouzounidou ve ark., 2013). Bahçe ürünlerinin raf ömrü, genetik ve çevresel etmenleri içeren hasat öncesi morfolojik ve fizyolojik özelliklere ve hasat sonrası işlemlere bağlıdır (Zou ve ark., 2019; Koukounaras ve ark., 2020). Raf ömrü yetiştirme sırasındaki çevre koşullarından büyük ölçüde etkilenir. Marul geniş yapraklı olması ve sulu dokusu nedeniyle hasat sonrası raf ömrü sınırlıdır. Marulda hasat sonrası kayıplar önlenerek önemli bir ticari kazanç elde edilebilir (Kongwong ve ark., 2019).

Yakın bir zamana kadar sebze üretimi dış ortamda öngörülemez iklim şartlarında böcek, yabancı ot, hastalık ve zararlılar gibi çevresel stres faktörlerinin etkisi altında yılın belirli dönemlerinde yapılmaktaydı (Paz ve ark., 2019). Günümüzde bu etmenlerinin kısmen kontrol edilebildiği veya edilmeye çalışıldığı örtüaltı sistemlerinde sebzecilik yapılmaktadır. Son yıllarda ürünün kalitesi ile birlikte girdileri düşürerek maliyetleri azaltan sürdürülebilir üretim sistemleri daha çok dikkate alınmaktadır

(Bulgari ve ark., 2015). Sebze üretiminde bitkilerin büyümesi üzerine en etkili faktörlerin başında gübreleme gelmektedir. Gübreleme bitkisel üretimde en çok araştırılan konulardan birisi olmuştur. Gübreleme ile ürünlerde sadece hasat dönemindeki kalite değil pazar istekleri doğrultusunda hasat sonrası raf ömrü ve depolama performansları da önem arz etmektedir. Kaliteli ürünlerin hasat sonrası performansları da yüksek olmaktadır. Bitkisel üretimin en temel hedeflerden birisi bitki büyümesini ve üretkenliğini etkileyen kök bölgesinde fiziksel, kimyasal ve biyolojik olarak bitkinin istediği bir ortamın sağlanmasıdır (Alagöz ve ark., 2006; Gruda ve ark., 2013). Organik madde toprakta katyonların yıkanmasını önleme, fonksiyonel grupları ile şelatlama yapma, biyolojik aktiviteyi artırma, strüktürü geliştirme, su tutma kapasitesini artırma gibi fiziksel ve kimyasal olumlu etkileri bulunmaktadır (Frank ve Roeth, 1996). Türkiye topraklarının büyük bir bölümünün organik madde içerikleri büyük oranda düşük seviyelerdedir (Eyüpoğlu ve ark., 1995). Topraklarda organik madde içeriğini artırmanın en kolay yolu humik maddelerin kullanılmasıdır (Chen ve Aviad, 1990; Unlu ve ark., 2011). Humik asitlerin de içerisinde yer aldığı biostimulantlar bitkilerde farklı fizyolojik süreçleri düzenlemekte ve abiyotik streslere karşı toleransı artırmaktadır (Bulgari ve ark., 2015). Tarımsal üretimde en yaygın kullanılan biostimulantlar deniz ekstraktı ve humik asitlerdir. Biyostimulantlar bitkilerde büyüme, çiçeklenme, meyve tutumu, verim ve gübre kullanım etkinliğini artırmakta ve abiyotik stresörlere karşı koruma sağlamaktadır (Rouphael ve Colla, 2020). Humik asitler toprakta bulunan besinleri şelatlama ve pH'ı tamponlama özelliğinde olan karmaşık makromoleküllü organik maddelerdir. İçermiş olduğu mineral maddeler ve fizikokimyasal etkileri nedeniyle besin alımı, toprak havalanması, toprak agregasyonu, bitki büyümesi ve kök gelişimine olumlu katkıları bulunmaktadır (Khaled ve Fawy, 2011). Humik maddeler bitkilerde karbonhidrat içeriğini azaltırken, nitrat redüktaz ve fenilalanin amonyak liyaz aktivitesinin indükleyerek toplam protein içeriği ve verimi artırmaktadır (Hernandez ve ark., 2015). Besin maddelerinin alınımı ve kök gelişimine olan etkileri ile doğrudan bitki gelişimini artırır (Lobartini ve ark., 1997). Humik asit marul bitkilerinde topraktan uygulama, sulama suyu şeklinde uygulama ve yapraktan uygulamada bitki azot ve potasyum içeriğini artırırken fosfor içeriği üzerine etkisiz bulunmuştur (Taha ve ark., 2016). Bununla birlikte yüksek oranda humik madde içeren kompost uygulamaları bitki kök bölgesinde arbusküler mikorizal oluşumunu da artırmaktadır (Solaiman ve ark., 2019).

Bor, normal bitki büyümesi ve gelişmesi için gerekli olan su ve topraklarda bulunan genellikle borik asit şeklinde alınan temel bir mikro besindir (Tanaka ve Fujiwara, 2008). Bitkilerde bulunan borun %90'ı hücre duvarında yer almaktadır (Goldbach ve Wimmer, 2007). Hücre duvarı ve zarın yapısına katılarak hücreye şekil vermekte güç ve sertlik sağlamaktadır (Brown ve ark., 2002). Bununla birlikte artan kanıtlar borun hücre duvarına katılımından daha ileri fonksiyonları olduğu yönündedir (Goldbach ve Wimmer, 2007). Nitekim borun çok sayıda iyon, metabolit ve hormonun taşıma reaksiyonlarına katıldığı ifade edilmektedir (O'Neill ve ark., 2004). Bitkilerin bor gereksinimleri büyük farklılıklar göstermekle birlikte eksiklik ve toksisite aralıkları dardır (Brdar-Jokanović, 2020). Marul sulama suyundaki bor içerikleri (2-4 mg L<sup>-1</sup>) açısından tolerant bitkiler grubuna girmektedir (Keren ve Bingham, 1985). Tarımsal üretimde verimlilik uygun gübreleme yönetimi ile sürdürülebilir (Sobucki ve ark., 2019). Marul kısa vejetasyonu ve raf ömrü kısa bir bitki

olduğu için gübrelemesi önem arz etmektedir. Marul bitkilerinde N, P ve K gibi makro besin elementleri ile birlikte bor, bakır, magnezyum ve mangan ihtiyacı belirgindir (Thompson ve Kelley, 1957). Humik asit ilave edilmiş yetiştirme ortamında farklı bor seviyelerinde marul çeşitlerinin verim ve kalitelerinin belirlenmesi önemlidir. Bu çalışmada üç farklı marul çeşidinde borlu gübre ve humik asidin verim ve bazı kalite özelliklerine etkileri belirlenmiştir.

## MATERYAL ve YÖNTEM

Marul çeşitlerinde humik asit ve bor uygulamalarının etkilerinin test edildiği çalışma ODÜ Ziraat Fakültesi Araştırma ve Uygulama arazisindeki ısıtmasız tünel tipi plastik örtülü serada yürütülmüştür.

### Materyal

Deneme materyali olarak Fırtına, Campania (AG Tohum, Antalya) ve Olenka (Rain Seeds, Ankara) kıvrıkcık yapraklı marul çeşitleri kullanılmıştır.

### Yöntem

#### Bitkilerin Yetiştirilmesi

Marul bitkileri 50x18x16 cm boyutlarındaki plastik saksılarda yetiştirilmiş ve 3:1 oranındaki torf-perlit karışımı yetiştirme ortamı olarak kullanılmıştır. Fide dikimi her saksıya üçer adet olacak şekilde 10 Ekim tarihinde yapılmıştır. Hasada kadar tüm kültürel işlemler eksiksiz yerine getirilmiştir (Vural ve ark., 2000).

#### Temel Gübreleme, Bor ve Humik Asit Uygulamaları

Çalışmada temel gübreleme olarak tüm parsellere 10-8-10 kg da<sup>-1</sup> dozunda NPK gübrelemesi yapılmıştır. Fosfor kaynağı olarak TSP (Triple super fosfat) dikimle birlikte 8 kg da<sup>-1</sup> olacak şekilde tek seferde uygulanmıştır. Azot kaynağı olarak Ca(NO<sub>3</sub>)<sub>2</sub> (Kalsiyum nitrat), potasyum kaynağı olarak Potasyum sülfat (K<sub>2</sub>SO<sub>4</sub>) gübreleri 10 kg da<sup>-1</sup> dozunda uygulanmıştır. Azot ve potasyum gübreleri 5 kg da<sup>-1</sup> olacak şekilde fide dikiminden sonra iki seferde (15. ve 25. günlerde) verilmiştir.

Çalışmada borlu gübre 0, 50, 100, 200 ve 400 g da<sup>-1</sup> dozlarında denenmiş ve her uygulama dozu fide dikiminden sonra eşit miktarlarda 2 seferde (15. ve 25. günlerde) sulama şeklinde yetiştirme ortamına uygulanmıştır. Borlu gübre (Na<sub>2</sub>B<sub>8</sub>O<sub>13</sub>4H<sub>2</sub>O-Disodyum oktaborat tetrahidrat) %67 oranında Bor oksit (B<sub>2</sub>O<sub>3</sub>) içermektedir.

Humik asit 0 (su) ve %0.2 dozlarında ve saksı başına 500 ml olacak şekilde fide dikiminden 15 gün sonra uygulanmıştır.

Marul bitkilerinde hasat işlemi fide dikiminden sonraki 60. günde (10 Aralık) yapılmıştır. Bitkiler kök boğazından keskin bir bıçak yardımı ile kesilerek hasat edilmiştir.

#### Bitki Analizlerinde Kullanılan Yöntemler

Hasat edilen marul bitkilerinde yaşlı ve sararmış yapraklar atılarak kaba temizlik yapılmış ve en dıştaki 2. veya 3. yapraklardan rastgele seçilmiş 5 yaprakta uzunluk (cm) ve en (cm) değerleri belirlenmiştir. Ayrıca bitki verimi (g bitki<sup>-1</sup>) ile toplam yaprak sayısı (adet bitki<sup>-1</sup>) tespit edilmiştir. Bitkilerin kök boyu değerleri, köklerin

gövdeden ilk çıktığı noktadan köklerin en uç noktasına kadar bir cetvel yardımıyla ölçülerek "cm" olarak belirlenmiştir.

### Deneme Deseni ve İstatistiksel Analizler

Tesadüf parselleri deneme desenine göre 3 tekerrürlü olarak yürütülen çalışmada her bir saksı uygulama tekerrürü kabul edilmiştir. Çalışmanın verileri JUMP istatistik paket programında değerlendirilmiş ve ortalamalar arası önemli görülen farklılıklar LSD çoklu karşılaştırma testi kullanılarak belirlenmiştir ( $P < 0.05$ ).

## BULGULAR ve TARTIŞMA

### Marul Çeşitlerinde Bitki Ağırlığı

Marul çeşitlerinde humik asit (HA) ve bor uygulamalarına göre bitki ağırlığı değerlerinin değişimi Çizelge 1'de verilmiştir.

**Çizelge 1.** Marulda borlu gübre ve humik asit uygulamalarının bitki ağırlığına etkisi (g bitki<sup>-1</sup>).

**Table 1.** Effect of boron fertilizer and humic acid applications on plant weight in lettuce (g plant<sup>-1</sup>).

Çeşit	Bor Dozları (g da <sup>-1</sup> )	HA Kontrol	HA %0.2	Ortalama
Campania	0	553.77 bcd	562.56 bc	558.16 b
	50	593.62 a	594.53 a	594.08 a
	100	544.39 def	565.57 b	554.98 b
	200	528.34 g-l	546.30 def	537.32 cd
	400	515.99 klm	544.35 def	530.17 c-g
	<b>Ortalama</b>	<b>547.22 B</b>	<b>562.66 A</b>	<b>554.94 A</b>
Fırtına	0	538.53 d-j	543.30 d-g	540.92 c
	50	539.69 d-ı	584.45 a	562.07 b
	100	549.09 cde	531.23 f-k	540.16 c
	200	533.97 e-j	527.93 g-l	530.95 c-f
	400	506.91 m	523.56 jkl	515.24 h
	<b>Ortalama</b>	<b>533.64 C</b>	<b>542.09 B</b>	<b>537.87 B</b>
Olenka	0	527.92 h-l	527.64 h-l	527.78 d-g
	50	541.72 d-h	531.94 f-j	536.83 cde
	100	524.91 r-l	514.84 lm	519.87 gh
	200	528.76 g-l	516.08 klm	522.42 fgh
	400	536.53 e-j	515.58 lm	526.05 e-h
	<b>Ortalama</b>	<b>531.97 C</b>	<b>521.22 D</b>	<b>526.59 C</b>
Bor	0	540.07	544.50	542.29 B
	50	558.34	570.31	564.32 A
	100	539.46	537.21	538.34 B
	200	530.36	530.10	530.23 C
	400	519.81	527.83	523.82 D
	<b>Ortalama</b>	<b>537.61 B</b>	<b>541.99 A</b>	

LSD<sub>bor</sub>: 6.27\*\*\* LSD<sub>çesit</sub>: 0.62\*\*\* LSD<sub>humik</sub>: 3.97\* LSD<sub>çesit\*humik</sub>: 6.87\*\*\* LSD<sub>çesit\*bor</sub>: 6.62\*\*\*

LSD<sub>bor\*humik</sub>: ö.d. LSD<sub>bor\*çesit\*humik</sub>: 15.37\*\*\*

öd: önemli değil, \*:  $P \leq 0.05$ , \*\*:  $P \leq 0.01$ , \*\*\*:  $P \leq 0.001$  Aynı satır ve sütündeki ortalamalar arasındaki istatistiksel farklılıklar harf veya harf grubu ile gösterilmiştir (Anova  $P \leq 0.05$  LSD Testi).

Marul çeşitlerinde bor ve humik asit dozlarının bitki ağırlığı değerlerindeki değişim istatistiksel olarak önemli bulunmuştur ( $P \leq 0.05$ ). Marul çeşitleri bitki ağırlığı bakımından üç farklı istatistik grupta yer almışlardır. En yüksek bitki verimi (554.94 g bitki<sup>-1</sup>) Campania çeşidinden elde edilmiştir (Çizelge 1). [Çağlar \(2014\)](#) tarafından yapılan çalışmada farklı fındık zurufu ve çay çöpü karışımlarından hazırlanan ortamlarda Campania çeşidinin bitki ağırlığının Fırtına çeşidinden daha yüksek olduğu tespit edilmiştir. [Uğur ve ark. \(2014\)](#) azotlu gübre uygulamalarının

marul çeşitlerinin verim özellikleri üzerine etkilerini inceledikleri araştırmada azotta doz artışı ile birlikte Fırtına çeşidinde bitki ağırlıklarının Campania çeşidine göre daha yüksek olduğunu bulmuşlardır. Çalışmada bitki gelişiminin daha uzun sürede gerçekleştiği, bu nedenle de yaprak sayısı fazla olan Fırtına çeşidinin bitki veriminin daha yüksek olduğu belirlenmiştir. [Öztürk \(2011\)](#) organik gübre ve dikim zamanlarının etkilerinin incelediği çalışmada kıvrıkcık marul çeşitlerinde bitki ağırlığının 299.20-894.43 g aralığında değiştiğini bildirmişlerdir. Bitki ağırlığı değerlerinin [Uluçay Çam \(2018\)](#)'ın bulgularından daha yüksek, [Şahin ve ark. \(2017\)](#)'nın bulgularından ise daha düşük olduğu bulunmuştur. Bu durum çalışmada kullanılan çeşitlerin, ekolojilerin ve dikim zamanlarının farklı olmasından kaynaklanmaktadır.

Borlu gübre dozlarının bitki ağırlığı üzerine etkilerine bakıldığında, 50 g da<sup>-1</sup> dozunda bitki ağırlığı en yüksek bulunmuş ve kontrole göre yaklaşık %4.06 oranında bir artış belirlenmiştir. Borlu gübre uygulamalarından 100 g da<sup>-1</sup> dozundan elde edilen bitki ağırlığı kontrole benzer bulunmuş, buna karşılık 200 ve 400 g da<sup>-1</sup> dozlarında bitki ağırlığı kontrole göre azalmıştır (Çizelge 1). [Şahin ve ark. \(2017\)](#) serada bahar döneminde borlu gübre uygulamaları ile Funly çeşidinde 100 g da<sup>-1</sup> dozunda bitki ağırlıklarını 950 g bitki<sup>-1</sup> olarak belirlemişlerdir. Değişik kültür bitkilerinde yapılan borlu gübre çalışmalarında, borlu gübre dozundaki artışın bitki ağırlık değerlerinde ve yaprak kalitesinde azalmalara neden olduğu bildirilmiştir ([Francois, 1991](#); [Chutichudet ve Chutichudet, 2009](#); [Samet ve ark., 2015](#); [Şahin ve ark., 2017](#)). [Zambi \(2015\)](#) borlu gübre uygulamalarının yeşil soğanda bitki verimini yaklaşık %10.36'ya varan oranlarda artırdığını bildirmiştir. Araştırmacı verim artışının 50, 100 ve 200 g da<sup>-1</sup> dozlarında olduğunu ve doz artışı ile verimde artışın sınırlandığını ifade etmiştir. Borlu gübre dozundaki artışla birlikte görülen bitki verimin azalması şeklindeki bulgular borun yüksek dozlarda toksik etkisine bağlı olabilir.

Marul çeşitlerinde humik asit uygulamasına bağlı olarak bitki ağırlıkları artmıştır (Çizelge 1). Bu artışın Campania ve Fırtına çeşitlerinde 50 g da<sup>-1</sup> dozunda görülen bitki ağırlıkları ile ilişkili olduğu görülmüştür. Olenka çeşidinde humus ve humik asit uygulama dozlarının etkilerinin inceleyen [Köse \(2015\)](#), bitki veriminde %232'ye varan oranlarda artış olduğunu belirlemiştir. [Baş Odabaş \(2019\)](#) marulda 400 mg kg<sup>-1</sup> humik asit uygulamalarının verimi %11.21 oranında artırdığını ifade etmiştir. [Uğur ve ark. \(2014\)](#) torf:perlit (3:1) karışımında yetiştirdikleri marul çeşitlerinde %0.2 oranında humik asit uygulamasında verim değerlerinin kontrol ile benzer olduğunu belirtmişlerdir. Verimde humik asit uygulamalarının etkinliğinin yüksek olmamasının nedeni çalışmanın bitki kök gelişimi açısından uygun torf:perlit karışımı ortamında yürütülmesi olabilir. Bitki özelliklerinde görülen değişimler borlu gübre dozları ve çeşit özellikleri ile daha çok ilişkilendirilmiştir. Çalışmada Campania çeşidinde humik asit etkilerine bakılmaksızın 50 g da<sup>-1</sup> borlu gübre uygulaması ile Fırtına çeşidinde humik asit uygulanmış 50 g da<sup>-1</sup> borlu gübre uygulamaları en yüksek verimi vermiştir.

### **Kıvrıkcık Marulda Yaprak Sayısı Değerleri**

Kıvrıkcık marul çeşitlerinde yaprak sayısı üzerine borlu gübre ve humik asit uygulamalarının etkileri Çizelge 2'de gösterilmiştir.

**Çizelge 2.** Marulda borlu gübre ve humik asit uygulamalarının yaprak sayısına etkisi (adet bitki<sup>-1</sup>).

**Table 2.** Effect of boron fertilizer and humic acid applications on the number of leaves in lettuce (number plant<sup>-1</sup>).

Çeşit	Bor Dozları (g da <sup>-1</sup> )	HA Kontrol	HA %0.2	Ortalama
Campania	0	24.89 h	24.95 h	24.92 d
	50	24.67 h	24.56 h	24.61 de
	100	25.00 h	23.44 ı	24.22 ef
	200	25.78 g	23.33 ı	24.56 de
	400	23.56 ı	24.55 h	24.06 f
	<b>Ortalama</b>	<b>24.78 C</b>	<b>24.17 D</b>	<b>24.47 B</b>
Fırtına	0	29.56 a	28.89 abc	29.22 a
	50	28.00 de	27.78 e	27.89 c
	100	28.83 bc	28.00 de	28.42 b
	200	29.44 ab	27.00 f	28.22 bc
	400	28.67 cd	27.78 e	28.22 bc
	<b>Ortalama</b>	<b>28.90 A</b>	<b>27.89 B</b>	<b>28.39 A</b>
Olenka	0	21.78 j	19.22 klm	20.50 g
	50	19.78 k	18.78 lm	19.28 h
	100	18.55 mn	17.67 o	18.11 ij
	200	17.44 o	17.89 no	17.67 j
	400	17.44 o	19.33 kl	18.39 ı
	<b>Ortalama</b>	<b>19.00 E</b>	<b>18.58 F</b>	<b>18.79 C</b>
Bor	0	25.41 a	24.35 b	24.88 A
	50	24.15 bc	23.70 d	23.93 B
	100	24.12 bc	23.04 ef	23.58 C
	200	24.22 bc	22.74 f	23.48 C
	400	23.22 e	23.89 dc	23.56 C
	<b>Ortalama</b>	<b>24.22 A</b>	<b>23.54 B</b>	

LSD<sub>bor</sub>: 0.28\*\*\* LSD<sub>çegit</sub>: 0.22\*\*\* LSD<sub>humik</sub>: 0.18\*\*\* LSD<sub>çegit\*humik</sub>: 0.31\* LSD<sub>çegit\*bor</sub>: 0.31\*\*\*

LSD<sub>bor\*humik</sub>: 0.40\*\*\* LSD<sub>bor\*çegit\*humik</sub>: 0.70\*\*\*

öd: önemli değil, \*: P≤0.05, \*\*: P≤0.01, \*\*\*: P≤0.001 Aynı satır ve sütündeki ortalamalar arasındaki istatistiki farklılıklar harf veya harf grubu ile gösterilmiştir (Anova P ≤ 0.05 LSD Testi).

Borlu gübre ve humik asit uygulamaları ile interaksiyon etkileri marul çeşitlerinin yaprak sayısında istatistiksel anlamda önemli değişimlere neden olmuştur (P≤0.05).

Her üç marul çeşidinin yaprak sayıları istatistiksel olarak farklı gruplarda yer almış, Fırtına çeşidi 28.39 adet bitki<sup>-1</sup> değeri ile en yüksek yaprak sayısını vermiştir. Campania ve Olenka çeşitleri diğer istatistiki grupları oluşturmuştur. Borlu gübre dozunun artışı ile birlikte marul çeşitlerinde bitki yaprak sayısı azalmıştır (24.88-23.56 adet bitki<sup>-1</sup>). Marulda organik atık karışımlarını inceleyen [Cağlar \(2014\)](#) Fırtına ve Campania çeşitlerinin yaprak sayısı değerlerinin sırasıyla 33.09 ve 25.55 olduğunu bildirmiştir. Azot gübre dozları ve humik asit uygulamasının marul çeşitlerinde etkilerini inceleyen [Uğur ve ark. \(2014\)](#) Campania çeşidinde 26.80 adet bitki<sup>-1</sup> olan yaprak sayısının Fırtına çeşidinde 30.40 adet bitki<sup>-1</sup> olduğunu tespit etmişlerdir. Yaprak sayısı bulgularının literatürle uyumlu olduğu görülmüştür.

Humik asit uygulamaları ile bitki yaprak sayısı değerleri azalmıştır. Marul çeşitlerinde humik asit kontrol uygulamasında 24.22 adet bitki<sup>-1</sup> olarak belirlenen bitki yaprak sayısı değeri 23.54 adet bitki<sup>-1</sup> değerine düşmüştür. [Köse \(2015\)](#) humus ve humik asit uygulamalarında doz artışı ile birlikte bitki yaprak sayılarının 13.7 adet bitki<sup>-1</sup>'den 27 adet bitki<sup>-1</sup>'ye kadar arttığını belirtmiştir. Diğer yandan [Uğur ve ark. \(2014\)](#) ise marul çeşitlerinde humik asit uygulamalarının yaprak sayısını etkilemediğini belirtmiştir. Daha önce humik asit uygulamalarının bitki ağırlığı değerleri üzerine etkilerinde ifade edildiği üzere humik asit uygulamasının açık tarla koşullarında toprağa uygulanması ile bitkilerde besin maddeleri alınımında



iyileşmelere bağlı olarak bitkilerde kalite özelliklerinde olumlu etkiler daha belirgin görülmektedir. Çalışmada humik asit uygulamaları ile bitki yaprak sayısı değerlerinde azalmaların olması şeklindeki bulgu, muhtemelen humik asit uygulaması ile birlikte bitkinin yeni yaprak oluşturma yerine daha çok yaprak kalitesini artırmaya yönelmesinin bir sonucudur.

Marul bitkilerinde borlu gübre ve humik asit uygulamalarının interaksiyon etkileri değerlendirildiğinde, yaprak sayılarının 17.44-29.56 adet bitki<sup>-1</sup> arasında olduğu belirlenmiştir. Fırtına çeşidinde kontrol uygulamasında 29.56 adet bitki<sup>-1</sup> ile en yüksek yaprak sayısı değeri elde edilmiş, en az yaprak sayısı ise, 17.44 adet bitki<sup>-1</sup> değerleri ile Olenka çeşidinde 200 ve 400 g da<sup>-1</sup> borlu gübre uygulamalarında belirlenmiştir. Borlu gübre uygulamaları ile Olenka çeşidinde yaşlı yapraklarda dökülme görülmüş, bu durumun borun toksik etkilerinden kaynaklandığı düşünülmüştür. [Francois \(1988\)](#)'in ifade ettiği yüksek doz borlu gübre uygulamaları ile yaprakların kıvrılarak sarılma şeklindeki görünüm Olenka çeşidinde daha çok görülmüştür. Bu görünüm daha çok yaprağın dip bölgelerinde tespit edilmiştir.

### Marul Çeşitlerinde Yaprak Uzunluğu

Marul çeşitlerinde borlu gübre ve humik asit uygulamalarının yaprak uzunluğu üzerine etkileri Çizelge 3'te verilmiştir.

**Çizelge 3.** Marulda borlu gübre ve humik asit uygulamalarının yaprak uzunluğuna etkisi (cm).

**Table 3.** Effect of boron fertilizer and humic acid applications on the leaf length in lettuce (cm).

Çeşit	Bor Dozları (g da <sup>-1</sup> )	HA Kontrol	HA %0.2	Ortalama
Campania	0	20.35 ı	20.70 hı	20.53 g
	50	20.70 hı	22.55 e	21.63 e
	100	21.12 gh	21.23 fg	21.18 f
	200	22.93 e	21.29 fg	22.11 d
	400	21.61 f	20.63 hı	21.12 f
	<b>Ortalama</b>	<b>21.34 C</b>	<b>21.28 C</b>	<b>21.31 B</b>
Fırtına	0	18.72 j	18.73 j	18.72 h
	50	18.43 j	17.64 k	18.04 ij
	100	17.81 k	17.82 k	17.81 j
	200	17.84 k	18.53 j	18.18 ı
	400	17.86 k	18.35 j	18.11 ij
	<b>Ortalama</b>	<b>18.13 D</b>	<b>18.21 D</b>	<b>18.17 C</b>
Olenka	0	27.85 a	27.86 a	27.85 a
	50	26.78 cd	27.57 ab	27.17 bc
	100	27.16 bc	27.61 ab	27.38 b
	200	26.69 cd	27.49 ab	27.09 bc
	400	26.49 d	27.33 b	26.91 c
	<b>Ortalama</b>	<b>26.99 B</b>	<b>27.57 A</b>	<b>27.28 A</b>
Bor	0	22.31 abc	22.43 ab	22.37 A
	50	21.97 d	22.59 a	22.28 AB
	100	22.02 cd	22.22 bcd	22.12 BC
	200	22.48 ab	22.44 ab	22.46 A
	400	21.99 d	22.11 cd	22.05 C
	<b>Ortalama</b>	<b>22.15 b</b>	<b>22.36 a</b>	

LSD<sub>bor</sub>: 0.19\*\*\* LSD<sub>çesit</sub>: 0.15\*\*\* LSD<sub>humik</sub>: 0.12\*\* LSD<sub>çesit\*humik</sub>: 0.21\*\*\* LSD<sub>çesit\*bor</sub>: 0.34\*\*\*

LSD<sub>bor\*humik</sub>: 0.28\* LSD<sub>bor\*çesit\*humik</sub>: 0.48\*\*\*

öd: önemli değil, \*: P≤0.05, \*\*: P≤0.01, \*\*\*: P≤0.001

Borlu gübre ve humik asit uygulamaları ile interaksiyon etkilerinde marul çeşitlerinin yaprak uzunluğu değerleri arasındaki değişim istatistiksel olarak önemlidir ( $P \leq 0.05$ ).

Borlu gübre ve humik asit uygulamalarına göre belirlenen yaprak uzunluk değerleri bakımından tüm çeşitler istatistiki olarak farklı grupta yer almışlardır. Yaprak uzunluğu en yüksek çeşit 26.69 cm ile Olenka çeşidi olmuştur. Campania çeşidinde yaprak uzunluğu 21.31 cm, Fırtına çeşidinde ise 18.17 cm olarak belirlenmiştir. Marul çeşitlerinin yaprak sayıları ile yaprak uzunlukları arasında ters yönlü bir ilişki belirlenmiştir. Yaprak uzunluk değerleri düşük olan Fırtına çeşidinde yaprak sayısı en yüksek bulunmuştur. Fırtına'da bitki yaprak uzunluğunu artırma yerine daha çok yeni yaprak oluşturma şeklinde davranış göstermektedir. Borlu gübre uygulamalarında en yüksek yaprak uzunluk değeri kontrol ve 200 g da<sup>-1</sup> dozundan elde edilmiş, en küçük yaprak uzunluk değeri ise 400 g da<sup>-1</sup> dozunda belirlenmiştir. Çalışmada interaksiyon etkileri irdelendiğinde, humik asidin Olenka marul çeşidinde yaprak uzunluk değerini artırdığı belirlenmiştir. Borlu gübrenin humik asit ile interaksiyon etkilerine göre yaprak uzunluk değerleri 17.64-27.86 cm arasında bulunmuştur. Yaprak uzunluk değerleri Campania çeşidinde 20.35 cm ile 22.93 cm arasında, Fırtına çeşidinde 17.64 cm ile 18.73 cm arasında, Olenka çeşidinde ise 21.23 cm ile 27.86 cm arasında değişmiştir. Yaprak uzunluk değerlerinde Fırtına çeşidi 100 g da<sup>-1</sup> dozunda en düşük, Olenka çeşidi kontrol parsellerinde en yüksek değerler belirlenmiştir.

Olenka çeşidinde humus ve humik asit uygulamaları ile yaprak uzunluğunun 16.89-28.20 cm arasında değiştiği tespit edilmiştir (Köse, 2015). Humik asit uygulama dozundaki artış yaprak uzunluk değerlerini artırmıştır. Gün (2019) farklı dozlardaki organik gübreleme ile yaprak boy uzunluk değerinin Olenka çeşidinde 21.0 cm, Campania çeşidinde 17.2 cm ve Fırtına çeşidinde ise 15.0 cm olduğunu tespit etmiştir. Humik asit uygulamasının kontrole göre yaprak uzunluk değerlerini değiştirmedeği ve Campania çeşidinde 16.70 cm, Fırtına çeşidinde ise 14.69 cm olduğu şeklinde bulgular da vardır (Uğur ve ark., 2014). Bu çalışmada birim alandaki bitki başına yetiştirme ortam miktarının daha az olması nedeniyle daha düşük yaprak uzunluk değerleri elde edilmiş olabilir. Yaprak uzunluk değerleri sonuçları önceki bulgular ile paralellik arz etmektedir.

### **Marul Çeşitlerinde Yaprak Eni**

Marul çeşitlerinde borlu gübre ve humik asit uygulamalarının bitki yaprak eni üzerine etkisi Çizelge 4'te gösterilmiştir.

**Çizelge 4.** Marulda borlu gübre ve humik asit uygulamalarının yaprak enine etkisi (cm).  
**Table 4.** Effect of boron fertilizer and humic acid applications on the leaf width in lettuce (cm).

Çeşit	Bor Dozları (g da <sup>-1</sup> )	HA Kontrol	HA %0.2	Ortalama
Campania	0	13.67 p	13.81 op	13.74 ı
	50	14.95 f <sub>1</sub>	15.44 b-e	15.19 de
	100	14.13 mno	14.73 h-l	14.43 gh
	200	14.33 lmn	14.82 h-k	14.57 g
	400	14.36 lmn	14.43 klm	14.40 gh
	<b>Ortalama</b>	<b>14.29 D</b>	<b>14.65 C</b>	<b>14.47 C</b>
Fırtına	0	15.91 a	15.91 a	15.91 a
	50	15.71 abc	15.87 a	15.79 ab
	100	15.75 abc	15.72 abc	15.74 ab
	200	15.26 d-g	15.81 ab	15.54 bc
	400	15.13 e-h	14.86 g-j	14.99 ef
	<b>Ortalama</b>	<b>15.55 A</b>	<b>15.64 A</b>	<b>15.59 A</b>
Olenka	0	14.01 nop	14.57 ı-l	14.29 h
	50	15.47 b-e	15.41 cde	15.44 cd
	100	15.54 a-d	14.93 f <sub>1</sub>	15.23 de
	200	15.30 def	14.66 ı-l	14.98 ef
	400	15.23 eh	14.52 j-m	14.88 f
	<b>Ortalama</b>	<b>15.11 B</b>	<b>14.82 C</b>	<b>14.88 B</b>
Bor	0	14.53 g	14.76 ef	14.65 C
	50	15.38 ab	15.57 a	15.47 A
	100	15.14 bc	15.12 cd	15.13 B
	200	14.96 cde	15.10 cd	15.03 B
	400	14.91 de	14.60 fg	14.76 C
	<b>Ortalama</b>	<b>14.98</b>	<b>15.03</b>	

LSD<sub>bor</sub>: 0.16\*\*\* LSD<sub>çesit</sub>: 0.12\*\*\* LSD<sub>humik</sub>: öd. LSD<sub>çesit\*humik</sub>: 0.18\*\*\* LSD<sub>çesit\*bor</sub>: 0.28\*\*\*  
LSD<sub>bor\*humik</sub>: 0.23\* LSD<sub>bor\*çesit\*humik</sub>: 0.40\*\*  
öd: önemli değil, \*: P<0.05, \*\*: P<0.01, \*\*\*: P<0.001

Borlu gübre ve humik asit uygulamaları ile interaksiyon etkileri marul çeşitlerinin yaprak eni değerlerinde istatistiksel anlamda önemli değişimlere neden olmuştur (P<0.05).

Her üç marul çeşidinin yaprak eni bakımından istatistiksel olarak farklı gruplarda yer almıştır. Borlu gübre uygulamalarında 400 g da<sup>-1</sup> dozu kontrolle benzer bulunmuş, en yüksek yaprak eni değerleri 50 g da<sup>-1</sup> borlu gübre uygulamalarında belirlenmiştir. Borlu gübre, çeşit ve humik uygulama interaksiyonlarına göre humik asit Campania'da yaprak eni değerlerine olumlu etkide bulunmuştur. Borlu gübre uygulamalarında en yüksek yaprak eni değeri 50 g da<sup>-1</sup> dozunda 15.47 cm olarak tespit edilmiştir. Campania çeşidinde bor ve humik asit kontrol uygulaması en düşük yaprak eni değerini verirken Fırtına çeşidinde kontrol uygulaması ve humik asit uygulanmış 50 g da<sup>-1</sup> borlu gübre dozu en yüksek yaprak eni değerini vermiştir. Humik asit uygulaması yaprak eni değerleri üzerine istatistiki anlamda önemsiz bulunmuştur (P>0.05).

[Köse \(2015\)](#) artan dozlarda humus ve humik asit uygulamaları ile Olenka çeşidinde yaprak eni değerlerinin 14.02-22.14 cm arasında olduğunu bildirmiştir. Çalışmada humik asit ve humus uygulama dozundaki artış ile birlikte yaprak eni değerleri de artmıştır. Farklı azot ve potasyum dozlarının etkilerini inceleyen [Uluçay Çam \(2018\)](#) kıvrıcık marulda yaprak eninin 11.33-16.69 cm aralığında bulunduğunu belirlemiştir. [Çağlar \(2014\)](#) ise fındık zürufu ve çay çöpü ortam karışımlarında yetiştirdiği kıvrıcık marul çeşitlerinde yaprak eninin Campania'da 14.68 cm ve Fırtına'da ise 12.56 cm olarak tespit etmiştir. [Gün \(2019\)](#) iki farklı organik gübre uygulanan kıvrıcık marul çeşitlerinde yaprak eni değerlerini Olenka çeşidinde 14.0 cm, Campania çeşidinde

13.2 cm ve Fırtına çeşidinde ise 13.8 cm olarak belirlemiştir. Yaprak eni değerleri önceki çalışma bulgularına benzer olup, Fırtına en yüksek yaprak eni değerini vermiş, Olenka ve Campania bu çeşidi takip etmiştir. Borlu gübre uygulamaları yaprak eni değerlerinde 50, 100 ve 200 g da<sup>-1</sup> dozlarında kontrole göre kadar arttırıcı yönde etkide bulunmuştur. En yüksek doz 400 g da<sup>-1</sup> borlu gübre dozu ise yaprak eni değerlerinde kontrole benzer bulunmuştur. Doz artışı ile muhtemelen borlu gübrenin toksik etkileri nedeniyle yaprak eni değerlerindeki artış sınırlı kalmıştır.

### Marul Çeşitlerinde Kök Boyu

Çalışmada kıvrıkcık marul çeşitlerinin borlu gübre, humik asit uygulamaları ile interaksiyon etkileri istatistiksel anlamda önemli bulunmuştur ( $P \leq 0.05$ ). Kıvrıkcık marullarda en yüksek kök boyu değeri 19.08 cm ile Olenka çeşidinde belirlenmiş olup, bunu 16.07 cm kök boyu ile Campania çeşidi ve 15.94 cm kök boyu ile Fırtına çeşidi izlemiştir. Borlu gübre dozları kontrol uygulamasına göre kök boyunu arttırmış, en yüksek kök boyu değerleri 50 g da<sup>-1</sup> ve 100 g da<sup>-1</sup> dozlarında belirlenmiştir. Kıvrıkcık marul çeşitlerinde kök boyunun Campania'da 14.84 cm ile 18.03 cm, Fırtına çeşidinde 13.82 cm ile 18.29 cm, Olenka çeşidinde ise 16.08 cm ile 23.16 cm aralığında olduğu belirlenmiştir. İnteraksiyon etkilerine bakıldığında en kısa kök boyu humik asit uygulanan Fırtına çeşidinin 100 g da<sup>-1</sup> borlu gübre uygulanmış parselinden 13.82 cm ile elde edilirken, en uzun kök boyu humik asit uygulanmayan Olenka çeşidinin 100 g da<sup>-1</sup> borlu gübre uygulanmış parselinden 23.16 cm ile elde edilmiştir (Çizelge 5).

**Çizelge 5.** Marulda borlu gübre ve humik asit uygulamalarının kök boyuna etkisi (cm).  
**Table 5.** Effect of boron fertilizer and humic acid applications on root length in lettuce (cm).

Çeşit	Bor Dozları (g da <sup>-1</sup> )	HA Kontrol	HA %0.2	Ortalama
Campania	0	15.28 k-o	17.02 gh	16.15 e
	50	15.48 k-n	14.84 n-o	15.16 f
	100	16.57 hij	15.71 klm	16.14 e
	200	17.98 ef	14.99 m-o	16.48 e
	400	18.03 ef	14.84 n-o	16.44 e
	<b>Ortalama</b>	<b>16.67 D</b>	<b>15.48 E</b>	<b>16.07 B</b>
Fırtına	0	16.84 ghı	15.21 l-o	16.02 e
	50	17.27 fgh	15.39 k-o	16.33 e
	100	18.29 e	13.82 p	16.06 e
	200	17.46 efg	14.57 o-p	16.01 e
	400	15.99 jkl	14.60 o-p	15.29 f
	<b>Ortalama</b>	<b>17.17 C</b>	<b>14.72 F</b>	<b>15.94 B</b>
Olenka	0	19.29 d	16.08 ijk	17.69 e
	50	22.30 b	19.71 cd	21.01 a
	100	23.16 a	18.08 ef	20.62 a
	200	20.42 c	20.07 cd	20.24 b
	400	19.48 d	19.71 cd	19.60 c
	<b>Ortalama</b>	<b>20.93 A</b>	<b>18.73 B</b>	<b>19.83 A</b>
Bor	0	17.14 d	16.10 fg	16.62 C
	50	18.35 b	16.65 de	17.50 A
	100	19.34 a	15.87 g	17.60 A
	200	18.62 b	16.54 ef	17.58 A
	400	17.83 c	16.39 ef	17.11 B
	<b>Ortalama</b>	<b>18.26 A</b>	<b>16.31 B</b>	

LSD<sub>bor</sub>: 0.34\*\*\* LSD<sub>çesit</sub>: 0.26\*\*\* LSD<sub>humik</sub>: 0.21\*\*\* LSD<sub>çesit\*humik</sub>: 0.37\*\*\* LSD<sub>çesit\*bor</sub>: 0.59\*\*\*

LSD<sub>bor\*humik</sub>: 0.48\*\*\* LSD<sub>bor\*çesit\*humik</sub>: 0.84\*\*\*

öd: önemli değil, \*:  $P \leq 0.05$ , \*\*:  $P \leq 0.01$ , \*\*\*:  $P \leq 0.001$

[Cağlar \(2014\)](#) Campania ve Fırtına kıvrıkcık marul çeşitlerinin kök boylarını sırasıyla 8.37 cm ile 8.04 cm olarak tespit etmiştir. [Bilgi \(2009\)](#) marulda biostimulant içerikli uygulamalar ile kök boyunun 24.0-35.4 cm arasında değiştiğini belirlemiştir. Marul bitkisinde yetiştirme ortamlarına organik madde ilavesi ile bitkilerde büyüme ve gelişmenin olumlu yönde etkilendiği görülmüştür. [Gün \(2019\)](#) torf:perlit karşımı yetiştirme ortamında organik gübre doz uygulamaları ile Campania çeşidinde 16.2-19.4 cm, Fırtına çeşidinde 14.3-26.5 cm, Olenka çeşidinde ise 14.1-27.5 cm aralığında kök boyu değerleri elde edildiğini tespit etmiştir. [Cağlar \(2014\)](#)'ın sonuçları muhtemelen yetiştirme ortamı farklılığı nedeniyle bu çalışmanın sonuçlarına göre biraz düşük bulunmuştur. Diğer çalışmalardaki kök boyu değerleri bulgulara benzer bulunmuştur.

## SONUÇ

Bu çalışma sonbahar yetiştirme döneminde ısıtmasız plastik örtülü sera şartlarında saksı denemesi şeklinde yürütülmüş, borlu gübre ve humik asit uygulamalarının üç farklı kıvrıkcık marul çeşidinde verim ve bazı bitki kalite özelliklerine etkisi belirlenmiştir. Çalışma sonuçlarına göre uygulamalar istatistiksel açıdan önemli bulunmuştur. Kıvrıkcık marul çeşitleri arasında genotipsel farklılıklar nedeniyle verim ve kalite özelliklerinde değişimler gözlenmiş olup, çeşitlerin borlu gübre dozları ve humik asit uygulamalarına verdiği tepkilerin de farklı olduğu tespit edilmiştir. Borlu gübre uygulamalarından 50 g da<sup>-1</sup> dozu incelenen verim ve bitki kalite özelliklerinde kontrol uygulamasına göre artırıcı yönde etkide bulunmuştur. Kıvrıkcık marul çeşitlerinden Olenka çeşidinde bitki yaprak uzunluk değerleri ve kök boyu değerlerinin diğer çeşitlere göre daha yüksek olduğu belirlenmiştir. Borlu gübre uygulamaları kök boyu değerlerini artırmıştır.

Çalışma bulgularına göre daha düşük dozlarda borlu gübrenin yaprak ve topraktan uygulanması, diğer bitki besin elementlerinin borlu gübre ile birlikte denenmesinin tarımsal verimlilik ve bitki kalite özellikleri açısından önemli olduğu düşünülmektedir. Topraksız kültür şartlarında humik asit uygulamalarının daha çok yaprak uzunluğu değerlerini artırdığı ve bu artışın bitki verimini olumlu etkilediği görülmüştür. Topraklı yetiştiricilikte ve yapraktan humik asit uygulama etkilerinin daha belirgin olabileceği düşünülmektedir.

## ÇIKAR ÇATIŞMASI

Yazarlar herhangi bir çıkar çatışması olmadığını beyan ederler.

## YAZAR KATKISI

**Özge Kurt:** Araştırmanın planlanması, literatür tarama, arazi çalışmasının yürütülmesi, laboratuvar çalışmalarının yapılması, verilerin elde edilmesi ve bilgisayara girişi, makalenin yazılması.

**Atnan Uğur:** Araştırmanın planlanması, arazi çalışması için malzeme temini, arazi çalışmasının yürütülmesi ve kontrolü, laboratuvar çalışmalarının kontrolü, verilerin analiz edilmesi, makalenin yazılması.

## ETİK KURUL KARARI

Bu makale Etik Kurul Kararı gerektirmemektedir.

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## Comparative Investigation of n-Hexane and Ethanol Solvents Used in *Eleais guinesis* Kernel Oil Extraction and Optimization via Two Computational Modelling

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### ABSTRACT

The global usages of oil seed products are on high demand; which gave rise to the need to optimize the extraction of *Eleais guinness* kernel oil. This work investigated the performance of n-hexane and ethanol as solvents for extraction and optimization of *Eleais guinesis* kernel oil via Response System Methodology (RSM) and Artificial Neural Networks (ANNs) computational modeling. The 5 days sun-dried *Eleais guinesis* Seeds collected were crushed, the oil was extracted from the powdered seed using a Soxhlet extractor, with n-hexane and ethanol as solvents. The result analyzed by average computation of 40min extraction time, 175 ml solvents, and 50g sample weight for both solvents shown that the average oil yield for n-hexane is 38.15% (w w<sup>-1</sup>) and 28.83% (w w<sup>-1</sup>) for ethanol. At the box-Behnken experimental design having the same averaged independent variables, the average predicted values of: RSM is 35.21; ANNs is 37.21 for n-hexane solvent, while for ethanol solvent, the average predicted values of: ANNs is 31.118; RSM is 30.80. The coefficients of determination (R<sup>2</sup>) for RSM were 99.94% for n-hexane and 99.89% (w w<sup>-1</sup>) for ethanol, and ANN has 99.99% (w w<sup>-1</sup>) for n-hexane and 99.899% (w w<sup>-1</sup>). As a result; n-hexane is better than ethanol in term of oil extraction, ANNs has higher predicted values for optimization in both solvents, therefore it is a better model for oil's optimization, it further proved that both models can be used adequately to represent the actual relationship of the chosen factors which can be applied for optimization simultaneously.

#### RESEARCH ARTICLE

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- *Eleais guinesis* kernel oil,
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## INTRODUCTION

The global utilization of oil is on increase and has led to increasing in demand for vegetable oil for both domestic and industrial use. In meeting this demand, oil is being extracted from the biological seeds like the groundnut, pawpaw, soya bean, and *Elaeis guinesis* seeds, which are vital in this regard due to the kind, volume, quality, and usefulness of oil derived from them. *Elaeis guinesis* (Oil palm) seed produces two major oils from the processing of Fresh Fruit Bunches (FFB) which are Crude Palm Oil and Crude Palm Kernel Oil (Otti *et al.*, 2014). The two kinds of oils produced are edible plant oils (Imoisi *et al.*, 2015), palm kernel oil is gotten from the kernel of the seed of oil palm (Poku, 2002). *Elaeis guinesis* kernel oil is used in manufacturing detergents, soaps, and as washing powders due to the lauric and myristic fatty acids present in it, it is used in the pharmaceutical industries, for the production of drugs for consumption purposes (Alander, 2004). According to Ijaola and Adepoju (2021b), *Elaeis guinesis* kernel oil is used as lubricants for steam engines, machinery, and as major raw material for soap manufacturing. Palm kernel oil is an excellent source of lauric acid, oleic acid, and myristic acid (Chandrasekharan *et al.*, 2000).

The quality of *Elaeis guinesis* kernel oil is determined by the physicochemical composition of the oil which is reported to contain highly saturated fatty acids which is semi-solid at room temperature, and several saturated and unsaturated fats in the forms of glyceryl laurate (Cottrell, 1991). It resists oxidative deterioration (Berger, 1992). This is recently corroborated by the discovery of Ijaola and Adepoju (2021b), who investigated the physicochemical properties of palm kernel oil and discovered that the physical state of the oil is yellowish-brown with the following chemical properties; FFA 11.08%, acid value 22.16 mg KOH g<sup>-1</sup> oil, saponification value 140.123 mg KOH g<sup>-1</sup> oil, iodine value 87.85 g I<sub>2</sub> 100g<sup>-1</sup> and higher heating value 30.51, the finding which supports that the composition of the oil confirmed its impotence mentioned earlier.

Having established the quality of *Elaeis guinesis* kernel oil and its usefulness, the extraction of the oil is very crucial to making it available for consumption, hence the need to investigate the better solvent to be used in solvent extraction methods among other methods like mechanical screw-press, and traditional methods (Jin, 2008). The solvents that have been used for vegetable oil seeds solvent extraction includes; hexane, heptane, isohexane, isopropanol, and ethanol (Connerton *et al.*, 1995; Baker and Sullivan, 1983, Senior *et al.*, 1998). These have been investigated on cottonseed (Abraham *et al.*, 1988), sunflower seed (Senior *et al.*, 1998) and soybean (Baker and Sullivan, 1983) and found to be appropriate as solvents in extraction. This work investigates and compares two solvents namely n-hexane and ethanol as a solvent for extraction of *Elaeis guinesis* kernel oil, having discovered the limitation of the hexane as it is identified to pollute the air when it is emitted during the oil extraction (Wan *et al.*, 1995a). For health and environmental safety, ethanol was researched as an adequate replacement for hexane to eliminate and or reduce the emissions of volatile organic compounds also potential traces of hexane in edible oils after extraction. Ethanol is reported to be non-toxic alcohol with fewer handling risks as compared to hexane in extraction (Suzana 2003; Ijaola and Adepoju, 2021b).

Because of the interest in extracting the optimum oil from the kernel; this work investigated the use of two computational models in optimizing oil extraction. Modeling

is a scientific approach that represents ideas about the natural phenomenon under investigation, presenting alternatives to the real phenomenon, by referring to the existing knowledge (Gendy *et al.*, 2020). One of the generally used models is mathematical modeling (Najafi *et al.*, 2019). And the two mathematical models used to predict experimental behavior in this research are Response Surface Methodology (RSM) and Artificial Neural Networks (ANNs) which are significant in the field of processes in optimization (Gendy *et al.*, 2020). These methods determine the relationship between the input and output variables through the data derived from the experiment. The models are used for the prediction of the optimum situations of independent variables (Ahmadpour *et al.*, 2018). RSM enables the estimation of desired responses from several independent variables with relationships between them. The major benefit of RSM is fewer experimental runs are sufficient to provide a statistically significant result, and because it has provided efficient solutions, it successfully used in engineering problems (Osman *et al.*, 2019; Selvan *et al.*, 2018). However, ANNs modeling is a statistical technique that solves problems that are not eligible for conventional statistical methods. It handles obscure, complex, incomplete problems; it is a model that produces predictions and generalizations at high speed (Gendy *et al.*, 2020).

Both RSM and ANN techniques do not need the precise expressions or the physical meaning of the system under investigation (Selvan *et al.*, 2018). Some existing scholars have compared the RSM and ANN in optimization like; Ahmadpour *et al.* (2018), discovered that the ANNs model is more accurate than the accuracy of RSM, Manda *et al.* (2019), showed that ANNs has better modeling accuracy than RSM, while Awolusi *et al.* (2019), stated RSM showed the supremacy over ANNs as a model that analyzes non-linear relationships of data sets, but ANNs provides good fitting for data and it is better for prediction. The findings from these scholars revealed that ANNs are better than RSM in optimization, though the cost of computation is high (Osman *et al.*, 2019). Since Ahmadpour *et al.*, 2018 employed caustic water waste for the comparison of the performance of the models, then, this work will study the performance of RSM and ANNs on optimization oil extraction, specifically the palm kernel oil.

The research work investigates the effectiveness of replacing hexane with ethanol as a solvent in extracting palm kernel oil by comparing the output of both solvents; it also seeks to find out which of the RSM and ANN perform better in optimizing the oil extracted through the two solvents considering the variable experimental inputs.

## MATERIALS and METHODS

Palm kernel oil extraction and optimization from the seeds collected from the fields follow the methods described as follows:

### Equipment and Reagent

The equipment that was used includes Muslim Bag, Soxhlet Extractor of 500 ml, Digital Weighing balance, Heating Mantle, Water Bath, and Oven. Flash Point Machine, Spectrometer as presented in figure 2 which is used to separate and measure the spectral component of the sample, Viscometer, Glassware which includes beakers, round bottom flask, conical flasks, pycnometer, Petri dish, Measuring cylinder and

burettes. The analytical reagents used are; Ethanol, Potassium Iodide (KI), Phenolphthalein, Iodine, Chlorine, HCL, KOH, NaOH, and they are all obtained from BDH Chemical Ltd., Poole England ([Ijaola and Adepoju, 2021b](#)).

### Seeds preparation

Palm kernel nuts were collected from Akwa Ibom State in July 2018, the nuts were cracked off the shells, and the broken shells were separated from the seed. Palm kernel seeds were sundried for two days and were later crushed and grounded.

### Extraction Procedures

Four 500 ml Soxhlet extractors as seen in Figure 1, were used for this study alongside two solvents which are n-hexane, and ethanol. A known weight of palm kernel seed powder which ranged from 40-60 g was put in a Muslim bag and then placed in Soxhlet apparatus, and a known volume of the solvent ranging from 150-200 ml in a round bottom flask was placed on the heating mantle. The soxhlet apparatus was placed on the flask, the condenser is fixed and the water inlet and outlet are connected and with the aid of the resort, the stand was placed to balance. The water tap was turned on, the heating mantle was turned on and it provides heat at 68-70°C a temperature below the boiling point of the solvents. After the process, the solvents are recycled and the oil was left in the round bottom flask which is later weighed using a weighing balance. The oil yield was evaluated as the ratio of the weight of the extracted oil to the weight of palm kernel oilseed grounded sample as expressed in 3.1; oil obtained was stored in a freezer at 40°C for further characterization.

$$\% \text{ Oil yield (w/w)} = \frac{\text{Weight of extracted oil in grams}}{\text{Weight of grinded samples in grams}} \times 100 \quad (1)$$

### Experimental Design

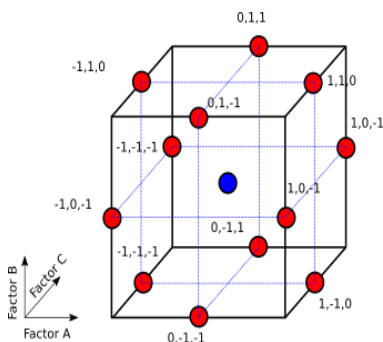
The experimental design used is Box-Behnken see Figure 3, employed by [Ijaola and Adepoju \(2021a\)](#), in the optimization of the extraction of oil from Moringa seed. Box-Behnken is a significant part of RSM in the design of experiments devised by George. E.P. Box and Donald Behnken in 1960 for three variable independent factors which coded as -1, 0, +1, design to fit quadratic model for the reasonable coefficient of the ratio of several experimental points ([Karmoker et al., 2019](#)). It is abbreviated as DoE, which is used for the model fitting of physical experiments, with numerical experiments. Its aim is the choice of the points where the response should be evaluated. The mathematical model of the process is needed for the optimal design of experiments; the model is usually polynomial with an unknown structure that is  $y = f(x_i)$  or  $y = f(x_1, x_2, x_3)$ , such that experiments are designed for the particular problem ([Box and Draper, 1987](#)). The research which is carried out in the year 2018; runs seventeen experiments with three independent variables which were: extraction time  $F_1$  which ranged from 30-50 min, solvent volume  $F_2$  which ranged 150-200 ml, and sample weight  $F_3$  which ranged 40-60 g.



**Figure 1.** Three sets of the Soxhlet Extractor.



**Figure 2.** Spectrometer.



**Figure 3.** Box-Behnke extraction design.

## RESULTS AND DISCUSSION

### Extraction and Optimization of *Elaeis guinesis* Kernel Oil Extraction

The extraction and optimization of *Elaeis guinesis* kernel oil, using n-hexane and ethanol in RSM and ANNs statistical computational models are employed, the experimental procedure valuation and determination of experimental results and values through design Expert Version 11.1.0.1 software for optimizing the extraction process. The formulas for the models are present as follows:

The experiment generated 17 experimental runs through a series of tests. The three variable independent factors employed were sample weight, solvent volume, and extraction time which are given in Table 1(a) and (b).

**Table 1a.** Independent variables and their levels for Box-Behnken design.

Variable	Symbol	Coded factor levels		
		-1	0	+1
Extraction time (min)	F <sub>1</sub>	30	40	50
Solvent volume ( ml)	F <sub>2</sub>	150	175	200
Sample weight (g)	F <sub>3</sub>	40	50	60

**Table 1b.** Box-Behnken experimental design for three independent variables.

Std run	F <sub>1</sub>	F <sub>2</sub>	F <sub>3</sub>
1	30	150	50
2	50	150	50
3	30	200	50
4	50	200	50
5	30	175	40
6	50	175	40
7	30	175	60
8	50	175	60
9	40	150	60
10	40	200	40
11	40	150	40
12	40	200	60
13	40	175	60
14	40	175	50
15	40	175	50
16	40	175	50
17	40	175	50

### Comparative Analysis of n-Hexane and Ethanol

A critical analysis of the values displayed in Table 2 (a) and (b) showed that the predicted values of *Elaeis guinesis* kernel oil yield for the two solvents were close to the experimented values obtained from the laboratory. In table 2a at 7<sup>th</sup> run (Std), with 175 ml of n-hexane, the highest yield of 37.60% (w w<sup>-1</sup>) was recorded at values of 37.68 and 35.732 for RSM and ANN respectively, in contrast with the same 7<sup>th</sup> run-in Table 2b for the ethanol solvent of 175 ml the oil yield a lower of 26.63% (w w<sup>-1</sup>) with 25.64 RSM predicted value which is lower to the predicted value of ANN at 29.581, at the same values for the remaining independent variables of 30 min extraction time and 60 g sample weight for both solvents. By average computation of 40 min extraction time 175 ml solvents (n-hexane and ethanol) and 50 g sample weight, the average *Elaeis guinesis* kernel oil yield for n-hexane is 38.15% (w w<sup>-1</sup>) and 28.83% (w w<sup>-1</sup>) oil yield for ethanol solvents. This shows that n-hexane is better in terms of oil extraction. However, [Capello et al. \(2007\)](#), opined that ethanol is better in terms of environmental health and renewability since ethanol is less toxic, and renewable.

**Table 2a.** Box-Behnken experimental design for three independent factors for n-hexane, oil yield, predicted, and residual values of RSM and ANN.

Std	F <sub>1</sub>	F <sub>2</sub>	F <sub>3</sub>	EKO Oil yield % (w w <sup>-1</sup> )	Predicted value (RSM)	Residue (RSM)	Predicted value (ANN)	Residue (ANN)
1	30	150	50	30.92	30.88	0.0425	36.311	0.0011654
2	50	150	50	36.86	36.86	-0.0037	32.986	0.003548
3	30	200	50	35.97	35.92	0.0463	34.608	0.012062
4	50	200	50	35.72	35.76	-0.0350	35.968	0.0019233
5	30	175	40	31.56	31.52	0.0350	30.923	0.0032996
6	50	175	40	37.47	37.48	-0.0060	31.572	0.012094
7	30	175	60	37.60	37.68	0.0812	35.732	0.011577
8	50	175	60	37.47	37.48	-0.0060	33.079	0.00077682
9	40	150	40	33.08	34.91	-0.0425	33.068	0.0021047
10	40	200	40	36.31	33.12	-0.0463	34.845	0.0050696
11	40	150	60	34.84	36.36	-0.0812	37.693	0.012968
12	40	200	60	34.62	34.92	0.0038	36.865	0.0046598
13	40	175	50	33.07	34.58	0.0038	37.47	0.030336
14	40	175	50	37.47	33.07	-0.0060	37.47	0.00033598
15	40	175	50	37.47	37.48	-0.0240	37.47	0.00033598
16	40	175	50	37.50	37.48	0.0240	37.47	0.00033598
17	40	175	50	32.99	33.03	-0.0387	37.47	0.00033598

**Table 2b.** Box-Behnken Experimental Design for Three Independent Factors for Ethanol, oil yield, predicted and residual values of RSM and ANN.

Std	F <sub>1</sub>	F <sub>2</sub>	F <sub>3</sub>	EKO Oil yield % (w w <sup>-1</sup> )	Predicted value (RSM)	Residue (RSM)	Predicted value (ANN)	Residue (ANN)
1	30	150	50	26.01	26.21	-0.1975	26.008	0.0016445
2	50	150	50	32.55	32.55	0.0025	32.552	0.0016984
3	30	200	50	33.30	33.51	-0.2050	33.299	0.0010548
4	50	200	50	29.57	29.57	0.0000	29.568	0.0018724
5	30	175	40	26.70	26.50	0.2050	26.702	0.0024643
6	50	175	40	29.57	29.57	0.0000	29.57	0.00041155
7	30	175	60	26.70	26.50	0.2025	26.705	0.0051787
8	50	175	60	29.57	29.57	0.0000	29.57	5.8986E-5
9	40	150	40	36.67	36.47	0.1975	36.663	0.0068047
10	40	200	40	36.42	36.42	0.0050	36.429	0.0085301
11	40	150	60	35.00	35.00	-0.0025	34.999	0.00051272
12	40	200	60	33.61	33.62	-0.0050	33.611	0.001284
13	40	175	50	33.11	33.10	0.0073	29.581	3.5289
14	40	175	50	30.03	30.23	-0.2023	29.581	0.4489
15	40	175	50	29.57	29.37	0.0000	29.581	0.0111
16	40	175	50	25.63	25.64	-0.0073	29.581	3.9511
17	40	175	50	29.57	29.57	0.0000	29.581	0.0111

### Variance Analysis of the Solvents

The equation for the response in terms of coded factors for the Box-Behnken (explained in the methodology) surface quadratic model is used in the computation is given as:

In Table 3(a) showed the regression coefficient and significance response surface quadratic for n-hexane while Table 3(b) showed that of the ethanol solvent. The coefficient of determination ( $R^2$ ) which is derived from the formula as  $(R^2) = (TSS - RSS) / TSS$ ; Where: TSS – Total Sum of Squares =  $\sum (Y_i - Y_m)^2$ , RSS – Residual Sum of Squares =  $\sum (Y_i - \hat{Y})^2$  and  $\hat{Y}$  is the predicted value of the model,  $Y_i$  is the value and  $Y_m$  is the mean value. The coefficient of determination derived for as n-hexane is 99.97%, and  $R^2$  (adj) is 99.92%, while for ethanol ( $R^2$ ) is 99.89% and  $R^2$  (adj) is 99.89% also; the coefficient of determination ( $R^2$ ) for n-hexane is 99.99% and 99.899% for ethanol (ANNs). The two ( $R^2$ ) for both n-hexane and ethanol in RSM and ANNs show a high consistency between the experimented values and the predicted values as seen in Table 4(b) and (d). The  $R^2$  for the two solvents showed average stability between the

experimented values and the predicted values. The lack of fit is the ratio MSLF to MSPE which is given as MSLF/MSPE; where MSLF Lack of fit mean square. MSPE Pure error mean square is 0.0013 for n-hexane and has known for ethanol i.e. the model is significant for the response for n-hexane only. Figures 4 (a) and (b) show the graphs of the predicted and the actual values for the solvents. It was observed that n-hexane gave the highest oil yields compared to ethanol solvent. The analysis in Tables 4(b) and 4(c), gives a clear significance due to the F-value for lack of fit which is 2384.79 for n-hexane and 703.86 for ethanol. This significance is confirmed by Tables 4 (a) and (d) which show the p-value of 0.0001 for the ANOVA of surface quadratic Model of Variance both n-hexane and ethanol respectively.

**Table 3 (a):** Regression coefficient and significance of response surface quadratic for hexane.

Factor	Coefficient estimate	df	Standard error	95%CL Low	95%CL High	VIF
Intercept	15.23	1	0.2615	14.61	15.84	1
F <sub>1</sub>	1.92	1	0.0947	1.70	2.15	1
F <sub>2</sub>	2.19	1	0.0947	1.97	2.41	1
F <sub>3</sub>	26.26	1	0.3528	25.42	27.09	1
F <sub>1</sub> F <sub>2</sub>	1.17	1	0.0312	1.09	1.24	1
F <sub>1</sub> F <sub>3</sub>	-1.60	1	0.0609	-1.74	-1.46	1
F <sub>2</sub> F <sub>3</sub>	-1.26	1	0.0609	-1.41	-1.12	1
F <sub>1</sub> <sup>2</sup>	-2.65	1	0.0304	-2.72	-2.58	1
F <sub>2</sub> <sup>2</sup>	0.1445	1	0.0304	0.0725	0.2165	1
F <sub>3</sub> <sup>2</sup>	-7.64	1	0.1157	-7.91	-7.36	1

**Table 3 (b):** Regression coefficient and significance of response surface quadratic for ethanol

Factor	Coefficient estimate	df	Standard error	95%CL Low	95%CL High	VIF
Intercept	29.57	1	0.0835	29.37	29.77	
F <sub>1</sub>	1.55	1	0.0660	1.40	1.71	1.00
F <sub>2</sub>	3.18	1	0.0660	3.02	3.34	1.00
F <sub>3</sub>	-1.95	1	0.0660	-2.11	-1.80	1.00
F <sub>1</sub> F <sub>2</sub>	0.8825	1	0.0934	0.6616	1.10	1.00
F <sub>1</sub> F <sub>3</sub>	3.01	1	0.0934	2.79	3.23	1.00
F <sub>2</sub> F <sub>3</sub>	1.22	1	0.0934	0.9966	1.44	1.00
F <sub>1</sub> <sup>2</sup>	0.1888	1	0.0910	0.0315	0.3990	1.01
F <sub>2</sub> <sup>2</sup>	-0.2662	1	0.0910	-0.4815	-0.0510	1.01
F <sub>3</sub> <sup>2</sup>	3.25	1	0.0910	3.04	3.47	1.01



**Table 4 (a).** ANOVA for a surface quadratic model of variance table for n-hexane solvent.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	82.43	9	9.16	2348.79	< 0.0001	significant
A-SW (g)	1.61	1	1.61	412.11	< 0.0001	
B-SV (ml)	2.08	1	2.08	534.70	< 0.0001	
C-ET (min)	21.61	1	21.61	5541.20	< 0.0001	
AB	5.45	1	5.45	1398.26	< 0.0001	
AC	2.69	1	2.69	689.77	< 0.0001	
BC	1.68	1	1.68	430.09	< 0.0001	
A <sup>2</sup>	29.52	1	29.52	7571.59	< 0.0001	
B <sup>2</sup>	0.0879	1	0.0879	22.55	0.0021	
C <sup>2</sup>	16.98	1	16.98	4353.90	< 0.0001	

**Table 4 (b).** Analysis of Variance (ANOVA) of regression for n-hexane solvent.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	82.43	9	9.16	2348.79	< 0.0001	Significant
Residual	0.0273	7	0.0039			
Lack of Fit	0.0266	3	0.0089	49.21	0.0013	Significant
Pure Error	0.0007	4	0.0002			
Cor Total	82.45	16		Cor Total	82.45	16
R <sup>2</sup> = 0.9997    AdjR <sup>2</sup> = 0.9992    Predicted R <sup>2</sup> = 0.9948						

**Table 4 (c).** ANOVA for surface quadratic model of variance table for ethanol solvent.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	221.04	9	24.56	703.86	< 0.0001	significant
A-SW (g)	19.28	1	19.28	552.61	< 0.0001	
B-SV (ml)	80.90	1	80.90	2318.50	< 0.0001	
C-ET (min)	30.50	1	30.50	874.05	< 0.0001	
AB	3.12	1	3.12	89.28	< 0.0001	
AC	36.18	1	36.18	1036.89	< 0.0001	
BC	5.93	1	5.93	169.93	< 0.0001	
A <sup>2</sup>	0.1422	1	0.1422	4.07	0.0833	
B <sup>2</sup>	0.2985	1	0.2985	8.55	0.0222	
C <sup>2</sup>	44.58	1	44.58	1277.52	< 0.0001	

Note: df is the degree of freedom

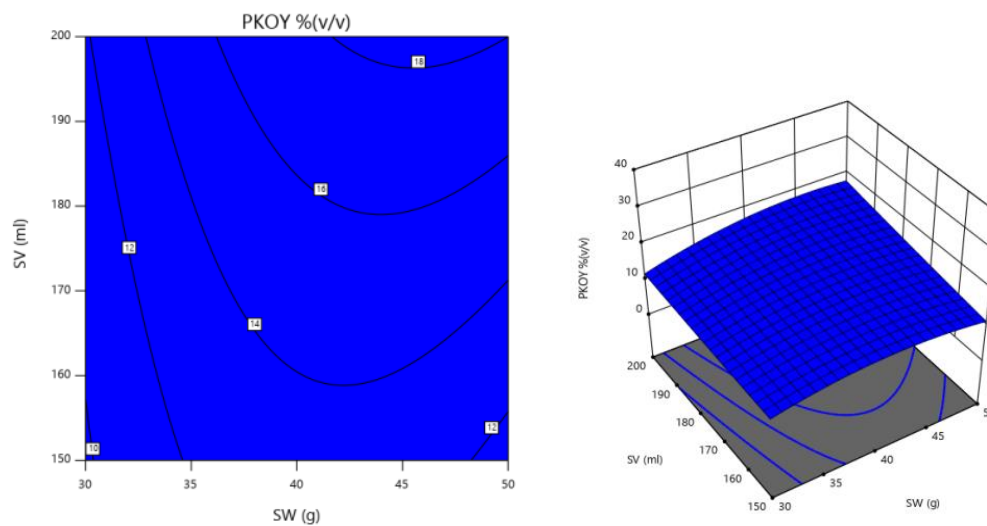
**Table 4 (d).** Analysis of Variance (ANOVA) of regression for ethanol solvent.

Source	Sum of Squares	df	Mean Square
Model	221.04	9	24.56
Residual	0.2443	7	0.0349
Lack of Fit	0.2443	3	0.0814
Pure Error	0.0000	4	0.0000
Cor Total	221.28	16	
R <sup>2</sup> = 0.9989    AdjR <sup>2</sup> = 0.9823    Predicted R <sup>2</sup> = 0.9989			

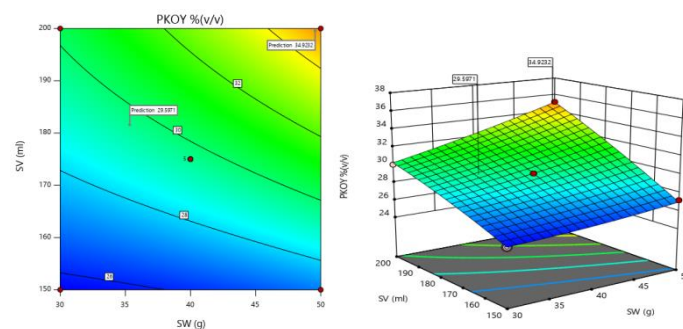
### Evaluation of RSM and ANN in the Oil Extraction Optimization

In Table 2 (a) and (b) the average values of independent variables for 17 experimental runs at an average of 40 min extraction time, 175 ml solvents, and 50 g sample weight

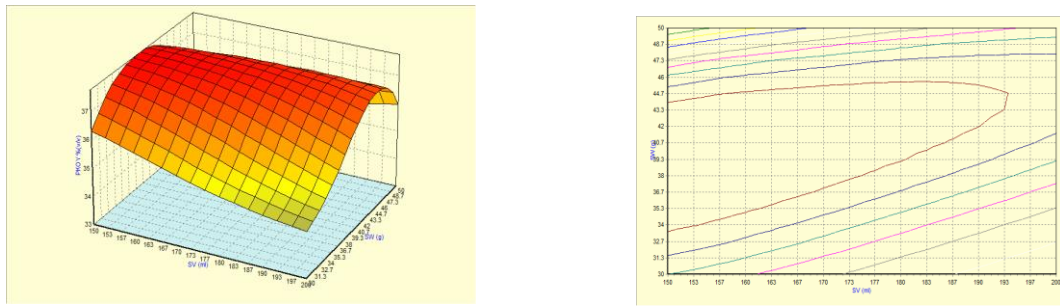
gives an average predicted value of RSM as 35.21 which is less than the average predicted value of ANN which is 37.21 for n-hexane solvent, likewise for ethanol solvent the average predicted value of 31.118 of ANN is higher than the average predicted value of 30.80, this means for both solvents ANN has a higher predicted value for optimization, which means it is a better model for optimization of *Elaeis guinness* kernel oil. Furthermore, the 2D contour and 3D response surface plots are graphic representations of the interactions between two or three variables. The nature of the curves shows that the relationship between the variables, where the elliptical shape is an indication of the good interaction of the two variables and a circular shape indicates no interaction. The contour and graph for both RSM and ANN are presented to evaluate the interactive effect of the three variables via both models. The 2D contour and 3D response surface plots for the two solvents are shown in Figures 3 (a) and (b) for RSM software, the chosen model equation shows the relationship between the independent and the dependent variables, as seen in Figures 4 (a) and (b) for ANN. Figures 3 (a) and 3(b) showed the highest oil yield observed at the lowest sample weight. It was also noted that the highest solvent volume was when the highest oil yield was gotten, meaning that solvents volume has so much significance in the percentage of oil extracted. The same trend is observed in Figures 4 (a) and (b) for the solvents.



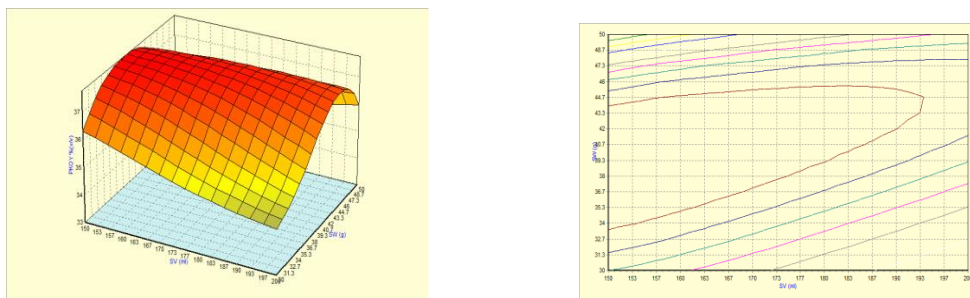
**Figure 3 (a).** The contour and 3D response surface plots for the effects of solvent volume, sample weight and their relationship to oil yield at zero solvent volume for n-hexane.



**Figure 3 (b).** The contour and 3D response surface plots for the effects of solvent volume, sample weight and their relationship to oil yield at zero sample weight for ethanol.



**Figure 4 (a).** The contour and 3D response surface plots for the effects of solvent volume, sample weight and their relationship to oil yield at zero sample weight for n-hexane (ANN).



**Figure 4 (b).** The Contour and 3D response surface plots for the effects of solvent volume, sample weight and their relationship to oil yield at zero solvent volume for ethanol (ANN).

Figure 5(a) and (b) for RSM model and Figure 6 ANNs: The plots showed the effect of extraction time and solvent volume at a reciprocal relation with oil yield when sample weight is at zero level. RSM showed that the optimal yield of palm kernel seed would be 37.68% for n-hexane, at the following optimized conditions: sample weight 40 g, the solvent volume of 175 ml and extraction time of 50 min and 36.67 for Ethanol with the solvent volume of 175 ml and extraction time of 50 min for the optimized conditions. Average values are calculated for optimal factor values in two independent replicates as; 37.038% for n-hexane and 36.045% for ethanol, and this value was well within the range predicted by the model and ANN gave the yield of 37.693% ( $w w^{-1}$ ) at the following conditions sample weight of 40 g, the solvent volume of 150 ml and extraction time of 60 min. This evaluation indicates that even at different independent variables levels and with different solvents that RSM and ANN software can be applied to the optimization.

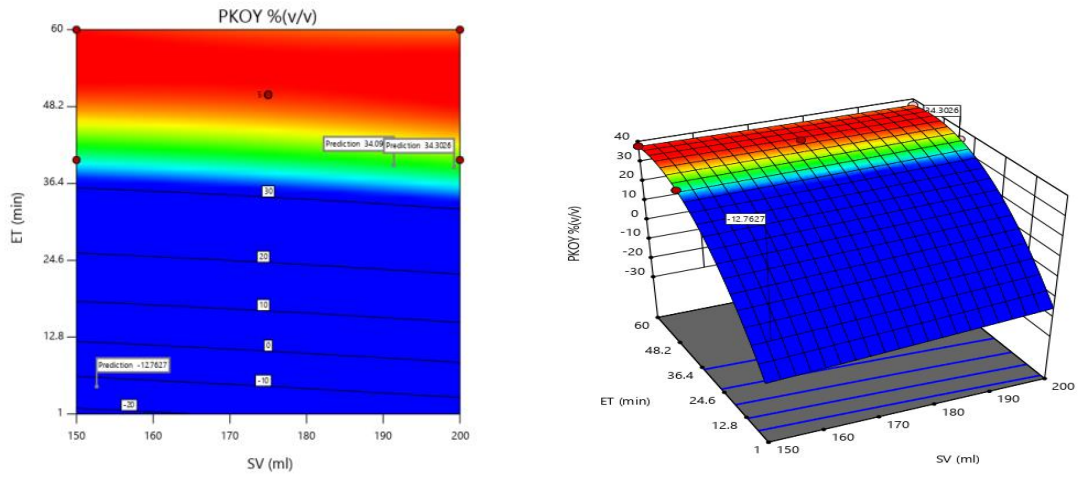


Figure 5 (a). The contour and 3D response surface plots for the effects of extraction time, solvent volume and their relationship to oil yield at zero sample weight for n-hexane.

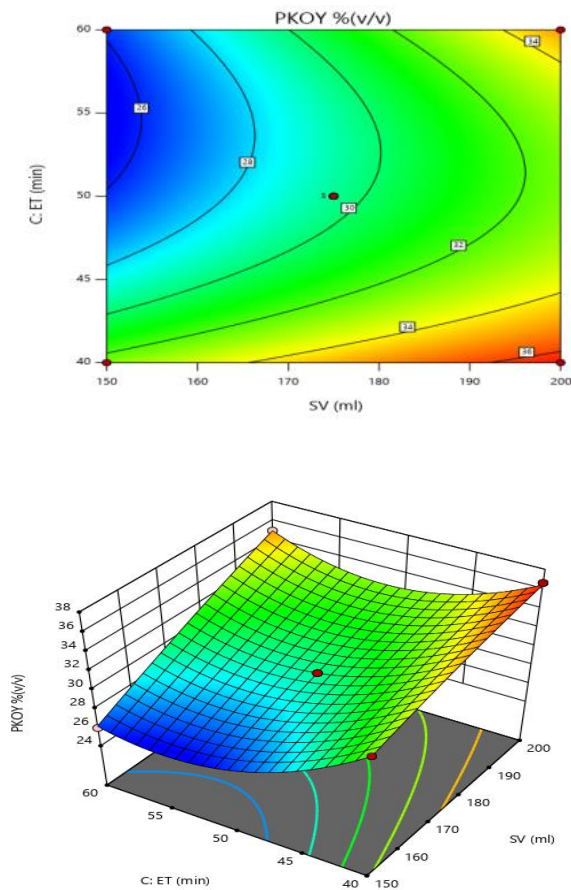
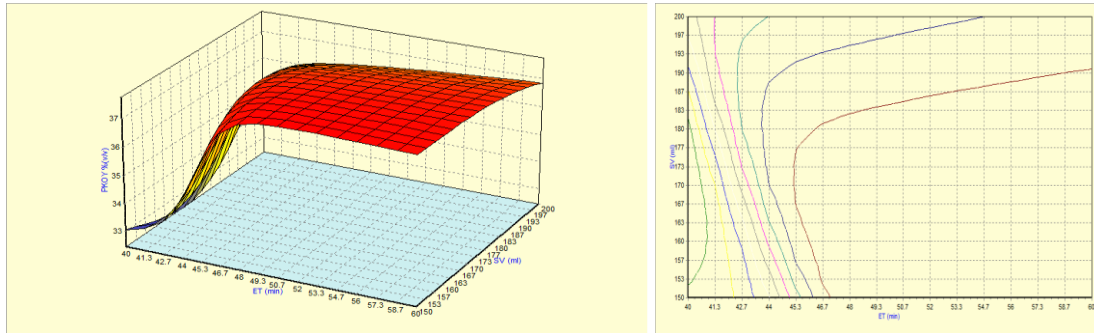


Figure 5 (b). The contour and 3D response surface plots for the effects of extraction time, solvent volume and their relationship to oil yield at zero sample weight for ethanol.



**Figure 6.** The contour and 3D response surface plots for the effects of solvent volume, extraction time, and their reciprocal interaction on oil yield keep sample weight constant at zero level for hexane (ANN).

## CONCLUSION

It is clear that to meet the global need for the use of vegetable oil, extraction and optimization of oilseed is inevitable, therefore this research is conducted using three variable independent factors which are sample weight, solvent volume, and extraction time with a specific investigation on the performance of n-hexane and ethanol as solvents. The n-hexane solvent better term of oil extraction than ethanol, however, [Capello \*et al.\* \(2007\)](#), opined that ethanol is better terms of environmental health and renewability since ethanol is less toxic, and renewable, but n-hexane is a pollutant when emitted during extraction and react with other air pollutants with the product that is hazardous to environmental health. It was also noted that the highest solvent volume was when the highest oil yield was gotten, meaning that solvents volume has so much significance in the percentage of oil extracted. The evaluation indicated that even at different independent variables levels and with different solvents RSM and ANNs software's can be adequately used for optimization in extracting oil, then proved that both models are appropriate for representing the actual relationship of the required factors, and it buttresses the fact that ANNs is better than RSM. It recommended that further research should be concentrate on finding non-toxic solvents that can equal or better yield than n-hexane.

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## DECLARATION OF COMPETING INTEREST

The author declares that she has no conflict of interest.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

The author is responsible for all parts of this article.

## ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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## Optimization of Some Physical and Functional Properties of Extruded Soybean Crud Residue-Base Floating Fish Feed

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### ABSTRACT

The effect of some extrusion factors on soybean crud residue-based floating fish feed was investigated. Extrusion was conducted at 20%, 25%, and 30% moisture content level, die size of 2 mm, 4 mm and 6 mm, and screw speed of 150 rpm, 200 rpm, and 250 rpm. Pearson square method of fish feed formulation was used to attain a 35% protein content of catfish feed protein requirement. Optimized value of extrusion factors moisture content, die size, and screw speed were 30%, 6 mm, and 150 rpm respectively and the optimized result of responses, expansion rate (ER) floatation rate (FR) sinking velocity (SV) specific mechanical energy (SME) swelling capacity (SC) water absorption index (WAI) water solubility index (WSI) hydration capacity (HC) and hydration index (HI) are 32.73%, 95.87%, 0.024 m s<sup>-1</sup>, 16.97 kJ kg<sup>-1</sup>, 1.73, 1.61, 2.76, 0.51, and 0.67 respectively. Feed moisture content and die size have the most significant effect on the physical and functional properties of the extrudate. Coefficient of determination R<sup>2</sup> ranges from 0.65 to 0.96, lack of fit not-significant, desirability in optimization of 0.806, suggesting adequacy of research. Soybean crud residue base floating fish feed has been formed and evaluated with an outcome of high efficacy. This extruded producing model can be used for both domestic and industrial scales of catfish feed production.

#### RESEARCH ARTICLE

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## INTRODUCTION

Legume and grain foods are well known for the supply of food classes to humans and livestock (Kocira, 2019). Soybean (*Glycine max* (L.) Merrill), popular among legume food and grains, with a high content of protein, fibers, fat, and starch (FAO, 2009). It originated from East Asia and is one of the most cultivated legumes in the temperate region (3000 BC) in China and is long recognized in surrounding countries like Japan, Korea, and Malaysia, where it is a long-established cultivated plant (Elvis *et al.*, 2015). Soybean of 40% protein content necessitates additional value chain in its usage on a domestic and industrial scale, being processed to soymilk, tofu, and other soy foods (Sharmila and Athmaselvi, 2017). Soybean Crud Residue (SCR) is the extract for every soybean being processed to soymilk or tofu, after the separation of the molten state (Kamble and Ran 2020). SCR contains 20% protein and fibers 9% inclusive of high moisture content (70%-80%), which makes its handling strenuous, and it is drying exuberant through conventional means. SCR of 1.2 kg was produced as a waste for every 1 kg of soybean processed to soymilk (Bo, 2008). Several tones of SCR were being produced annually in Nigeria with its high nutritional value but being used as a landfill on the refuse dump.

Extrusion cooking has gained recognition in the food processing industry with several operations affecting the physicochemical and functional properties of the food produced for consumer safety (Hongyuan and Alan, 2010). High-Temperature Low Retention Time (HTLT) of extrusion process as making it suitable for processing ready to eat foods mostly made from cereal and leguminous grains like soybean, cowpea, maize, and cassava flour, etc. and production of animal feeds like floating fish feed (Adeleye *et al.*, 2020). Extrusion process operations convert starchy and protein-rich food into viscoelastic resin after gelatinization of starch components, forced out of high temperature and high pressure in the barrel aided by the shear forces generated from the rolling screw. The abrupt reduction in pressure at the die surface allows the quick evaporation of moisture content in the food and expansion of the food, uniform sizing of food was attained at the cutting point on the die orifice (Alam *et al.*, 2016; Nagaraju *et al.*, 2021). Large quantity of soybean crud residue (SCR) being produced from the processing of soybean as being a serious concern in the food manufacturing industry due to the management of this waste and the nuisance it constitutes to the environment (Pelembé *et al.*, 2002). In other to reduce this degradation in our surrounding food engineers and technologist are seeking a management model of the agro-by product (Kamble and Ran, 2020). Production of floating fish feed from this by-product will reduce the cost of fish production since the feeding of fish constitutes 60%-80% of total management costs (Olomola, 1990).

The aim of this research is to investigate the effect of some extrusion condition on the physical and functional properties of soybean crud residue (SCR) base floating fish feed. Response surface methodology (RSM) was used to analysed the statistical result of this experiment. RSM is a collection of statistical techniques for experimentat design, model development, evaluating impact and effect of factors on responses, optimization of factors condition, and level of desirability was used to assess the aim and objectives of this research work.

## MATERIALS and METHODS

### Procurement of Materials

Soybean (*Glycine max* (L.) Merrill) was purchased at the Oja-Oba market in Akure. Cassava starch, fishmeal was purchased in Farm support feed mill Akure.

### Preparation of Soybean Crud Residue

The first operation was to clean about 22 kg of soybean grains using a laboratory aspirator (Vegvari Ferenc Type OB 125, Hungary) to remove dirty and unwanted materials like stalks, leaves, and other foreign matter. It was then soaked in distilled water for 12 hours in an aluminum can of about 35 litres at room temperature to improve its handling. Dehulling was the next stage of preparation this was done manually in a bowl of 50 litres capacity with treated water, it chaffs and some dirty materials have been removed leaving behind well-soaked soybean grains. It was milled using an attrition milling machine (Imex GX 160, Japan) the grain was processed to molten state soybean. The molten state was further mixed with water and sieved by using mesh number 25 (BSI, 1985) to separate the liquid and solid soybean. Soybean crud residue is the wet solid pulverized soybean, it was then poured into a sack bag for dewatering by placing it under a screw jack (3031.14 series, England) pressure increase was consistent for quick and adequate moisture reduction for about three hours. Pulverization of soybean crud residue was achieved using a pulverizing machine (FPP-300, India) and later sun-dried on a mat for a day (at 33°C and 28.21% relative humidity) before packing to polythene bag for further use.

### Formulation of Soybean Crud Residue Base Floating Fish Feed

Pearson square method for determining protein content in fish feed formula was used, The predicted protein content of the feed was 35% after identifying the amount of protein in each of the constituents through proximate analysis, maize contains 10% protein, soybean crud residue 30%, cassava starch 3.6%, wheat bran 17%, soybean meal 40%, fish meal 65%. The constituent was ground properly and mixed thoroughly using a mechanical mixer (335 LP china). Calculation of arithmetic procedures in the Pearson square method is as summaries (Orire and Sadiku, 2015; Enwemiwe 2018).

**Table 1.** Arithmetic result of Pearson square method of fish feed formulation.

	First Group	Second Group
Average	17.3%	37.3%
Percentage of the group in 35% protein.	11.85%	88.30%
First group constituent percentage.	3.95%	29.43%
Percentage of	Components of	
Maize	0.39%	Soybean crud residue 8.82%
Soybean's meal	1.58%	Wheat bran 4.98%
Cassava starch	0.14%	Fish meal 19.11%
Summation of group	2.11%	32.91
<b>Total</b>		<b>35.02%</b>

First group average =  $10 + 40 + 3.6 = \frac{53.6}{3} = 17.9\%$

Second group average =  $17 + 30 + 65 = \frac{112}{3} = 37.3\%$

Component of these feeds were readily locally available food materials, the proportion of each component in the feed are maize 14%, soybean crud residue 35%, wheat bran 25%, soybeans meal 10%, cassava starch 10%, fish meal 5%, vitamin C 0.3%, Methionine 0.4%, antioxidant 0.3% make 100% proportion of every 1 kg of fish feed formulated.

### Extrusion Cooking

Feed formulated was divided into 20 specimens, each was fed into the machine with the aid of a feeder attached to the upper end of the barrel at  $2.0 \text{ kg min}^{-1}$ , and a single screw extruder in the Department of Agricultural and Environmental Engineering Federal University of Technology Akure was used. Moisture content, die size, and screw speed was varied based on experimental design. The screw performs functions of mixing, transporting, and compression of feed, heated by heat generated in the barrel causing cooking effect, gelatinizing feed, improving its physicochemical and biochemical qualities. Experimental samples were collected after gelatinization was attained and were being left in the open air for some minutes to cool, before packing into a polythene bag before further analysis.

### Moisture Content

Moisture content (*wet basis*) of the feed was calculated and determined with the use of modified Equation (1) used by [Twum and Akash \(2018\)](#)

$$\text{Moisture content wet basis} = \frac{W_i - W_f}{W_i} \times 100 \quad (1)$$

Where,  $W_i$  is the initial weight (g),  $W_f$  is the final weight of the sample (g). The amount of water to be added was determined with the use of a modified Equation of [20] in Equation 2

$$Q = \frac{W(A-B)}{100-A} \quad (2)$$

Where,  $Q$  is the amount of water required ( $m^3$ ),  $A$  is initial moisture content (%),  $B$  is final moisture (%) to be attained,  $W$  is the initial weight of feed (g).

### Determination of Physical and Functional Properties

#### Expansion Rate

This is the rate at which the feed expanded immediately after extrusion at the die orifice. Vernier caliper was used to measure the diameter of the die and the diameter of extruded feed. The ratio of the diameter of die and diameter of extruded feed was used to express expansion. The expansion rate was determined using Equation 3 ([Twum and Pare, 2018](#)).

$$Er = \frac{D_1 - D_2}{D_2} \times 100 \quad (3)$$

Where;  $Er$  is the expansion rate,  $D_1$  is the diameter of extruded feed,  $D_2$  is the diameter of the die hole.

### Flotation Test

A flotation test was performed using a transparent conical flask for each treatment. A specific amount of extruded feed was immersed in water and at the end of every observation the number of extruded feed afloat both the initial number and the final number of feeds were recorded; the flotation rate was determined using equation 4 from [Solomon \*et al.\* \(2011\)](#) with slight modification.

$$\text{Flotation rate} = \frac{\text{final number of feed afloat}}{\text{initial number of feed afloat}} \times 100 \quad (4)$$

### Sinking Velocity Test

A sinking velocity test was conducted using a transparent conical flask and some amount of water. The water was filled into the flask to a height of 30 cm and several feeds were poured in it, this was kept constant while the time spent by each sample to reach the bottom of the flask was recorded. Sinking velocity was determined using Equation 5 below.

$$\text{Sinking velocity (m s}^{-1}\text{)} = \frac{h}{t} \quad (5)$$

Where;

$h$  is the height of the water column (m),

$t$  is the time (s) taken by the extrude to reach the bottom of the container.

### Specific Mechanical Energy (SME)

The amount of energy generated from the machine and being converted to energy that transforms the extrude – the work done by the machine on the extrude is called the specific mechanical energy of the machine. This was determined with the use of a mathematical Equation from [Ojo \*et al.\*, 2015](#)

$$\text{SME} = \frac{2\pi \times \tau \times \frac{S_s}{60}}{F_r} \times 3.6 \text{ kJ/k} \quad (6)$$

Where;  $\tau$  is the torque (N m),  $S_s$  is screw speed (rpm),  $F_r$  is feed rate (kg min<sup>-1</sup>).

The calculation of machine torque was done with the use of Equation 7.

$$\tau = \frac{60p}{2\pi N} \quad (7)$$

### Water Absorption Index (WAI)

It was determined by the method used by [Filli \*et al.\* \(2010\)](#) and modified by [Sharmila and Athmaselvi \(2017\)](#) with slight modification. A 2 g sample of each of the feed was mixed with 20 ml distilled water in a centrifuge tube, allowed to stand at the ambient temperature of 25°C for 30 min, and then centrifuged for 30 min at 2,000xg. Decantation of supernatant was done into a stainless-steel pan of known mass and dried to a constant weight at 105°C. The weight of gel in the centrifuge tube was recorded and equation 8 was used to determine WAI

$$\% \text{WAI} = \frac{W_1}{W_2} \times 100 \quad (8)$$

Where,  $W_1$  is the weight of gel in grams (g)  $W_2$  is the weight of extrudate (g)

### Water Solubility Index (WSI)

The water solubility index determines the amount of polysaccharides or polysaccharides released from the granule on the addition of an excess of water. WSI was the weight of dry solids in the supernatant from the water absorption index test expressed as a percentage of the original weight of the sample it was determined with the use of modified method used by [Beuchat \(1977\)](#) and [Gbenyi \*et al.\* \(2016\)](#)

$$(\%) \text{ WSI} = \frac{W_s}{W_d} \times 100 \quad (9)$$

Where,  $W_s$  is Weight (g) of dissolved solids in the supernatant,  $W_d$  Weight of dry solids (g)

### Hydration Capacity and Hydration Index

These were determined by the method used by [Brachet \*et al.\* \(2015\)](#) with some modifications. One hundred feeds were counted and weighed. The feeds were then transferred into a measuring cylinder. About 100 ml of distilled water was added. The cylinder was then covered with aluminum foil and allowed to stay for 12-18 h at room temperature. The water was decanted; superfluous water was removed with the aid of filter paper. The feeds were then weighed, and the hydration capacity was calculated using the following expressions.

$$HC = \frac{W_2 - W_1}{n} \text{ (g feed}^{-1}\text{)} \quad (10)$$

Where,  $W_1$  is the weight of feeds before soaking,  $W_2$  is the weight of feeds after soaking  $n$  is the number of feeds. Hydration index ( $HI$ ) was calculated using the formula below:

$$HI = \frac{HC}{W} \text{ (feed}^{-1}\text{)} \quad (11)$$

Where,  $HC$  is Hydration Capacity per feed and  $W$  is Weight of one feed (g)

### Experimental Design

The statistical analysis of this research was designed using central composite face-centered design (CCFC) in Design Expert 11 Stat-Ease Microsoft Window ([Minneapolis MN USA, 2018](#)) statistical software. Table 2 shows the design procedure and levels of variables used in this experiment. The matrix of this experiment was shown in Table 3 is developed from a central composite face-centered design. Fourteen-star points and six central points made up the experimental space, adding up to 20 runs of the experiment. The model obtained from the experiment was fitted to a second-order polynomial regression model ([Annor \*et al.\*, 2009](#)).

$$Y = P_0 + P_1X_1 + P_2X_2 + P_3X_3 + P_{11}(X_1)^2 + P_{22}(X_2)^2 + P_{33}(X_3)^2 + P_{12}X_1X_2 + P_{13}X_1X_3 + P_{23}X_2X_3 + \varepsilon$$

Where,  $X_1, X_2$  and  $X_3$  are operating parameters, moisture content, die size, and screw speed respectively  $P_0$  is the regression constant;  $P_1, P_2$  and  $P_3$  are linear regression terms,  $P_{11}, P_{22}$  are  $P_{33}$  quadratic regression terms;  $P_{12}, P_{13}$  are  $P_{23}$  the cross-product regression terms;  $\varepsilon$  is the error term.

**Table 2.** Independent variables and their levels of replication.

Factors	Replicate			
	-1	0	+1	
Moisture Content (%),	$X_1$	20	25	30
Die Size (mm),	$X_2$	2	4	6
Screw Speed (rpm),	$X_3$	150	200	250

**Table 3.** Central Composite Face Centered (CCFC) design matrix and the independent factors in their coded and actual values.

Runs	$X_1$	$X_2$	$X_3$	Moisture Content (%)	Die (mm)	Size	Screw (rpm)	Speed
1	0	0	0	25	4		200	
2	0	0	1	25	4		250	
3	0	1	0	25	6		200	
4	0	0	0	25	4		200	
5	0	0	0	0	4		200	
6	0	0	0	25	4		200	
7	0	0	0	25	4		200	
8	-1	-1	+1	20	2		250	
9	+1	+1	-1	30	6		200	
10	+1	-1	+1	30	2		250	
11	0	0	0	25	4		200	
12	+1	+1	+1	30	2		150	
13	0	0	-1	25	2		150	
14	+1	+1	+1	30	6		250	
15	-1	+1	+1	20	6		250	
16	+1	0	0	30	4		200	
17	-1	0	0	20	4		200	
18	0	+1	0	25	6		200	
19	-1	-1	-1	20	2		150	
20	-1	+1	-1	20	6		150	

Key:  $X_1$  = Moisture content,  $X_2$  = Die size,  $X_3$  = Screw speed

### Statistical Analysis

Design expert 11 Stat-Ease Microsoft Window ([Minneapolis MN USA, 2018](#)) statistical analysis software was used in the statistical analysis of data. Analysis of variance (ANOVA) was used to validate the significance of the experimental result, interaction of predicted and observed value was established using correlation analysis of response surface methodology (RSM). RSM gives a three factorial graph combining two factors, showing the effect on each response. Numerical optimization and interactive graphs were used to optimize the various input variables and responses. It generated models for each of the responses with accurate validation to make a further prediction, evaluation, and postulation. The level of desirability shows the reliability of results, this level ranges from 0 to 1. The nearness to 1 the level of desirability the more reliable the result got from the research.

## RESULTS AND DISCUSSION

### Experimental Result Obtained for Statistical Analysis

The central composite face-centered design (CCFC) of design expert 11 developed the experimental design used in inputting and analyzing experimental result data

statistically, Table 4 shows varying levels of dependent variables, moisture content, die size, and screw speed, the inclusion of result gotten for responses. Analysis of this data developed models of quadratic and linear regression, ANOVA result indicates good fit of models with lack of fit not significant and close range between the predicted and observed coefficient of determination  $R^2$ .

**Table 4.** The experimental result was obtained for statistical analysis.

Run	Moisture Content	Die Size	Screw Speed	ER	FR	SV	SMER	SC	WAI	WSI	HC	HI
	%	mm	rpm	%	%	$m s^{-1}$	$kJ kg^{-1}$		$g g^{-1}$	%	$g feed^{-1}$	$feed^{-1}$
1	25	4	200	28	86	0.0245	22	2.2	1.4	2.9	0.3	0.42
2	25	4	250	28	86	0.0245	22	2.2	1.4	2.9	0.3	0.42
3	25	6	200	31	85	0.0232	18	1.7	1.5	2.7	0.4	0.66
4	25	4	200	28	86	0.0245	22	2.2	1.4	2.9	0.3	0.42
5	25	4	200	28	86	0.0245	22	2.2	1.4	2.9	0.3	0.42
6	25	4	200	28	86	0.0245	22	2.2	1.4	2.9	0.3	0.42
7	25	4	200	28	86	0.0245	22	2.2	1.4	2.9	0.3	0.42
8	20	2	250	22	84	0.0262	24	2.1	1.7	2.4	0.4	0.60
9	30	6	150	32	97	0.0233	17	1.8	1.6	2.8	0.5	0.68
10	30	2	250	22	85	0.0264	24	2.1	1.9	2.4	0.4	0.57
11	25	4	200	28	86	0.0245	22	2.2	1.4	2.9	0.3	0.42
12	30	2	150	22	85	0.0264	24	2.1	1.9	2.4	0.4	0.57
13	25	4	150	28	86	0.0245	22	2.2	1.4	2.9	0.3	0.42
14	30	6	250	32	97	0.0233	17	1.8	1.6	2.8	0.5	0.68
15	20	6	250	22	90	0.0231	17	1.8	1.5	2.9	0.5	0.67
16	30	4	200	29	88	0.0244	19	1.6	1.5	2.8	0.3	0.39
17	20	4	200	22	87	0.0245	21	2.0	1.4	2.7	0.2	0.43
18	25	6	200	23	83	0.0261	21	2.1	1.8	2.5	0.4	0.56
19	20	2	150	22	84	0.0262	23	2.1	1.7	2.4	0.3	0.6
20	20	6	150	30	90	0.0231	17	1.8	1.5	2.8	0.5	0.67

### Model Description and Development

Physical and functional properties of resin data generated a regression equation model after statistical analysis was conducted on it and were being presented in Table 5. The independent Factors in the equation model  $X_1$ ,  $X_2$  and  $X_3$  moisture content, die size, and screw speed respectively, significantly affect the responses, expansion rate, floatation rate, sinking velocity, specific mechanical energy, swelling capacity, water absorption index, water solubility index, hydration capacity, and hydration index. Coefficient of determination ( $R^2$ ) of all these dependent variables are 0.77, 0.88, 0.65, 0.94, 0.75, 0.90, 0.88, 0.91 and 0.96 respectively. Lack of fit was not significant for all responses except for floatation rate and water solubility index which is still acceptable.  $R^2$  of 0.60 was used for a good of fit, on the contrary: [Joglekar and May \(1987\)](#) agued that 0.80 value of  $R^2$  for a good fit could be excesive for validation. Also, [Annor \(2009\)](#) narrated that 0.80 (80%) value of  $R^2$  is outrageous in the preliminary study, therefore recommended an  $R^2$  value of 0.60 (60%) as an adequate  $R^2$  value for validation. The predicted and observed value were very close in all the responses indicating a good fit of the models. These are the fitted model of responses below:

$$ER = 27.38 + 1.9X_1 + 3.2X_2 - 0.800X_3 - 0.9483X_1^2 - 2.72X_2^2 + 1.55X_3^2 + 1.5X_1X_2 + 1.00X_1X_3 - 1.000X_2X_3 \quad (R^2 = 0.7750)$$

$$FR = 84.90 + 1.7X_1 + 4.9X_2 + 0.000X_3 + 4.26X_1^2 - 3.33X_2^2 + 2.76X_3^2 + 1.50X_1X_2 + 0.0000X_1X_3 + 0.0000X_2X_3 \quad (R^2 = 0.8823)$$

$$SC = 2.16 - 0.00380X_1 - 0.1722X_2 - 0.0000X_3 - 0.3017X_1^2 - 0.0145X_2^2 + 0.0983X_3^2 - 0.0250X_1X_2 + 0.000X_1X_3 + 0.0000X_2X_3 \quad (R^2 = 0.7530)$$

$$HC = 0.2793 + 0.0200X_1 + 0.0547X_2 + 0.0100X_3 + 0.0017X_1^2 + 0.0970X_2^2 + 0.0517X_3^2 - 0.0125X_1X_2 - 0.0125X_1X_3 - 0.01125X_2X_3 \quad (R^2 = 0.9118)$$

$$HI = 0.4097 - 0.0080X_1 + 0.0411X_2 + 0.000X_3 + 0.0159X_1^2 + 0.1747X_2^2 + 0.0259X_3^2 + 0.0100X_1X_2 + 0.0000X_1X_3 + 0.0000X_2X_3 \quad (R^2 = 0.9680)$$

A quadratic model was formed for all the responses except sinking velocity which has a linear model.

$$SV = 0.0247 - 0.0001X_1 - 0.0013X_2 + 0.000X_3 \quad (R^2 = 0.6523)$$

$$SME = 22.07 - 0.1000X_1 - 3.35X_2 + 0.1000X_3 - 2.17X_1^2 + 0.676X_2^2 - 0.1772X_3^2 - 0.125X_1X_2 - 0.1250X_1X_3 - 0.1250X_2X_3 \quad (R^2 = 0.9415)$$

$$WAI = 1.42 + 0.0700X_1 - 0.1172X_2 + 0.000X_3 - 0.0017X_1^2 + 0.3155X_2^2 - 0.0517X_3^2 - 0.0250X_1X_2 + 0.000X_1X_3 + 0.00X_2X_3 \quad (R^2 = 0.9026)$$

$$WSI = 2.85 + 0.000X_1 + 0.1931X_2 + 0.0100X_3 - 0.0207X_1^2 - 0.3638X_2^2 - 0.293X_3^2 - 0.0125X_1X_2 - 0.01250X_1X_3 + 0.025X_2X_3 \quad (R^2 = 0.8881)$$

**Table 5.** The regression coefficient for physical and functional properties of extrudates.

Coefficients	ER	FR	SV	SME	SC	WAI	WSI	HC	HI
P <sub>0</sub>	27.38	84.90	0.0247	22.07	2.16	1.42	2.85	0.2793	0.4097
P <sub>1</sub>	1.9	1.70	0.0001	-0.1000	-0.0380	0.0700	0.0000	0.0200	-0.0080
P <sub>2</sub>	3.27	4.90	-0.0013	-3.35	-0.1722	-0.1172	0.19311	0.0547	0.0411
P <sub>3</sub>	-0.8000	0.000	0.0000	0.1000	0.0000	0.0000	0.0100	0.0100	0.0000
Quadratic									
P <sub>11</sub>	-0.9483	4.26	-----	-2.17	-0.3017	-0.0017	-0.0207	0.0017	0.0159
P <sub>22</sub>	-2.72	-3.33	-----	0.6767	-0.0145	0.3155	-0.3638	0.0970	0.1747
P <sub>33</sub>	1.55	2.76	-----	-0.1724	0.0983	-0.0517	0.1293	0.0517	0.0259
Interaction									
P <sub>12</sub>	1.50	1.50	-----	-0.1250	-0.0025	-0.0250	-0.0125	-0.0125	0.0100
P <sub>13</sub>	1.00	0.000 0	-----	-0.1250	0.0000	0.0000	-0.0125	-0.0125	0.0000
P <sub>23</sub>	-1.0000	0.000 0	-----	-0.1250	0.0000	0.0000	0.0125	-0.0125	0.0000
Lack of Fit	NS	Sig.	NS	NS	NS	NS	Sig.	NS	NS
R <sup>2</sup>	0.7750	0.882 3	0.6523	0.9415	0.7530	0.9026	0.8881	0.9118	0.9680
Adjusted R <sup>2</sup>	0.5725	0.776 3	0.5871	0.8888	0.5308	0.8150	0.7874	0.8323	0.9392
S. D	2.37	1.79	0.0007	0.7001	0.1384	0.0744	0.0927	0.0361	0.0277

$$Y = P_0 + P_1X_1 + P_2X_2 + P_3X_3 + P_{11}(X_1)^2 + P_{22}(X_2)^2 + P_{33}(X_3)^2 + P_{12}X_1X_2 + P_{13}X_1X_3 + P_{23}X_2X_3 + \varepsilon;$$

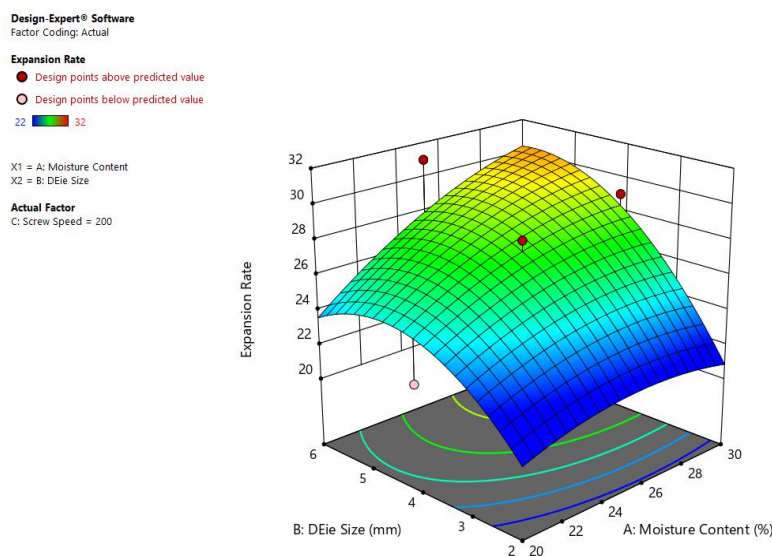
ER = expansion rate, FR = floatation ratio, SV = sinking velocity, SME = specific mechanical energy, SC = swollen capacity, WAI = water absorption index, WSI = water solubility index, HC = hydration capacity, HI = hydration index, NS = not significant, sig. = significant, S.D = standard deviation.

### Expansion Rate

Change in pressure from high rate to low-rate atmospheric pressure in the barrel to the exit lead to flash – off of internal moisture content and vapour pressure being replaced



with air caused a puff called expansion (Emmanuel *et al.*, 2004). Sectional expansion of extrudate is mostly high in the extrusion of starchy constituents than protein foods, soybean has a low expansion rate (Yatin *et al.*, 2015). The expansion rate of extruded soybean crud residue-based floating fish feed was affected positively by moisture content and die size, there was an increase in the expansion rate from 22% to 32% as the die size varied from 2 mm to 6 mm, likewise; moisture content was directly proportional to expansion rate, though; there is the tendency of decrease in expansion rate if moisture content increases further, as it is shown on the graph between expansion range of 22% to 28% at the moisture content of 30%. Most extruded product expansion rate increases with an increase in variation of moisture content level coupled with variation in screw speed level, with the presence of adequate water for expansion of the resin (Nagaraju *et al.*, 2021; Geetha *et al.*, 2016). The die hole on the die head fixed to the barrel of the extruder has a more significant effect on the expansion of the resin than moisture content, this was in line with (Azam and Singh, 2017) in their research on extrusion of Kodo based extrudate. The highest expansion rate was 32% recorded at 6 mm die size and 30% moisture content, ANOVA result shows that the effect of die size and moisture content on expansion rate was significant at both  $p < 0.05$  and  $p < 0.01$  level of significance, lack of fit of the model generated was not significant which validate good fit of the model.

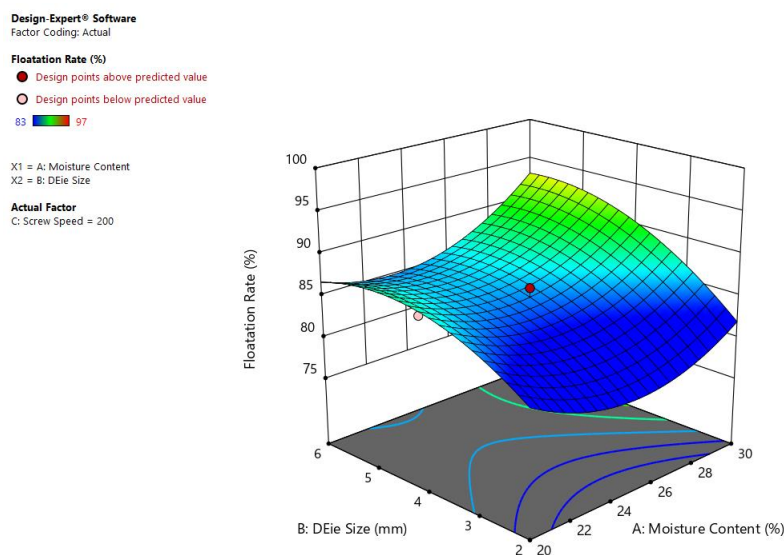


**Figure 1.** Effect of some extrusion factors on the expansion rate of soybean crud residue-based floating fish feed extrudate.

### Floataion Rate

Floataion is the ability of the feed to obey the law of buoyance, using the void created during expansion and reduced bulk density. Airspace formed in the feed during extrusion process being dependent of extrusion factors aided the rate of floataion of extrudate and duration of floating, this was in line with (Orire *et al.* (2015) in the test of buoyance for fish feed. It serves as a device to know how healthy and hardy the fish(s) (Orire and Emine, 2019). Floataion of soybean crud residue-based floating fish feed has the highest value of floataion of 97% being observed at 6 mm die size and 30% moisture content. This was in line with (Olaowale and Oluniyi (2019) research on comparative analysis of sinking time index and water stability of the different levels of cassava flour

and brewer yeast. Floatation was significantly affected by moisture content, figure one showed a decrease in floatation rate between 24% to 28% moisture content. The highest level of floatation was 97% at 30% moisture content and 6 mm die size. [Solomon \*et al.\* \(2011\)](#) Recorded 97% floatation in feed with cassava starch has binder. [Orire and Sadiku \(2019\)](#) also observed a similar reaction in their quest for farm-made floating fish feed. ANOVA result revealed that floatation rate was significant at a p-value of  $p < 0.01$  level of significance, coefficient of determination  $R^2$  was 0.88. 88% of the coefficient of determination  $R^2$  was considered enormous, however; 60%  $R^2$  was used for a good fit in this research work, in other words; 88% coefficient of determination  $R^2$  indicates a very good fit. The close range of our predicted value to our observed authenticate a good fit of the model generated.

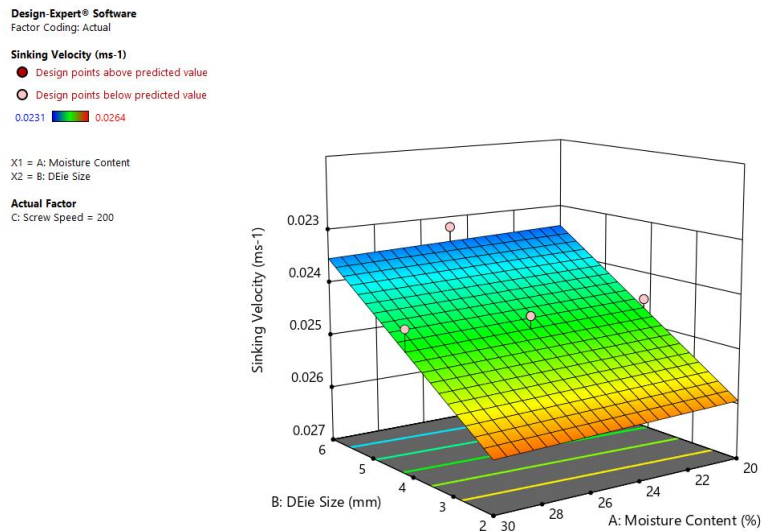


**Figure 2.** Effect of some extrusion factors on floatation rate of soybean crud residue-based floating fish feed extrudate.

### Sinking Velocity

Sinking feed is a challenge that bedeviling aquaculture production over the years, feeds of high bulk density were majorly found to have a fast-sinking rate due to a low level of expansion for void creation during extrusion to aid its floatation ([Efren and Damian, 2018](#)). Its high rate of dissociation in water contaminates the water, giving rise to bad odor, increase in microbial activities and deteriorating biochemical 20, 30 standards of water content, high protein content feeds are in this category soybeans fish feeds are not excluded ([Yatin, 2015](#)). Moisture content and die size have a significant effect on sinking velocity at a p-value of 95%. It was observed that die size and sinking velocity was inversely proportional, 2 mm die size has the highest sinking velocity of  $0.0264 \text{ m s}^{-1}$ , while 6 mm die size has the lowest sinking velocity of  $0.023 \text{ m s}^{-1}$ . [Solomon \(2011\)](#) Also made a similar observation in cassava starch binding agent of formulated floating fish feed. [Olaowale and Oluniyi \(2019\)](#) made a similar inference that sinking velocity could be affected by the type of binding agent and rate of expansion coupled with the quantity of void created during expansion to aid buoyancy and reduce sinking velocity. Die size has more effect on sinking velocity than moisture content, this may be as a result of vapour evaporation at the exit of the die during extrusion. Coefficient of determination  $R^2$  of 0.65 was recorded from ANOVA result, and lack of fit of not

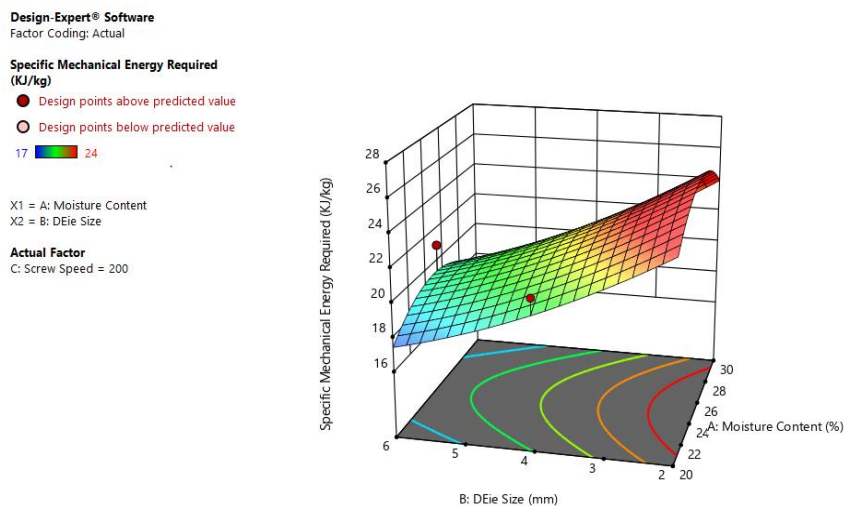
significant was postulated, validating the good fit of the linear regression model formed. However, of all the responses only sinking velocity generated linear model while others were a quadratic model.



**Figure 3.** Effect of some extrusion factors on sinking velocity of soybean crud residue-based floating fish feed extrudate.

### Specific Mechanical Energy

The energy required to process soybean crud residue-based floating fish feed in an extruder is dependent on the moisture content of the resin, die size, and screw speed of the machine. The low moisture content of the resin in the barrel caused clogging at the dying opening, overstressing the screw auger leading to breakage of the power transmission unit, the chain. The hole of the die played a significant role in the flow rate of extrudate we observed that at low orifices there was an accumulation of stress while at bigger die sizes flow rate was high due to allowance of die size. This was corroborated by [Shuyang \(2018\)](#) in his research on the effect of extrusion temperature and moisture on physical, functional, and nutritional properties of kabuli chickpea, sorghum, maize, and their blends. The highest specific mechanical energy was 24 kJ kg<sup>-1</sup> at 26% moisture content and 2 mm die size, while the least was 17 kJ kg<sup>-1</sup>, 6 mm die size and 20% moisture content. The coefficient of determination R<sup>2</sup> of 0.94 coupled with lack of fit not significant validates the good fit of quadratic equation model generated and authenticate the reliability of this work.



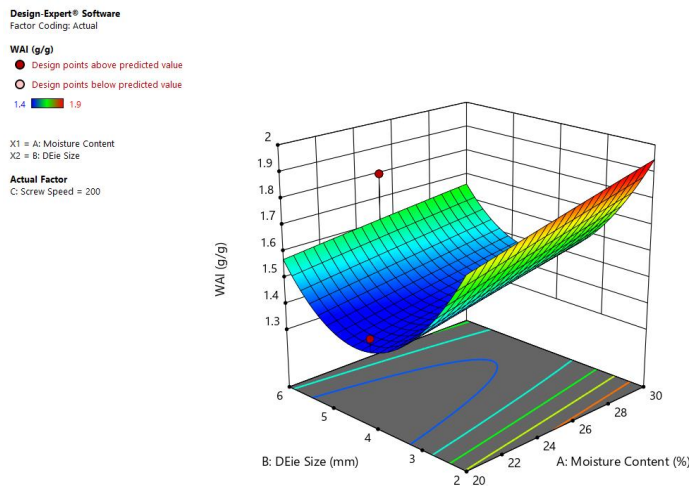
**Figure 4.** Effect of some extrusion factors on Specific mechanical energy of soybean crud residue-based floating fish feed extrude.

### WAI and WSI

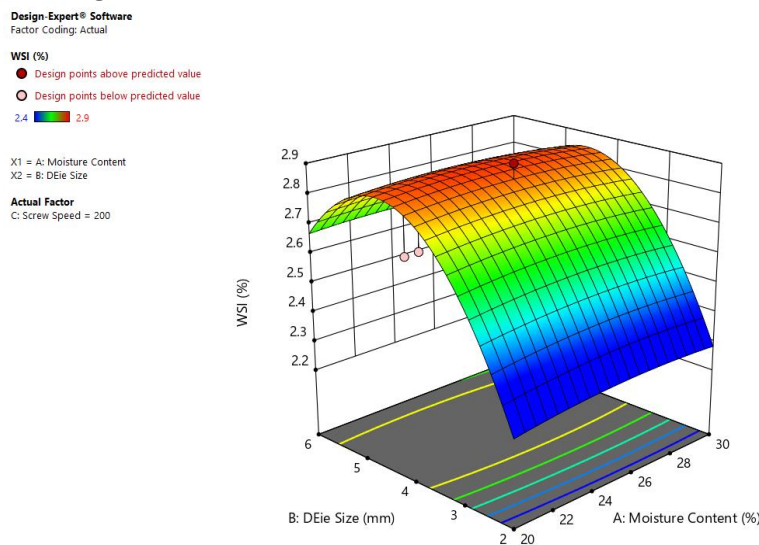
The water absorption index is one dependent feature used to evaluate the quality of extruded products. WAI may influence the hydration capacity of such extruded products. (Sharmila and Athmaselvi, 2017). Water absorption of this extrudate varied from 1.4 to 1.9 g H<sub>2</sub>O g<sup>-1</sup>, this low WAI was also observed by Singh *et al.* (2007) in the extrusion of soybeans flour. Soybeans being an oilseed will have low WAI, the reason being that its oil layer will prevent absorption of water into its layer decreasing WAI of soybeans-based extruded feed (Filli, 2010; Gbenyi, 2016). Starch-based feeds tend to have high WAI, enhanced during gelatinization properly formed by barrel temperature and feed moisture content during the extrusion process (Nagaraju *et al.*, 2021; Gbenyi, *et al.*, 2016). The highest value of WAI 1.9 g g<sup>-1</sup> was obtained at 30% moisture content and 2 mm die size, this may be as a result of an increase in variation of moisture content of the feed. This is in concord with Gbenyi *et al.* (2016) report, increase in moisture content of feed have a significant effect on WAI, moisture acting as a catalytic agent of plasticizer in extrusion cooking affects starch granule and improve water absorption capacity. The coefficient of determination R<sup>2</sup> of 0.902 was obtained from the ANOVA result, indicating a good fit for this research work. Moisture content, die size and screw speed have a significant effect on WAI at the p-value of both p<0.05 and p<0.01. A quadratic model was generated to navigate through the work and lack of fit was insignificant validating the model. The water absorption index serves as a measuring device in determining volume occupied by resin starch after swelling in water, justifying the integrity of starch behavior in aqueous dispersion (Nagaraju, 2021). Swelling capacity, hydration capacity, and their indices were being affected by water absorption index WAI.

WSI: The water solubility index (WSI) of extrudate in this work ranged from 2.4% to 2.9%. WSI of the resin increase with a decrease in die size and later decrease as it reduces in Figure 6. This was interned with Marcin *et al.* (2020) in the extrusion cooking of red and white beans, soybeans as a protein seed were significantly affected with screw speed, die size, and moisture content. Hamada *et al.* (2017) Recorded a decrease in WSI which is a function of the degree of gelatinization and dissolution of starch content. Water solubility index WSI is a function of barrel temperature

influenced by screw speed causing more shear force and tear aided by die size. The extrusion process is high torque low speed and high-temperature low retention time process (Alam *et al.*, 2016). Variation in die size influences clogging during extrusion cooking, low die sizes tend to have clogging than high sizes leading to accumulation of forces consequential of high temperature, which affects the quantity water solubility index (WSI). Pelembe (2002) identifies that starchy food content is not only responsible for WSI but other soluble content like protein. This affects the water solubility index (WSI) of soybeans crud residue-based floating fish feed, moisture content during extrusion has a significant effect on WSI at the p-value of  $p < 0.01$ , coefficient of determination of  $R^2 = 0.888$ , and closeness to the predicted value of  $R^2$  intensify the good fit of the result gotten. WSI was an indicator of the degree of gelatinization of starch components and degradation of molecular components as well as the measurement of the quantity of soluble polysaccharides from the starch of extruded product. The quadratic equation generated has a lack of fit of not significant validating the good fit of the model.



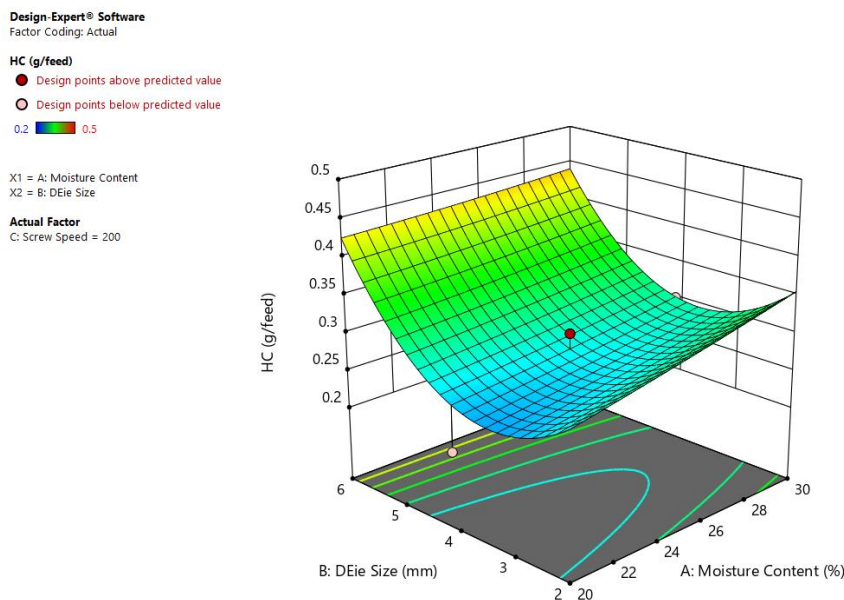
**Figure 5.** Effect of some extrusion factors on WAI of soybean crud residue-based floating fish feed extrudate.



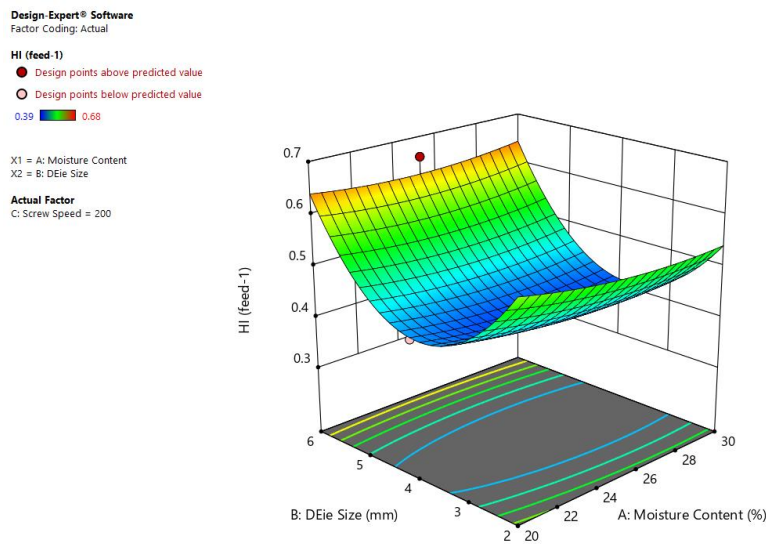
**Figure 6.** Effect of some extrusion factors on WSI of soybean crud residue-based floating fish feed extrudate.

## Hydration Capacity and Hydration Index

Water affinity of plant-based foods enhances the evaluation of hydration capacity, water absorption, and water solubility indices of their functional properties. The polymeric component starch material determines the increase or decrease range of hydration capacity, hydration index, water absorption capacity, water solubility index, and swelling all these are known as water activities of extruded feed (Adeleye *et al.*, 2020). The hydration capacity and hydration index of soybeans crud residue base floating fish feed are shown in figures 8 and 9 hydration capacity of extruded food or feed indicates the potency of feed layers to bind and retain water molecules within their matrix. The highest value of HC is  $0.5 \text{ g feed}^{-1}$  at 30% moisture content, 6 mm die size, and 150 rpm, while HI highest value is  $0.68 \text{ feed}^{-1}$  obtained at moisture content of 30% screw speed of 150 rpm and die size of 6 mm. Extrusion factors have a significant effect on HC and HI at the p-value of  $p < 0.05$ , coefficient of determination  $R^2$  of 0.9118, 0.9680, and a predicted  $R^2$  of 0.8323, 0.9392 respectively. A quadratic model with a lack of fit not significant was obtained from the ANOVA result validating the essentiality of the model and ability to navigate through the model.



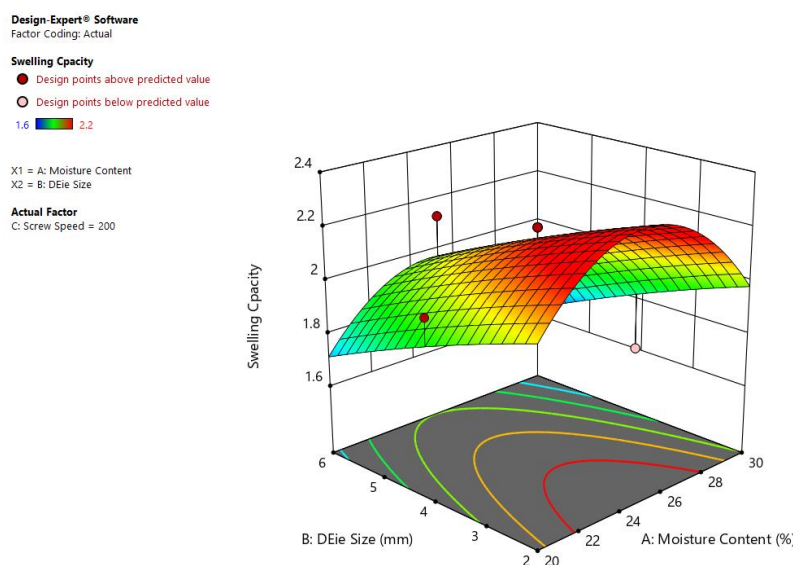
**Figure 7.** Effect of some extrusion factors on HC of soybean crud residue-based floating fish feed extrudate.



**Figure 8.** Effect of some extrusion factors on HI of soybean crud residue-based floating fish feed extrudate.

### Swelling Capacity

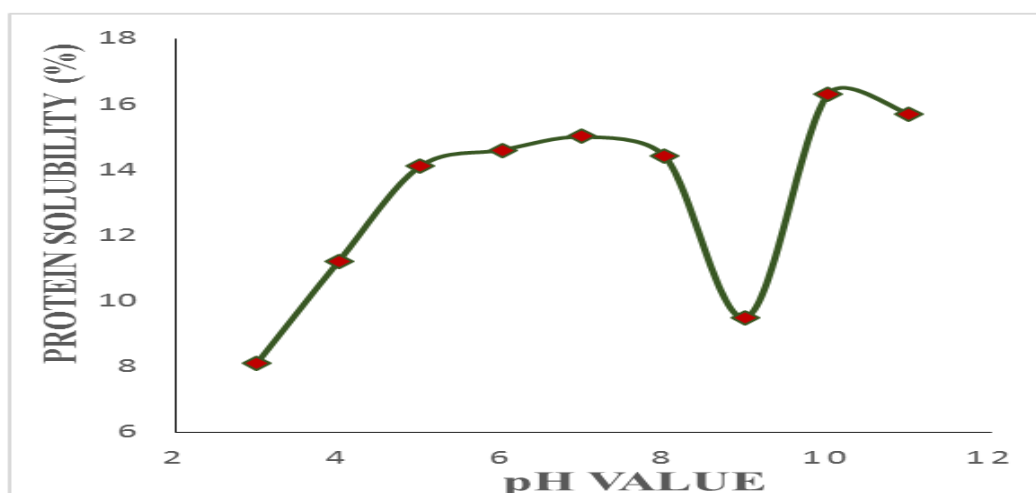
The swelling capacity of extrudate increases inversely to die size increase, but moisture content initially increases with increase in moisture content and has the highest value of swelling capacity 26% moisture content, 2 mm die size, and screw speed of 250 rpm, it later decreases at higher moisture content values. Extrusion factors have a significant effect on swelling capacity at  $p < 0.01$  coupled with the coefficient of determination  $R^2 = 0.7530$ .  $R^2$  of 75% is very appreciable since the bench mark for this study is 60% and the showed a good fit for our experiment. A quadratic model embedded with interactions showed a lack of fit not significant validating the good fit of the model. Swelling capacity is dependent on the water absorption index and both were a function of the starch component in the feed. Feeds with high protein content have a low swelling capacity, soybeans crud residue is excluded from this class, however; this is shown from the result of this experiment.



**Figure 9.** Effect of some extrusion factors on the swelling capacity of soybean crud residue-based floating fish feed extrudate.

### Protein Solubility

The highest percentage of protein solubility was 16.3% at a pH value of 10 while the lowest was 8.1% at a pH value of 3. This graphical representation indicates that protein solubility was of high performance at alkaline medium, due to good digestibility that was obtained at pH of 11, which is of 15.7% value, pH 8 was 18.4%, pH 7 was 15%, pH 6 was 14.6% and pH 5 was 14.1. protein solubility decreases as the pH decreases, only the pH 9 result contravenes this, having a result of 9.5% digestibility. This was in line with what [Pelembé, \(2002\)](#) in their research on protein solubility of fish meal. Solubility of protein in soybean crud residue in both acidic and alkaline medium could be a function of its aggregate. [Shuhong \*et. al.\* \(2013\)](#) corroborate this statement as factual due to the impact of aggregate, size, and fractions, on protein recovery from soybean. The functionality of this feed in the food system is largely dependent on its protein solubility, the more the solubility the better its performance in the food system. The good protein solubility of this feed in an alkaline medium is an indication that the protein isolates for the feed could be extracted by alkaline extraction followed by precipitation at their isoelectric pH. The feed is soluble both in acidic and alkaline mediums, indicating that it can be given to fish in both acidic and non-acidic water.



**Figure 10.** Effect of moisture content and die size on HI of soybean crud residue-based floating fish feed extrudate.

**Table 6.** Laboratory result of protein solubility.

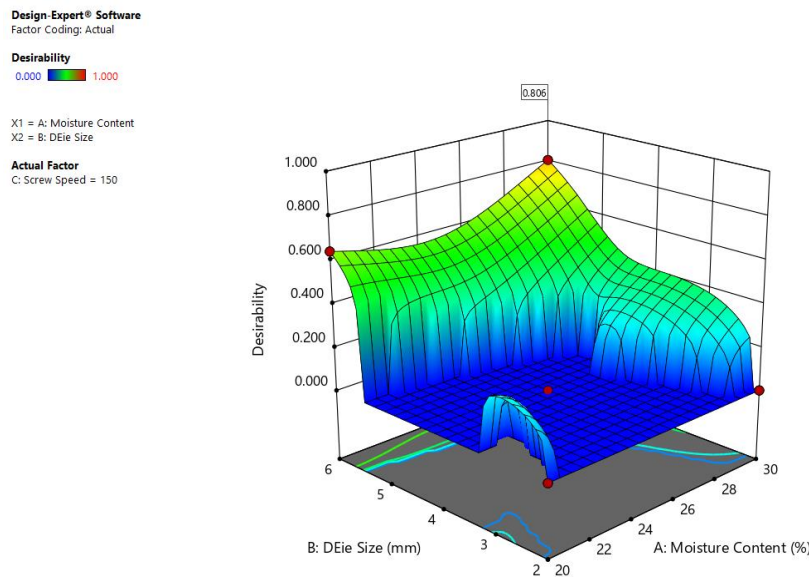
Ph Value	3	4	5	6	7	8	9	10	11
Protein Solubility	8.1	11.2	14.1	14.6	15.0	14.4	9.5	16.3	15.7

### Optimization

Optimization of input variables and responses were carried out numerically and interactive graphs were used in interpreting optimized variables and responses. Variables level was adjusted from interactive optimization plots unit, highest desirability value possible was obtained for each responses. Optimized values of input variables, moisture content, die size and screw speed, are 30%, 6 mm, and 150 rpm respectively. Optimize the value of responses Expansion Rate, Floatation Rate, Sinking Velocity, Specific Mechanical Energy, Swelling Capacity, Water Absorption Index, Water Solubility Index, Hydration Capacity and hydration index were 32.73%, 95.87%,



0.024 ms<sup>-1</sup>, 16.97 kJ kg<sup>-1</sup>, 1.73, 1.61, 2.76, 0.51, and 0.67 respectively. A desirability level of 0.806 out of 1.0 was obtained from the numerical optimization. Desirability showed the idealness of models generated in this research work. Figure 6. Is the desirability graph interpreting the relationship between maximized and the minimized responses during optimization and giving validity of desirability level.



**Figure 11.** Desirability graph of soybean crud residue-based floating fish feed.

## CONCLUSION

A well expanded, instantized soybean curd residue base floating fish feed with good water absorption index, protein digestibility, and low sinking velocity was produced. Soybean crud residue is not waste anymore, but a useful material. The Pearson square method was applied to get the feed formulation. The optimum level of all responses was established from numerical optimization. Feed moisture content and die size have a distinct effect on functional and physical properties followed by screw speed during the extrusion process. Application of central composite face-centered design in Response Surface Methodology (RSM) brought about the 3D graphical representation which shows the relationship of all the three factors and each response at every instance. Postulation made from the result analysis indicates that soybean crud residue base floating fish feed is a good fit for fish feed production. A cheap source of fish feed has been obtained through this research work and an added value to the aqua-cultural sector for the integral production of fish feed and fish.

## DECLARATION OF COMPETING INTEREST

The authors declare that they have no conflict of interest.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

**Saheed Abiola Olaoye:** Investigation, methodology, conceptualization, formal analysis, data curation, validation and review, I made noble contributions.

**Olanrewaju Temitope Owoseni:** Investigation, methodology, conceptualization, formal analysis, data curation, validation and review, and I can be question for my contribution evaluation.

**Ayoola Patrick Olalusi:** I supervised each and every section of this research work and I can be communicated for and cloudy area.

## ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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## Farmers' Participation in Watershed Management in Sodo Zuria District of Southern Ethiopia

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### ABSTRACT

This examination aimed to survey the farmers' participation in watershed management and distinguish significant factors deciding the farmers' participation and cooperation in watershed management practices in the investigation territory. Descriptive statistics and econometric models were utilized for investigation purposes to meet the expressed targets. The sampled farmer families were classified low, medium, and highly dependent on their support score esteems the 4-8, 9-12, and 13-16 participation run separately. The farmers' participation in problem identification and decision making, planning and monitoring and evaluation is shallow in contrast with their participation in the execution level. The Ordered Logit Model outcome uncovered that among the 17 variables estimated to influence the farmers' participation in watershed management, 8 variables were measurably noteworthy with the speculated sign as determinants of farmers' participation in the watershed the executives. Consequently, the family size is positive and significantly influence the farmers' participation; dependency ratio negatively and significantly influences the farmers' participation; more dependency ratio diminishes time, work, and enthusiasm to partake in watershed management practices, education positive and fundamentally influence the farmers' participation, farm size is positive and significantly affect the farmers' participation, the distance of parcel of land from residence positive and altogether influence the farmers' participation, soil fertility positive and altogether influence the farmers' participation, extension contact positive and altogether influence the farmers' participation, farmers households who approach credit were found to have negative and fundamentally influence the farmers' participation.

#### RESEARCH ARTICLE

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- Farmers participation,
- Ordered Logit Model,
- Participation index,
- Slope of land,
- Soil erosion,
- Watershed management practices



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## INTRODUCTION

Ethiopia is one of the creating nations in which land assets are getting progressively scant, and the nature of assets, for example, soil water, plants, and creatures are diminishing because of misuse and management. Watershed degradation: land degradation as soil disintegration, sedimentation, depletion of soil supplements, deforestation, and overgrazing are essential issues confronting the farmers in Ethiopia. This restricts their capacity to increase agricultural production and decrease poverty and food insecurity (Temesgen, 2012).

To address the watershed degradation, the comprehensive watershed management practices were dispatched in Ethiopia, particularly after the starvation of the 1970s. Starting now and into the foreseeable future, immense regions have made sure about terraces, bunds, and a massive number of trees have been planted. Even though various watershed management techniques have acquainted with battle watershed degradation, the adoption of these practices stays underneath desires (Yeraswork, 1998).

Regardless of considerable advancement in watershed management in southern Ethiopia, watershed degradation is as yet proceeding. Watershed management is fundamental for monitoring water, land, and biodiversity, redesigning neighborhood jobs, improving the economy of the inhabitants' people.

The impact of watershed degradation is severe in the highlands of the country (areas that lie above 1500 m), which constitute less than half of the country (43 percent of the country). Because of its good atmosphere for production and presence of moderately more fertile soils just as less sickness rate, the Ethiopian high lands have about 88% of the national populace (FAO, 2000).

Watershed degradation especially erosion and the decrease in humus substance of soils lessen penetration limit of soils and soil moisture and storage capacity. This way, reduction in infiltration and moisture storage capacity of soils diminishes the limit of yields to withstand drought. Thus, manageable variations in rainfall become catastrophic events with watershed degradation. Many exploration concentrates in Ethiopia credited the chronic poverty, fundamental food insecurity and repeating starvation halfway to the natural catatropical damages finally (Woldeamlak, 2003).

Perceptive of these issues, watershed management practices have actualized in numerous pieces of the highlands during the 1970s and 1980s. They were introduced in some degraded and food insufficiency districts fundamentally through food-for-work motivating forces. The significant kinds of management practices introduced were essential, and the most notable was the bench terraces and ordinary bunds (Bekele, 2007).

The examination's overall goal was to assess the farmers' participation in watershed management in Sodo Zuria Woreda, Wolaita Zone. The study's specific targets were to look at the farmers' current status in the watershed management in the examination

region and recognize the factors impacting the farmers' cooperation in watershed management in the assessment zone.

## MATERIALS AND METHODS

In this investigation, to have a fair portrayal of the populace and catch a representative sample; a multi-stage sampling procedure has utilized to choose sample family units. Ethical Committee approval was obtained for the study.

In the main stage, out of the 12 rural woredas in Wolaita Zone, one Woreda (Sodo Zuria) and from 31 Kebeles of the Woreda, 3 Kebeles have picked purposively. In the second stage, 3 watersheds have selected from one from every 3 Kebeles by purposively. In the third stage, sampling frame (complete watershed farmer household lists) was obtained from each Kebele Administrative Office and then by using the Probability Proportional to Sample Size methods the sample households from each Kebele have selected according to the number of household in it. Finally, 90 respondents have drawn by using a systematic random sampling technique (Figure 1).

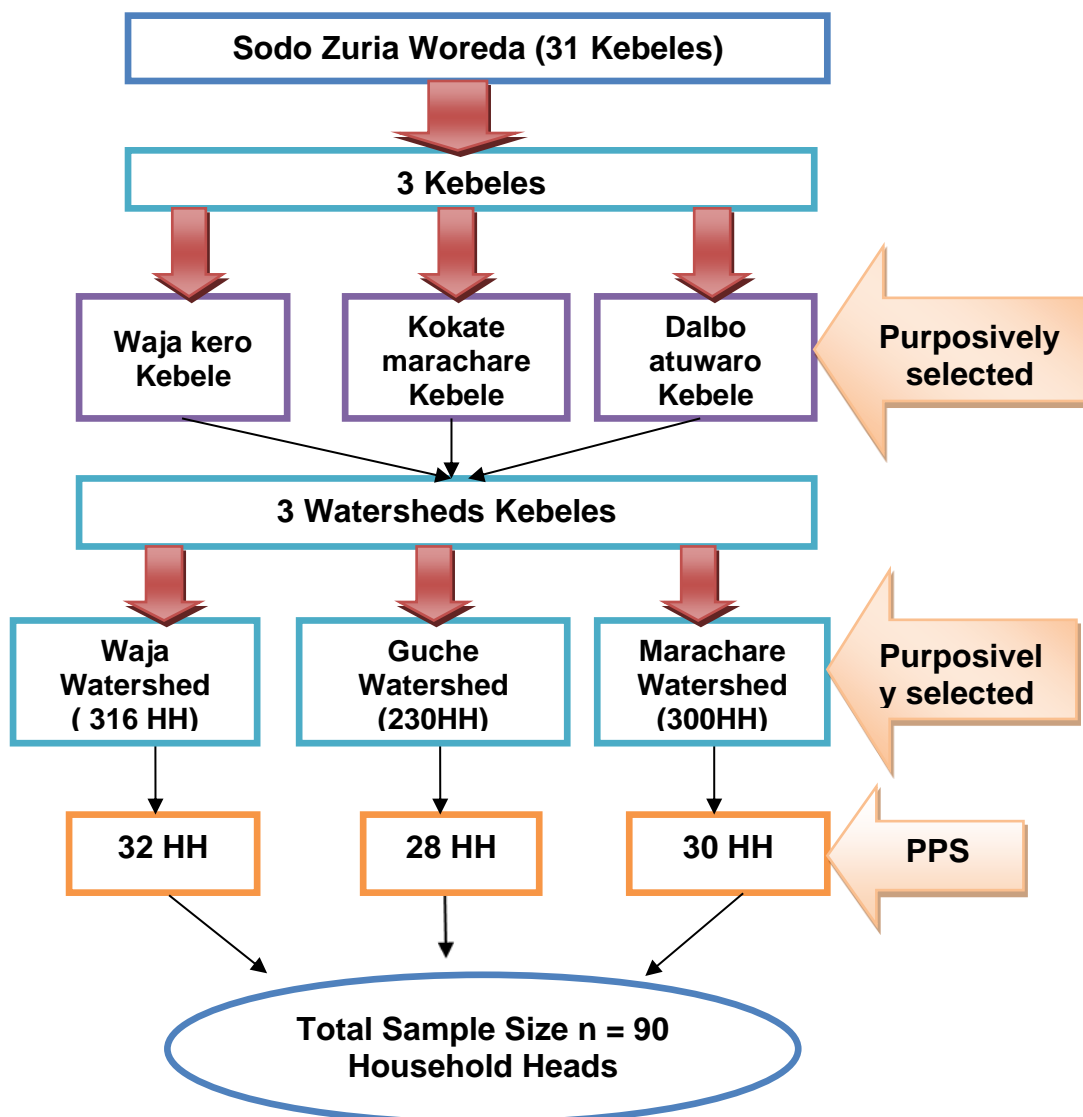


Figure 1. Random sampling technique.

### Sample Size Determination

A significant choice that has made while choosing a sampling technique is about the size of the sample. Suitable example size relies upon different variables identifying with the subject under investigation, including time, cost, and precision level. When sample size is minimal; the goals of our research may not be tended to definitely. So proper sample size was applied to get a good representative data.

Because of its easiness work with the simplified formula for proportions suggested by [Yamane \(1970\)](#) has used to calculate the sample size. It assumes that a 90% confidence level, e is the error margin between (5-10) percent.

Assuming  $e = 10\%$

$$n = \frac{N}{1 + N(e)^2}$$

Where n is the sample size, N is the Universe of the population size of 3 watersheds which have been 846 HHs, e is the extent of precision that assumes  $e = 0.10$ . When the formula has applied to the above sample, the sample size is necessary for the study has shown below as follows.

$$n = \frac{846}{1 + 846(0.10)^2} = 90$$

The farmers' participation in watershed management activities has estimated by putting the indicator exercises with their score assessment of frequencies.

The respondents have asked the amount they were sharing an interest in those exercises. This relies upon their intervention as Frequently, Occasionally, Seldom, and Never and the focuses have granted for each response with good scoring esteems as 4, 3, 2, and 1 independently. The repeat counts of reactions have recorded to handle the Participation Index (PI) of a farmer for all of them picked works out, so the index built by appointing equivalent loads to every reaction.

**Table 1.** Indicators to measure the farmers' participation and their given score values.

S. No.	Types of Indicators	Nature of Participation	Value Given
1	Problem Identification and Decision	Never	1
2	Planning	Seldom	2
3	Implementation	Occasionally	3
4	Monitoring and Evaluation	Rare	4

The respondent's score could be extended from 4 to 16, where 4 exhibits all the farmers are not participating in some random action and 16 shows the significant interest of all farmers in that development that infers all are frequently partaking ([Tilahun, 2008](#)).

The recurrence checks of responses have recorded to enlist the Participation Index (PI) of a farmer for all of them picked works out. By then the Participation Index for each activity has been figured by using the going with the equation;

$$\text{Participation Index (PI)} = (N1 \times 4) + (N2 \times 3) + (N3 \times 2) + (N4 \times 1) \quad (1)$$

Where:

PI=Participation Index for different activities of Participation in the watershed management

N1 = Farmer who participate **Frequently**

N2 = Farmer who participate **Occasionally**

N3 = Farmer who participate **Seldom**

N4 = Farmer who **Never** Participate

To attain the first objective, this is "to analyze the current status of watershed management practice in the investigation area." the study employed participation index. The index has determined from important indicators of farmer Participation in watershed management found from the literature review. The scores of these exercises were resolved for each respondent and changed over them into critical record esteem a motivating force as [Roman \(2010\)](#) used a similar strategy to gauge the admittance to and use of family arranging information among country women and the strengthening status of the rural women by calculating the scores obtained from the different pointers.

So as to accomplish the second objective, "to recognize the factors that impact the farmers' support in watershed management in the examination region", ordered logit model has utilized. Since the Dependent variable accepts ordinal nature, the ordered logit is generally favoured when contrasted with different models.

The ordered logit model was used due to the arranged idea of the dependent variable. The usage of a reasonable model has commonly directed by the concept of the dependent variable or elements. In this examination, the dependent variable is categorical or ordered nature. At that point, the Ordered Linear Regression hasn't adequately given the variable's non-span nature, and the scattering of the outcome choices can't be uniform. Ordinal logit and probit models have been commonly used to look at such kinds of information ([Liao, 1994](#)).

Some polychotomous Dependent factors have unavoidably requested. Even though the outcome is discrete, the multinomial logit or probit models would disregard to speak to the ordinal idea of the Dependent variable ([Greene, 2008](#)). The arranged probit and logit models have come into wide use to separate such reactions ([Zavoina and MacElvey, 1975](#)). Accordingly, the Ordered Logit Model has used to overview the farmers' Participation's determinant having three particular classes. That is Low, Medium, and High support classes.

By following [Greene \(2008\)](#) and [Liao \(1994\)](#), the utilitarian form of the ordinal logit model has indicated as follows:

$$y^* = \sum_{k=1}^k \beta_k + \varepsilon \quad (2)$$

$y^*$  is in secret and along these lines can be the idea of as the hidden propensity of an observed phenomenon

$\varepsilon$  is accepted where it follows a specific symmetric distribution with zero methods, for example, standard or logistic appropriation. What has noticed is

$$\begin{aligned} y &= 1 && \text{if } y^* \leq \mu_1 \\ y &= 2 && \text{if } \mu_1 < y^* \leq \mu_2 \\ y &= 3 && \text{if } \mu_2 < y^* \leq \mu_3 \\ y &= j && \text{if } \mu_{j-1} < y^* \end{aligned} \quad (3)$$



Where  $y$  is observed in  $j$  number of ordered categories, are unknown threshold parameters separating the adjacent categories to be estimated with The general form of the probability that the observed  $y$  falls into category  $j$  and and the are to be estimated with an ordinal logit model is:

$$\text{Prob}(y = j) = 1 - L \left( \mu_{1-1} - \sum_{k=1}^k \beta_k x_k \right) \quad (4)$$

Where  $L(\cdot)$  speaks to the total logistic appropriation

[Tilahun \(2008\)](#) and [Roman \(2010\)](#) have identified three categories of the respondents: low, medium, and high dependent on their different score values. The current investigation depended on their order that is low, medium, and high. Relies upon considering the mean worth score assessment of the respondents got for the whole exercises. Consequently, the respondents' classification dependent on their participation score value was 4-8, 9-12 and 13-16 for low, medium, and high classifications independently.

## RESULTS AND DISCUSSION

To decide the sample respondents' participation status, Participation Index has set up by utilizing four indicators of investment in Watershed management found from the literature survey. The ranchers' investment level has estimated by figuring the score estimations of every respondent based on the allotted indicators exercises.

The base score esteem was 4, and the prominent score esteem was 16 for each respondent. The results from the indicators in Table 3 revealed that 26 (28.9%) of the inspected respondents are under low-interest level, 53 (58.9%) are under medium support level, and 11(12.2%) of the respondents are under the high level of participation.

**Table 2.** Participation level and their score ranges.

Categories of Participation	Number	%	Participation Score
Low	26	28.9	4-8
Medium	53	58.9	9-12
High	11	12.2	13-16
<b>Total</b>	<b>90</b>	<b>100</b>	<b>4-16</b>

**Ordered Logit model** has used to recognize the elements influencing the farmers' in watershed management in the investigation region. Subsequently, factors theorized to affect the Farmers' participation in watershed management have tested in the model and out of 17 explanatory variables 8 of them have discovered to be significant. Among those factors tested into the model; educational level, dependency ratio, family size, land size, distance to the land from residence, soil fertility, extension contact and access to credit have discovered to be significant at 1%, 5% and 10% likelihood levels.

**Table 3.** Output of the Ordered Logit Regression Model.

Explanatory variables	Estimate	Std. Error	Wald	Sig.	Odds
AGE	0.37	0.40	0.896	0.344	1.038
EDULVL	0.644*	0.337	3.656	0.056	1.904
DEPNDCY	-4.045**	1.971	4.212	0.040	0.017
FMSIZE	0.322*	0.170	3.596	0.058	1.379
FARMSIZE	1.116***	0.342	10.631	0.001	3.053
TLU	0.016	0.124	0.016	0.899	1.016
SLOPE	-0.590	0.479	1.519	0.218	0.554
DISTANCEPCL	-5.291***	1.372	14.866	0.000	0.005
OFFFARM	0.852	0.666	1.639	0.200	2.344
FERTLTY	1.660*	0.884	3.523	0.061	5.259
EXTCONT	1.849***	0.555	11.107	0.001	6.353
SOURCELND	0.454	0.863	0.277	0.599	1.575
LANDSECUR	1.798	1.197	2.257	0.133	6.037
TRAINING	0.681	0.887	0.590	0.442	1.976
CRDTACCESS	-3.880***	1.139	11.606	0.001	0.021
PERCNSEP	1.288	1.704	0.572	0.449	3.625
NEWTECHAS	-0.854	1.044	0.668	0.414	0.426

Note: \*, \*\* and \*\*\* = significant at 10%, 5% and 1% probability levels respectively.

Dependent variable: Farmers' Participation in Watershed Management

-2Log likelihood= 165.554

Chi-square = 86.613

Significant level: 0.000

The result of the Ordered logit regression on farmers' participation in watershed management has summed up in Table 3. From 17 theorized logical factors, eight of them are a significant effect on the farmers' participation in watershed management. These eight variables are educational level, dependency ratio, family size, land size, distance to the land from home, soil fertility, extension contact, and access to credit. However, the other nine explanatory variables, for example, the age of the respondents, number of livestock holding, the slope of the land, off-farm employment, source of land, land tenure security, training, perception of soil erosion as a problem and new technology acceptance do not have significant support on farmers' participation in watershed management.

**Education** influences farmers' decision to accept new technologies of watershed management by enhancing farmers' ability to obtain, understand and utilize the practice and improve the overall managerial ability of farmers. Therefore, more education to a farmer means more participation in watershed management practices. Education has estimated to have a positive effect on farmers' participation in watershed management. The model yield additionally underpins the theory. It shows that an expansion in long periods of education would bring about a 1.904 figure increment the ordered log-odds for being in a higher interest level. Simultaneously, various elements in the model has held consistent. The eventual outcome of this finding is dependable with the results of [Long \(2003\)](#).

**Dependency Ratio** indicates the number of people in a family who are not monetarily dynamic affects farmers' participation in watershed management negatively at 5% significant level. This is because in the families with a more massive consumer to worker ratio, an economically active member of the household shoulders the

responsibility of feeding many people. To fulfill this responsibility, they engage in other activities which divert the resource (attention, labour, time...) away from their watershed management practice in general. Hence, they fail to give attention to watershed management practices. The model yield likewise underpins the theory and shows that the dependant family size has negatively influenced farmers' participation in watershed management practices. It shows that a unit increase in the number of dependents in given households would bring about a 0.017-factor decline in the ordered log-odds for being in a higher participation level. Interestingly, different elements in the model are held steady. The consequence of this finding is predictable with [Bekele's \(1998\)](#) results and [Wagayehu and Lars \(2003\)](#).

**Family Size** is the number of household members living together in terms of adult equivalent. It has estimated that it has a positive relationship with the dependent variable. This could be because managing watershed is labour-intensive if household labor is the only source of labor; households with larger household sizes participate in watershed management in general. The model yield also influences the hypothesis and shows that family size has positively affected the farmers' participation in watershed management at 10% likelihood level. It shows that when one unit of adult joins in the family would bring about a 1.379-factor increment in the ordered log-odds for being in a higher participation level, the other factors in the model are held constant. The aftereffect of this finding is predictable with the results of [Woldeamlak \(2003\)](#). However, it disagrees with the results of [Bekele \(1998\)](#). This states that family size is negatively related to farmer participation in watershed management.

**Farm Size:** Taking conservation measures in the watershed can be expensive and risky ([Long, 2003](#)) as the Physical Conservation measures impose a higher cost in terms of the land they put out of production ([Wagayehu and Lars, 2003](#)).

In various investigations led in Ethiopia, it has accounted for that conservation estimates take 10-20% of development land through embankments and earthen channels ([Campbell, 1991](#)) and land removed from cultivation increases quickly with expanding slope ([Belay, 1992](#)). This makes the advantage that will be acquired from saving the soil in little homesteads to be less disposed to compensate for diminishing yield on account of actual protection measures ([Wagayehu and Lars, 2003](#)). It was hypothesized that it has a positive effect on farmers' participation in watershed management. This is because the farmers' huge landholding size can hold up under danger of loss of productive land from conservation structures of the watershed. The model yield also underpins the theory, what's more, it shows that ranch size has influenced the support of the farmers' in watershed management emphatically huge at 1% likelihood level. It shows that a farmer holds more hectare of land would bring about a 3.053-factor increment in the ordered log-odds for being in a higher participation level, while the other factors in the model have held consistent. The outcome of this finding is reliable with the consequences of ([Belay, 1992](#)).

#### **Distance of a Parcel of Land from Farmers' Residence:**

The separation of a land package from farmers' habitation speaks to how far the bundle of land has arranged from home, as indicated by farmers' evaluations.

Farmers residing close to their cultivation land invest more on watershed management practices than their counterparts living at a distance. This is because land cultivation closer to the residences receives more attention and supervision than land situated at the farthest distance. Farmers also want to invest more in the field that requires the least effort. It was hypothesized that distance influences farmers' participation negatively. The model yield likewise bolsters the theory and shows that the land's distance affects the farmers' participation in watershed management negatively significant at 1% likelihood level. The negative association implied that the farmers' cultivating land at the farthest distance is less likely to take part in watershed management practices. It shows that the distance of a parcel of land becomes far from the farmers' residence would bring about a 0.005 figure decline the Ordered log-odds for being in a higher support level, while various model elements have held consistent. The result of this finding is unsurprising with the results of [Kessler \(2006\)](#).

The model output also supports the hypothesis and shows that distance has influenced farmers' participation in watershed management negatively and significantly at 1% probability level. The negative association implies that farmers cultivating land at the farthest distance are less likely to participate in watershed management practices. It shows that the length of a parcel of land becomes far from the farmers' residence would result in a 0.005-factor decrease in the ordered log-odds in favor of being in a higher participation category, while the other variables in the model are held constant. The result of this finding is consistent with the results [Kessler \(2006\)](#).

**Soil Fertility:** Farmers' perception about soil fertility status of the land they cultivate influenced farmers' participation in watershed management positively and significantly at 10% probability level. Even though it was hypothesized that soil fertility influences farmers' participation negatively, the model output results that soil fertility has a positive association with farmers' participation in watershed management practice. This shows that the farmers who cultivate black soil (intermediary to rich soil in the zone) participate more in watershed management practices. The model yield also bolsters the speculation and shows that soil fertility has affected the farmers' cooperation in watershed management. It shows that an additional increase in soil fertility would result in a 5.259-factor increment in the Ordered log-odds for being in a higher cooperation level. In contrast, different factors in the model have held consistent. The consequence of this finding is predictable with the results of [Wagayehu and Lars \(2003\)](#). However, it contrasts with the conclusions of [Osgood \(1992\)](#), [Valk and Graff \(1995\)](#). They found that farmers invest more on none fertile land.

**Extension Contact:** The recurrence of extension contact with Development Agents has positively influenced the farmers' participation in the watershed management at 1% likelihood level. The positive association indicates that the farmers having close contact with DAs appear to better partake in watershed management practices. This is because; farmers reduce the risk associated with watershed management practices by obtaining adequate information. The frequent extension contact with DAs makes accurate and timely information readily available to farmers. It has hypothesized that extension contact influences farmers' participation positively. The model yield also underpins the theory and shows that extension contact has influenced the farmers' participation in watershed management. It shows that an increase in extension contact would bring

about a 6.353 factor increment in the Ordered log-odds for being in a higher cooperation level, while different model components have held constant. The research finding results are reliable with the results of [Benin \(2002\)](#), [Wagayehu and Lars \(2003\)](#).

**Access to Credit:** Access to Credit represents a means by which a farmer accessed credit to invest in watershed management practices. Access to finance, both saving and credit, helps give the aggregates needed to put resources viz., land, housing, health and education into the fundamental family ([Bekele, 1998](#)). It was estimated to impact the farmers' participation in watershed management. The model output shows that access to credit has negatively influenced farmers' participation in watershed management and significantly at 1% likelihood level. The negative association implies that farmers who have access to credit are participate in other off-farm employment activities other than watershed management practices. This likewise infers better access to off-farm activities diminishes farmers' motivations to contribute on watershed management practices. This is because association in off-farm activities swarms out assets (time, labor, interest) required for the watershed's management practices, Gould (1989). The model output shows that an increase in credit access would bring about a 0.021 calculate decline in the requested log-chances for being in a higher support level, while various model elements have held consistent. The aftereffect of this finding is steady, with the results of [Bekele \(1998\)](#).

## CONCLUSION

Watershed degradation is a danger to Ethiopia's economic improvement as it influences the rural area of the nation essentially. It has brought about by deforestation and improper use and the administration of the essential resources, (soil and water). It prompts both indigenous horticulture creation and extended risks of cataclysmic flooding, sedimentation and avalanches. Henceforth, effective watershed management becomes very crucial. In the area, the watershed management scope was presented with the objective of monitoring, creating, restoring damaged watersheds and expanding food security through expanded food creation/accessibility. These measures can orchestrate into three reliant on the land use type in which they have introduced. These are preservation measures on farmlands, insurance measures on slants and protection measures on slopes, and protection measures on corrupted grounds (to reestablish crevasses). Even though a lot of effort has done to manage watersheds, success has not been comparable with the effort made. It was found that farmers responded to the action by destroying watershed management practices fully or partially in their different reasons. Accordingly, about 28.9%, 58.9%, and 12.2% of farmers interviewed to participate in low, medium, and high watershed management, respectively. It was also found that farmers' participation in problem identification and decision making, planning and monitoring and evaluation is very low compare to their participation in the implementation level.

Different factors to farmers' participation in watershed management were assessed using an ordered logit model. The model has good predictive power. The model outcome uncovered that among the 17 variables were estimated to influence the farmers' participation in watershed management; eight variables were measurably noteworthy with the speculated sign as determinants of farmers' participation in the watershed the executives.

Consequently, the family size is positive and significantly influence the farmers' participation; the more prominent family members have the likelihood to more significant work commitment to watershed management. Dependency ratio negatively and influence the farmers' participation substantially; more dependency ratio diminishes time, work, and enthusiasm to partake in watershed management practices. Education is positive and fundamentally influence the farmers' participation. The distance of parcel of land from residence positive and altogether influence the farmers' participation. Soil fertility is positive and overall influence the farmers' participation. Extension contact is positive and entirely influence the farmers' participation. Moreover, farmer households who approach credit were found to have negative and fundamentally affect the farmers' participation in watershed management.

### **Recommendations**

For effective watershed management, farmers must be interested in recognizable issue proof and dynamic, planning, implementation and monitoring and evaluation levels. Moreover, policymakers and the extension service should pay due attention to farmers' participation in every level of watershed management.

**Education** has decidedly and essentially identified with the participation of watershed management. Hence, the government or policymakers should give due attention to expand informal and formal education like primary and secondary schools, Farmers Training Center (FTC) and like.

**Dependency ratio** has found to affect farmers' cooperation in watershed management. Hence, the government and NGOs working on this area need to create a strategic approach for the utilization of family planning services.

**Farm size** was found to affect the farmers' cooperation in watershed management. The farmers who have an enormous farm sizes contribute their resources mainly time and labor on their land, and the farmers who have fewer farm size are investing their resources on their other employment practices. Therefore, the government and NGOs working in this area need to have created awareness to undertake watershed management.

**Distance of a parcel of land** from farmers' residence has affected the farmers' interest in watershed management practice. Hence, Government and NGOs organizations working in this area need to make proper awareness creation activities through training for the farmers who have a parcel of land far from their residence.

**Soil fertility** was found to affect the farmers' participation in watershed management positively. Therefore, the government and NGOs working on this area need to make training on the skills required to undertake watershed management practice for the farmers who have less fertile soil.

**Extension contact** was found to have a positive effect on the farmers' participation in watershed management. Hence, the agricultural extension should be strengthened with a view of educating farmers on watershed management.

**Access to Credit** has found to affect the farmers' cooperation in watershed negatively. Hence, due attention is needed from the government and/or other concerned parties in providing adequate extension service in terms of training, etc.

### Ethiopia Technical Terms

Kebele means Village

Woreda means District

## DECLARATION OF COMPETING INTEREST

The authors declare that they have no conflict of interest.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

The authors declared that the following contributions are correct.

**Daniel Balta:** Data collection, write up, SPSS analysis.

**Marisennayya Senapathy:** Research guidance, manuscript grammatical and technical corrections.

**Marisennayya Pandikumar:** Statistical analysis, Table and Figure correction, write up.

**Chinnaza Gods Will:** Statistical Analysis, citation correction.

## ETHICS COMMITTEE DECISION

This article requires an ethical committee decision. Ethical committee decision has been given from Wolaita Sodo University, and added to the Materials and Methods section.

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## Characterization and Classification of Soil on Varying Lithology in Okigwe Imo Southern Eastern, Nigeria

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### ABSTRACT

The field study was carried out in Okigwe South-eastern Nigeria. Three profiles were sunk in soils of each of each parent material. Soil samples were subjected to routine and standard laboratory analysis for selected physic- chemical properties. The morphological and physic-chemical properties of the soils varied widely, sand size particles dominated other particle sizes with the mean values of 448, 538 and 648 g kg<sup>-1</sup> on sandstone, while soils derived from Imo clay shale was 583 g kg<sup>-1</sup> for Umuna. Clay in Imo Clay Shale and bulk density recorded highest values at NIHORT 1, 416 and 15 g kg<sup>-1</sup> respectively. All pedons exhibited sandy clay loam on topsoil and relatively more clayey subsoil. Total nitrogen correlated positively and significantly (0.5) with organic matter in both soils. Clay correlated negatively with ECEC in sandstone and positively in clay shale, and both were not significant. The soils derived from false bedded sandstone (NIHORT 1, 2 and 3) were classified according to USDA soil Taxonomy as Inceptic Paleudults which translate to Dystric Nitisols in WRB system. Soils from Umuna were classified as Typic Hapludalf USDA and soils from NIHORT 1, 2 and 3 were classified as class II of the USDA capability classification system. Umuna was classified in class III. In conclusion the soils of these two parent materials sustain farm produce, if proper land use practices and special conservation for selected crop production are adopted.

#### RESEARCH ARTICLE

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## INTRODUCTION

Soil is an important resource for the production of food and fibre necessary for the sustenance of an increasing world population (Papendick and Parr, 1992). Soil is a dynamic resource, hence it supports plant life. It is made of different sized mineral particles, organic matter, and numerous species of living organisms. Thus, soil has biological, chemical, and physical properties, some of which are dynamic and can change in response to how the soil is managed. Soils are classified as natural bodies on the basis of their profile characteristics (Brady and Weil, 1999). Conserving and improving soil quality during the cultivation activities are the basics of sustainable agriculture (Celik *et al.*, 2017). Soil quality is the capacity of a specific kind of soil to function, within natural or managed ecosystem boundaries, to sustain plant and animal productivity, maintain or enhance water and air quality, and support human health and habitation. Changes in the capacity of the soil to function are reflected in soil properties that change in response to management or climate. Its production can be limited by the factors such as the soil characteristics, agro-ecological factors, topography, parent material, land use and management among others. To avert this limitation, the need for a systematic appraisal of the soil resource with respect to their extent, distribution characteristics, behavior and nutrient status is crucial for developing a productive and sustainable agricultural system.

Most farmers in south-eastern Nigeria regard the soil to be the same in every aspect because they are all the same based on geographical location. Igwe (2003) posit that most soils in southeastern Nigeria are not classified but are utilized in land use activities leading to water erosion. Onweremadu (2007) stated that characterization and classification of soil of any given location help in generating soil related data which are useful in proper and sustained use of soil resource. Presently, there is an increased request of information on soils with respect to food production. Agriculture overtime as become the major economic activity in Nigeria, because of the development in agriculture and its increased demand for experimental data much work is carried out on soil characterization in Nigeria. This makes available elementary information essential to generate efficient schemes for the classification of soils as well as assessing the fertility of the soil so as to unpick certain exceptional complications of soil in an ecology (Lekwa, 1998). The combination of soil mapping, characterization and classification of soil offers a potent source to the advantage of mankind, specifically to ensure adequate environmental sustenance and food security. Knowledge acquired from the characterization of soils would as well assist agriculturalists to reasonably plan the development and the use of lands accordingly, so as to put accessible agricultural lands to their optimum uses for sustainable production of food. Varying soil characteristic requires dissimilar management practices, land use activities for best and sustainable performance. The possibility lies on adequate information with respect to the physicochemical parameters of the soil category under investigation. In addition, several, agriculturalists and most land users in Nigeria; especially the south easterners have handled the soils of this sub-region in a similar method, erroneously considering that all the soils are the same. The low return on investment can be attributed to the erroneous concept and methodology used in both for agricultural production and other land use categories. Based

on the notion, the study therefore focused on the characterization and classification of soil on varying lithology in Southeastern Nigeria for sustainable agriculture for food security. The major focus of this study is to characterize and classify some soils derived from two different parent materials. The USDA Soil Taxonomy and World Reference Base (WRB) were utilized, and the specific objectives includes the estimation of the degree of variability of some soil properties among the different soil groups and to establish the amount of relationship between various soil properties in the study area.

## **MATERIAL and METHODS**

Okigwe, Southeastern Nigeria is the experimental area. It is situated on latitudes 5° 45'N - 6° 00'N and longitudes 7° 15'E - 7° 30'E with an altitude of approximately 300 m and above. Okigwe has a humid tropical climate, having a mean annual rainfall range of 2000-2250 mm. Mean annual temperature range of 27-28°C and relative humidity varies with seasons as 80 -90% range occurs at 10 am in the rainy season while 60-80% relative humidity is recorded at 10 am during the dry season. Okigwe has a humid tropical climate, having a mean annual rainfall range of 2000-2250 mm. Mean annual temperature range of 27 -28°C and relative humidity varies with seasons as 80-90% range occurs at 10 am in the rainy season while 60-80% relative humidity is recorded at 10 am during the dry season. The soils of the area are derived from false bedded sandstones (Ajali Formation) of the Maastrichtian geologic era and proximal to the upper coal measures (Nsukka Formation) of the Danvan geologic age and Imo Clay shale.

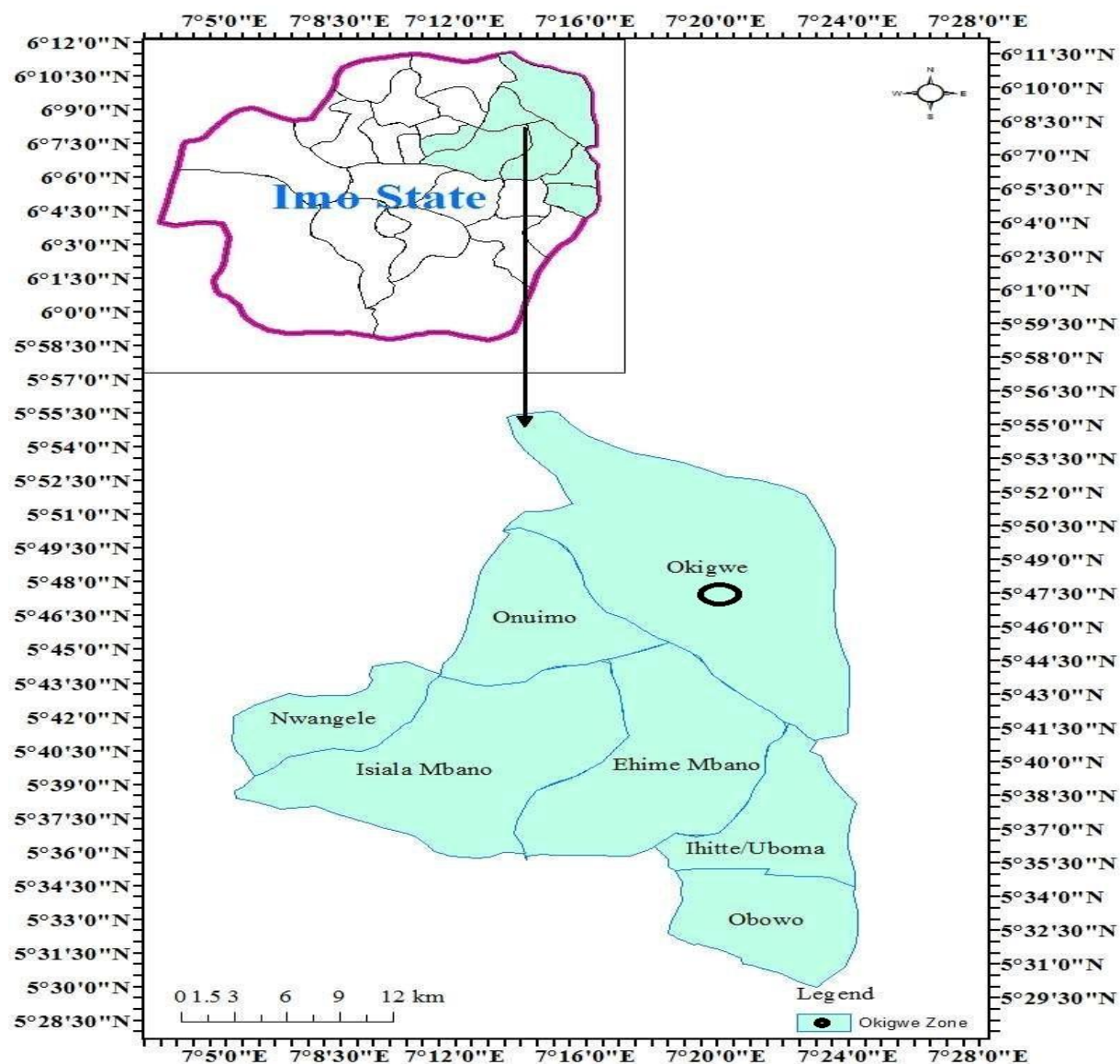


Figure 1. Location map of the study area.

### Geology of The Experimental Area

The experimental zone is a complicated geological location in Southeastern Nigeria. Hence, the following stratigraphic components inspire the area: Benin, Ogwashi-Asaba, Bende-Ameki, Imo Shale, Nsukka and Ajali Formations (Nwosu *et al.*, 2010). The Benin Formation is superimposed by lateritic overburden and underlain by the Ogwashi-Asaba Formation which is in turn underlain by the Ameki Formation of Eocene to Oligocene age. The Benin Formation consists of coarse-grained gravelly sandstones with minor intercalations of shales and clay. The sand soil units which are usually coarse grained; pebbly and poorly arranged comprise lenses of fine-grained sands (Onyeaguocha, 1980). The raining period commences in April and terminates in October with a peak in the month of June and July whereas, dry period last from November to March.

## Field Work

An inspection visit was carried out on the experimental zone, materials such as location map, topographic map, geology maps and munsell colour chart were used handheld Global Positioning System (GPS) receiver (Gamin Ltd Kansas) was utilized to geo-reference sampling site. A free-soil survey technique was used to locate the sampling point from the two geological formations of the study area. The following were the study site located, False bedded sandstone (NIHORT 1, NIHORT 2, NIHORT 3) and Imo Clay shale (Umuna). The study area is a complex geological environment in Imo State. The following stratigraphic units underlie the area; the Benin formation, the Ogwashi-Asaba formation, the Bende – Ameki formation, Imo state formation, Nsukka formation and Ajali formation.

**Table 1.** Geo-location of sampled sites.

Position	Latitude(N)	Longitude (E)	Elevation (m)
NIHORT 1	5° 52. 67'N	7° 18.47'	156
NIHORT 2	5° 52. 68'N	7° 18.54'	153
NIHORT 3	5° 52. 87'N	7° 18.54'	153
UMUNA	5° 46. 08'N	7° 14.97'	129

## Sample Collection and Preparation

Soil profiles were described according to [FAO \(1983\)](#) procedure. A total of five profiles were sunk, three on each parent material. Delineation of horizon boundary was accomplished before actual sample collection. Soil samples collection was based on horizon differentiation. The samples collected were stored in polythene bags and labeled. Samples collected were air-dry and 2 mm sieve was used in the preparation to various laboratory analyses.

## Laboratory Analyses

The soil samples were analyzed for certain selected properties that are necessary for proper scientific classification of the soils. These include physical properties such as particle size distribution, bulk density and hydraulic conductivity. Chemical properties such as soil pH, exchangeable bases (Ca<sup>2+</sup>, Mg<sup>2+</sup>, K<sup>+</sup>, Na<sup>+</sup>) exchangeable acidity (Al<sup>3+</sup> and H<sup>+</sup>), total nitrogen, available phosphorus and organic carbon. The hydrometer method was utilized in the determination of the particle size distribution as posited by [Gee and Or \(2002\)](#). Silt clay ratio was obtained by calculation, soil moisture content was determined gravimetrically by weighing an oven drying soil sample collected from the field at 105°C until a constant weight was attained ([Obi,1990](#)), Bulk density was determined using the core samplers as noted by ([Grossman and Reinsch, 2002](#)). Soil pH was determined by the use of distilled water on Beckman Zeromtic pH meter using a glass electrode at a 1: 2: 5 soil water ratio ([Thomas, 1996](#)), exchangeable acidity and organic carbon (OC) was determined by an unbuffered saturated solution such as in KCL at the pH of the soil ([Mclean, 1982](#)) and Walkey and Black wet oxidation method ([Nelson and Sommer, 1982](#)) respectively. Ca, Mg, K, Na (exchangeable bases) were determined from the soil in 1 m ammonium acetate solution ([Thomas, 1996](#)), soil organic matter was determined by calculating

$$\% \text{organic matter} = \% \text{organic carbon} \times 1.724,$$

Where: 1.724 = correlation factor (Van Bemelies constant factor), exchangeable potassium and sodium in the extract was determined by the use of flame photometric method while calcium and magnesium was determined using ethylene diamine tetra acetic acid titration method (EDTA).

The Micro-Kjedahl digestion method was used in the determination of total nitrogen and available phosphorus ([Bremner and Mulvaney, 1982](#)) and Bray 11 method ([Olsen and Sommers, 1982](#)) respectively. Summation of all exchangeable bases and acidity was used to determine effective cation exchange capacity (ECEC) ([Carter, 1993](#)), total exchangeable base (TEB) was determined by summation of all the exchangeable bases. Percentage base saturation was determined using the following formula:

$$\text{Percentage base saturation} = \frac{TEB}{CEC} \times 100 \quad (1)$$

$$\text{Aluminum saturation was calculated using the formula } \frac{AL}{CEC} \times 100 \quad (2)$$

USDA soil Taxonomy and world reference base for soil resources (WRB) was used in the soil classification and soil properties.

### Data and Statistical Analyses

Simple descriptive statistics, means, standard deviation and co-efficient of variation (CV) was used during the data calculation. Variability of soil properties on the two parent materials was estimated using coefficient of variation. Ranking of variability and sample regressions was performed using ([Wilding, 1985](#)).

Correlation analyses of the model  $[D=i+s_1 X_1+ s_2 X_2 + s_3 X_3 + \dots + s_n X_n + U]$  was used.

Where,

D = dependent variable,

i = intercept (constant),

s = slope,

X = independent variable,

U = stochastic factor.

Co- efficient of variation as ranking according to [Wilding \(1985\)](#).

**Table 2.** The coefficient of variation as ranked by [Wilding \(1985\)](#).

Level %	Ranking
CV < 15	Low Variation
CV 15 – 35	Moderate Variation
CV > 35	High Variation

## RESULTS and DISCUSSION

The soil texture result is shown in Table 4. This indicates that the soil texture ranges from sandy loam to sand clay loam. Umuna soil had sandy loam on the top soil and sandy clay down the profile. This is in line with the work of [Eshett \(1985\)](#), who observed that the texture of surface horizons of soils in humid tropics is dominated by loamy sand to sandy loam. The matrix colour at the top soil (A horizon) was darker than the subsoil. This is shown in Table 3.

**Table 3.** The soil morphological characteristics of the experimental site.

Horizon	Depth (cm)	Matrix colour	Texture	Structure	Consistency	Drainage
<b>UMUNA</b>						
<b>A</b>	0 – 15	Dark Reddish Brown (5YR $3/4$ )	SCL	Granular	Loose	wd
<b>AB</b>	15 -40	Dark Brown (7.5YR $3/4$ )	C	Sbk	Firm	wd
<b>Bt1</b>	40 – 80	Brown (7.5YR $4/4$ )	SC	Sbk	Firm	wd
<b>Bt2</b>	80 – 120	Strong Brown (7.5YR $4/6$ )	SC	Sbk	Firm	wd
<b>Bt3</b>	120 - 150	Strong Brown (7.5YR $4/6$ )	LS	Sbk	Firm	Pd
<b>NIHORT 1</b>						
<b>A</b>	0 - 15	Reddish Brown (5YR $4/3$ )	SCL	Sbk	Loose	wd
<b>AB</b>	15 - 45	Yellowish Red (5YR $4/6$ )	SCL	Sbk	Firm	wd
<b>Bt1</b>	45 - 80	Red (2.5YR $4/8$ )	C	Sbk	Firm	wd
<b>Bt2</b>	80 - 120	Red (2.5YR $5/8$ )	SL	Granular	Firm	wd
<b>Bt3</b>	120 - 180	Red (10 YR $4/8$ )	SL	Sbl	Firm	wd
<b>NIHORT 2</b>						
<b>A</b>	1 - 18	Strong Dark Brown (7.5YR 2.5/3)	SCL	Sbk	Loose	wd
<b>AB</b>	18 - 50	Dark Brown(7.4YR $3/4$ )	SCL	Sbk	Firm	wd
<b>Bt1</b>	50 - 75	Red (10YR $4/6$ )	SL	Granular	Firm	wd
<b>Bt2</b>	75 - 120	Red (2.5YR $8/8$ )	SCL	Sbk	Firm	wd
<b>NIHORT 3</b>						
<b>A</b>	1 - 20	Very Dark Brown(2.5YR $2.5/2$ )	SCL	Sbk	Firm	wd
<b>AB</b>	20 - 60	Very Dark Brown(7.5YR $4/3$ )	SCL	Sbk	Firm	Pd
<b>Bt1</b>	60 - 90	Brown (7.5YR $4/3$ )	SCL	Granular	Firm	Pd

SL = sandy loam, C = clay, LS = loamy sand, SCL = sandy clay loam, sbk = sub angular blocky, wd = well drained, pd = poorly drained.

### Physical Properties of Soil Derived from Imo Clay Shale and False Bedded Sandstone

Tables 4.1. and 4.2 shows the result of the physical properties of Imo clay shale and false bedded sandstone in the study areas, the soil moisture content at Umuna improved via depth and has a mean value of 85 g kg<sup>-1</sup> in clay shale. [Salako \(2003\)](#) reported similar increase in water retention and clay content down the profile in Guinea Savana zone of Nigeria. Soil from sandstone (NIHORT 1, 2 and 3) has no definite distribution pattern of soil moisture content. Clay content was high generally in the subsoil of all the profile of the two parent materials but low at the top, increasing down the profile before decreasing in the last horizon as shown in depth function of clay in the pedons. [Eshett \(1985\)](#) noted that the increment in

the content of clay down the profile could be diagnostic of existence of illuviation. Silt content ranges from low to moderate. The highest mean silt value was recorded at NIHORT 1 (136 g kg<sup>-1</sup>). Bulk density ranges from 12.9 g kg<sup>-1</sup> to 16.9 g kg<sup>-1</sup> on both soil types. It would be observed that the bulk density on the two parent materials was lower, hence it oppose the quoted value of the minimum bulk density at which root – constraining situations occur in the sandy loam soils (175-180 g kg<sup>-1</sup>) (USDA NRCS, 1996) sand (16 g kg<sup>-1</sup>) and clay (1.4 g kg<sup>-1</sup>). The low amount in bulk density shows that the soils were not compacted. Hence, it may attribute to the improvement of soil tilt and porosity through the continuous addition of organic matter from decay plants residues in soil surfaces.

**Table 4.1.** Physical properties of soil derived from false bedded sand stone.

Horizon	Depth (cm)	Soil moisture content (smc)	Sand →	Silt g kg <sup>-1</sup>	Clay ←	Textural class	Silt/Clay ratio (g kg <sup>-1</sup> )	Bulk density (mg m <sup>-3</sup> )	Soil hydraulic (kgs m <sup>-3</sup> )
<b>A</b>	0 - 15	62.6	628	120	252	SCL	0.48	14.7	0.3
<b>AB</b>	15 - 45	96.5	508	160	332	SCL	0.48	14.9	0.2
<b>B1</b>	45 - 80	80.9	328	80	592	C	0.13	16.9	0.2
<b>B2</b>	80 - 120	62	388	140	472	SC	0.29	14.2	0.9
<b>B3</b>	120 - 160	64.4	388	180	432	SC	0.41	15.5	1.4
<b>Mean</b>		73.7	448	136	416		0.36	15	0.7
<b>A</b>	1-18	118.1	648	80	272	SCL	0.29	13.4	0.19
<b>AB</b>	18 - 50	95.2	468	100	432	SC	0.23	15.1	0.12
<b>B1</b>	50 - 75	76	368	160	472	SC	0.33	13.9	0.13
<b>B2</b>	75 - 120	113.3	668	20	312	SCL	0.38	15	0.23
<b>Mean</b>		100	538	90	372		0.3	14	0.2
<b>A</b>	1-20	73.7	628	100	270	SCL	0.37	14.3	0.12
<b>AB</b>	20 - 60	92	728	60	210	SCL	0.28	14	0.24
<b>B</b>	60 - 90	112.1	588	140	272	SCL	0.51	13.9	0.13
<b>Mean</b>		93	64.8	100	251		0.44	14	0.2

SL = Sandy Loam, C = Clay, LS= Loamy Sand, SCL = Sandy Clay Loam

**Table 4.2.** Physical properties of soil derived from Imo Clay Shale.

Horizon	Depth (cm)	Soil moisture content (smc)	Sand →	Silt (g kg <sup>-1</sup> )	Clay ←	Textural class	Silt/Clay ratio	Bulk density (mg m <sup>-3</sup> )	Soil hydraulic conductivity (g kg <sup>-1</sup> )
<b>UMUNA</b>									
<b>A</b>	0 - 15	64.6	588	180	232	SCL	0.77	13.9	1.1
<b>AB</b>	15 - 40	95.2	328	160	512	C	0.31	15.8	0.5
<b>B1</b>	40 - 80	87	388	160	452	SC	0.35	14.6	0.1
<b>B2</b>	80 - 120	73.0	808	110	820	SC	1.34	14.8	0.2
<b>B3</b>	120 - 150	103	820	60	112	LS	0.53	12.8	1.2
<b>Mean</b>		85	588	134	278		0.4	14	0.4

SL = Sandy Loam, C = Clay, LS = Loamy Sandy, SCL = Sandy Clay Loam



### Chemical Properties of Soil Derived from Imo Clay Shale and False Bedded Sandstone

The result of the chemical properties of the two areas under study is as stated in Tables 5 and 6 respectively. The pH was reported to be moderately acidic (4.63-5.74). The pH in H<sub>2</sub>O was higher than the pH in KCL in the soil, this is in accordance with [Ukaegbu and Akamigbo \(2004\)](#). The organic carbon ranges from 3.9-18.6 g kg<sup>-1</sup> and has a mean value of 14 g kg<sup>-1</sup>, the highest value was recorded at the surface. This is in line with [Igwe \(2003\)](#), who observed that when soils erode negative soil properties such as sealing, crusting, nutrient depletion and loss of organic matter occur. Also in sand stone the highest mean value was recorded at the surface horizon of NIHORT 1 and 2, except NIHORT 3 where the highest value was at the subsoil AB horizon and decreases down profile. Total nitrogen and organic matter ranges from 0.3-2.1 g kg<sup>-1</sup> and 6.8-42.3 g kg<sup>-1</sup>, while their mean values are 1.2 g kg<sup>-1</sup> and 24 g kg<sup>-1</sup> respectively. The C/N ratio of clay shale have a mean value of 12.3%, this is in line [Onweremadu and Anikwe \(2007\)](#) who reported a C/N ratio range from 9% to 16% in the top soil of the shale soils of Isienyi Ibeku and [Igbokwe et al. \(1982\)](#) which ranged from 8.2 to 12% at Afikpo. Similarly there was low C/N ratio in sandstone which ranges from 13.0 %, 12.3% and 12.6% in NIHORT 1, 2 and 3 respectively the C/N ratio has irregular distribution pattern on all the pedons. The mean values of exchangeable calcium and total exchangeable bases were 2.30 g kg<sup>-1</sup> and 4.5 g kg<sup>-1</sup>; while available phosphorous was 3.1 mg kg<sup>-1</sup> respectively. The highest value of magnesium was recorded at the topsoil (A horizon) in clay shale 2.40 cmol kg<sup>-1</sup> and the mean value 1.76 cmol kg<sup>-1</sup>. The potassium value of Umuna (clay shale) ranges from 0.18-0.31 cmol kg<sup>-1</sup>, while, NIHORT 1 (sandstone) has a potassium value lowest 0.15 cmol kg<sup>-1</sup>. Exchangeable sodium was generally low in both soils of different parent materials as shown in the tables 4.1 and 4.2. The mean values of ECEC in sand stone were 5.5, 6.8 and 6.9 cmol kg<sup>-1</sup> they had irregular distribution pattern at all the physiographic position with the highest value recorded in NIHORT 3 (8.53 cmol kg<sup>-1</sup>). The Ca/Mg ratio ranges from 0.5-1.71 on the Imo clay shale, while the ration on false bedded sandstone ranges from 1.2-2.5. This indicates an increasing unavailability of Mg and increases availability of Ca required. According to [Landon \(1991\)](#); Ca: Mg ration less than 3.0 negatively influences calcium and phosphorus availability in the soil. Sandy soils have relatively high bulk density since total pore space in sands is less than that of clay soils, hence a high clay content achieved a higher bulk density.

**Table 5.** Chemical properties of soils derived from false bedded sand stone of NIHORT 1, 2 and 3.

Horizon	Depth cm	pH H <sub>2</sub> O	KCL →	O.C g kg <sup>-1</sup>	OM ←	T.N	C:N Ratio	TE A	AL →	H	Ca cmol kg <sup>-1</sup>	Mg ←	K	Na	TEB	CEC	BS%	Ca:Mg Ratio	K:Mg Ratio	Avl.P g kg <sup>-1</sup>
<b>NIHORT 1 (false bedded sand stone)</b>																				
<b>A</b>	0 -15	5.08	3.71	30.3	52.2	0.26	11.6	2.4	0.9	1.5	3.5	1.4	0.22	0.11	5.23	7.31	73.3	2.5	0.15	9.37
<b>AB</b>	15 -45	4.96	3.6	10.4	17.8	0.08	13.0	1.4	1.0	0.4	3.2	1.6	0.16	0.09	5.05	6.45	78.2	2.0	0.11	2.82
<b>Bt<sub>1</sub></b>	45 -80	5.67	3.47	6.8	11.2	0.05	13.6	2.7	1.4	1.3	1.6	1.0	0.15	0.12	2.87	5.61	51.8	1.6	0.15	1.49
<b>Bt<sub>2</sub></b>	80-120	4.34	3.72	6.7	11.6	0.05	13.4	1.5	0.9	0.7	2.6	1.2	0.26	0.19	4.25	5.75	73.9	2.1	0.12	2.34
<b>Bt<sub>3</sub></b>	120- 160	5.79	3.59	6.7	11.6	0.05	13.4	0.6	0.4	0.2	2.2	1.2	0.15	0.07	4.25	4.2	85.7	1.8	0.12	0.96
<b>Mean</b>		5.1	3.6	12.0	24.0	0.1	13.0	1.6	0.9	0.8	2.6	1.2	0.2	0.1	4.3	5.8	59.7	2.0	0.13	3.3
<b>NIHORT 2 (false bedded sandstone)</b>																				
<b>A</b>	1- 18	5.11	3.72	14.6	25.1	0.12	12.6	2.2	1.2	1.0	3.0	1.2	0.24	0.1	4.54	6.74	67.3	2.5	0.2	4.68
<b>AB</b>	18 – 50	5.74	4.41	7.4	12.7	0.06	12.33	1.8	1.2	0.6	1.8	1.2	0.25	0.12	3.37	5.17	65.1	1.5	0.2	1.69
<b>Bt<sub>1</sub></b>	50 – 75	5.67	3.57	14.9	25.7	0.12	12.41	2.0	1.2	0.8	3.8	2.2	0.24	0.15	6.39	8.39	76.1	1.7	0.1	2.16
<b>Bt<sub>2</sub></b>	75 – 120	5.13	3.72	12.4	21.3	0.1	12.41	1.9	0.9	1.0	3.2	1.6	0.26	0.12	5.18	7.17	73.5	2.0	0.16	2.87
<b>Mean</b>		5.4	3.8	12.0	21.0	6.1	12.3	1.9	1.1	0.8	2.9	1.5	0.2	0.1	3.8	6.8	70.5	1.9	0.2	2.2
<b>NIHORT 3 (false bedded sandstone)</b>																				
<b>A</b>	1 – 20	4.33	3.27	5.5	9.6	0.04	13.75	0.8	0.3	0.5	1.8	1.4	0.23	0.14	3.57	4.37	81.6	1.2	0.16	1.42
<b>AB</b>	20 – 60	5.21	4.73	14.2	24.4	0.12	11.8	0.9	1.0	0.9	3.5	2.6	0.32	0.21	6.63	8.53	77.7	1.3	0.14	2.66
<b>Bt<sub>1</sub></b>	60 – 90	5.28	4.89	12.4	21.3	0.1	12.4	1.7	1.1	0.6	3.66	2.2	0.29	0.22	6.37	8.01	78.7	1.66	0.13	2.54
<b>Mean</b>		4.9	4.3	12.0	13.0	0.08	12.6	0.6	0.8	0.5	2.9	2.0	0.2	0.2	5.5	6.9	79.3	1.3	0.14	2.2

OC =organic content, OM= organic matter, TN= total nitrogen, TEA=total exchangeable acidity, AL= aluminum, H= hydrogen,Ca= calcium, Mg= magnesium, potassium, Na= sodium, TEB= total exchangeable base, CEC= cation exchangeable capacity, BS=base saturation, Avl. P = available phosphorus

**Table 6.** Chemical properties of soil derived from Imo Clay Shale.

Horizon	Depth (cm)	pH (H <sub>2</sub> O)	KCL →	O.C g kg <sup>-1</sup>	O.M ←	T.N	C:N ratio	TEA	AL	H	Ca →	Mg	K cmol kg <sup>-1</sup>	Na ←	TEB	CEC	BS%	Ca:Mg ratio	K:Mg ratio	Avl. P g kg <sup>-1</sup>
<b>UMUNA Imo clay shale</b>																				
<b>A</b>	0 -15	5.43	4.84	24.5	42.3	2.1	11.6	0.5	0.4	0.1	1.2	2.4	0.31	0.2	4.11	7.41	93.0	0.5	0.12	3.86
<b>AB</b>	15 -40	5.46	4.74	3.9	6.8	0.3	13	2.0	1.2	0.8	1.8	1.2	0.24	0.16	3.4	4.6	56.5	1.5	0.2	0.66
<b>Bt<sub>1</sub></b>	40 -80	5.74	4.54	14.9	25.7	1.2	12.4	1.9	1.1	0.8	3.3	2.0	0.3	0.27	5.87	7.7	75.5	1.65	0.15	4.06
<b>Bt<sub>2</sub></b>	80 - 120	5.68	4.73	18.6	31.9	1.5	12.4	0.8	0.3	0.5	2.75	1.6	0.18	0.12	4.65	5.45	85.3	1.71	0.11	5.46
<b>Bt<sub>3</sub></b>	120 - 150	4.63	3.63	8.7	15.1	0.7	12.4	1.0	0.7	0.3	2.6	1.6	0.27	0.18	4.65	5.65	82.3	1.62	1.6	1.51
<b>Mean</b>		5.3	4.3	14.0	24.0	1.2	12.3	1.2	0.8	2.5	2.3	1.76	0.26	0.18	4.5	6.1	78.5	1.4	0.4	3.1

OC =organic content, OM= organic matter, TN= total nitrogen, TEA=total exchangeable acidity, AL= aluminum, H= hydrogen, Ca= calcium, Mg= magnesium, potassium, Na= sodium, TEB= total exchangeable base, CEC= cation exchangeable capacity, BS=base saturation, Avl. P = available phosphorus

**Table 7.** Variability of some selected soil properties of false bedded sandstone.

Soil properties	NIHORT 1	Ranking CV %	NIHORT 2	Ranking CV %	NIHORT 3	Ranking CV%	UMUNA	Ranking CV %
<b>Sand</b>	27.0	MV	27.0	MV	11.0	LV	39.6	HV
<b>Silt</b>	28.0	MV	64.0	HV	40.0	HV	36.2	HV
<b>Clay</b>	31.0	MV	26.0	MV	14.0	LV	70.0	HV
<b>S/clay ratio</b>	42.0	HV	21.0	MV	30.0	MV	64.0	HV
<b>SHC</b>	21.0	MV	20.0	MV	21.0	MV	19.0	MV
<b>BD</b>	7.5	LV	6.5	LV	1.4	LV	8.1	LV
<b>pH (H<sub>2</sub>O)</b>	11.0	LV	6.5	LV	11.2	LV	8.1	LV
<b>TEB</b>	22.0	MV	26.0	MV	31.2	HV	20.0	MV
<b>ECEC</b>	20.0	MV	19.0	MV	33.0	MV	54.0	HV
<b>BS</b>	53.0	HV	7.5	LV	3.2	LV	18.0	MV
<b>OM</b>	85.0	HV	28.0	MV	42.0	HV	57.0	HV
<b>Ca : Mg ratio</b>	17.0	MV	23.0	MV	17.0	MV	36.2	HV

MV= Moderate variation, HV= High variation, LV= Low variation, SHC= Soil moisture content, BD= Bulk density, TEB= Total exchangeable base, TEA= Total exchangeable acidity, ECEC= Exchangeable cation exchange capacity, BS= Base saturation, OM= Organic matter

The result of the bulk density, pH and base saturation as shown in Table 7, indicates low variation. This might be accredited to the high incidence of leaching of base cation under high annual rainfall saturation. TEA and ECEC, Ca:Mg ratio and hydraulic conductivity had a moderate variation. Sand and clay had a moderate variation in NIHORT 1, 2 and NIHORT 3 recorded a low variation. Hence, it could be ascribed to the existence of argillic horizons in most of the study area. This argillation in soils of southeastern Nigeria has been reported by [Onweremadu and Anikwe \(2007\)](#). Organic matter contents were high in NIHORT 1 and 3 but moderate in NIHORT 2 as this could be attributed to the land sue of the area, while the moderate variation is ascribed to leaching of organic carbon under annual precipitation. The relationship among selected physico-chemical properties of the studied soils at the two soils shows that sand correlated negatively with clay (-0.973\*\* and -0.943\*\*) and highly significant in the two parent materials. Clay correlated negatively with ECEC in sandstone (-0.519<sup>NS</sup>) and positively to clay shale (0.337<sup>NS</sup>).

### **Taxonomy Classification**

Taxonomy classification of the selected soils around Okigwe L.G.A. based on the physical, chemical and morphological characteristics and it was classified using USDA soil taxonomy system ([Soil survey staff, 2003](#)) and World reference base system (W.R.B). The soils at NIHORT 1, 2 and 3 had argillic (Bt) horizon as determined by the distribution of clay down the profile and low silt/clay ration indicating advanced stage of weathering. In addition, percentage base saturation greater than 35% is recorded in the soil of false bedded sand stone, therefore the soil belong to the soil order Alfisol, suborder Udalf, great group Hapludaf, sub-group Typic Hapludaf, W.R.B soil class Luvisols, W.R.B soil unit Arenic luvisols. Soils of Umuna had argillic horizons and low silt / clay ratio and it was classified as Alfisol, suborder Udalf, great group Hapludaf, sub-group Typic Hapludaf, W.R.B soil class Luvisols, W.R.B soil unit Arenic luvisols.

### **Land Capacity Classification of Soil According to USDA System**

The physicochemical properties of the soils, the nature of the landscape and the soil depth, the soils of NIHORT 1, 2 and 3 were grouped as class II of the USDA capability classification system. They are moderately suitable due to uneven plain of the slope which ranges from 2-6% and are derived from sand stone. The moderate susceptibility to water erosion, soil stoniness of 20-27%, depth to root limiting layer of about 60 cm and slow subsoil permeability resulted in the placement of Umuna in class III.

## **CONCLUSION**

An unrestricted soil survey techniques used was aided with geological map of the area. Three (3) soil profiles were sunk, profile pit was defined based on the FAO procedure and samples were collected according to the horizon differentiation. The samples were exposed to laboratory analysis for the selected physicochemical properties and the results were analyzed using suitable statistical tools. The result from various soils as shown in the tables suggests that soils vary not only in parent materials but then again in many key physicochemical properties. Consequently their capability to support varying categories/kinds of land use is

anticipated to vary. The soil properties from the characterization of the selected soil on the study area were utilized in the classification of the soil according to the USDA soil taxonomy and WRB soil classification systems. Hence it is observed that the false bedded sand stone were in class II and Umuna (Imo clay shale) in class III. Finally, it is found that soils from the selected areas are suitable for agricultural practices, lowland crops like rice can be grown on about 42% of the soils due to the seasonal fluctuations of water table. This study will go a long way to assist researchers, farmers and agriculturist who will want to embark on massive agricultural production in different countries of the world that has similar land terrain. This study can also be expanded in the future to accommodate other regions of the country and the world at large.

## DECLARATION OF COMPETING INTEREST

We author hereby affirm that there is no conflicting of interest whatsoever.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

The authors hereby declare that the contributions given are correct.

**Christopher Ikechi Obineche:** Writing original draft and Investigation

**Patricia Akunna Oriaku:** Validation, Review and Methodology.

## ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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## Microbiological Comparison of Wet and Dry Plucked Chicken Meat Sold in Adana Province

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### ABSTRACT

This study was carried out in order to clarify the debates on the hygiene of plucking methods in slaughter in today's poultry. For this purpose, in order to determine the microbiological quality of drumstick and breast meat obtained from the wet and dry plucked carcass for sale in the market Total Mesophilic Aerobic Bacteria (TMAB), Total Mold-Yeast levels and *Salmonella* ssp. levels were investigated. In this context, a total of 24 meat samples purchased from 12 different markets were used. As a result of the analysis, it was determined that there was no significant difference between the groups of Total Mesophilic Aerobic Bacteria and Total Mold-Yeast levels in wet and dry plucked drumstick and breast samples. 1 *Salmonella* ssp. was detected in wet plucked drumsticks and also 1 dry plucked breasts. The results obtained show that the wet or dry plucking method does not have an effect on the microorganism level in the meat.

#### RESEARCH ARTICLE

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- *Salmonella*,
- Total Mesophilic Aerobic Bacteria,
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- Feather plucking methods

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## Adana İlinde Satışa Sunulan Yaş ve Kuru Yolum Yapılmış Tavuk Etlerinin Mikrobiyolojik Yönden Karşılaştırılması

### ÖZET

Bu çalışma, günümüz tavukçuluğunda kesimdeki tüy yolma yöntemlerinin hijyeni konusundaki tartışmalara bir açıklık getirmek amacıyla yapılmıştır. Bu amaç doğrultusunda, yaş ve kuru tüy yolma yöntemleri uygulanarak satışa sunulan karkaslardan elde edilen but ve göğüs etlerinde mikrobiyolojik kaliteyi belirlemek amacıyla Toplam Mezofilik Aerobik Bakteri (TMAB), Toplam Küf-Maya düzeyleri ve *Salmonella* ssp. durumları araştırılmıştır. Bu kapsamda 12 farklı marketten satın alınan toplam 24 et örneği kullanılmıştır. Araştırma sonucunda yaş ve kuru yolum yapılan but ve göğüs örneklerinde Toplam Mezofilik Aerobik Bakteri ve Toplam Küf-Maya düzeyleri bakımından gruplar arasında farkın önemli olmadığı tespit edilmiştir. Sulu yolum uygulanmış butlarda 1 adet ve kuru yolum uygulanmış göğüslerde 1 adet *Salmonella* ssp. tespit edilmiştir. Elde edilen sonuçlar kesimde sulu veya kuru tüy yolma yöntemi uygulamasının etlerde bulunan mikroorganizma düzeyi üzerine bir etkisinin bulunmadığını göstermiştir.

#### ARAŞTIRMA MAKALESİ

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#### Anahtar Kelimeler:

- *Salmonella*,
- Toplam Küf-Maya,
- Toplam Mezofilik Aerobik Bakteri,
- Tüy yolma yöntemleri

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### GİRİŞ

Tavuk etinin çiftliklerde üretiminden sofrada tüketimine kadar olan süreçte mikrobiyolojik yükü üzerine birçok faktör etki etmektedir. Hayvanların yetiştirilme aşamasında yediği yem, içtiği su, yetiştirildiği ortamın havası ve koşulları, çevrede temas halinde bulunduğu vektörler; kesim için araçlara yükleme, taşıma ve boşaltma esnasında maruz kaldığı koşullar ile kesimhane ortamı; kesim sonrası etin soğutma, parçalama, paketlenme, saklama ve dağıtım esnasındaki koşullar tavuk etinin mikrobiyolojik kalitesini etkileyen faktörler arasındadır. Kesim için kesimhaneye getirilen hayvanlar ayakları, derisi, tüyü, tırnakları ve bağırsaklarında birçok mikroorganizma barındırmaktadır. Canlı hayvanlardaki enfeksiyonların dışında hayvanların taşınması, kesimi, ıslatılması, tüylerin yolunması, iç organların çıkartılması, etlerin soğutulması, parçalanması ve paketlenmesi gibi aşamalarındaki çapraz kontaminasyon ile saklama sıcaklığı ve süresinin de patojen mikroorganizmaların etlerle kontaminasyonunda önemli rol oynadığı bilinmektedir. Diğer yandan yemlerin üretim yerlerinde, işletmelerde ve perakende satış noktalarında da bulaşma riskinin olduğu bildirilmektedir ([Sarımehmetoğlu ve ark., 1996](#); [Erol, 2007](#); [Sırıken ve Türk, 2013](#); [Şireli, 2017](#)). Etlik piliçlerin kesimi esnasında uygulanan klasik yaş yolma yönteminde, ıslatma (suya daldırma), tüylerin yolunması, iç organların çıkarılması aşamalarında ve yeterli tedbirler alınmadığında yolma ekipmanlarından ve diğer hayvanlardan çapraz kontaminasyon riski oldukça yüksektir ([Rivera-Perez ve ark., 2014](#); [Perez-Arnedo ve Gonzalez-Fandos, 2019](#)). Özellikle iç organların mekanik olarak çıkarılması esnasında bağırsakların makine tarafından



zarar görmesi halinde karkas dışkı ile kontamine olabilmektedir. Diğer taraftan sıcak suda ıslatma ve tüy yolma işlemleri derinin epidermis tabakasını kaldırmakta, bundan dolayı bağırsakların çıkarılması ve karkasların soğutulması işlemleri sırasında karkas yüzeyinde bakterilerin artarak koloni oluşturmasına zemin hazırlanarak etin raf ömrü etkilenmektedir ([Erginkaya ve Yurdakul, 2010](#); [Var ve ark., 2011](#)). Yapılan birçok araştırma sonucunda sulu yolum yöntemi uygulanan karkaslarda mikroorganizma düzeyinde artış olduğu bildirilmektedir ([Pool, 1954](#); [Essary ve ark., 1958](#); [Mallmann, 1958](#); [Fromm, 1959](#); [Goresline ve Haugh, 1959](#); [Kinsley ve Mountnery 1966](#); [Klose, 1971](#); [Grossklaus ve ark., 1979](#); [Anıl ve ark., 1989](#)).

Etlik piliçlerde kullanılan bir diğer yöntem kuru yolum yöntemidir. Piliçlerin tüylerini yumuşatmak için sıcak su kazanında ıslatma işlemi uygulanmadan nemli sıcak hava tünellerinden geçirilerek tüy diplerinin gevşetilip, tüylerin otomatik yolum makineleri tarafından el değmeden temizlendiği bir yöntemdir. Bu yöntemin tüy yolumundan önce hayvanların iç organları ile birlikte sıcak su kazanına daldırılarak ıslatma işlemi uygulanmadığı için patojen mikroorganizmalarla kontaminasyon riskinin daha az ve daha hijyen bir yöntem olduğu bildirilmektedir ([Riggs ve ark., 2011](#); [Anonim, 2021](#)). Öte yandan günümüzde bazı çevreler tarafından geleneksel etlik piliç endüstrisinde uygulanan ıslak yolma yöntemi ile yolunmuş tavuk etlerinin helal ve tüketimin uygun olmadığı algısı nedeniyle tercih edilmemektedir ([Batuhan, 2022](#)). Oysaki ülkemizde Aralık 2011'de Türk Standartları TS OIC/SMIIC 1 isimli standart belirlenmiş ve İslami kurallara göre helal gıda ve mamullerinin her aşamasında uyulması gereken genel kuralları kapsamaktadır. Uygulanan her iki sistem de helal kesim standardı ile kontrol edilmektedir ([Sarıca, 2021](#)).

Etlik piliçlerde uygulanan yaş ve kuru yolum yöntemlerinin etin mikroorganizma düzeyi üzerine olan etkileri konusunda yeterince araştırma bulunmamaktadır. Kuru yolum yapılmış piliç etinin daha sağlıklı olduğu düşüncesi ile tüketiminin giderek artması, bu konuda yeni araştırmaların yapılmasını gerekli kılmaktadır. Bu çalışmada, Adana ilinde yaş ve kuru tüy yolma yöntemleri uygulanarak satışa sunulan karkaslardan elde edilen but ve göğüs etlerinde mikrobiyolojik test sonuçlarının karşılaştırılması amaçlanmıştır.

## MATERYAL ve YÖNTEM

### Materyal

Mevcut araştırmada 2015 yılı yaz mevsiminde (Temmuz-Ağustos) Adana ilinde 7 adet yaş ve 5 adet kuru yolum uygulanmış ve piyasada satışa sunulan farklı firmalara ait 12 ayrı marketten satın alınan ve son kullanma tarihine 4-5 gün kalmış 12 adet but ve 12 adet göğüs toplamda 24 adet et örneği steril koşullarda mikrobiyolojik analizleri yapılmak üzere Çukurova Üniversitesi, Zootekni Bölümü Hayvan Besleme Laboratuvarı'na getirildi ve aynı gün analizleri yapıldı.

### Yöntem

#### Et örneklerinin hazırlanması

Her bir but ve göğüs etinden (deri+et) steril poşetler içine 10 g tavuk örneği alınarak tartıldı. Örnekler üzerine 90 ml Tamponlu Pepton Suyu (TPS) eklenerek 200 devirde 2 dakika süreyle stomacher kullanılarak karışım homojenize edildi ve  $10^{-1}$  seyrelti çözeltisi hazırlandı. Hazırlanmış olan homojenizattan TPS (%0.1) kullanılarak dilüsyonlar hazırlandı.

### Toplam Mezofilik Aerobik Bakteri (TMAB) Sayımı

Hazırlanan seyreltimlerden 1 ml steril pipet ile alınarak Plate Count Agar (PCA) (Oxoid CM 325)'a dökme plak yöntemi ile ekimi yapılarak petriler 37°C'de 24-48 saatlik inkübasyona bırakıldı. İnkübasyon süresi sonunda koloniler sayılarak gram başına logaritmik Koloni Oluşturma Birimi (log kob/g) ile hesaplandı.

### Küf-maya sayımı

Toplam küf-maya sayımı için homojenize seyreltimlerden 0.5 ml steril pipetler ile alınarak drigalski spatula ile Potato Dextrose Agar (PDA) üzerine yayma plak tekniği ile uygulandı. Plakalar 25°C'de beş gün inkübe edildi, koloniler sayıldı ve sonuçlar gram başına logaritmik Koloni Oluşturma Birimi (log kob/g) olarak hesaplandı.

### Salmonella spp. Aranması

Salmonella izolasyonunda geleneksel yöntem kullanıldı (FAO, 1992).

### İstatistiksel Analizler

Araştırmadan elde edilen veriler IBM SPSS 19.0 (2010) paket programı kullanılarak analiz edildi. Ortalamalar arasında fark olup olmadığı varyans analizi (ANOVA) ile incelendi ve gruplar arasındaki anlamlılığı bulmak için Tukey testi uygulandı. İstatistiksel önem derecesi  $P \leq 0.05$  olarak kabul edildi.

## BULGULAR ve TARTIŞMA

Standart yaş ve kuru yolma ile tüyleri yolunmuş karkaslardan elde edilen ve piyasada satışa sunulan but ve göğüs etlerinde yapılan mikrobiyolojik analiz sonuçları Çizelge 1'de verilmiştir. Elde edilen sonuçlara göre, toplam mezofilik aerobik bakteri ve toplam küf-maya düzeyleri yolma yöntemi, karkas bölümü ve yolma yöntemi x karkas bölümü açısından istatistiki olarak önemsiz bulunmuştur ( $P < 0.05$ ). Buna rağmen toplam mezofilik aerobik bakteri miktarı kuru yolum uygulanan gruba ait butlarda (4.89) ve göğüslerde (5.34) rakamsal olarak daha yüksek olma eğiliminde olduğu belirlenmiştir.

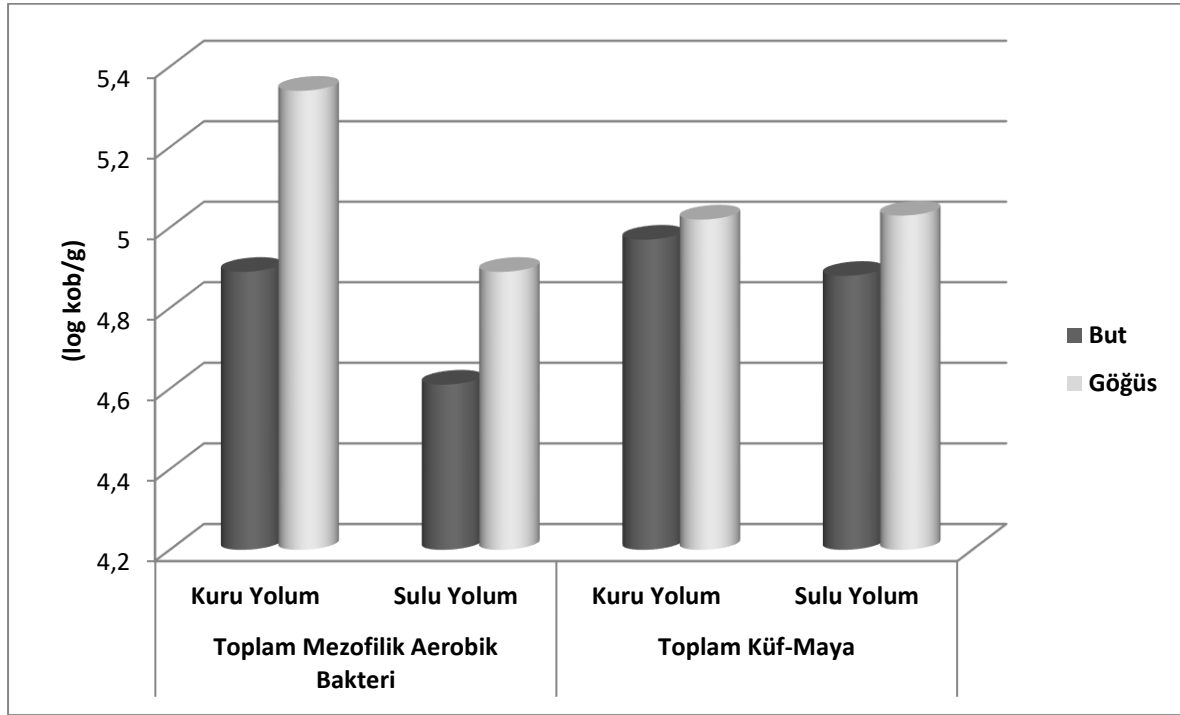
**Çizelge 1.** Sulu ve kuru yolum uygulanmış but ve göğüs etlerinde toplam mezofilik aerobik bakteri ve küf-maya düzeyleri (log kob/g).

**Table 1.** Total mesophilic aerobic bacteria and mold-yeast levels in wet and dry plucked drumstick and breast meats (log CFU/g).

Mikroorganizma	Yolma Yöntemi (Grup)	Karkas Bölümü (KB) (Ort. x Standart Sap.)		Grup	P	
		But	Göğüs		KB	Grup X KB
TMAB	Kuru Yolum	4.89±0.91	5.34±1.210	0.441 öd	0.443 öd	0.854öd
	Sulu Yolum	4.61±1.26	4.89±0.710			
Tolam Küf-Maya	Kuru Yolum	4.97±1.06	5.02±0.63	0.918 öd	0.795 öd	0.905 öd
	Sulu Yolum	4.88±1.30	5.03±0.59			

öd: önemli değil

Toplam küf-maya düzeyi ise kuru yolum grubunun butlarında rakamsal olarak yüksek bulunurken (4.97), kuru (5.02) ve sulu (5.03) yolum gruplarının göğüs etlerinin yaklaşık aynı seviyede olduğu tespit edilmiştir (Şekil 1). *Salmonella* ssp. ile ilgili yapılan analiz sonuçlarında sulu yolum uygulanmış butlarda 1 adet ve kuru yolum uygulanmış göğüslerde 1 adet *Salmonella* ssp. tespit edilmiştir.



**Şekil 1.** Yaş ve kuru yolum uygulanmış but ve göğüs etlerinde mikroorganizma düzeyleri (log kob/g).

**Figure 1.** Microbial load (log CFU/g) of wet and dry plucked drumstick and breast meat.

Sulu yolum yöntemi uygulanan karkaslarda yolumdan önce 50-60°C'ler arası sıcak suya (en yaygın kullanım 53°C) daldırma işlemi (ıslatma) yapıldığından mikroorganizma düzeyinde artış olduğu bildirilmektedir (Pool, 1954; Essary ve ark., 1958; Mallmann, 1958; Fromm, 1959; Goresline ve Haugh, 1959; Kinsley ve Mountnery 1966; Klose, 1971; Grossklaus ve ark., 1979; Anıl ve ark., 1989; Sarıca, 2021). Ayrıca iç organların çıkarılması esnasında, özellikle iç organların mekanik yolla uzaklaştırılması sırasında makine tarafından bağırsaklara zarar verilmekte ve karkasların dışkı ile kontaminasyonuna sebep olmaktadır. Karkas soğutma işlemi soğuk su kazanları içerisine sokularak sağlanıyor ise bu risk daha da artmaktadır. Islatma ve tüy yolma işlemleri derideki epidermis tabakasının hasarına neden olarak, bağırsakların çıkarılması ve karkasın soğutulması sırasında karkas yüzeyinde bakterilerin çoğalarak koloni oluşturmasına zemin hazırladığı belirtilmektedir (Erginkaya ve Yurdakul, 2010; Var ve ark., 2011). Literatür bilgilerinin aksine, mevcut çalışmada sulu ve kuru yolum uygulamasının etlerdeki mikroorganizma düzeyi üzerine bir etkisi olmamıştır.

Kuru yolum yönteminde, tüy yolumundan önce hayvanlar iç organları ile birlikte sıcak su kazanına daldırılarak ıslatma işlemi uygulanmadığı için toz, dışkı ve patojen mikroorganizmalarla kontaminasyon riskinin daha az olduğu, aynı zamanda karkas soğutma işlemi de hava akımı (70-80°C) ile yapıldığından çapraz bulaşmadan kaynaklı karkasın mikroroganizma yükünü artırıcı sebeplerin ortadan kaldırılmasından dolayı daha hijyenik bir yöntem olduğu bildirilmektedir (Riggs ve ark., 2011; Anonim, 2021). Anıl ve ark. (1989), kuru yolumun sulu yolumdan daha hijyenik bir yöntem olduğunu ve su uygulamasının mikrobiyal kontaminasyona yol açabileceğini belirtmiştir. Bu bildirimlerin aksine araştırma bulgularımıza göre her iki yolum yöntemi arasında mikroorganizma düzeyleri bakımından bir farklılık olmadığı gözlenmiştir.

Değişik arařtırmacılar tarafından yapılan birçok çalıřma ile satıřa sunulan tavuk etlerinde TMAB, toplam küf-maya ve *Salmonella* ssp. ile birlikte deęiřik mikroorganizmaların varlıęı tespit edilmiřtir (Saęun ve ark., 1996; Baydur, 2006; Süzme, 2012; Yıldırım ve ark., 2015; řahin ve ark., 2017; Kılıç Altun ve Atasever., 2018). Mevcut arařtırmada benzer řekilde TMAB, küf-maya ve *Salmonella* ssp. varlıęı tespit edilmiřtir.

Elliott ve Heiniger (1965), *Salmonella* ssp. çoęalmasını sınırlayan minimum sıcaklıęın 46.2°C olduęunu bildirmiřtir. Buna göre *Salmonella* ssp. yükünün artmasının önlenmesi için ıslatma suyu sıcaklıęının 47°C'den yüksek olması yeterli olmalıdır (Buhr ve ark., 2014). Türk Gıda Kodeksi Mikrobiyolojik Kriterler Yönetmelięinde (2011) mekanik olarak ayrılmıř tavuk etinde aerobik koloni sayısı limiti  $5 \times 10^5 - 5 \times 10^6$  log kob/g (ISO: 4833) ve *Salmonella* ssp. sayısı ise 0/25 g-mL (En/ISO: 6579) olarak verilmiřtir. Bu arařtırmada sulu yolum uygulanmıř butlarda ve kuru yolum uygulanmıř göęüslerde birer adet *Salmonella* ssp. ye rastlanmıř olması tavuk etinin çiftlikten sofraya gelinceye kadarki süreçte hijyen řartlarına titizlikle uyulması gereklilięini ortaya koymuřtur.

## SONUÇ

Mevcut arařtırma sonuçlarına göre, yař ve kuru tüy yolma yöntemi uygulanmıř ve piyasada satıřa sunulmuř but ve göęüs örneklerinde toplam mezofilik aerobik bakteri ve toplam küf-maya tespit edilmiř ancak düzeyleri bakımından önemli bir farklılık gözlenmemiřtir. Sulu yolum uygulanmıř but örneklerinde ve kuru yolum uygulanmıř göęüs örneklerinde 1'er adet olmak üzere 2 adet *Salmonella* ssp. tespit edilmiřtir. Sonuç olarak kesimde uygulanan tüy yolma yönteminin etlerdeki mikroorganizma düzeyine bir etkisinin olmadıęı, ancak *Salmonella* ssp. varlıęına rastlanmıř olması tavuk eti üretimi ile tüketimi arasındaki basamaklarda hijyen kurallarına uymanın ne denli önemli olduęunu ortaya çıkarmıřtır.

## ÇIKAR ÇATIřMASI

Yazar herhangi bir çıkar çatıřması olmadıęını beyan ederler.

## YAZAR KATKISI

Yazar, bu makaledeki tüm bölümlerin tamamını kendisi geliřtirdięini beyan eder.

## ETİK KURUL KARARI

Bu makale Etik Kurul Kararı gerektirmemektedir.

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## Development of Olive Harvesting Machine by Shaking

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### ABSTRACT

Olive harvesting machine by shaking was developed and fabricated using local materials with required specifications. The developed elements were frame and hitch, gear assembly, input and transmission shafts, vibrating unit, connecting rod, limb clamp, and transmission system. This research work aimed to evaluate the developed olive harvester capable to perform harvesting operation in the proper time using the tractor as the available economic source of power. Measurements covered the properties of olive fruit, stem, and limb for five olive varieties: Agizi, Manzanillo, Picholine, Kalamata and Arbiquen. The developed harvester was tested at three levels of frequency; 3.3, 6.7, and 10 Hz, four levels of stroke; 40, 80, 120, and 160 mm, and three levels of shaking time 60, 120 and 180 s. Results indicated that the effective range to attach the clamp on the olive limb were 30 to 40% of limb length. The average value of the maximum bending stress affecting the limb and limb deflection were 16.5 MPa and 196.6 mm respectively. In addition, results have provided the suitability of the developed shaker to harvest olive fruit. The suitability of the developed machine was judged through the fruit removal percentage. The values of performance parameters of olive harvester were 10 Hz optimum shaking frequency, 120 mm of stroke, and 120 s of shaking time.

#### RESEARCH ARTICLE

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- Stroke,
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## INTRODUCTION

Olive crops are considered one of the main crops all over the world. Olive cultivation increased considerably during the last two decades due to the great efforts paid for expanding olive cultivated areas with new cultivars in reclaimed areas ([FAOSTAT, 2018](#)). Olive harvesting is the most important operation among all production operation, and the manual harvesting is the traditional method in some countries. The cost of manual harvesting ranged from 30 to 60% of the total production costs and about 30% from the total price of crop production ([Cicek, 2011](#)). Mechanical harvesting of olives is a very important aspect in olive growing both to reduce the costs of production and to assure oil quality ([Testa \*et al.\*, 2014](#)). The mechanical harvesting of olives is performed either by shaking or combing the tree ([Nasini and Proietti, 2014](#)). [Almeida \*et al.\* \(2015\)](#) listed several factors that affect the mechanical harvesting of olive trees such as, tree shape, pruning methods, canopy density, orchard management, fruit removal force, fruit weight and the ratio between fruit removal force and fruit weight. [Ferguson \*et al.\* \(2010\)](#) stated that the factors affecting the mechanical harvesting of tree fruits by shaking are frequency, eccentricity, direction of shaking, fruit size, shaking time and detachment force to fruit weight ratio. Fruit detachment force (FDF) and fruit fresh weight were used to predict harvesting efficiency, although during harvesting process, fruit is subjected to bending and twisting movement besides pulling forces simulated by FDF measurements ([Ruiz \*et al.\*, 2018](#)). [Babanatsas \*et al.\* \(2019\)](#) derived a mathematical relationship to predict the power required for vibration. The derived equation correlates the modulus of elasticity at olive tree, rupture module on the olive tree, trunk diameter and length, correction module and shaking amplitude. [Ghonimy \(2006\)](#) derived a mathematical relationship to predict the suitable-shaking amplitude of limb tree shaker. The derived equation correlates the pulling force to fruit mass ratio, stem length, shaking frequency and damping ratio with the shaking amplitude. [Bernardi \*et al.\* \(2018\)](#) found that the work capacities varied between 5 tons of harvested olives per day when employing mechanical harvest aids and 18 tons per day when employing trunk shakers. [Guirado \*et al.\* \(2016\)](#) developed and tested a continuous lateral canopy shaker harvester on large olive trees in order to analyze the operating harvester parameters and tree properties to improve mutual adaptation. They found that the 77.3% of removal efficiency was achieved during 28 s shaking duration, 0.17 m amplitude vibration and 12 rod drum. This result was obtained reporting 0.26 s of accumulative shaking time over 200 m s<sup>-2</sup> resultant acceleration. [Morad and El-Termezy \(2020\)](#) evaluate the performance of manufactured olive harvester and they found that the harvester productivity, 26.7 tree/h, harvesting losses, 1.8%, specific energy, 0.674 kWh/tree, and operational cost, 3.152 L.E./tree were achieved at 300 rpm PTO speed, 15 cm vibration amplitude, and 1.0 m vertical height clamp position on the tree. [Zipori, \*et al.\* \(2014\)](#) compared between the final product quality and harvesting efficiency of the manual picking and trunk shaking mechanical harvesting for four different cultivars of green table olive. They found that elimination of rod beating significantly reduced harvesting efficiency, they also reported that the final product quality of the mechanically harvested olives of cv. Manzanilla was inferior to those picked manually. [Alzoheiry \*et al.\* \(2020\)](#) estimated the natural frequency (FN) of olive fruit stem system using one and two degrees of freedom models. Their result indicated that the FN value of full mature stage was 33.9 Hz, half-ripe olive was



31.9 Hz, and 28.0 Hz for full-ripe olive. They found that the maximum fruit removal percentage value, 90.6%, could be achieved at a frequency of 35 Hz and amplitude of 25 mm. A handheld olive harvester for small farms was developed and evaluated by Ghonimy *et al.* (2020). They found that a 1600 rpm of head rotating speed gave the optimal machine productivity, and fruit removal percentage. Thus, the aim of this study is to evaluate the developed olive harvester capable to perform harvesting operation in the proper time using the tractor as the available economic source of power.

## MATERIALS and METHODS

The plan of realizing the objective of this research was based upon designed the functional parts of the limb tree shaker, assembling of these functional parts in a compacted machine, and testing the developed machine in the field under normal operating conditions.

### Design considerations of the olive harvesting machine

1. It should be simple and should be constructed by locally available materials.
2. It should be small and realize reasonable capacity.
3. It should use standard components to save time and money.
4. It should be to minimize the mechanical damage of olives tree and fruits.

### The components of the developed olive harvester

The developed olive harvester consists of seven functional subsystems, frame and hitch, gear assembly, input and transmission shafts, vibrating unit, connecting rod, limb clamp, and transmission system.

### Frame and hitch

The frame and three-point hitch of the developed harvester were manufactured of steel pipe having 2.0 inch (5.08 cm) outside diameter. The frame, Figure 1, has two parts; the first part manufactured of steel pipe 5.08 cm diameter and 3 mm thickness, and includes the three-point hitch. The second part of the frame was manufactured of steel plate to fix the shaker by six screw bolts in the shaker base. Rubber pads were imbedded under the shaker base to reduce the vibrations resulted from the tractor.

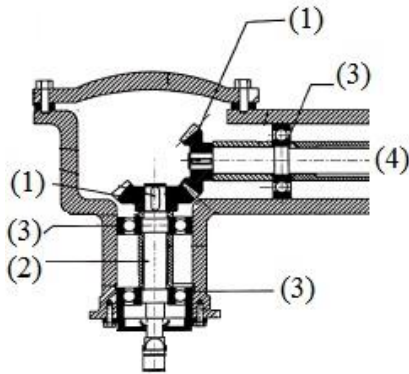


Figure 1. The frame and hitch points of olive harvester.

### Gear assembly

The gear assembly, Figure 2, of the olive harvester consisted of two bevel gears, two ball bearings, and input shaft. According to the design calculations, the kinematics of gears were 15 teeth, 5 mm module, 75 mm diameter, 20° pressure angle, and 41.42° pitch

angle. In addition, the forces (tangential, axial, and radial forces) and bending stress applied on gear tooth were 832.24 N, 227.15 N, 200.40 N, and 54.39 MPa respectively. The gears material is made of steel 50. The endurance limit ( $S_e$ ) of gear material is 235.5 MPa, and the total resulted factor of safety ( $F_s$  total) is  $1.73 > 1$ .



(1) Bevel gears                      (2) Input shaft                      (3) Ball bearing                      (4) Transmission shaft

**Figure 2.** The gear assembly of olive harvester.

The second part of gear assembly was input shaft. Input shaft receives the motion from the hydraulic motor and transmits it to gear assembly. According to the design calculations, the dimensions of input shaft are 274 mm length and 30 mm diameter. The input shaft material is steel 50. The mechanical properties of steel 50 included tensile strength, yield strength, elastic modulus, and poisson's ratio were 450 MPa, 345 MPa, 190-210 GPa and 0.27-0.30 respectively. Ball bearings (SKF) are usually used with some combination of radial and thrust (axial) load. The bearing 6006 satisfies safety.

The motion transmits from gear assembly to vibrating unit using transmission shaft, Figure 2. The length of transmission shaft is 840 mm. The transmission shaft material is steel 50. According to the design calculations, the minimum shaft diameter is 30 mm. Using transmission shaft of diameter equal to 40 mm satisfies safety.

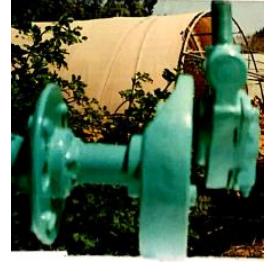
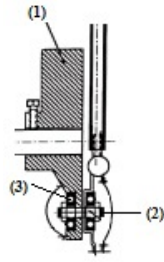
### Vibrating unit

Vibrating unit convert the rotating speed of the transmission shaft to a reciprocating movement (shaking stroke) to the connecting rod.

Vibrating unit, Figure 3, was designed from a circular disk ended with eccentric pin. The ball bearings are connected with a pin, which is fastened on the vibrating unit. The rotating disk of the shaker had four holes that were used to adjust the tested strokes.

To determine the suitable shaking stroke, five limbs for each olive variety were selected and balance hanged at the point, which represents 40% of the limb length, as recommended by [Erdoğan \*et al.\* \(2003\)](#). A test was run to measure the maximum limb deflection with load, Figure 4. A tree trunk was held vertically by a support the initial position of the limb attachment location where the load was applied was marked by using a pin on the leveling staff. Loads were added gradually at a rate of 5 kg and the limb deflection was measured. The loading was continued until the limb breaking occurred.

- (1) Vibrating unit.
- (2) Pin.
- (3) Ball bearing.



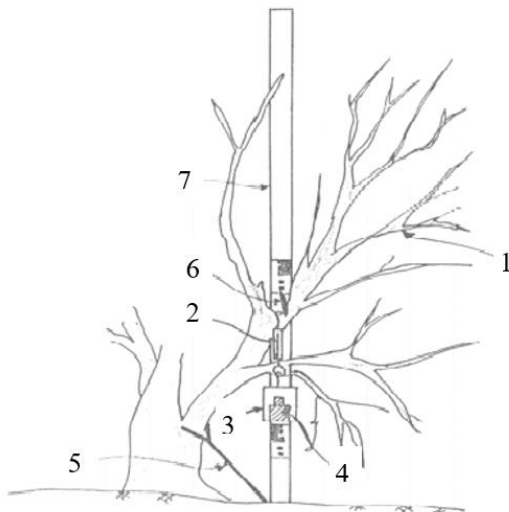
**Figure 3.** Vibrating unit.

### Connecting rod

The dimensions of the connecting rod, Figure 5, are two meter long and 19 mm diameter.

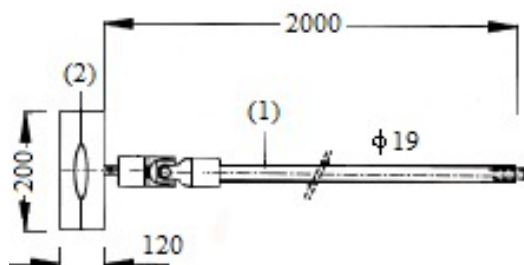
### Limb clamp

The limb clamp, Figure 5, consists of two parts of wood. The dimensions of the limb clamp were 200 mm length, 120 mm width and 70 mm depth. The inside phase of the clamp is covered with a layer of sponge which is covered with a layer of leather. The two inside parts of the clamp are called pad. The function of the pad system is to transmit shaking force from the shaker to the limb, and to distribute the shaking and clamping force over a layer area to minimize stresses in the contact area.



1. Limb of olives tree.
2. Pocket balance.
3. Load corer.
4. Variable load.
5. Support.
6. Pin.
7. Leveling staff.

**Figure 4.** Schematic diagram of loading procedures.



- (1) Connecting rod      (2) Limb clamp      Dimensions in mm

**Figure 5.** Connecting rod and limb clamp.

### Transmission system

To transmit the motion from the tractor to olive harvester, hydraulic transmission system was used. The transmission system of the olive harvester consists of three main elements.

**a) Hydraulic hose**

The function of the hydraulic hose is to convey hydraulic fluid to hydraulic components, valves, actuators, and tools. SAE 100R1 hydraulic hose were used to convey the hydraulic oil from the tractor to flow control valve, oil hydraulic motor and returns to the tractor. This hose is a high-pressure hose is used with petroleum or water-based fluids designed to power general industrial applications. The hose is single steel, wire-braided tubing that will operate in temperatures ranging from -40°C to 100°C.

**b) Flow control valve**

The VRFB 90-series flow control valve is used to adjust the speed of an actuator in both directions, which enable controlling on the rotating speed of the Input shaft and bevel gears. The specifications of the used control valve were 35 l min<sup>-1</sup> maximum flow rate, 350 bar maximum pressure, and 0.4 kg weight.

**c) Hydraulic motor**

The oil hydraulic motor MP 40 is used. The specifications of the used hydraulic motor are shown in Table 1.

**Table 1.** The specifications of the hydraulic motor.

Type	MP 40		
	Continous	Intermittierend	Peak
Max. Speed, rpm	1500	1750	--
Max. Torque, daNm	6.2	8.2	10.7
Max. Output, kW	8.4	11.6	--
Max. Oil Flow, l min <sup>-1</sup>	60	70	--
Max. Pressure Drop, kPa	12000	15500	22500
Max. Inlet Pressure, kPa	17500	20000	22500
Max. Return Pressure, kPa	17500	20000	22500
Weight, kg	5.7		

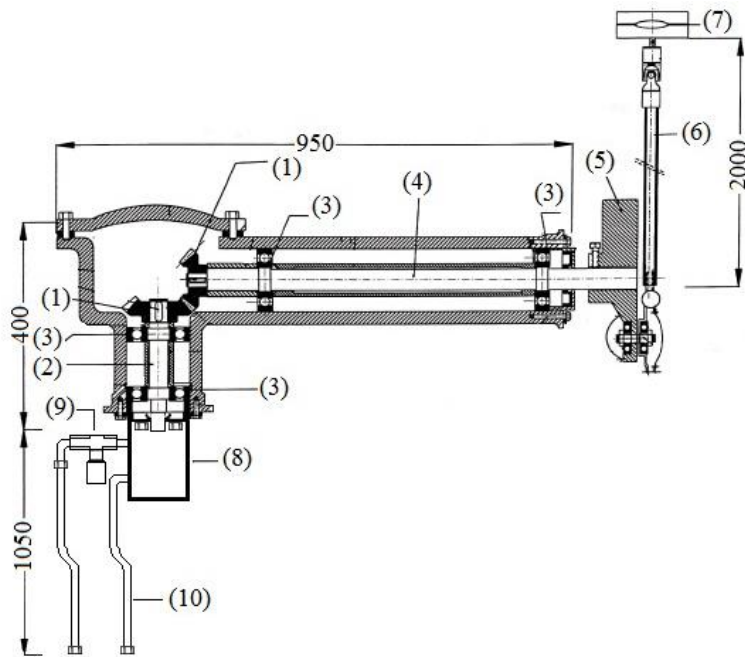
These subsystems were assembled in the compacted machine, Figures 6 and 7.

**Olive varieties**

The tested five olive varieties were Agizi, Manzanillo, Picholine, Kalamata and Arbiquen.

**Treatments**

The olive harvester was tested at three low tested values of frequencies were 3.3, 6.7 and 10 Hz ([Leone \*et al.\*, 2015](#)), four tested values of stroke 40, 80, 120, and 160 mm. While the period to shake any limb were 60, 120 and 180 s ([O'brien \*et al.\*, 1983](#)).



- |                        |                     |                        |
|------------------------|---------------------|------------------------|
| (1) Bevel gears        | (2) Input shaft     | (3) Ball bearing       |
| (4) Transmission shaft | (5) Vibrating unit  | (6) Connecting rod     |
| (7) Limb clamp         | (8) Hydraulic motor | (9) Flow control valve |
| (10) Hydraulic hose    |                     |                        |
- Dimensions in mm

**Figure 6.** Sectional plan of the developed olive harvester.



**Figure 7.** Olive-harvester during operation.

## Laboratory and experimental measurements

### Laboratory measurements

The laboratory measurements included some properties of fruit, stem and limb. The properties of fruits were mass, volume, length, maximum diameter, density, moisture content, oil content, flesh thickness of and detachment force. The properties of stem were stem length, diameter and the moisture content.

### Bending stress ( $\sigma_b$ ) of limb tree

The applied stress ( $\sigma_b$ ) on the limb was calculated from equation (1) (Shigley and Mitchell, 1983),

$$\sigma_b = \frac{10^{-6} \times F \times L \times d/2}{\frac{\pi}{64} d^4} \quad (1)$$

Where:

$\sigma_b$  = Bending stress, MPa,

$F$  = Applied load, N,

$L$  = Distance between load location and the base of limb, m,

$d$  = Limb diameter, m.

### Determine the point to attach the machine clamp on the limb length

Five limbs for each olive's variety were selected and shacked under constant frequency 6.7 Hz, 40 mm stroke, 60 s shaking time and different attachment locations (10, 20, 30, 40, and 50% of limb length).

### Experimental tests

The olive limbs were vibrated using the developed harvester. These limbs were chosen in a critical stage of maturity (contains full-ripe, half-ripe and full mature stage). Nylon nets were fixed on a stand to collect the removed fruits. The fruit removal percentage (*FRP*) was calculated from equation (2) according to [Polat \*et al.\* \(2007\)](#).

$$FRP = \frac{N_1}{N_2} \times 100 \quad (2)$$

Where:

$FRP$  = Fruit removal percentage, %

$N_1$  = Number of harvested olive fruits from one limb

$N_2$  = The total number of olive fruits from one limb

Also, the effect of mechanical harvesting on the trees were determined. The status of mechanically harvested trees was monitored through two seasons. These observations included leaf status (yellowing or falling leaves), main and subsidiary branches (fractures in the main or subsidiary branches of the tree), tree productivity, and bruises to the spines at the contact point of the vibrator clutch with the limb.

### Statistical analysis

The measured data for all variables were statistically analyzed by microcomputer program (CoStat ver. 6.400, 2008) via analysis of variance using randomized complete block design, three factors model. The means of treatments were obtained, and differences were assessed with Student-Newman-Keuls at 5% level of probability.

## RESULTS AND DISCUSSION

### The properties of olive fruit, stem, and limb

The mean values of some physical and mechanical properties of fruits for five full-ripe olive varieties are shown in Table 2.

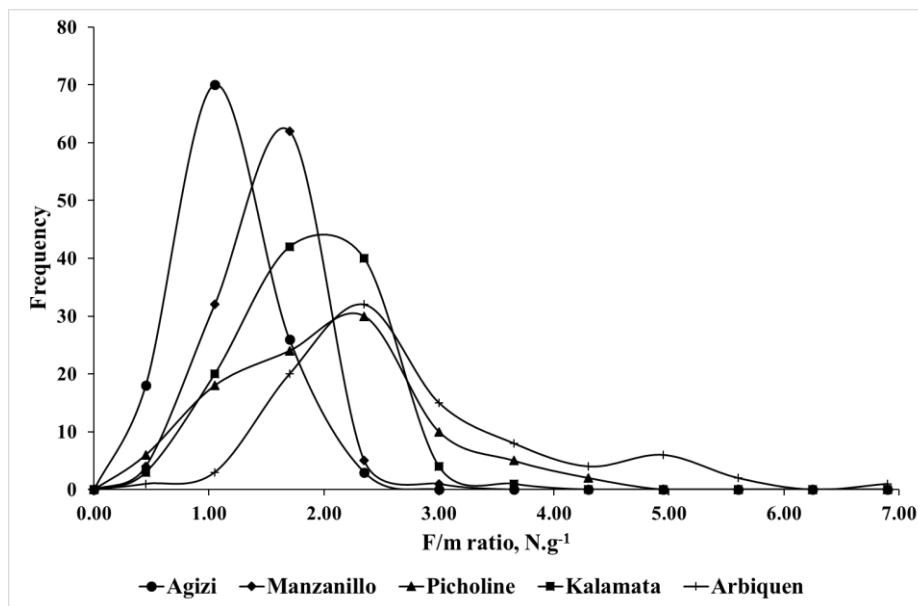
**Table 2.** Mean values of some properties of olive fruits for five varieties.

Characteristics	Olive varieties				
	Agizi	Manzanillo	Picholine	Kalamata	Arbiquen
Length, mm	26.5	20.3	22.5	23.3	13.6
Diameter, mm	18.4	16.2	14.4	11.8	11.2
Volume, cm <sup>3</sup>	4.95	3.06	2.11	1.96	0.97
Density, g cm <sup>-3</sup>	1.02	1.00	1.20	1.04	1.04
Moisture content, %	69.97	66.78	66.47	67.16	63.18
Oil content, %	4.72	20.29	15.30	15.10	15.55
Flesh thickness, mm	5.62	3.84	3.51	3.33	1.87
Detachment force ( $F$ ), N	6.65	5.81	5.36	4.36	2.40
Mass ( $m$ ), g	5.04	3.05	2.53	2.03	1.01
$F/m$ , N g <sup>-1</sup>	1.32	1.90	2.12	2.15	2.38

It is clear that a noticeable difference in the characteristics among the tested varieties existed. Detachment force of fruit was thoroughly examined as it plays an important role in the performance of shaking machine. The detachment force of great number olive limbs of the five different varieties of olives was determined. The frequency distribution of these measurements shown in Figure 8.

Analysis of these data proves that there is a direct relationship between the value of the detachment force of olive fruit and its mass. As the mass of olive fruit decreases the required detachment force decreases.

The stem and the limb of the olives tree for the five tested varieties occupied a significant interest as they are the affected parts of the tree and they determine the limits of the shaking action. The results of the physical properties of stem for different varieties were measured and are given in Table 3. Also, Table 3 shows the results of the physical properties of stem such as stem length, diameter and moisture content of different varieties. The minimum stem length is 24.7 mm for Arbiquen variety, and the maximum stem diameter is 43.5 mm for Agizi variety.

**Figure 8.** The frequency distribution of the F/W ratio.

**Table 3.** Mean values of some physical properties of stem and limb of five olive varieties.

Characteristics	Olive varieties				
	Agizi	Manzanillo	Picholine	Kalamata	Arbiquen
Stem length, mm	43.5±2.13 <sup>a</sup>	37.7±0.85	33.5±1.44	30.4±2.16	24.7±1.75
Stem diameter, mm	1.3±0.93	1.4±0.47	1.3±0.23	1.1±0.32	1.2±0.85
Stem MC, %	48.57±4.54	50.84±3.19	63.40±2.18	59.30±2.16	79.22±3.20
Limb length, m	2.71±0.80	2.27±0.23	2.34±0.14	2.43±0.15	2.17±0.41
Limb MC, %	12.50±1.85	13.00±2.05	12.00±1.85	16.50±3.15	19.00±2.65

<sup>a</sup> Standard deviation (SD); difference between two means  $\geq$ SD indicates significant difference.

The results of the limb diameter of five olive varieties are given in Table 4. In this experiment, five limbs were chosen for each of the tested varieties, and the limb diameter was estimated at five locations on the limb; 10, 20, 30, 40, and 50% of the limb length from its base. The results in table 4 show that the limb diameter increased by decreasing the location at the limb. The minimum value of limb diameter was 25.2 mm for Picholine variety at 50% of limb length while the maximum value of limb diameter was 50.3 mm for Arbiquen variety at 10% of limb length. In addition, the mean value of limb diameter is 35.7 mm. The results in Table 4 affect the dimensions of the padding area of the developed limb clamp of the harvester.

**Table 4.** Mean values of limb diameter of five olive varieties.

Characteristics	Limb diameter, mm					
	Agizi	Manzanillo	Picholine	Kalamata	Arbiquen	Average
10% Limb length	37.6± 0.58*	34.5± 0.93	37.5± 0.75	41.4± 0.68	50.3± 0.93	<b>40.3± 5.48</b>
20% Limb length	36.0± 0.40	32.1± 0.37	34.3± 0.93	39.9± 0.58	47.6± 1.03	<b>38.0± 5.45</b>
30% Limb length	34.3± 0.51	29.7± 0.55	31.5± 1.02	37.8± 0.75	44.4± 0.86	<b>35.5± 5.21</b>
40% Limb length	32.1± 1.12	28.1± 1.05	28.5± 0.51	36.0± 0.58	43.1± 0.40	<b>33.6± 5.56</b>
50% Limb length	29.9± 0.40	26.6± 0.66	25.2± 0.81	33.0± 0.97	41.1± 1.51	<b>31.2± 5.66</b>
<b>Average</b>						<b>35.7</b>

\* STDEV standard deviation based on the entire population

## Performance Parameters and Preliminary Experiments Analysis

### a) Effect of load on the limb deflection

Figure 9 shows the relation between the load in kg and the limb deflection in mm for each olive's variety. The maximum limb deflection causing a limb breaking was 235 mm for Picholine variety at 70 kg load. While the minimum limb deflection causing a limb breaking was 160 mm for Arbequien variety at 60 kg load. Thus, it can be considered that the deflection of the limb at which a breaking occurs is 160 mm. If a safety factor of 50% is used, then the maximum deflection of the limb was 80 mm, thus the maximum stroke was 160 mm. Thus, the tested values of stroke were 40, 80, 120 and 160 mm.



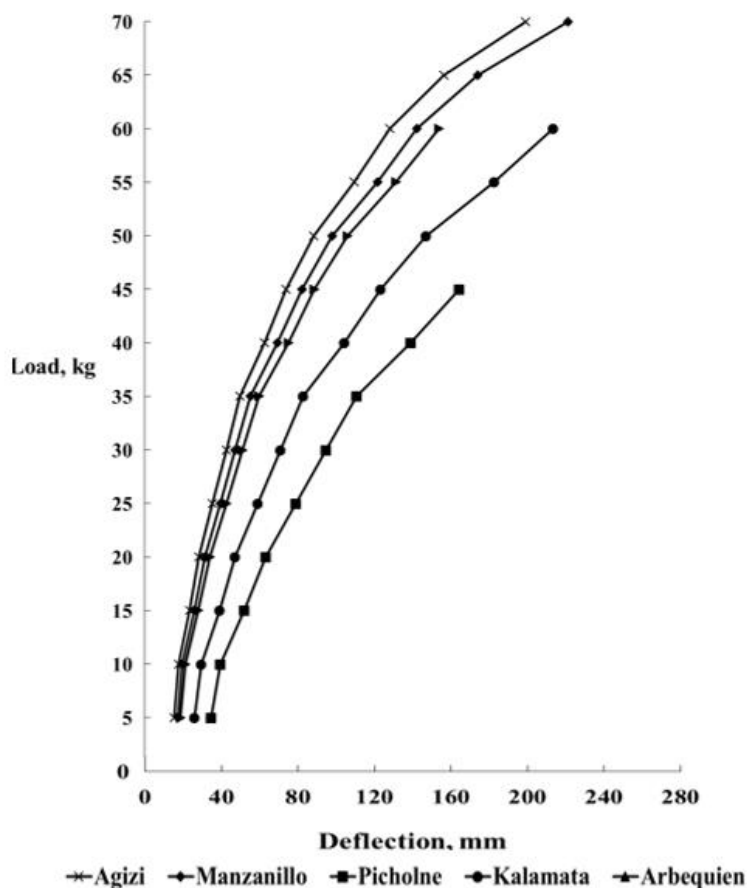


Figure 9. The effect of load on the olive limb deflection.

#### b) Effect of the point to attach the machine clamp with the limb on the fruit removal

The results of this experiment are tabulated in Table 5. The results show that increasing the attachment locations from 10% to 40% tends to increase the removal percentage by 8%, but the increase of attachment location from 40 to 50%, decreased the removal percentage by 4%. Thus, the optimum point to attach the clamp of the machine ranged from 30 to 40% of the limb length. These results are close to the results found by [Erdoğan et al. \(2003\)](#).

Table 5. The removal percentage under different location of olives varieties.

Location	Olive varieties					Average
	Agizi	Manzanillo	Picholine	Kalamata	Arbiquen	
10 % L	26.70±1.21 <sup>a</sup>	23.72±0.66	24.85±0.61	23.03±0.95	20.04±1.76	23.67
20 % L	29.25±1.05	26.26±1.21	25.91±1.01	26.38±1.00	22.64±0.63	26.09
30 % L	32.07±0.92	30.22±1.00	27.48±1.82	28.83±1.01	24.14±1.07	28.55
40 % L	34.98±1.16	33.29±1.66	32.01±1.16	31.87±1.44	26.76±1.54	31.78
50 % L	31.82±0.82	29.89±1.01	25.89±0.46	26.36±1.18	24.44±0.90	27.68

<sup>a</sup> Standard deviation (SD); difference between two means  $\geq$ SD indicates significant difference.

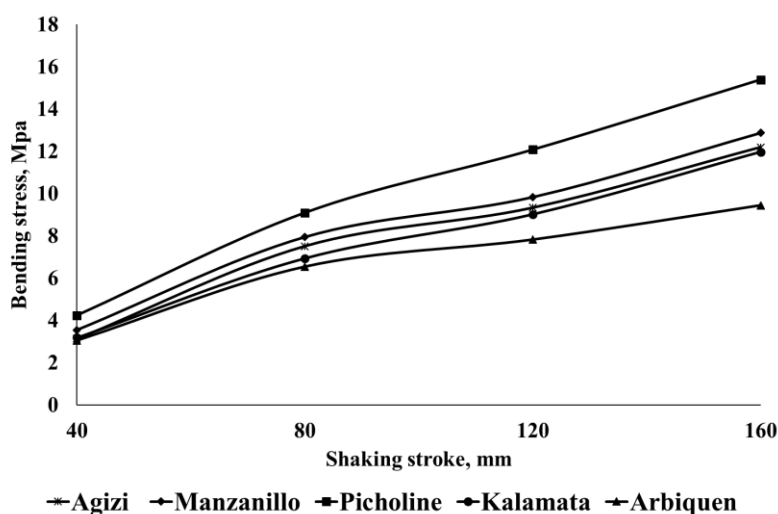
#### c) Relationship between shaking stroke and bending stress ( $\sigma_b$ ) affecting the olive limb

The results of bending stress ( $\sigma_b$ ) are presented in Table 6 and Figure 10. Table 6 shows the maximum stress on the limb causing breaking and the maximum deflection. The average value of maximum bending stress on the limb is 16.5 MPa with SD  $\pm$  3.626 MPa. The average value of maximum limb deflections for all varieties is 196.6 mm with SD  $\pm$  0.964 mm, this value were between (-) 36.6 mm and (+) 38.4 mm

around the average. Figure 10 shows the bending stress on the limb at four chosen strokes. It is clear that the affected stress at the different strokes ranged between 20.85% and 75.03% of the maximum stress causing breaking of the limb.

**Table 6.** The maximum bending stress ( $\sigma_b$ ) affecting the limb and maximum deflection

Varieties	Maximum ( $\sigma_b$ ), MPa	Maximum deflection, mm
Agizi	17.8	200
Manzanillo	17.7	164
Picholine	20.8	235
Kalamata	14.8	224
Arbiquen	11.2	160
Average	16.5	196.6
S.D.	3.626	0.694



**Figure 10.** Relation between shaking stroke and bending stress affecting the olive limb.

### Fruit removal percentage (*FRP*)

The average values of fruit removal percentage (*FRP*) for five olive varieties are shown in Figure 11. It is clear that the *FRP* increased by increasing applied frequency, shaking stroke and shaking time.

#### a) Effect of applied frequency on the fruit removal percentage

For Agizi variety at 40 mm stroke and 60 s shaking time, the *FRP* increased by 7.3 and 19.39% when the frequency increased from 3.3 Hz to 6.7 and 10 Hz respectively. Also, the *FRP* increased by 8% when the applied frequency increased from 6.7 Hz to 10 Hz. The same trend found at 80, 120 and 160 mm of shaking stroke. Thus, for Agizi variety, the increasing rate of *FRP* was 3% for each 1 Hz shaking frequency.

For Manzanillo variety at 40 mm stroke and 60 s shaking time, the *FRP* increased by 6.7 and 19.2% when the frequency increased from 3.3 Hz to 6.7 and 10 Hz respectively. Also, the *FRP* increased by 12.5% when the applied frequency increased from 6.7 Hz to 10 Hz. The same trend found at 80, 120 and 160 mm of shaking stroke.

For Picholine variety at 40 mm stroke and 60 s shaking time, the *FRP* increased by 7.95 and 22.39% when the frequency increased from 3.3 Hz to 6.7 and 10 Hz respectively. Also, the *FRP* increased by 14.44% when the applied frequency increased from 6.7 Hz to 10 Hz. The same trend found at 80, 120 and 160 mm of shaking stroke.

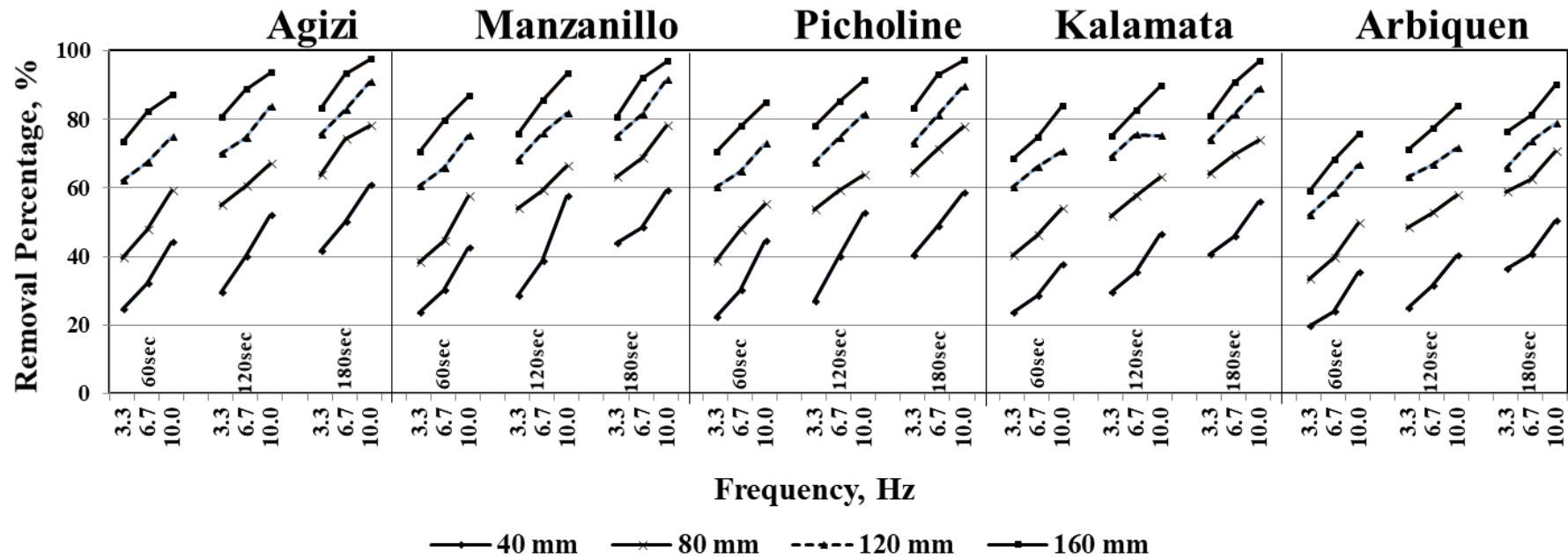
For Kalamata variety at 40 mm stroke and 60 s shaking time, the *FRP* increased by 4.96 and 13.91% when the frequency increased from 3.3 Hz to 6.7 and 10 Hz respectively. Also, the *FRP* increased by 8.95% when the applied frequency increased from 6.7 Hz to 10 Hz. The same trend found at 80, 120 and 160 mm of shaking stroke.

For Arbiquen variety at 40 mm stroke and 60 s shaking time, the *FRP* increased by 4.14 and 15.65% when the frequency increased from 3.3 Hz to 6.7 and 10 Hz respectively. Also, the *FRP* increased by 11.51% when the applied frequency increased from 6.7 Hz to 10 Hz. The same trend was found at 80, 120 and 160 mm of shaking stroke.

The detachment of the fruits can be attributed to the increase in the forces acting to detach the fruit as the frequency and the stroke increases the 10 Hz frequency with 160 mm stroke gave the highest fruit removal percentage for all olive varieties while, the 3.3 Hz frequency with 40 mm stroke was the smallest treatment for fruit removal percentage for olive varieties. These results are similar to those found by [Sola-Guirado \*et al.\* \(2019\)](#), who reported that the optimum frequency to operate the vibrator at 7.8 Hz. [Ghonimy \*et al.\* \(2021\)](#) found that the highest values of fruit removal, 81%, was performed at 27 Hz frequency and 60 mm or 70 mm stroke. [Younis \*et al.\* \(2017\)](#) found that the highest harvesting productivity was achieved at 1600 rpm and 3 min. Low damage percent were evaluated at 900 rpm and 3 min, machine achieved highest productivity and Low damage with Kornaki variety.

#### **b) Effect of shaking stroke on the fruit removal percentage**

For Agizi variety at 3.3 Hz frequency and 60 s shaking time, the *FRP* increased by 14.92, 37.88 and 48.58 % when the shaking stroke increased from 40 mm to 80, 120, and 160 mm respectively. The *FRP* increased by 14.92%, 22.96%, and 10.7% when shaking stroke increased (from 40 mm to 80 mm), (from 80 mm to 120 mm), and (from 120 mm to 160 mm) respectively. The same trend was found at 6.7 and 10 Hz of applied frequency. Thus, the rate of increase of the *FRP* was 0.4% for each 1 mm shaking stroke. The same trend was found for varieties Manzanillo, Picholine, Kalamata, and Arbiquen. It was noted that the use of 160 mm stroke caused some bruising to the tree limbs. Therefore, the suitable stroke is 120 mm.



**Figure 11.** The effect of applied frequency, shaking stroke and shaking time on olive removal percentage for Agizi, Manzanillo, Picholine, Kalamata, and Arbiquen varieties.

### c) Effect of shaking time on the fruit removal percentage

For Agizi variety at 3.3 Hz frequency and 40 mm shaking stroke, the *FRP* increased by 4.76 and 16.96% when the shaking time increased from 60 to 120, and 180 s respectively. Also, the *FRP* increased by 12.2 % when the shaking time increased from 120 s to 180 s. The same trend was found at 80, 120, and 160 mm shaking stroke. Thus, the rate of increase of the *FRP* was 0.14% for each one s shaking time. The same trend was found for the varieties Manzanillo, Picholine, Kalamata, and Arbiquen.

The results of the average values of *FRP* for all treatments indicated that the existence of sufficient variability among the five olive varieties under three frequencies; 3.3, 6.7, and 10.0 Hz and four strokes; 40, 80, 120, and 160 mm for fruit removal percentage, Figure 11. The 10 Hz frequency with 160 mm stroke gave the highest fruit removal percentage for all olive varieties while, the 3.3 Hz frequency with 40 mm stroke was the smallest treatment for fruit removal percentage for olive varieties. However, the Arbiquen olive variety gave the lower percent of fruit removal compared to the other olive varieties.

### The status of mechanically harvested trees

Harvested limbs were examined for signs of damage or breaking. Small bruising was found on the limbs when precisely examined. This bruising may increase the flowering and stimulate the floral buds. This may be because the bruising block the transportation of hormones other materials through the phloem and cause these materials to be diffused into the xylem and carried upward to the leaves, also the ethylene produced by bruising induces the flowering processes. An increase in flower initiation following possible phloem blockage due to vibrating action, is usually evident in the season following treatment ([Gawankar \*et al.\*, 2019](#)).

## CONCLUSION

The following conclusion can be made from the study:

1. At 30% to 40% of limb length were effective range to attach the clamp on the limb.
2. The minimum deflection which causes damage to the limb was 160 mm. The corresponding stroke was 320 mm. using safety factor 50%, the chosen range of stroke was 40, 80, 120 and 160 mm.
3. The fruit removal percentage (*FRP*) increased by increasing both stroke, shaking frequency and shaking time.
4. The increasing rate of *FRP* was about 3% for each 1 Hz shaking frequency for the range of applied frequency from 3.3 Hz to 10 Hz.
5. The *FRP* increased by 48.58% when the shaking stroke increased from 40 to 160 mm.
6. The *FRP* increased about 16.96% by increasing shaking time from 60 to 180 s.

Therefore, the olive harvesting machine can be fabricate using local materials with required specifications. The values of performance parameters of olive harvesting machine are 10 Hz shaking frequency, 120 mm of stroke, and 120 s of shaking time.

## DECLARATION OF COMPETING INTEREST

There is no conflict of interest between authors.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

This work was carried out in collaboration among all authors. All authors contributed equally in various roles.

## ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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## Effects of Alginate Based Edible Coating Applications on Physicochemical Quality of Minimal Processed Melon Slices

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### ABSTRACT

In this study, it was aimed to produce ready-to-eat melon slices by applying sodium alginate (SA) based edible film formulations on sliced “Kırkağaç” variety melons and to determine their quality under passive modified atmosphere conditions. In the study, sliced melons, first treated with 0.5% calcium chloride (CaCl<sub>2</sub>) solution, were coated with edible film solutions containing 1%, 1.5% and 2% sodium alginate (SA), then packed with polyethylene (30µm-PE) films and a group of samples that without packaging were stored for 6 days at +5°C and subjected to O<sub>2</sub>%, CO<sub>2</sub>%, total phenolic, antioxidant capacity (ABTS), pH, water-soluble dry matter, weight loss, texture and color (L\*, a\*, b\*) analyzes every 2 days period. As a result, it was determined that melon slices coated with in different concentrations of sodium alginate had not statistical differences in total phenolic content (54.66-58.03 GA mg kg<sup>-1</sup>), antioxidant capacity (256.2-295.8 mg kg<sup>-1</sup>), texture (0.433-0.444 N), water-soluble dry matter (9.70-9.88%) and weight loss (0.40-0.54) values on the 6th day of storage. As a result, it was determined that melon slices coated with sodium alginate had better properties in terms of antioxidant capacity, water soluble dry matter, weight loss and O<sub>2</sub> - CO<sub>2</sub> % values on the 6<sup>th</sup> day of storage.

#### RESEARCH ARTICLE

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#### Keywords:

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- Quality

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# Aljinat Bazlı Yenilebilir Kaplama Uygulamalarının Minimal İşlem Görmüş Kavun Dilimlerinin Fizikokimyasal Kalitesine Etkileri

## ÖZET

Bu çalışmada, sodyum aljinat (SA) bazlı yenilebilir film formülasyonlarının taze dilimlenmiş “Kırkağaç” çeşidi kavunlara uygulanması ile tüketime hazır kavun dilimlerinin üretilmesi ve pasif modifiye atmosfer koşullarında ürün kalitesinin belirlenmesi amaçlanmıştır. Çalışmada ilk olarak %0.5 kalsiyum klorür (CaCl<sub>2</sub>) çözeltisiyle muamele edilen kavun dilimleri %1, %1.5 ve %2 sodyum aljinat (SA) içeren yenilebilir film çözeltileriyle kaplandıktan sonra polietilen (30µm-PE) ambalajla paketlenmiş ve bir grup örnek de kaplama işlemine tabi tutulmadan ambalajsız olarak +5°C’de 6 gün depolanarak 2 günde bir %O<sub>2</sub>, %CO<sub>2</sub>, toplam fenolik madde, antioksidan kapasite (ABTS), pH, suda çözünür kuru madde, ağırlık kaybı, doku ve renk (L\*, a\*, b\*) analizlerine tabi tutulmuştur. Sonuçlar değerlendirildiğinde sodyum aljinat ile kaplanan kavun dilimlerinin depolamanın 6. gününde toplam fenolik madde (54.66-58.03 GA mg kg<sup>-1</sup>), antioksidan kapasite (256.2-295.8 mg kg<sup>-1</sup>), doku (0.433-0.444 N), suda çözünür kuru madde (%9.70-9.88) ve ağırlık kaybı (0.40-0.54) değerleri açısından kaplama konsantrasyonlarındaki değişimin istatistiksel olarak fark yaratmadığı belirlenmiştir.

### ARAŞTIRMA MAKALESİ

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### Anahtar Kelimeler:

- Modifiye atmosferde paketlenme,
- Soğukta muhafaza,
- Kalite

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## GİRİŞ

Günümüzde değişen beslenme alışkanlıkları besin değeri yüksek, tüketime hazır ve tazeliğini koruyan gıdaları daha fazla tercih edilir hale getirmiştir. Tüketime hazır gıda grupları içinde minimal işlenmiş meyve ve sebzelere olan talep de artmıştır. Minimal işlenmiş meyve ve sebzeler kabuk soyma, kesme/dilimleme, yıkama gibi sadece fiziksel işlemler uygulanarak üretilmektedirler ([Özyürek ve ark., 2013](#)). Bu ürünlerin işlenmesinde sadece fiziksel işlemlerin uygulanması nedeniyle ortaya çıkan kısıtlı raf ömrünü uzatmak ve besin değerini korumak amacıyla yenilebilir film ya da kaplama uygulamalarının yanında, modifiye atmosferde paketlenme (MAP) ve soğukta muhafaza uygulamalarının ayrı ayrı ya da bir arada kullanılma gerekliliği ortaya çıkmaktadır.

Yenilebilir kaplamalar, gıdaları korumak ve raf ömürlerini uzatmak amacıyla bir gıdanın yüzeyinde ince tabaka şeklinde oluşturulan, gıdayla beraber yenilebilir nitelikte olan, doğal kaynaklardan elde edilen ambalaj materyalleridir ([Keleş, 2002](#)). Yenilebilir kaplamaların gıdalara uygulanması, gıdaların kalitesini iyileştirme potansiyeline sahiptir, çünkü bunlar oksijen, karbondioksit ve aroma bileşenlerine seçici bariyerler oluşturabilirler. Aynı zamanda antimikrobiyal maddeler, antioksidanlar, aroma maddeleri, enzimler, fonksiyonel bileşenler (probiyotikler) veya besin maddeleri (mineraller ve vitaminler) gibi çeşitli aktif bileşenlerin taşıyıcıları olarak görev yapabilirler. Bu nedenle, yenilebilir kaplamalar gıdanın güvenliğini, besin değerini ve duyu özelliklerini arttırabilmektedirler ([Ribeiro ve ark., 2007](#); [Falguera ve ark., 2011](#); [Avena-Bustillos ve McHugh 2012](#); [Zhao, 2012](#)).

MAP tekniği ise gıdalarda mikrobiyolojik gelişimi azaltmak, raf ömrünü uzatmak ve enzimatik bozulmayı engellemek için ambalaj içindeki gaz atmosferinin değiştirilerek ürünün yapısına uygun özellikteki ambalaj malzemeleri ile ürünün ambalajlanması işlemidir (Erkan ve ark., 2000). Modifiye atmosfer ya gıda tarafından pasif olarak (pasif-MAP) ya da ambalaj içindeki gaz atmosferinin dışarıdan değiştirilmesiyle aktif olarak (aktif-MAP) oluşturulur.

Ülkemiz kavun yetiştiriciliğinde dünyada önemli bir yere sahiptir. TÜİK verilerine göre 2018 yılı kavun üretim miktarı 1.753.942 ton olarak gerçekleşmiştir (TÜİK, 2018). Çalışmada kullanılan Kırkağaç çeşidi kavun nakliyye ve depolamaya elverişlidir ve ülkemizde en fazla Manisa, Bursa, Balıkesir, Trakya ile kısmen Orta Anadolu'nun bazı yerlerinde geniş ölçüde yetiştirilmektedir. Kavun çoğunlukla taze olarak tüketilmektedir. Bunun yanı sıra tatlılara, sorbelere, dondurmalara ve kokteyllere ilave edilerek tüketim alanı bulmaktadır. Ancak sodyum aljinatın minimal işlem görmüş kavunlarda kullanımının araştırıldığı kısıtlı sayıda çalışma literatürde yer almaktadır. Bu çalışmalar aşağıda özetlenmiştir.

Senturk Parreidt ve ark. (2018), taze kesilmiş kantelop kavunlarını, sodyum aljinat bazlı yenilebilir film çözeltisi (%1.25 a a<sup>-1</sup>) kullanarak daldırma ve vakum emdirme yöntemleriyle kaplamış ve en iyi kaplama prosesi parametrelerini (daldırma süresi, vakum periyodu uzunluğu, vakum basıncı, atmosferik restorasyon süresi) tanımlamak amacıyla, farklı kaplama parametrelerinin, kaplanmamış ve kaplanmış örneklerin fiziksel kalite parametreleri (ağırlık değişimi, renk ve doku) üzerine etkisini belirlemişlerdir. Her iki sürecin de kavun dilimlerinin sertliğini artırdığı ancak vakum emdirme uygulamasında daha yüksek sertlik ve daha az ağırlık kaybı sonuçlarının olduğunu tespit etmişlerdir. Başka bir çalışmada ise kantalup türü kavunlar %2 tarçın kabuğu yağı ve %0-0.5 soya fasulyesi yağı içeren ve soya fasulyesi yağı içermeyen, %1 sodyum aljinat karışımı ile kaplanmıştır. Kaplama uygulamasının kavunların 21°C'lik ortamda depolama boyunca kalitesine etkisi ve inoküle edilmiş bazı patojenlerinin, doğal olarak oluşan küflerin ve mayaların canlılığına etkisi incelenmiştir. Örneklerin toplam çözünür kuru madde içeriğinde ve ağırlık kaybında, kaplama çözeltileri arasında önemli farklılık oluşmadığı ifade edilmiştir. Ayrıca, incelenen antimikrobiyal kaplama sisteminin, kantalup türünün mikrobiyolojik kalitesi ve güvenliğini artırma potansiyelini ortaya koyduğu belirtilmiştir (Zhang ve ark., 2015). Raybaudi-Massilia ve ark. (2008) taze kesilmiş "Piel de Sapo" kavununu malik asit ve tarçın, palmarosa ve limonotunun esansiyel yağları ile (%0.3-0.7) bunların aktif bileşenlerini (eugenol, geraniol ve sitral-%0.5) içeren aljinat bazlı yenilebilir filmle kaplayarak pasif MAP koşullarında depolanmışlar ve uygulanan işlemlerin kavunun raf ömrü ve güvenliği üzerindeki mikrobiyolojik ve fizikokimyasal etkisini araştırmışlardır. Pasif MAP koşullarında depolanan kaplanmamış kavun dilimlerinin mikrobiyolojik raf ömrü 3.6 güne kadar ve fizikokimyasal raf ömrü 14 günden daha az olduğu tespit edilmiştir. Pasif MAP ile malik asitin kombine etkisinin, kaplanmamış taze kavun dilimlerine kıyasla, kaplanmış taze kavun dilimlerinin mikrobiyolojik (9.6 güne kadar) ve fizikokimyasal (>14 gün) açıdan raf ömrünü arttırmada daha etkili olduğu belirlenmiştir.

Literatürde yer alan çalışmalar incelendiğinde farklı konsantrasyonlarda sodyum aljinat içeren yenilebilir film kaplama uygulamasının literatürde yer almadığı görülmektedir. Bu nedenle araştırma kapsamında %1, %1.5 ve %2 sodyum aljinat (SA) içeren yenilebilir film çözeltileriyle kaplanmış ve pasif MAP uygulanmış kavun

dilimlerinin depolanmasında ortaya çıkan bazı fizikokimyasal kalite değişimlerinin belirlenmesi amaçlanmıştır.

## MATERYAL ve YÖNTEM

### Materyal

Çalışmada yerel bir pazardan temin edilen “Kırkağaç” çeşidi kavun (12 adet) kullanılmış ve işlem görene kadar +10°C’de depolanmıştır. Film formülasyonları için orta viskoziteli sodyum aljinat (2.000 cp, %2, Sigma-Aldrich, Almanya) kullanılmış ve plastikleştirici olarak gliserol (Sigma-Aldrich, Almanya) ilave edilmiştir. Ayrıca kavunlarda ortaya çıkacak yumuşamayı engellemek ve film kaplamanın yüzeye daha iyi tutunmasını sağlamak için CaCl<sub>2</sub> (Sigma-Aldrich, Almanya), MAP uygulamasında ise polietilen-30 µm (Poliner plastik, Bursa) ambalaj materyali kullanılmıştır.

### Üretim Yöntemleri

Çalışma kapsamında; ilk olarak sodyum aljinat (SA) bazlı yenilebilir film formülasyonları ön denemeler sonucunda üç farklı konsantrasyonda (%1 SA, %1.5 SA ve %2 SA; m v<sup>-1</sup>) oluşturulmuştur. Bu amaçla 45°C’ye getirilmiş saf su içerisinde belirtilen konsantrasyonlarda sodyum aljinat ilave edilerek film çözeltileri hazırlanmış ve plastikleştirici olarak ön denemelerle belirlendiği şekilde %5 (v v<sup>-1</sup>) oranında gliserol ilave edilmiştir. Hazırlanan kaplama çözeltileri ultrasonik banyo (37 kHz) içerisinde ortam sıcaklığında (25°C) 30 dakika boyunca degaz işlemine tabi tutulmuştur (Karagöz, 2018). Kavunlar film kaplama öncesinde yıkanmış, kurulanmış, çekirdek evleri çıkarılmış ve kabuklarından ayrılarak 3x6 cm boyutlarında dilimlenmiştir. Dilimlenen kavunlar ön denemelerle belirlenmiş olan 4 dakikalık süre boyunca %0.5 (m v<sup>-1</sup>) konsantrasyondaki CaCl<sub>2</sub> çözeltisinde bekletildikten sonra süzülerek çıkarılmış ve %1, %1.5, %2 sodyum aljinat içeren film formülasyonları içerisine ön denemelerle belirlenmiş olan 5 dakikalık süre boyunca daldırılmış, film materyalinin fazlasının süzülmesi için 10 dakika bekletilmiş ve 1 saat ortam sıcaklığında kurumaya bırakılmıştır. Kaplama işlemi uygulanan ve uygulanmayan kavun dilimleri 30 µm polietilen (PE) film materyali pasif-MAP koşullarında paketlenerek depolanmıştır. Bir grup örnek de PE ambalajın etkinliğini ölçmek için kaplama yapılmadan ambalajsız olarak depolanmış, ayrıca kontrol grubu olarak kaplanmamış ve MAP uygulanmamış örnek grubu oluşturulmuştur. Her bir ambalaj 300 g örnek içerecek şekilde strafor tabaklar içerisinde paketlenmiştir. Hazırlanan beş örnek grubuna ait tüm materyaller kalite değişimlerinin belirlenmesi amacıyla +5°C’de %80 bağıl nemde 6 gün boyunca depolanmış ve 48 saatte bir fizikokimyasal analizlere tabi tutulmuştur.

### Analiz Yöntemleri

Örneklerin ambalaj içi gaz konsantrasyonları gaz analizatörü (Gaspacer-2, İngiltere) ile belirlenmiştir. Cihazın enjeksiyon iğnesi önceden paket üzerine yapıştırılmış kauçuk bir mantar içerisine batırılarak paketin tepe boşluğundaki %O<sub>2</sub> ve %CO<sub>2</sub> oranları ölçülmüştür (Demirdöven ve Batu, 2003). Kavunların toplam fenolik madde içerikleri Franke ve ark. (2004)’nın tanımladığı spektrofotometrik yöntemle, antioksidan kapasite değerleri ise Re ve ark. (1999) tarafından geliştirilen yöntemle belirlenmiştir. pH değeri ise pulp haline getirilmiş örneklerde WTW Inolab pH-Level-1 (Almanya) model pH-metre kullanılarak ölçülmüştür (Anonim, 1995). Ayrıca homojenize edilmiş

örneklerden Abbe refraktometresiyle (CETI 8200, İngiltere) suda çözünür kuru madde değerleri (SÇKM) belirlenmiştir. Kavunların başlangıç ve depolama sonundaki ağırlık değişimleri belirlenerek Tokatlı (2016)'da belirtildiği şekilde ağırlık kayıpları (%) hesaplanmıştır. Kavunlardaki dokusal (sertlik) değişimi belirlemek içinse kavunları dikey boyutundan 10 mm delmek için gereken maksimum kuvvet Newton cinsinden ölçülmüştür. Ölçümde, 10 mm çapında paslanmaz çelik başlığa sahip Zwick/Z0.5 (Almanya) test cihazı kullanılmıştır (Anonim, 2002). Kavunların meyve eti renkleri, Minolta renk ölçüm cihazı (Chroma meter, CR-300, Japonya) kullanılarak beyaz ve siyah standart bir plakada kalibre edildikten sonra, Hunter renk ölçüm parametreleri ile L\* (parlaklık), a\* (kırmızı/yeşil), b\*(sarı/mavi) değerleri kavun üzerinde üç farklı noktada ölçülerek belirlenmiştir (Anonim, 1995). Çalışmada kullanılan tüm analiz yöntemlerine ait detaylar Demir (2019)'da yer almaktadır.

### İstatistiksel Analiz

Verilere ait istatistiksel değerlendirmeler SPSS-21 paket programı kullanılarak yürütülmüştür (SPSS, 2013). Tüm analizler dört tekerrürlü olarak yürütülmüştür, n=4. Uygulamalara ait farklılıklar, örnek grupları ile depolama süresi-örnek gruplarının ortalamalarına ait karşılaştırmalar “tek yönlü ANOVA” analizine göre değerlendirilmiştir. Ortalama değerler %95 güven düzeyinde DUNCAN testi ile karşılaştırılmıştır.

## BULGULAR ve TARTIŞMA

**%O<sub>2</sub> ve %CO<sub>2</sub> konsantrasyonlarındaki değişimler:** Depolama süresince örneklerin bulunduğu ambalajlardaki %O<sub>2</sub> ve %CO<sub>2</sub> konsantrasyon değerleri Çizelge 1 ve 2'de verilmiştir. Örnek gruplarında depolama boyunca beklediği şekilde ambalaj içerisindeki oksijen oranları düşerken (P<0.05), karbondioksit oranları artmıştır (P<0.05). Depolama sonunda en düşük oksijen içeriği kaplama uygulanmadan paketlenen MAP grubunda %6.43, en yüksek oksijen içeriği ise %1 sodyum aljinatla kaplanan %1 SA+MAP grubunda %17.15 olarak tespit edilirken oksijen içerikleri arasındaki fark istatistiksel açıdan önemli bulunmuştur (P<0.05). Depolama sonunda en yüksek karbondioksit içeriği kaplama uygulanmadan paketlenen MAP grubunda %10.23, en düşük karbondioksit içeriği ise %1 sodyum aljinatla kaplanan %1 SA+MAP grubunda %3.0 olarak tespit edilmiştir; Karbondioksit içerikleri arasındaki fark istatistiksel açıdan önemli bulunmuştur (P<0.05).

**Çizelge 1.** Paket içi %O<sub>2</sub> konsantrasyon değerleri.

**Table 1.** O<sub>2</sub>% concentration values in the package.

Örnek Grupları	Depolama periyodu (Gün)			
	0	2	4	6
MAP	21.00±0.00 <sup>A</sup>	18.12±0.64 <sup>Bab</sup>	15.48±0.42 <sup>Cd</sup>	6.43±1.50 <sup>Dd</sup>
%1 SA+MAP	21.00±0.00 <sup>A</sup>	18.97±0.99 <sup>Ba</sup>	18.68±0.12 <sup>Ba</sup>	17.15±0.60 <sup>Ba</sup>
%1.5 SA+MAP	21.00±0.00 <sup>A</sup>	17.75±0.27 <sup>Bb</sup>	17.20±0.11 <sup>Bc</sup>	11.70±0.27 <sup>Cc</sup>
%2 SA+MAP	21.00±0.00 <sup>A</sup>	18.20±0.21 <sup>Bab</sup>	17.95±0.16 <sup>Bb</sup>	14.53±0.15 <sup>Cb</sup>

n=4, ±SD, <sup>A,B...</sup> harfleri aynı satırdaki örneklere,

<sup>a,b...</sup> harfleri aynı sütundaki örneklere ait istatistiksel farklılıkları göstermektedir, (P<0.05).

Sipahi ve ark, (2013) geliştirdikleri çok katmanlı antimikrobiyal aljinat bazlı yenilebilir kaplamayı karpuz dilimlerine uyguladıkları çalışmada, kaplama formülasyonunun tepe boşluğu kompozisyonunu etkilemediğini ( $P>0.05$ ) ancak çok tabakalı antimikrobiyal yenilebilir kaplamanın solunum sırasında  $CO_2$  üretimini önlemeye yardımcı olduğunu ve bunun nedeninin, solunum için gerekli olan gazlara daha iyi bariyer görevi yaptığını bildirmişlerdir. Bir diğer çalışmada ise Oms-Oliu ve ark. (2008c) tarafından taze kesilmiş armut dilimlerine N-asetilsistein ve glutation içeren aljinat bazlı, pektin ve jellan içeren yenilebilir kaplamaların uygulandığı çalışmada paketlerin tepe boşluğunda bulunan gaz değişimindeki genel eğilimin  $O_2$  konsantrasyonlarının %5-8'e,  $CO_2$  konsantrasyonlarının ise %15-20'ye yükselmesi şeklinde olduğu tespit edilmiştir. N-asetilsistein ve glutatyon içeren formülasyonlarla kaplanmış armut dilimlerinde N-asetilsistein ve glutatyonun, meyve dokusunun kaplama ortamında  $O_2$ 'i absorbe etme yeteneğini azaltmasının ve ambalaj atmosferinde daha fazla  $O_2$  bırakılmasının antiesmerleşme ajanları ile muamele edilmeyen kaplanmış numunelerden bir miktar yüksek olduğu bildirilmiştir. Bu çalışmada ise 6 günlük depolama sonunda örneklerin % $O_2$  içerikleri %6.43-17.15,  $CO_2$  içeriklerinin ise %3.0-10.23 arasında oldukları belirlenmiştir. Bu nedenle elde edilen sonuçların literatürle kısmen uyumlu olduğu denge gaz bileşiminin yenilebilir film kaplanmamış örnek grubunda daha kısa sürede oluştuğu saptanmış ve kavunlarda aljinat bazlı yenilebilir film ve pasif-MAP kombinasyonunun uygulanmasında denge gaz bileşiminin oluşması için 6 günden fazla bir depolama gerektiği belirlenmiştir.

**Çizelge 2.** Paket içi % $CO_2$  konsantrasyon değerleri.

**Table 2.**  $CO_2$  % concentration values in the package.

Örnek Grupları	Depolama periyodu (Gün)			
	0	2	4	6
MAP	0.03±0.00 <sup>D</sup>	3.23±0.76 <sup>Ca</sup>	5.03±0.15 <sup>Ba</sup>	10.23±1.50 <sup>Aa</sup>
%1 SA+MAP	0.03±0.00 <sup>D</sup>	1.57±1.35 <sup>Cb</sup>	2.55±0.08 <sup>BCc</sup>	3.00±0.13 <sup>Bd</sup>
%1.5 SA+MAP	0.03±0.00 <sup>D</sup>	2.85±0.05 <sup>Cab</sup>	3.02±0.13 <sup>Cb</sup>	6.30±0.35 <sup>Bb</sup>
%2 SA+MAP	0.03±0.00 <sup>D</sup>	2.55±0.43 <sup>Cab</sup>	2.70±0.06 <sup>Cc</sup>	4.90±0.11 <sup>Bc</sup>

n=4, ±SD, \*A,B... harfleri aynı satırdaki örneklere,

a,b... harfleri aynı sütundaki örneklere ait istatistiksel farklılıkları göstermektedir, ( $P<0.05$ ).

**Toplam Fenolik Madde ve Antioksidant Kapasite Değerleri:** Örneklere ait depolama başlangıcı toplam fenolik madde değerleri 55.9-61.1 GA mg kg<sup>-1</sup> aralığında belirlenmiştir (Çizelge 3). Depolama sonunda ise 54.66-79.30 GA mg kg<sup>-1</sup> olarak belirlenmiştir. Depolama boyunca MAP, %1 SA+MAP ve %1.5 SA+MAP örneklerinin toplam fenolik madde içeriklerindeki değişim istatistiksel olarak önemsiz bulunurken ( $P>0.05$ ), kontrol ve %2 SA+MAP örneklerinin toplam fenolik madde içeriklerindeki değişim istatistiksel olarak önemli bulunmuştur ( $P<0.05$ ). Kontrol örneğinin toplam fenolik madde içeriği 6.günde önemli düzeyde artarken ( $P<0.05$ ), %2 SA+MAP grubunun toplam fenolik madde içeriği azalmıştır ( $P<0.05$ ), 0. ve 2. günlerde tüm örnek grupları arasındaki farkın ise istatistiksel olarak önemsiz olduğu belirlenmiştir ( $P>0.05$ ). Kontrol ve MAP grubundaki örneklerin fenolik madde değerleri depolama boyunca artış göstermiştir. Oms-Oliu ve ark. (2008a) taze kesilmiş kavunların fenolik bileşik miktarlarındaki artışın antioksidan kapasitenin artması ile ilgili olduğunu bildirmişlerdir. Ayrıca kontrol örneklerindeki artışın daha yüksek olduğu

görülmektedir. Bu durumun suyun buharlaşmasıyla birim ağırlıktaki kuru madde oranının artışıyla kaynaklı olduğu düşünülmektedir. Sodyum aljinatla kaplı örneklerin toplam fenolik madde değerlerinin 4. güne kadar azaldığı, 6. gün ise artış gösterdiği belirlenmiştir. Oms-Oliu ve ark. (2008b) yaptıkları çalışmada aktif-MAP koşullarında taze kesilmiş 'Piel de Sapo' kavununu depolamışlar ve daha yüksek O<sub>2</sub> içeriği bulunan atmosferlerin daha fazla fenolik bileşik üretimine neden olduğunu belirlemişlerdir. Bu durumun paket içinde bulunan çok düşük O<sub>2</sub> ve yüksek CO<sub>2</sub> konsantrasyonlarının yol açtığı ve oksidatif stres ile ilgili olduğunu ve bu nedenle aljinatla kaplanan kavun dilimlerinde artan fenolik birikimin hem meyve hem de ambalaj tepe boşluğundaki atmosferlerin önemli ölçüde modifikasyonuna bağlı olabileceğini bildirmişlerdir. Oms-Oliu ve ark. (2008c) tarafından yapılan bir diğer çalışmada da depolama sonunda sadece aljinatla kaplanmış armut dilimlerinin toplam fenolik madde miktarının ortalama 400 mg GA 100g<sup>-1</sup> değerlerinden 350 mg GA 100g<sup>-1</sup> değerlerine düştüğü, kaplanmamış örneklerin ise 360 mg GA 100g<sup>-1</sup> değerlerine düştüğü bildirilmiştir.

Tüm örneklerin antioksidan kapasite değerlerinde depolamanın 4.gününde kadar bir artış, 6.gününde ise azalma belirlenmiştir (Çizelge 4). %1 SA+MAP ve %1.5 SA+MAP örneklerinde 0. günden 4. güne kadar antioksidan kapasite değerlerinde istatistiksel açıdan önemsiz düzeyde bir artış gözlemlenirken (P>0.05), 6. günde istatistiksel olarak önemli düzeyde bir azalma meydana geldiği saptanmıştır (P<0.05). Kontrol ve %2 SA+MAP örneklerinin ise 4. gün antioksidan kapasite değerlerinde diğer günlerde elde edilen verilere göre istatistiksel açıdan önemli düzeyde artış meydana gelmemiştir (P<0.05). Ancak depolama boyunca tüm örnek gruplarının arasındaki fark istatistiksel açıdan önemsiz bulunmuştur (P>0.05). Robles-Sanchez ve ark. (2012) tarafından yapılan çalışmada sadece aljinatla kaplanan ve kaplama uygulanmayan taze mango dilimlerinin antioksidan kapasite değerlerindeki değişim mevcut çalışmaya benzer sonuçları içermektedir.

**Çizelge 3.** Toplam fenolik madde (GA mg kg<sup>-1</sup>) içeriklerindeki değişimler.

**Table 3.** Changes in total phenolic (GA mg kg<sup>-1</sup>) content.

Örnek Grupları	Depolama periyodu (Gün)			
	0	2	4	6
Kontrol	55.89±2.26 <sup>Ba</sup>	62.01±4.28 <sup>Ba</sup>	65.53±3.02 <sup>Ba</sup>	79.30±8.67 <sup>Aa</sup>
MAP	55.89±2.26 <sup>Aa</sup>	57.26±4.31 <sup>Aa</sup>	56.65±1.61 <sup>Ab</sup>	58.03±1.94 <sup>Ab</sup>
%1 SA+MAP	57.57±3.65 <sup>Aa</sup>	56.50±2.67 <sup>Aa</sup>	54.36±2.18 <sup>Ab</sup>	55.58±1.80 <sup>Ab</sup>
%1.5 SA+MAP	58.64±4.09 <sup>Aa</sup>	54.66±3.69 <sup>ABa</sup>	52.06±1.83 <sup>Bb</sup>	54.66±2.37 <sup>ABb</sup>
%2 SA+MAP	61.10±2.40 <sup>Aa</sup>	57.42±2.50 <sup>ABa</sup>	52.82±3.95 <sup>Bb</sup>	54.66±0.79 <sup>Bb</sup>

n=4, ±SD, \*A,B... harfleri aynı satırdaki örneklere,

a,b... harfleri aynı sütundaki örneklere ait istatistiksel farklılıkları göstermektedir, (P<0.05).

**Çizelge 4.** Depolama süresince antioksidan kapasite ( $\text{mg kg}^{-1}$ ) değerleri.  
**Table 4.** Antioxidant capacity ( $\text{mg kg}^{-1}$ ) values during storage.

Örnek Grupları	Depolama periyodu (Gün)			
	0	2	4	6
Kontrol	282.3±16.8 <sup>Ba</sup>	305.1±9.7 <sup>Ba</sup>	413.9±8.4 <sup>Aa</sup>	279.3±27.1 <sup>Ba</sup>
MAP	282.3±16.8 <sup>Aa</sup>	311.6±18.6 <sup>Aa</sup>	344.0±81.6 <sup>Aa</sup>	266.9±19.2 <sup>Aa</sup>
%1 SA+MAP	297.0±22.5 <sup>ABa</sup>	318.0±15.9 <sup>Aa</sup>	337.0±17.7 <sup>Aa</sup>	273.2±20.6 <sup>Ba</sup>
%1.5 SA+MAP	288.4±6.5 <sup>ABa</sup>	304.0±7.4 <sup>ABa</sup>	331.9±46.1 <sup>Aa</sup>	256.2±6.0 <sup>Ba</sup>
%2 SA+MAP	312.1±12.6 <sup>ABa</sup>	313.0±5.4 <sup>Ba</sup>	345.0±16.6 <sup>Aa</sup>	295.8±18.1 <sup>Ba</sup>

n=4, ±SD, \*A,B... harfleri aynı satırdaki örneklere,

a,b... harfleri aynı sütundaki örneklere ait istatistiksel farklılıkları göstermektedir, (P<0.05).

Antioksidan kapasitedeki en önemli artış ambalajsız kontrol örneklerinde gözlenmiştir. Bu sodyum aljinat bazlı kaplamaların, kaplanmış kavun dilimlerinde antioksidan bileşiklerin tüketimini yavaşlatabilen, oksijen iletimine karşı etkili bir bariyer görevi yaparak, depolama sırasında fenolik bileşiklerin kaybını geciktirebildiği ve bu nedenle antioksidan kapasite değerlerinde benzer dalgalanmaların olacağı düşünülmektedir (Aloui ve ark., 2014). Oms-Oliu ve ark. (2008a)'na göre, jellan ile kaplanmış taze kesilmiş kavunların antioksidan aktivitesindeki artış, meyve çevresindeki gaz bileşiminin modifikasyonu ile açıklanmıştır. İncelenen meyve türü, olgunlaşma aşaması ve kaplama materyalinin tipi gibi birçok faktör, antioksidan aktiviteyi güçlü bir şekilde etkileyebilmekte ve bu nedenle antioksidan aktivite için elde edilen farklı sonuçları açıklayabilmektedir.

**Doku değerleri:** Meyve sertliği, tüketiciler tarafından kabul edilebilirliği güçlü bir şekilde etkileyen kritik bir faktördür ve hasat sonrası meyvenin kalitesini korumak ve ekonomik kayıpları önlemek için doku kaybının kontrolü önemlidir (Aloui ve ark., 2014). Kavun dilimlerinin kalsiyum klorür çözeltisine daldırıldıktan sonra sodyum aljinat filmi ile kaplanması işlemi depolamanın başında doku değerlerinde artışa neden olmuştur (Çizelge 5). Depolamanın 4. gününde örnekler arasındaki fark istatistiksel olarak önemli bulunurken (P<0.05), en yüksek doku değeri 0.552 N olarak %1.5 SA+MAP örneğinde tespit edilmiştir. Depolama sonunda kontrol grubunun doku değeri 0.371 N, MAP ile depolanan kavun dilimlerinin doku değeri 0.296 N, %1 SA+MAP grubunun doku değeri 0.441 N, %1.5 SA+MAP grubunun doku değeri 0.433 N ve %2 SA+MAP grubunun doku değeri ise 0.444 N olarak tespit edilmiştir.

**Çizelge 5.** Doku (N) değerlerindeki değişimler.

**Table 5.** Changes in texture (N) values.

Örnek Grupları	Depolama periyodu (Gün)			
	0	2	4	6
Kontrol	0.384±0.09 <sup>Ba</sup>	0.516±0.09 <sup>Aa</sup>	0.368±0.10 <sup>Bbc</sup>	0.371±0.08 <sup>Ba</sup>
MAP	0.384±0.09 <sup>ABa</sup>	0.507±0.14 <sup>Aa</sup>	0.478±0.18 <sup>Aab</sup>	0.296±0.11 <sup>Ba</sup>
%1 SA+MAP	0.444±0.12 <sup>Aa</sup>	0.467±0.12 <sup>Aa</sup>	0.409±0.09 <sup>Abc</sup>	0.441±0.20 <sup>Aa</sup>
%1.5 SA+MAP	0.483±0.16 <sup>ABa</sup>	0.531±0.13 <sup>Aa</sup>	0.552±0.08 <sup>Aa</sup>	0.433±0.14 <sup>ABa</sup>
%2 SA+MAP	0.485±0.11 <sup>Aa</sup>	0.451±0.15 <sup>Aa</sup>	0.335±0.09 <sup>Ac</sup>	0.444±0.18 <sup>Aa</sup>

n=4, ±SD, \*A,B... harfleri aynı satırdaki örneklere,

a,b... harfleri aynı sütundaki örneklere ait istatistiksel farklılıkları göstermektedir, (P<0.05).

Depolamanın 0., 2. ve 6. gününde örnekler arasındaki farklılık istatistiksel olarak önemsiz bulunmuştur ( $P>0.05$ ). Depolama boyunca en az değişim %1 SA+MAP ve %2 SA+MAP örnek gruplarında görülmüş ve bu örnek gruplarındaki kavun dilimlerinin depolama periyodları arasındaki farklılık istatistiksel olarak önemsiz bulunmuştur ( $P>0.05$ ). Bunun sonucu olarak da tüm örnek grupları ile karşılaştırıldığında %1 SA+MAP ve %2 SA+MAP örnek gruplarının kavun dilimlerinin sertliğinin korunmasında daha etkili oldukları belirlenmiştir. [Raybaudi-Massilia ve ark. \(2008\)](#) tarafından yapılan çalışmada çapraz bağlama için çözeltiye eklenen kalsiyumun bir sonucu olarak kaplama uygulamasından hemen sonra, kaplanmamış ve kaplanmış taze kesilmiş kavun dilimleri arasında önemli farklılıklar bulunmuştur. Bu anlamda, diğer araştırmacılar da kalsiyum klorürün yenilebilir kaplamalarda kullanıldığında meyvelerdeki sertliğini koruyabildiğini bildirmişlerdir ([Olivas ve Barbosa Cánovas, 2005](#); [Rojas-Graü ve ark., 2008](#)). [Maftoonazad ve ark. \(2008\)](#) şeftalileri sodyum aljinat ve metil selülozla kaplayarak  $15^{\circ}\text{C}$ 'de ve %40 bağıl nemde depolamışlardır. Şeftalilerin doku değerleri hem kaplanmış hem de kaplanmamış meyveler için depolama süresi ilerledikçe doku yumuşaması göstererek azalmıştır. Bununla birlikte, meyvelerin kaplanması, sertliğin korunumu üzerinde anlamlı bir etki göstermiştir.

**pH, Suda Çözünür Kuru madde ve Ağırlık Kaybı Değerleri:** Depolama başlangıcında tüm örneklerin pH değerlerinde (Çizelge 6.) istatistiksel açıdan herhangi bir fark bulunmazken ( $P>0.05$ ), diğer tüm depolama periyodlarındaki fark istatistiksel olarak önemli bulunmuştur ( $P<0.05$ ). Depolama sonunda en yüksek pH değeri kontrol grubunda 5.94 olarak tespit edilirken, en düşük pH değeri MAP grubunda 5.30 olarak belirlenmiştir. MAP ve SA+MAP grupları arasında depolama sonunda istatistiksel açıdan fark yoktur ( $P>0.05$ ). Depolama sonunda tüm örnek gruplarının pH değerinde düşüş saptanmıştır. Depolama boyunca bu değerler arasındaki fark kontrol grubunda istatistiksel olarak önemsiz bulunurken diğer örnek gruplarında ise istatistiksel olarak anlamlı bulunmuştur ( $P<0.05$ ). [Mannozi ve ark. \(2017\)](#) yaban mersini meyvesini sodyum aljinat, pektin ve sodyum aljinat+pektin ile kaplayarak  $4^{\circ}\text{C}$ 'de 14 gün muhafaza etmişlerdir. Dikkate alınan her bir saklama süresinde kaplanmamış ve farklı filmlerle kaplanmış örnekler arasında pH değerleri arasında anlamlı bir fark tespit edilmemiştir.

**Çizelge 6.** pH değerleri.

**Table 6.** pH values.

Örnek Grupları	Depolama periyodu (Gün)			
	0	2	4	6
Kontrol	5.94±0.08 <sup>Aa</sup>	6.01±0.01 <sup>Aab</sup>	6.08±0.03 <sup>Aa</sup>	5.94±0.01 <sup>Aa</sup>
MAP	5.94±0.08 <sup>Aa</sup>	6.14±0.01 <sup>Aa</sup>	5.40±0.09 <sup>Bc</sup>	5.30±0.01 <sup>Bd</sup>
%1 SA+MAP	6.04±0.03 <sup>Aa</sup>	5.90±0.06 <sup>ABbc</sup>	5.89±0.03 <sup>ABab</sup>	5.69±0.06 <sup>Bb</sup>
%1.5 SA+MAP	5.87±0.01 <sup>Aa</sup>	5.93±0.14 <sup>Ac</sup>	5.73±0.12 <sup>Ab</sup>	5.33±0.06 <sup>Bd</sup>
%2 SA+MAP	5.93±0.03 <sup>Aa</sup>	5.85±0.01 <sup>Ac</sup>	5.85±0.01 <sup>Aab</sup>	5.52±0.03 <sup>Bc</sup>

n=4, ±SD, \*A,B... harfleri aynı satırdaki örneklere,

a,b... harfleri aynı sütündeki örneklere ait istatistiksel farklılıkları göstermektedir, ( $P<0.05$ ).

Kontrol, MAP, %1 SA+MAP ve %2 SA+MAP gruplarının SÇKM içerikleri 4. güne kadar azalmış, 6. gün ise artmıştır (Çizelge 7). %1.5 SA+MAP grubunun SÇKM içeriğinde ise dalgalanma gözlemlenmiştir. SÇKM'de düşüşün görülmesi solunumun



etkisiyle meyvedeki şekerlerin CO<sub>2</sub> ve H<sub>2</sub>O'ya dönüşümünden; artışın meydana gelmesi ise hücre duvarı polisakaritlerinin hidrolizinden, su kaybindan ve nişastanın şekerlere parçalanmasından dolayı kuru madde miktarının artmasından kaynaklanabilmektedir (Martinez-Romero ve ark., 2006; Dang ve ark., 2010; Díaz-Mula ve ark., 2012; Petriccione ve ark., 2015; Vieira ve ark., 2016). Depolama sonunda kontrol örneğinin SÇKM içeriğinde kurumayla birlikte daha konsantre olması sonucunda artış meydana gelirken, diğer örnek gruplarının SÇKM içeriğinde azalma görülmüştür. Kontrol ve %1 SA+MAP örneklerinin depolama boyunca SÇKM içerikleri arasındaki farklılık istatistiksel açıdan önemli bulunurken (P<0.05), diğer örnek gruplarının SÇKM içerikleri arasındaki farklılık istatistiksel açıdan önemsiz bulunmuştur (P>0.05). Maftoonazad ve ark. (2008) şeftalileri sodyum aljinat ve metil selülozla kapladıkları çalışmada, bütün örnek gruplarının SÇKM değerlerinde depolama boyunca küçük dalgalanmaların olduğunu ve depolama sırasında istatistiksel olarak anlamlı bir değişiklik olmadığını belirtmişlerdir.

**Çizelge 7.** Suda çözünür kuru madde (%) değerleri.

**Table 7.** Water-soluble dry matter (%) values.

Örnek Grupları	Depolama periyodu (Gün)			
	0	2	4	6
Kontrol	11.70±0.14 <sup>Ca</sup>	14.75±0.64 <sup>BCa</sup>	18.15±1.34 <sup>Ba</sup>	26.45±2.47 <sup>Aa</sup>
MAP	11.70±0.14 <sup>Aa</sup>	10.50±1.27 <sup>Ab</sup>	9.55±0.07 <sup>Ab</sup>	9.80±0.28 <sup>Ab</sup>
%1 SA+MAP	11.25±0.07 <sup>Aa</sup>	9.90±0.42 <sup>Bb</sup>	9.65±0.07 <sup>Bb</sup>	9.85±0.07 <sup>Bb</sup>
%1.5 SA+MAP	10.05±0.07 <sup>Ab</sup>	9.65±0.07 <sup>Bb</sup>	9.85±0.07 <sup>ABb</sup>	9.70±0.14 <sup>ABb</sup>
%2 SA+MAP	10.25±0.21 <sup>Ab</sup>	9.80±0.14 <sup>Ab</sup>	9.75±0.21 <sup>Ab</sup>	10.10±0.28 <sup>Ab</sup>

n=4, ±SD, \*A,B... harfleri aynı satırdaki örneklere,

a,b... harfleri aynı sütundaki örneklere ait istatistiksel farklılıkları göstermektedir, (P<0.05).

Depolama sonu verileri incelendiğinde en az ağırlık kaybı %0.40 olarak %1.5 SA+MAP grubu örneklerde hesaplanmışken, kontrol grubu örnekler %55.19, MAP örneklerinde %1.18, %1 SA+MAP örneklerinde %0.54, %2 SA+MAP örneklerinde ise %0.51 olarak tespit edilmiştir (Çizelge 8). Depolama boyunca kontrol grubunun ağırlık kaybı değerleri arasındaki fark istatistiksel olarak önemli bulunurken (P<0.05) diğer örnek gruplarının ağırlık kaybı değişimleri önemsiz bulunmuştur (P>0.05).

**Çizelge 8.** Ağırlık kaybı (%) değerleri.

**Table 8.** Weight loss (%) values.

Örnek Grupları	Depolama periyodu (Gün)		
	2	4	6
Kontrol	19.88±1.23 <sup>Ca</sup>	37.05±1.89 <sup>Ba</sup>	55.19±5.29 <sup>Aa</sup>
MAP	1.05±0.02 <sup>Ab</sup>	1.15±0.12 <sup>Ab</sup>	1.18±0.07 <sup>Ab</sup>
%1 SA+MAP	0.39±0.01 <sup>Ab</sup>	0.43±0.09 <sup>Ab</sup>	0.54±0.08 <sup>Ab</sup>
%1.5 SA+MAP	0.26±0.02 <sup>Ab</sup>	0.38±0.02 <sup>Ab</sup>	0.40±0.03 <sup>Ab</sup>
%2 SA+MAP	0.55±0.11 <sup>Ab</sup>	0.51±0.02 <sup>Ab</sup>	0.51±0.10 <sup>Ab</sup>

n=4, ±SD, \*A,B... harfleri aynı satırdaki örneklere,

a,b... harfleri aynı sütundaki örneklere ait istatistiksel farklılıkları göstermektedir, (P<0.05).

Depolamanın tüm aşamalarında, kaplama uygulanmış grupların ağırlık kaybı değerleri kaplama uygulanmamış örnek gruplarınınkinden düşük bulunmuştur. Ağırlık kaybındaki bu azalma, aljinat kaplamaların nem ve çözünme hareketini

geciktirebilen ve solunumu azaltabilen yarı geçirgen bir bariyer olarak etki göstermesinden kaynaklanmaktadır (Ali ve ark., 2010; Xiao ve ark., 2010). Rößle ve ark. (2011) taze kesilmiş elma dilimlerine oligofruktoz ve inülin gibi prebiyotikler içeren ve içermeyen sodyum aljinat yenilebilir filmiyle kapladıkları çalışmada en fazla ağırlık kaybı değerinin, çalışmamızdaki gibi kaplama yapılmamış kontrol grubunda olduğunu bildirmişlerdir. Aloui ve ark. (2014) sofralık üzümleri greyfurt çekirdeği ekstresi veya greyfurt esansiyel yağı içeren ve içermeyen %1 ve %2 sodyum aljinat bazlı biyobozunur filmlerle kapladıkları çalışmada %1 ve %2 kaplama uygulamasının, kontrole kıyasla depolama sırasında üzüm meyvesi taze ağırlık kaybını önemli ölçüde geciktirdiğini ve ağırlık kaybını azaltmak için en etkili kaplamalar olduğunu tespit etmişlerdir. Mevcut çalışmada da sodyum aljinat konsantrasyonundaki artışa bağlı olarak ağırlık kayıplarının azaldığının belirlenmiş olması ticari açıdan önemli bir avantaj olarak değerlendirilmiştir.

**Renk Değerleri:** Örnekler için renk değerleri Çizelge 9'da verilmiştir. Genel olarak tüm örnek gruplarının L\* değerlerinde depolamanın 4. gününe kadar artış olduğu, ardından kontrol ve %1 SA+MAP grubunun L\* değerlerinin düştüğü; MAP, %1.5 SA+MAP ve %2 SA+MAP uygulananların ise L\* değerlerinin 6. gün azaldığı gözlenmiştir. Kaplanmış ve kaplanmamış örnekler için artan L\* değerleri, kavunların buzdolabından oda sıcaklığına (21°C) taşınmasından sonra daha hızlı bir solunum hızına bağlanabileceği bildirilmiştir (Serrano ve ark., 2008). Depolama sonunda kontrol grubunun L\* değeri değişimi istatistiksel olarak önemli bulunurken (P<0.05), diğer örnek gruplarının L\* değeri değişimleri önemsiz bulunmuştur (P>0.05). Depolama sonunda örnekler arasında belirlenen farklılıkların istatistiksel olarak önemli olduğu tespit edilmiştir (P<0.05).

Renk a\* değerleri incelendiğinde ise depolama boyunca örnek gruplarının a\* değerlerinde dalgalanmalar olduğu görülmüştür. Depolamanın 2. gününde tüm örnek gruplarının a\* değeri artış göstermiş, ardından depolama sonuna kadar MAP, %1 SA+MAP ve %2 SA+MAP gruplarının a\* değerleri sürekli azalırken kontrol ve %1 SA+MAP gruplarının a\* değerleri 6. güne kadar azaldıktan sonra tekrar artmıştır. Depolamanın 6. gününde kontrol ve MAP örnek grupları arasındaki farklılık önemli bulunurken (P<0.05) diğer günlerde tüm örnekler arasındaki farklılık önemsiz bulunmuştur (P>0.05). Ayrıca depolama sonuna kadar her bir örnek grubunun a\* değerindeki değişimi istatistiksel olarak önemsiz bulunmuştur (P>0.05).

Çizelge 9. L\*, a\*, b\* değerleri.

Table 9. L\*, a\*, b\* values.

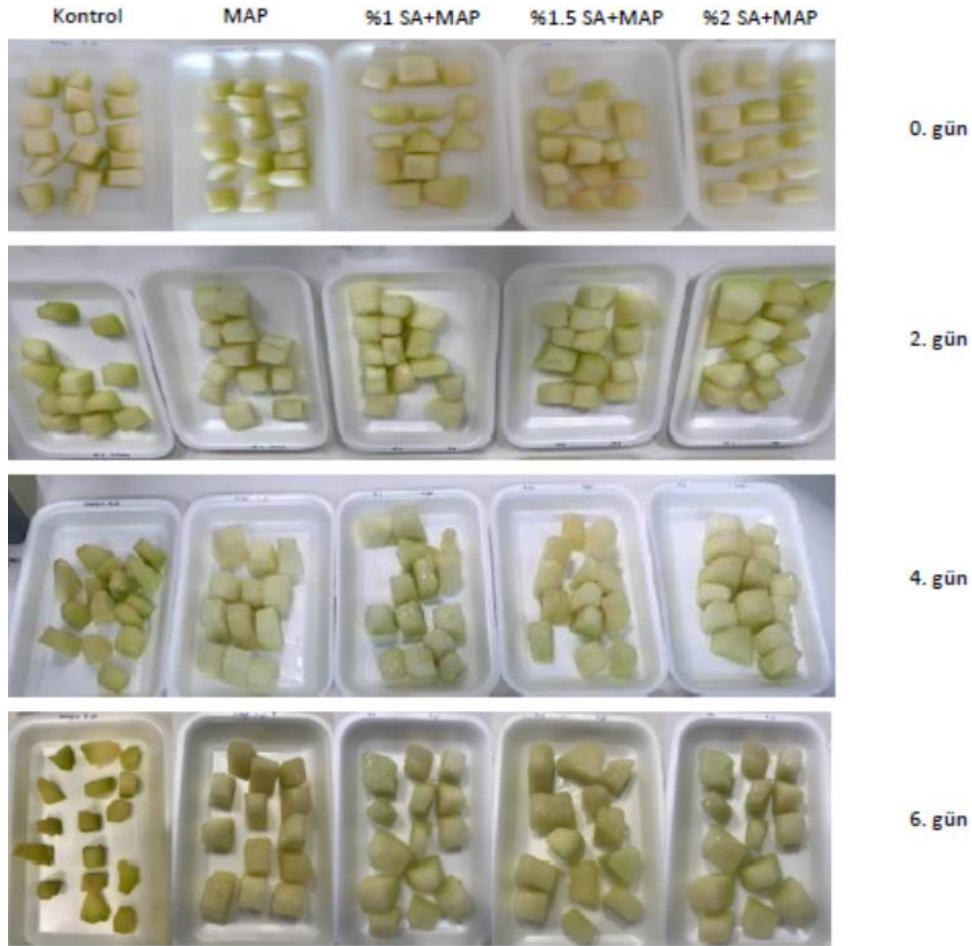
Örnek Grupları	Depolama periyodu (Gün)			
	0	2	4	6
<b>L*</b>				
Kontrol	60.98±5.99 <sup>Ba</sup>	84.59±9.99 <sup>Ab</sup>	92.06±3.02 <sup>Ab</sup>	53.85±5.46 <sup>BCa</sup>
MAP	60.98±5.99 <sup>Ba</sup>	95.36±8.32 <sup>Aa</sup>	94.78±8.18 <sup>Aa</sup>	48.76±3.34 <sup>Ca</sup>
%1 SA+MAP	57.35±6.49 <sup>Ba</sup>	92.48±11.58 <sup>Aab</sup>	96.93±10.98 <sup>Aab</sup>	56.82±5.07 <sup>Ba</sup>
%1.5 SA+MAP	56.48±7.59 <sup>Ba</sup>	95.15±10.81 <sup>Aab</sup>	93.04±6.51 <sup>Ab</sup>	51.61±7.74 <sup>Ba</sup>
%2 SA+MAP	39.47±7.64 <sup>Cb</sup>	96.78±8.25 <sup>Aab</sup>	98.70±8.51 <sup>Aab</sup>	54.28±4.00 <sup>Ba</sup>
<b>a*</b>				
Kontrol	-4.14±2.52 <sup>Ba</sup>	0.43±2.82 <sup>Aa</sup>	-3.65±4.49 <sup>Ba</sup>	-4.86±0.86 <sup>Bb</sup>
MAP	-4.14±2.52 <sup>Ba</sup>	1.75±1.61 <sup>Aa</sup>	0.30±1.95 <sup>Aa</sup>	-2.67±0.48 <sup>Ba</sup>
%1 SA+MAP	-3.31±1.72 <sup>Ba</sup>	1.00±1.72 <sup>Aa</sup>	-1.49±2.00 <sup>Ba</sup>	-3.76±1.45 <sup>Bab</sup>
%1.5 SA+MAP	-3.34±1.29 <sup>Ca</sup>	1.84±2.10 <sup>Aa</sup>	-0.82±1.08 <sup>Ba</sup>	-3.41±1.73 <sup>Cab</sup>
%2 SA+MAP	-2.32±0.57 <sup>Ba</sup>	2.37±1.11 <sup>Aa</sup>	0.09±3.03 <sup>Aa</sup>	-3.37±1.14 <sup>Bab</sup>
<b>b*</b>				
Kontrol	12.90±4.24 <sup>Aa</sup>	2.90±4.34 <sup>Ba</sup>	9.83±7.68 <sup>Aa</sup>	14.20±2.77 <sup>Aa</sup>
MAP	12.90±4.24 <sup>Aa</sup>	0.89±2.90 <sup>Ba</sup>	1.54±4.24 <sup>Bb</sup>	8.42±1.36 <sup>Ab</sup>
%1 SA+MAP	11.53±3.69 <sup>Aa</sup>	1.97±2.96 <sup>Ca</sup>	5.51±4.13 <sup>BCab</sup>	11.14±2.82 <sup>Aab</sup>
%1.5 SA+MAP	11.41±3.03 <sup>Aa</sup>	-0.07±3.15 <sup>Ca</sup>	5.03±2.28 <sup>Bab</sup>	10.17±3.81 <sup>Ab</sup>
%2 SA+MAP	8.96±1.18 <sup>Aa</sup>	-0.10±2.71 <sup>Ba</sup>	2.80±5.45 <sup>Bab</sup>	10.99±2.60 <sup>Aab</sup>

n=4, ±SD, \*A.B... harfleri aynı satırdaki örneklere,

a,b... harfleri aynı sütundaki örneklere ait istatistiksel farklılıkları göstermektedir, (P<0.05).

Depolama boyunca tüm örnek gruplarının b\* değerlerinde de benzer dalgalanmalar meydana gelmiştir. Depolamanın 2. gününde tüm örnek gruplarının b\* değeri düşüş göstermiş, ardından depolama sonuna kadar MAP, %1.5 SA+MAP ve %2 SA+MAP gruplarının b\* değerleri sürekli artarken kontrol ve %1 SA+MAP gruplarının b\* değerleri 6. güne kadar artmıştır. Depolamanın 4. ve 6. gününde örnek grupları arasındaki farklılık önemli bulunurken (P<0.05) diğer günlerde tüm örnekler arasındaki farklılık önemsiz bulunmuştur (P>0.05). Ayrıca depolama sonuna kadar her bir örnek grubunun b\* değerlerindeki değişim istatistiksel olarak önemsiz bulunmuştur (P>0.05).

Farklı konsantrasyonlarda sondum aljinat ile kaplanmış kavun dilimlerinin 6 günlük depolanmalarına ait görseller Şekil 1'de verilmiştir.



**Şekil 1.** Depolama süresi boyunca örneklere ait görseller.

**Figure 1.** Images of the samples during the storage period.

## SONUÇ

Çalışma kapsamında elde edilen verilere göre depolamanın ilk gününde filmle kaplanmış kavun dilimlerinin fenolik madde miktarlarının daha yüksek olduğu belirlenmiştir. Bu sonuca göre yenilebilir film kaplamanın toplam fenolik madde miktarını arttırdığı söylenebilir. Kontrol grubunun fenolik madde miktarının artması yoğun bir şekilde su kaybından dolayı birim ağırlıktaki kuru madde oranının artmasından kaynaklanmaktadır. Tüm örnek gruplarının antioksidan kapasite değerleri depolama sonunda azalmıştır. Ancak depolamanın 4. gününde tüm örnek gruplarında artış gerçekleşmiştir. Bu kaplanmış kavun dilimlerinde sodyum aljinat bazlı kaplamaların oksijen iletimine karşı etkili bir bariyer olduğunu göstermektedir. Kaplanmış örneklerin kaplanmadan önce kalsiyum klorür çözeltisine daldırılması meyve dokusunu olumlu yönde etkilemiştir. Kaplanmış kavun dilimleri depolamanın 6. gününe kadar sertliğini korumuş ve en iyi doku değeri %2 SA+MAP grubunda elde edilmiştir. Depolama sonunda paket içi en yüksek oksijen oranı %1 SA+MAP grubunda belirlenmiştir. Bu sonuç, kavun dilimlerinin %1 oranındaki sodyum aljinat çözeltisiyle daha etkin bir şekilde kaplanabildiğini göstermektedir. Tüm örnek gruplarının depolama sonunda pH değerleri azalmıştır. MAP ve SA+MAP grubu örneklerin pH değerleri benzerlik gösterirken kontrol grubununki daha yüksektir. SÇKM değerleri açısından yine depolama sonu itibariyle kaplanmış örnekler arasında en yüksek

%2 SA+MAP grubunda belirlenmiştir. Depolama boyunca en fazla ağırlık kaybı kontrol grubundadır. Kaplanmış gruplarda daha düşük ağırlık kaybı değerleri görülmüştür. En az ağırlık kaybı %1.5 SA+MAP grubunda elde edilmiştir. Genel olarak değerlendirildiğinde depolama sonunda başlangıç değerlerine en yakın L\* ve b\* değerleri %2 SA+MAP grubunda, a\* değeri ise kontrol grubunda gözlenmiştir. Sonuç olarak film kaplama umut vaat etmektedir. Film çözeltisinin etkinliğini iyileştirmek ve kaplanan gıda ürününün kalitesini artırmak için pasif-MAP uygulaması yerine aktif-MAP koşullarının kullanılması ve film çözeltisine antimikrobiyal maddeler gibi yardımcı maddeler ilave edilebilmesinin çalışılan 6 günlük depolama süresini uzatabileceği düşünülmektedir. Ancak çalışmanın ticarileşmesinden önce mikrobiyolojik açıdan da incelenmesi ile fizikokimyasal raf ömrüne ilaveten mikrobiyolojik raf ömründe belirlenmesi gerekmektedir.

## ÇIKAR ÇATIŞMASI

Yazarlar herhangi bir çıkar çatışması olmadığını beyan ederler.

## YAZAR KATKISI

**Bengü Demir:** Analizleri yürütmüş, sonuçları analiz etmiş, yorumlamıştır.

**Aslıhan Demirdöven:** Araştırma fikrini belirlemiş, Araştırma yöntemlerini önermiş, analiz sonuçlarını kontrol etmiş, makaleyi yazmıştır.

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## Fuel Properties of Sandbox (*Hura crepitans* Linn.) Methyl Ester and its Blends

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### ABSTRACT

Fuel properties of Sandbox Methyl Ester (SBME) blends were experimentally determined. Pure SBME designated B100 was blended with diesel at 5, 10, 15, 20, 25 and 50% volume designated B5, B10, B15, B20, B25 and B50 respectively. The fuel properties were determined according to American Society for Testing and Materials (ASTM) standards. Automotive Gas Oil (AGO) was used as a reference fuel. Fuel properties of the SBME were modified with the addition of diesel percentage in the blend from B100-B5: the density ranged from 891-865 kg m<sup>-3</sup>. The specific gravity ranged from 0.87-0.82. The kinematic viscosity ranged from 5.8-4.0 mm<sup>2</sup> s<sup>-1</sup>. The flash and fire point decreased from 160-90°C and 230-140°C, while the cloud and pour points ranged from 4.6-3.4°C and -5.8-(-9.2)°C respectively. The heating value ranged from 40.50-41.45 MJ kg<sup>-1</sup>. The Cetane number for the SBME was 46.71. The saponification value ranged from 200-143 mgKOH g<sup>-1</sup>. The acid value varied from 2.7-2.2 mgKOH g<sup>-1</sup>, while the FFA content varied from 1.33-1.10 mg g<sup>-1</sup>. The Iodine value was in the range of 108-94 gI<sub>2</sub> 100g<sup>-1</sup>. The pH value varied from 6.80-5.60. The carbon content increased from 80.02-85.62 wt%, while the ash content increased from 0.01-0.16 g 100g<sup>-1</sup>. In comparison to the (AGO) and the ASTM standards, SBME was found to possessed good flow, ignition and combustion characteristics to power diesel engines.

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- Diesel

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## INTRODUCTION

Petroleum fuel is still one of the major energy sources worldwide. However, the global concern of petroleum fuel depletion and potential extinction, in addition to environmental pollution from emissions from its combustion exhaust are facilitating researches into other energy sources to complement or totally substitute petroleum fuels ([Reyes and Spulveda, 2006](#); [Fangrui and Hanna, 1999](#); [Haas, 2005](#); [Kulkarni et al., 2006](#)). Biodiesel from organic lipids has become the available substitute to petroleum diesel. Biodiesel is a product of a displacement reaction known as transesterification between organic lipids such as vegetable oils, animal fats, waste oils and reprocessed lubricants and alcohol, mainly methanol or ethanol to form fatty esters such as methyl or ethyl ester ([Goering et al., 1982](#)). As a result of pressure on other uses of vegetable oils, there is need for exploration and exploitation into discovering of more sources of non-edible vegetable oil to augment for the gap. One of such feedstocks is the sandbox seed oil. Sandbox is an underutilized plant, which is planted as a shade tree in villages and cities ([Adewuyi et al., 2012](#)). Indigenous to tropical zones of North and South America, sandbox (*Hura crepitans* Linn.), is a tree of the (*Euphorbiaceae*) family recognizable by the shady, piercing backs and even brown exterior cover ([Feldkamp, 2006](#)). Sandbox seed has been found to contain oil and a number of important nutritional properties while the leaves can be used for medicinal purposes ([Idowu et al., 2012](#)). There is no specific use of the oil from the sandbox seed at present as the seeds are discarded as waste ([Adewuyi et al., 2012](#)). According to [Basumatary \(2013\)](#), sandbox seed contains about 53% oil. Biodiesel fuel properties are close and similar to that of common diesel, and therefore, can be used as fuel in its unadulterated state or a blend with diesel to drive ignition compression engines with minor or no modifications ([Van Gerpen, 2005](#); [Ma and Hanna, 1999](#)). Biodiesel or its blend offers a variety of advantages over diesel fuel. As a biological source from plant and animal makes it is renewable, non-toxic, and its oil origin, density and viscosity give it better lubrication ability for engine parts ([Demirbas, 2007](#)). Biodiesel standards have been established in most countries in an effort to ensure that only high-quality biodiesel is available in the marketplace. The two most important fuel standards are the American Standard Testing and Measurement (ASTM) D6751 in the United States and EN 14214 (European Committee for Standardization, CEN) in the European Union. Several factors such as the type of catalyst used; either acid or base, the concentration of the catalyst, molar ratio of alcohol to vegetable oil, the reaction temperature, free fatty acid (FFA) content of the vegetable oil, and the purity level of the reactant especially the water content have great influence on the quantity and quality of methyl esters produced from vegetable oils ([Zhang et al., 2003](#); [Ma and Hanna, 1999](#); [Meher et al., 2006](#); [Van Gerpen, 2005](#)). Various works have been reported on the fuel properties of so many vegetable oil methyl esters as shown in (Table 1). In characterizing fuel properties of sandbox seed methyl ester, [Adewuyi et al. \(2012\)](#) used two-step acid catalyzed pretreatment before the basic methanol transesterification. Also, the fuel properties of sandbox seed oil as reported by [Adewuyi et al. \(2012\)](#); [Adepoju et al. \(2013\)](#) and [Igbum et al. \(2012\)](#) were on the pure biodiesel and not comprehensive as some fuel properties were not determined. The present work produced methyl ester from sandbox seed by direct oil-methanol transesterification and evaluated essential fuel properties on the pure methyl ester and it blends with (AGO).

**Table 1.** Properties of vegetable oil methyl esters.

Vegetable oil methyl ester	Property								
	D (kg m <sup>-3</sup> )	KV (mm <sup>2</sup> s <sup>-1</sup> )	FLP (°C)	FIP (°C)	CP (°C)	PP (°C)	CN	CV (MJ L <sup>-1</sup> )	AN (mgKOH g <sup>-1</sup> )
Sandbox	860-920	2.8-4.0		152	6	-15	45-50	40.30	0.21-2.34
Oil palm	835-880	5.7	183				62		
Linseed	870	4.2	161				48	37.5	
Jatropha	840-880	4.7	170	8			51	35-41	5.31
Sun flower	860	4.6	126	0	-2		49	38	
Soybean	680	3.2-4.5	118				45	42	
Sesame	700	5.3	170					44	
Rapeseed	882	4.2	157	10	-1		54	38	
Cottonseed	880	4.0	150	161				39	
Neem seed	820	3.8	245				47	40	
Rubber seed	850	5.8	130					33	

Adopted from [Adewuyi et al. \(2014\)](#); [Adepoju et al. \(2013\)](#); [Sivaramakrishnan and Ravikumar \(2012\)](#); [Habibullah et al. \(2015\)](#); [Kumar and Sharma \(2016\)](#); [Cherng-Yuan Lin and Yi-Wei Lin \(2012\)](#); [Sathiyagnanam and Saravanan \(2011\)](#). D: Density, KV: Kinematic Viscosity; FLP: Flash Point; FIP: Fire point; CP: Cloud point; PP: Pour point; CN: Cetane number; CV: Calorific value; AN: Acid number.

## MATERIALS and METHODS

### Preparation of Sandbox Seed Biodiesel

About 100 kg of mature sandbox fruits were collected from under the trees in Uyo metropolis, Akwa Ibom State, Nigeria between 2016-2018. The fruits (Figure 1) were cracked to remove the seeds (Figure 2) and the seeds peeled to get the kernel (mesocarp) (Figure 3).

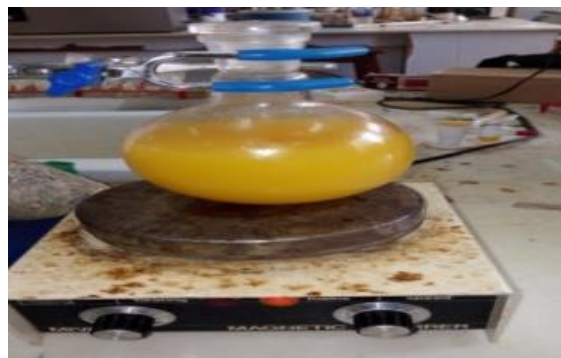


**Figure 1.** Sandbox fruits.      **Figure 2.** Sandbox seeds.      **Figure 3.** Sandbox kernels.

Sandbox oil (Figure 4) was extracted from the sandbox seed by solvent extraction method using AOCS 5-04 standard procedure. Methanol was used as the alcohol in the transesterification (Figure 5) of the oil in the presence of potassium hydroxide catalyst. The transesterification reaction was conducted at 60°C reaction temperature, alcohol-oil ratio of 1:5, catalyst concentration 0.9 g weight of oil and reaction time of 90 min. The methyl ester produced was separated from the glycerol phase and washed thoroughly. The SMBE (Figure 6) was blended with (AGO) at varying proportion, 5, 10, 15, 20, 25, 50 and 100%: diesel ratios, denoted as B100, B5, B10, B15, B20, B25, B50 and B100.



**Figure 4.** Sandbox seed oil.



**Figure 5.** Transesterification.



**Figure 6.** SBME.

#### Determination of fuel properties of sandbox methyl ester

The properties of the SBME produced were characterized using the ASTM standards (Table 2). The following properties of the biodiesel and its blends were determined: Density, Specific Gravity, Cloud Point, Pour Point, Kinematic Viscosity, Saponification Value, Acid Value, Free Fatty Acid Value, Iodine Value, pH value, Flash Point, Fire Point, Cetane Number, and Higher Heating Value (HHV). Automotive Gas Oil was used as control.

**Table 2.** Biodiesel standards and their corresponding ASTM Standards.

S/N	Property	ASTM Standard
1	Density	D941
2	Specific gravity	D1298
3	Cloud point	D2500
4	Pour point	D97
5	Flash and fire points	D93
6	Kinematic Viscosity	D445
7	Saponification value	D5558
8	Acid value	D664
9	Heating value	D40

#### Free Fatty Acid

The free fatty acid (FFA) of the fuel was obtained by the titration method used by [Buhain and Guo \(2013\)](#) for fresh vegetable oil and waste frying oil biodiesel. Two drops of phenolphthalein were added to 10 ml of isopropyl alcohol, 1 ml of fuel sample was

then added and titrated with 1 g l<sup>-1</sup> of NaOH in distilled water. A blank isopropyl alcohol was titrated and the volume of NaOH used recorded as V<sub>1</sub>. The volume of NaOH used on that with fuel samples were recorded as V<sub>2</sub>. The FFA content was calculated from Equation 1.

$$FFA (mg/g) = (V_2 - V_1) \times \frac{1.4}{d_{oil}} \quad (1)$$

Where  $d$  = density of fuel sample

### Iodine value

A volume of chloroform with 2% volume of the fuel sample were titrated with Wiji's solution (5 ml), stirred carefully and kept in the dark for 3 min. After this, Potassium iodide solution (5 ml; 7.5%) was added and titrated using 0.1 N sodium thiosulphate solution until a color change was observed. Starch indicator (3 drops) was afterward added, and titration progressed to a colourless or milky. Results were calculated as I<sub>2</sub>/100. Iodine values of the oil samples were calculated by the expression used by ([Ogbunugafor et al., 2011](#)) as shown in Equation 2.

$$\text{Iodine value} = \frac{(V_b - V_s) \times 1.269}{M} \quad (2)$$

Where  $V_b$  = Titre value for blank,  $V_s$  = Titre value for sample and  $M$  = mass of sample in gram.

### Cetane Number

The Cetane number of the biodiesel was determined by standard method from chromatography analysis of the free fatty acid contents of the biodiesel. The Philip's Pye-Unicam PU 4500 gas chromatography equipped with 30 m × 0.32 mm HP<sup>-5</sup> column, stationary phase coating 0.50 μm. The column temperature was kept at 250°C for 2 min, with increase at 5°C per min up to injector temperature of 250°C, split ratio of 1:35, and carrier gas (Helium) flow rate of 1.8 ml min<sup>-1</sup>. The SBME FFA compositions were identified by the Gc-Ms intensity and Equation 3, developed by ([Bamgboye and Hansen \(2008\)](#)) was used to calculate the Cetane number of the fuel.

$$CN = 61.1 + 0.088x_2 + 0.133x_3 + 0.152x_4 - 0.101x_5 - 0.039x_6 - 0.243x_7 - 0.395x_8 \quad (3)$$

Where  $x$  represents the free fatty acid compositions of the fuel

## RESULTS AND DISCUSSION

Fuel properties of the sandbox seed methyl ester and blends are presented in Table 3. The fuel properties and the control AGO are presented in Table 4. The comparison of the SBME and ASTM standards are shown in Table 5. The free fatty acid composition of the SBME is as shown in Table 6.

### Specific Gravity

The specific gravity increases with addition of SBME to AGO blend (B5-B100) from 0.87-0.82 (Table 3). The specific gravity of the SBME was 0.87, which was higher than the control (AGO) of 0.82 (Table 4). The specific gravity values were similar to  $0.860 \pm 0.015$  obtained by [Adepoju et al. \(2013\)](#) for sandbox seed. [Bamgboye and Oniya \(2012\)](#) reported 0.89 for Loofah seed oil, and 0.85 for groundnut oil methyl ester ([Oniya and Bamgboye, 2014](#)). The specific gravity values obtained for the SBME was within the limit specified by various international standards, ASTM (Table 5) and EN14214 (0.85-0.90), ONC1191 (0.86-0.90), CSN656507 (0.85-0.89), Journal Officiel (0.87-0.89), DINV51606 (0.87-0.90), UN110635 (86-0.90) and SS155436 (0.87-0.90) respectively for biodiesel fuels ([Bamgboye and Oniya, 2012](#)). The low value of the specific gravity indicated a good ignition property for the sandbox seed biodiesel ([Bello and Daniel, 2015](#)).

**Table 3.** Characteristics of sandbox seed biodiesel.

S/N	Characteristics	Biodiesel blends						
		B5	B10	B15	B20	B25	B50	B100
1	Density, kg m <sup>-3</sup>	865	878	880	884	890	888	891
2	Specific gravity	0.82	0.83	0.85	0.86	0.86	0.87	0.87
3	Kinetic viscosity, mm <sup>2</sup> s <sup>-1</sup>	4.0	4.1	4.1	4.2	4.4	5.0	5.8
4	Flash point, °C	90	93	98	102	108	128	160
5	Fire point, °C	140	142	143	145	150	218	230
6	Pour point, °C	-9.2	-8.4	-7.5	-7.3	-7.0	-6.9	-6.8
7	Cloud point, °C	3.4	3.8	3.9	4.0	4.1	4.4	4.6
8	Cetane number							46.71
9	Heating value, MJ kg <sup>-1</sup>	41.45	41.00	40.98	40.85	40.70	40.55	40.5
10	Saponification value, mgKOH g <sup>-1</sup>	143	150	156	170	182	188	200
11	Acid value, mgKOH g <sup>-1</sup>	2.2	2.4	2.4	2.5	2.5	2.5	2.7
12	FFA, mg g <sup>-1</sup>	1.1	1.2	1.2	1.25	1.25	1.25	1.33
13	Iodine value, gI <sub>2</sub> 100g <sup>-1</sup>	94	98	101	102	104	105	108
14	pH value	5.6	5.8	5.9	6.0	6.4	6.5	6.8
15	Ash content, g 100g <sup>-1</sup>	0.16	0.12	0.10	0.06	0.06	0.02	0.01
16	Carbon content, wt%	85.62	85.21	84.56	84.01	83.55	81.99	80.02

**Table 4.** Properties of sandbox seed biodiesel and diesel (AGO).

S/N	Properties	Biodiesel	Diesel (AGO)
1	Density, kg m <sup>-3</sup>	891	861
2	Specific gravity	0.87	0.82
3	Kinetic viscosity, mm <sup>2</sup> s <sup>-1</sup>	5.8	3.8
4	Flash point, °C	160	88
5	Fire point, °C	230	135
6	Pour point, °C	-6.8	-9.6
7	Cloud point, °C	4.6	1.8
8	Cetane number	46.71	42
9	Heating value, MJ kg <sup>-1</sup>	40.5	41.5
10	Saponification value, mgKOH g <sup>-1</sup>	200	140
11	Acid value, mgKOH g <sup>-1</sup>	2.7	2.0
12	FFA, mg g <sup>-1</sup>	1.33	0.9
13	Iodine value, gI <sub>2</sub> 100g <sup>-1</sup>	104.4	88
14	pH value	6.8	5.6
15	Ash content, g 100g <sup>-1</sup>	0.01	0.08
16	Carbon content, wt%	80.02	86.88

**Table 5.** Sandbox seed biodiesel characteristics against ASTM Standards.

S/N	Properties	SBME	ASTM Standards Biodiesel B100 D6751	ASTM Standards Diesel D975
1	Density, kg m <sup>-3</sup>	891	820-845	
2	Specific gravity	0.87	0.88	0.85-0.90
3	Kinetic viscosity, mm <sup>2</sup> s <sup>-1</sup>	5.8	1.9-6.0	1.3-4.1
4	Flash point, °C	160	130-170	60-80
5	Fire point, °C	230	315-350	180-340
6	Pour point, °C	-5.8	-15-10	-35-(-15)
7	Cloud point, °C	4.60	-3-12	-15-5
8	Cetane number	46.71	47-65	40-55
9	Heating value, MJ kg <sup>-1</sup>	40.5	37.27	
10	Saponification value, mgKOH g <sup>-1</sup>	200		
11	Acid value, mgKOH g <sup>-1</sup>	2.7	0.5-0.8 max	
12	FFA, mg g <sup>-1</sup>	1.33		
13	Iodine value, gI <sub>2</sub> 100g <sup>-1</sup>	104.4	120	
14	pH value	5.10		
15	Ash content, g 100g <sup>-1</sup>	0.01	0.05 max	
16	Carbon content, wt%	80.02		
17	Heat Capacity, MJ K <sup>-1</sup>	5.20		

Source: American Society for Testing and Materials, Standard Specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels, Designation D6751-07 (2007)

### Kinematic viscosity

The kinematic viscosity of the sandbox methyl ester was 5.8 mm<sup>2</sup> s<sup>-1</sup> and decreased with the addition of diesel in the blend (B100-B5) from 5.8-4.0 mm<sup>2</sup> s<sup>-1</sup> (Table 3). In comparison, the viscosity values are within the range of 3.8 obtained for the control (AGO) (Table 4). The viscosity was higher than the 3.95 ± 0.20 obtained by [Adewuyi et al. \(2012\)](#) and 2.78 ± 0.02 mm<sup>2</sup> s<sup>-1</sup> obtained by [Adepoju et al. \(2013\)](#) respectively for sandbox seed oil methyl ester. [Indhumathi et al. \(2014\)](#) obtained 9.1 mm<sup>2</sup> s<sup>-1</sup> for methyl ester from green micro algae, while [Bello and Daniel \(2015\)](#) and [Oniya and Bamgboye \(2014\)](#) obtained 4.4 mm<sup>2</sup> s<sup>-1</sup> and 7.60 mm<sup>2</sup> s<sup>-1</sup> respectively for

groundnut oil methyl ester. [Kumar and Kant \(2013\)](#) obtained  $3.237\text{mm}^2\text{ s}^{-1}$  from sorghum oil biodiesel. [Aladetuyi et al. \(2014\)](#) obtained the values; 3.97, 4.03, 4.76 and  $5.48\text{ mm}^2\text{ s}^{-1}$  respectively, for biodiesel from fresh palm kernel oil and palm kernel oil recovered from spent bleaching earth. The kinematic viscosity values of the biodiesel and its biodiesel blends are within the ASTM D6751 standard range of  $1.9\text{-}6.0\text{ mm}^2\text{ s}^{-1}$  (Table 5). The range of viscosity obtained for sandbox, is an indication of high injection performance, as it would be easy to pump, atomize and to achieve finer droplets ([Sivaramakrishnan and Ravikumar, 2012](#)), as high kinematic viscosity values causes poor fuel atomization, incomplete combustion, and carbon deposition in injectors ([Knothe and Steidley, 2005](#)).

### Flash Point

The flash point of the sandbox biodiesel was  $160^\circ\text{C}$  and decreased with the addition of diesel from B100 to  $90^\circ\text{C}$  for B5 (Table 3). The value was higher than  $142^\circ\text{C}$  obtained from the control (AGO) (Table 4). The flash point value was higher than  $152 \pm 1.1^\circ\text{C}$  and  $112^\circ\text{C}$  respectively obtained by [Adewuyi et al. \(2012\)](#) and [Adepoju et al. \(2013\)](#) for sandbox seed methyl ester. These differences could be influenced by prevailing weather and environmental conditions. [Bello and Daniel \(2015\)](#) obtained  $178^\circ\text{C}$  for groundnut oil methyl ester, while [Oniya and Bamgboye \(2014\)](#) obtained  $200^\circ\text{C}$  for groundnut oil methyl ester. [Sathiyagnanam and Saravanan \(2011\)](#) obtained  $150^\circ\text{C}$  as flash point for cottonseed oil biodiesel. [Rao \(2011\)](#) obtained flash point of  $170^\circ\text{C}$  for *Jatropha* biodiesel. [Varathan and Karuppasamy \(2015\)](#) obtained  $142^\circ\text{C}$  as flash point for *Calophyllum inophyllum* (Honne) oil biodiesel. [Neva Voca et al. \(2008\)](#) obtained flash point of  $157^\circ\text{C}$  for rapeseed,  $149^\circ\text{C}$  for sunflower and  $126^\circ\text{C}$  for waste edible oil. Also obtained are: babassu;  $127^\circ\text{C}$  soybean;  $178^\circ\text{C}$ , sun flower;  $96^\circ\text{C}$ , peanut;  $176^\circ\text{C}$ , and palm;  $183^\circ\text{C}$  ([Sivaramakrishnan and Ravikumar, 2012](#)) and mustard seed oil;  $120^\circ\text{C}$ , coconut oil;  $116^\circ\text{C}$ , *Jatropha*;  $185^\circ\text{C}$ , micro algae;  $115^\circ\text{C}$ , soybean;  $96^\circ\text{C}$ , sesame;  $170^\circ\text{C}$ , karanja;  $180^\circ\text{C}$ , linseed oil;  $161^\circ\text{C}$ , rubber seed oil;  $130^\circ\text{C}$  and Neem seed oil;  $245^\circ\text{C}$  ([Habibullah et al., 2015](#)). The sandbox seed biodiesel flash point is within the ASTM D6751 standard of ( $130\text{-}170^\circ\text{C}$ ) (Table 5). This flash point temperature of the fuel would guarantee safety during handling and transportation of the fuel.

### Fire Point

The fire point of the sandbox seed oil methyl ester was  $230^\circ\text{C}$  and decreased with the addition of diesel in the blend (B100-B5) to  $140^\circ\text{C}$  (Table 3). In comparison, the fire point value was higher than that the control (AGO),  $135^\circ\text{C}$  (Table 4). The value is below the ASTM D9751 standards of ( $315\text{-}350^\circ\text{C}$ ) for biodiesels, but within the range for AGO ( $180\text{-}340^\circ\text{C}$ ) (Table 5). The low values of the fire point are indication of the combustion quality of the sandbox seed oil methyl ester. [Sathiyagnanam and Saravanan \(2011\)](#) obtained  $161^\circ\text{C}$  as fire points for cottonseed oil biodiesel.

### Cloud Point

The cloud point of the sandbox seed oil methyl ester was  $4.6^\circ\text{C}$  and biodiesel decreased with the addition of diesel in the blend (B100-B5) from  $4.6\text{-}3.4^\circ\text{C}$  (Table 3). The cloud point value was higher than  $1.8^\circ\text{C}$  obtained for the control (AGO) (Table 4). The cloud point value was lower than  $6^\circ\text{C}$  obtained by [Adepoju et al. \(2013\)](#) as cloud point for sandbox seed methyl ester. [Rao \(2011\)](#) obtained  $8^\circ\text{C}$  for *Jatropha* seed oil biodiesel.

Cloud and pour points obtained by [Neva Voca et al. \(2008\)](#) for various biodiesel sources are rapeseed;  $-1^{\circ}\text{C}$  and  $-7^{\circ}\text{C}$ , sunflower;  $0^{\circ}\text{C}$  and  $-2^{\circ}\text{C}$  and waste edible oil;  $-1^{\circ}\text{C}$  and  $-5^{\circ}\text{C}$  respectively. The cloud point is within the ASTM standards (Table 5), indicating a good cold temperature quality of sandbox seed methyl ester.

### Pour Point

The pour point of the sandbox seed oil methyl ester was  $-6.8^{\circ}\text{C}$  and decreased with the addition of diesel in the blend (B100-B5) to  $-9.2^{\circ}\text{C}$  (Table 3). The pour point value was lower than  $-9.6^{\circ}\text{C}$  obtained for the control (AGO) (Table 4). The value of the pour point obtained was lower than  $-15^{\circ}\text{C}$  reported by [Adepoju et al. \(2013\)](#) for sandbox seed biodiesel. The effect of diesel addition to the cloud and pour point was the same as noted by [Bamgboye and Oniya \(2012\)](#) for loofah seed oil. The higher cloud and pour points of the biodiesels may entail a number of adverse effects on diesel engine when used during cold seasons. Cold temperature behavior of biodiesel is a vital quality criterion, as frozen fuel may cause obstruction of the fuel hoses and filters and limit fuel flow to the engine ([Bello and Agge, 2012](#)). The pour point is within the ASTM standards (Table 5), indicating a good cold temperature quality of sandbox seed methyl ester.

### Heating Value

The heating value of the sandbox seed oil methyl ester was  $40.5 \text{ MJ kg}^{-1}$  and increased with the addition of diesel in the blend (B100-B5) to  $41.45 \text{ MJ kg}^{-1}$  (Table 3). In comparison, the value was so close to  $41.5 \text{ MJ kg}^{-1}$  obtained for the control (AGO) (Table 4) and above the ASTM standard (Table 5). Similarly, [Adepoju et al. \(2013\)](#) obtained  $40.30 \text{ MJ kg}^{-1}$  for sandbox seed oil methyl ester. The calorific value of biodiesel is what establishes it as suitable alternative to diesel fuels as fuel heating value is a determinant of its energy content releasable for doing work ([Sivaramakrishnan and Ravikumar, 2012](#)). The heating value as obtained for sandbox seed biodiesel positions it as a suitable fuel to run diesel engine on its own as a blend of diesel fuel. The calorific value shows a comparative advantage over known biodiesel sources such as *Jatropha* oil ( $34.7 \text{ MJ L}^{-1}$ ) ([Barminas et al., 2001](#)), rapeseed ( $38.3$ ), sunflower ( $38.2$ ) and waste edible oil ( $37.5$ ) ([Neva Voca et al., 2008](#)),  $38.51 \text{ MJ kg}^{-1}$  and Neem seed oil;  $40.1 \text{ MJ kg}^{-1}$  ([Habibullah et al., 2015](#)).

### Cetane Number

The Cetane number for the sandbox methyl ester was  $46.71$  (Table 3). The approximated value of  $47$  fell within the ASTM D6751 standard (Table 5). The Cetane number value was higher than  $42$  obtained for the control (AGO) (Table 2). The Cetane number is within the range of  $45.62$  and  $50.40$  obtained by [Adewuyi et al. \(2012\)](#) and [Adepoju et al. \(2013\)](#) respectively for sandbox methyl ester. According to [Bamgboye and Hansen \(2008\)](#), Cetane number is an essential factor in determining the fuel quality of biodiesel. With  $82.32\%$  unsaturation (oleic,  $23.10\%$  and linoleic,  $56.42\%$ ) (Table 6), the Cetane number of the sandbox biodiesel was within the Cetane number reported for pure linoleic acid,  $36.8$  and that of oleic acid,  $57.2$  ([Bamgboye and Oniya, 2012](#)). This is in agreement with findings made by [Bamgboye and Hansen \(2008\)](#) that Cetane numbers of esters of cottonseed, sunflower, rapeseed, soybean, canola, oil palm, peanut, lard and tallow oils were within the range of Cetane numbers of the dominating fatty acid content. The Cetane number of sandbox



seed biodiesel is within the range of values reported for vegetable oils by [Moreno \*et al.\* \(1999\)](#); [Bamgboye and Hansen \(2008\)](#) for soybean (45-60), rapeseed (44-59), cottonseed (45-55).

**Table 6.** Fatty acid composition of Sandbox seed oil

S/N	Fatty acids	Sandbox seed oil methyl ester (% wt)	Saturation
1	Lauric (12:0)	-	Saturated
2	Myristic (14:0)	-	Saturated
3	Palmitic (16:0)	13.5	Saturated
4	Palmitoleic (16:1)	-	Saturated
5	Stearic (18:0)	4.13	Saturated
6	Oleic (18:1)	23.10	Unsaturated
7	Linoleic (18:2)	56.42	Unsaturated
8	Linolenic (18:3)	2.80	Unsaturated
9	Others	0.05	

### Saponification Value

The saponification value of the sandbox seed oil methyl ester was 200 mgKOH g<sup>-1</sup> and decreased with the addition of diesel in the blend (B100-B5) from 200-143 mgKOH g<sup>-1</sup> (Table 3). However, with the addition of diesel fuel in the blend, the saponification value narrows closer to 140 mgKOH g<sup>-1</sup> obtained for the control (AGO) (Table 4). The value is higher than the saponification value of 180.20 ± 0.10 mgKOH g<sup>-1</sup> obtained by [Adepoju \*et al.\* \(2013\)](#) for sandbox seed oil methyl ester, 165 mgNaOH g<sup>-1</sup> by [Kumar and Kant \(2013\)](#) for Sorghum oil, 191.32 and 170 mgKOH g<sup>-1</sup> for groundnut oil by [Bello and Daniel \(2015\)](#) and [Oniya and Bamgboye \(2014\)](#) respectively.

### Acid Value

The acid value of sandbox methyl ester was 2.7 mgKOH g<sup>-1</sup> and biodiesel decreased with the addition of diesel in the blend (B100-B5) from 2.7-2.2 mgKOH g<sup>-1</sup> (Table 3). The value is higher than 2.0 mgKOH g<sup>-1</sup> obtained for the control (AGO) (Table 4). The value is higher than 0.21 ± 0.00 and 2.34 ± 0.15 mgKOH g<sup>-1</sup> obtained by [Adewuyi \*et al.\* \(2012\)](#) and [Adepoju \*et al.\* \(2013\)](#) respectively for sandbox seed methyl ester. 0.43 and 5.31 mgKOH g<sup>-1</sup> acid value was obtained for Sorghum and Jatropha biodiesel respectively ([Kumar and Kant 2013](#)). The value fell short of ASTM standard (0.5-0.8) (Table 5).

### Other Parameters

The FFA content was 1.33 (Table 3). The value is higher than 1.17 ± 0.02 mg g<sup>-1</sup> obtained by [Adepoju \*et al.\* \(2013\)](#) for sandbox seed biodiesel. [Aladetuyi \*et al.\* \(2014\)](#) reported 6.21 and 0.80 mg g<sup>-1</sup> for fresh and recovered spent bleaching earth palm kernel oil. The Iodine value was 108 gI<sub>2</sub> 100g<sup>-1</sup> (Table). The value was below the ASTM standard of 120 gI<sub>2</sub> 100g<sup>-1</sup> (Table 5). It is also lower than 119.50 ± 0.50 and 116.40 ± 1.40 gI<sub>2</sub> 100g<sup>-1</sup> obtained for sandbox seed methyl ester by [Adewuyi \*et al.\* \(2012\)](#) and [Adepoju \*et al.\* \(2013\)](#) respectively. Other properties determined were pH value of 6.80, ash content; 0.01 g 100g<sup>-1</sup> and carbon content; 80.02 wt%.

## CONCLUSION

Fuel properties of sandbox seed methyl ester (SBME) and its blends with diesel (AGO) have been determined. The properties of the SBME were found to fall within the ranges specified by various international standards for biodiesel fuels. The properties determined, indicate that SBME possesses good flow, ignition and combustion characteristics and cold temperature qualities that would guarantee safety during cold seasons, handling and transportation of the fuel. Relatively to diesel, SBME can thus be employed either in its pure form or blends with diesel to power compression ignition engines with little or no modifications.

## DECLARATION OF COMPETING INTEREST

The author declares that she has no conflict of interest.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

The author is responsible for all parts of this article.

## ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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## Evaluation of Aqua-Crop Model using Onion Crop under Deficit Irrigation and Mulch in Semi-arid Nigeria

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### ABSTRACT

The Aqua-Crop simulation model has been playing a crucial role in assessing the performance of the existing strategies for the management of irrigation schemes for improving agricultural water use efficiency. This study evaluated the Aqua-Crop model using Onion crops under deficit irrigation and mulch practices in semi-arid Nigeria. Measurements were taken from the experimental plots which consisted of irrigation and mulch each at 4 levels were used to evaluate the Aqua-Crop model using canopy cover, biomass, yield, actual crop ET, and water productivity of Onion during the 2021 irrigation season. The simulated results from the Aqua-Crop model were evaluated and statistically compared with the experimental results. The model simulated canopy cover with the highest degree of correlation coefficient ( $0.74 \leq r \leq 0.94$ ). The model perfectly predicted Onion yield and biomass under full irrigation irrespective of the mulching. However, the model underestimated Onion yield and biomass at deficit irrigation. The model has perfectly estimated the seasonal actual crop evapotranspiration at different irrigation levels and mulch materials while underestimating water productivity in most of the treatments except at 100% irrigation under white synthetic mulch. However, both model and experimental water productivity were better at white synthetic mulch plots. Therefore, the Aqua-Crop model has proven to be a good Onion crop growth and yield predictor under different irrigation levels and mulch materials which can help improve Onion productivity in water-stressed areas like semi-arid Nigeria.

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## INTRODUCTION

Effective management of limited water in a water-stressed area like the semi-arid is a major challenge for irrigation water managers as agriculture consumes more than 70% of the total global freshwater (FAO, 2011; Shanono and Ndiritu, 2020). Therefore, effective and sustainable agricultural water management in such water-stressed areas is crucial. For example, managing water resources at both allocation and field levels in semi-arid Nigeria is very important and unavoidable (Tagar *et al.*, 2012; Heris *et al.*, 2014; Nasidi *et al.*, 2015; Shanono, 2019). Thus, on-farm strategy for irrigation water management should be conducted using irrigation techniques that conserve water such as deficit irrigation (DI) coupled with other on-farm management practices including mulching (Chukalla *et al.*, 2015). The concept of DI is a practice by which crops are deliberately exposed to a certain degree of water stress by irrigating below their crop water requirements. Although adopting DI practice, farmers might lose a certain proportion of yield. But, a considerable amount of water can be saved which could be used to develop and put additional land into cultivation thereby, increasing food production (Shanono *et al.*, 2014).

Mulching practices have been used all over the world to increase crop water use efficiency and yield, especially in water-stressed regions. The main aim was to protect the soil surface from solar radiation thereby, modifying the soil temperature, reduce the rate of evaporation and thus ensure more soil water availability for crops growth and hence, higher crop water use efficiency and yields. Mulching involves the placing of organic (e.g. crop residues and grasses) or inorganic material (e.g. polyethene sheets etc.) on the surface of soil under cultivation. The effects of mulching on crop yield and water use efficiency have been reported by many studies (Igbadun *et al.*, 2012; Liu *et al.*, 2014). Khonok (2013) reported an improvement of about 33% in Bean yield when mulch was spread compared to no mulch. Liu *et al.* (2014) reported a 20 to 35% increase in the yield of most grain crops and a 20 to 60% increase in the yield of most of the cash crops when the crops were planted under plastic mulch as compared to No mulch conditions.

Crop simulation models have been developed and applied in agricultural water management (Nazeer, 2009; Zakari *et al.*, 2015). Such crop models contributed immensely in testing and developing alternate strategies for obtaining maximum crop yield with less irrigation water particularly in water-stressed regions (Toumi *et al.*, 2016). Moreover, simulation models are mainly used as prediction tools to make the right decision for future scenarios (Shanono *et al.*, 2012). The models are tools used for gaining insights into the crop characteristics, growth, yield, physiological mechanisms and data extrapolation and prediction (Rauff and Bello, 2015). It simulates the behaviour of a plant including growth parameters such as roots, leaves, stems and yield, as well as other processes concerning the growth stages of a crop on a timely basis, climatic factors and management practices (Darko *et al.*, 2013). Thus crop modelling can provide powerful tools for investigating the dependence and nature of relationships among the variables of crop production. Many crop models have been developed and used to predict crop growth parameters and yield as a response to varying agro-climatological environments for different categories of crops. Some of these models include Crop Syst (Stöckle *et al.*, 2003), EPIC (Williams *et al.*, 1989), the APSIM models (Keating *et al.*, 2003), the DSSAT model (Jones *et al.*, 2003) etc. However, when large

number of input parameters are demanded, advanced skill are needed before calibration and operation which render their application very difficult for the users and policy-makers for irrigation scheme planning, operation and management ([Fererres, 2011](#)).

To tackle these concerns and to achieve an optimal balance between accuracy, simplicity, and robustness, FAO's crop-water model Aqua-Crop has been developed to solve these limitations. This resulted from a series of scientific experiments designed to quantify and understand crop growth in relation to water. The model simulates the yield of several herbaceous crops under the following four conditions, rain-fed, deficit, supplemental, as well as full irrigation condition ([Steduto \*et al.\*, 2009b](#); [Steduto \*et al.\*, 2009a](#)). Compared with other models, Aqua-Crop is easire to operate and allows the simulation of the performance of crops using many scenarios. Moreover, it is characterised with a higher level of accuracy and requires few input parameters ([Steduto \*et al.\*, 2009a](#)). The Aqua-Crop model has the capacity of predicting water requirements, water use efficiency and crop productivity under water-stressed conditions ([Raes \*et al.\*, 2009](#)). Thus, applying Aqua-Crop Model in semi-arid regions could improve crop water productivity and water use efficiency. The main objective of this research was to calibrate and validate Aqua-Crop Model for simulating Onion growth/yield and water used parameters under different irrigation levels and mulch materials in the semi-arid region of Nigeria.

## MATERIALS and METHODS

### Site Location and Experimental Design

This study was conducted at Dala Alamderi Irrigation Project, Maiduguri, Borno State, Nigeria. The Irrigation project is located between Latitudes 11°05' and 11°55' N, Longitudes 13°02' and 13°16' E and altitude 345 m above mean sea level. The mean annual rainfall of the study location is about 625 mm and the temperature range of 28.5°C-40.5°C ([Adeniji \*et al.\*, 2013](#)). The climate of Maiduguri is generally semi-arid with moderate variation in temperatures. The soils in the study location is predominantly sand to sandy-loam having low moisture retention and high permeability, and few places with clay to clay-loam.

The field experiments consisted of two factors (water application depth and mulch practice) each at four levels. The 4 levels of water applications are 100, 85, 70, and 55% of weekly reference evapotranspiration (WRET), while the 4 levels of mulch materials which include No Mulch (NM); Rice Straw Mulch (RM), Wood Shaving Mulch (WM) and White Synthetic Plastic Mulch (SM). The treatments were replicated 3 times making  $4 \times 4 \times 3 = 48$  experimental plots. The experiment was laid out using a split-plot design (SPD). The block was separated by 0.5 m and the basins in each block were also separated by a distance of 0.5 m. Such separation aims to minimize the water lateral movement from one plot to another.

### Land Preparation, Agronomic Operations and Water Application

A land with an area of 36 m by 15 m was cleared and prepared into levelled basins of 2.0 m x 2.0 m and Onion seedlings were transplanted on 1<sup>st</sup> December 2020. The variety of Onion used was a red creole, which is commonly grown in the study area. The crop was transplanted with a spacing of 20 cm between plants and 25 cm between rows resulting in a crop density of 80 plants per plot. The mulch were placed two weeks after

transplanting after which the transplanted Onion is fully established and recovered. All other operations were conducted using the standard agronomic procedure (Igbadun *et al.*, 2012, Sinnadurai, 1992; Sen *et al.*, 2006). The surface irrigation method which is not uncommon in the study location was used.

The surface irrigation method which is common in the study location was used. The major source of water in the study area are tube well. During the early growth stage, all experimental plots were irrigated at full irrigation to ensure proper plant establishment. Different irrigation water levels were applied to the developmental, mid and late growth stages. The water applied at every irrigation period was recorded during the entire cropping season using the reference ET amount for the days of irrigation and for each experimental treatment plots. The average weekly reference ET for December, January, February and March were 25 mm, 37 mm, 53 mm and 58 mm, for treatment Irrigated at 100% respectively. The seasonal water applied for the treatments irrigated at 100%, 85%, 70%, 55% WRET were 577, 490, 404, and 317 mm respectively throughout the crop growing season. Reference ETo of the site was calculated using the FAO-Penman-Monteith method which is incorporated in the CROPWAT model (FAO, 1977). The weather data for the calculation of ETo was obtained from the Meteorological Station (NIMET) situated in Maiduguri International Airport, Maiduguri. The consumptive use of the crop (CWU) of the treatments irrigated at 100% WRET ( $I_{100}$ ), was regarded as actual crop consumptive use (ACWU) while the crop CWU of the deficit irrigated treatments ( $I_{85}$ ,  $I_{70}$ ,  $I_{55}$ ) was regarded as deficit consumptive use (DCWU).

### Crop Data Collection

To ascertain the Onion response to the deficit irrigation and mulch conditions, number of leaves per plant and plant height were measured at 2, 4, 6, 8 and 10 weeks after transplanting. The canopy cover, leaf area, crop biomass, and harvest index were computed at 2, 4, 6, 8 and 10 weeks after transplanting using equations 1, 2, 3 and 4 respectively (Hsiao *et al.*, 2009; Corcoles *et al.*, 2015).

$$CC = \frac{LA_m N}{A} \times 100 \quad (1)$$

$$LA = 0.000199 + 1.277L \times A_{25} \quad (2)$$

$$CB = BB + LB \quad (3)$$

$$HI = \frac{Y}{B} \times 100\% \quad (4)$$

Where;  $CC$  = canopy cover in %,  $LA_m$  = average leaf area in  $m^2$ ,  $N$  = number of leaves and  $A$  = area occupied by crop in  $m^2$ .  $L$  = total leaf length and  $A_{25}$  is leaf width taken from the distance of 25% from the base of the leaf.  $HI$  = harvest index,  $Y$  = Onion yield in  $kg\ ha^{-1}$ ,  $CB$  = crop biomass,  $BB$  = bulb biomass,  $LB$  = leaves biomass and  $B$  is the total onion biomass in  $kg\ ha^{-1}$ .

The Onion yield and crop water use efficiency were calculated for each of the experimental plots using equations 5 and 6 expressed by Igbadun *et al.* (2012) and Bagg and Turner (1976) respectively.

$$Y = \frac{W}{A} \quad (5)$$

$$CWUE = \frac{Y_a}{ET_a} \quad (6)$$

Where;  $Y$  = Onion bulb yield in  $\text{kg ha}^{-1}$ ,  $W$  = crop weight in kg and  $A$  = experimental plot in ha.  $CWUE$  = Crop Water Use efficiency,  $ET_a$  = Actual crop evapotranspiration ( $\text{m}^3$ ) and  $Y_a$  is the crop yield ( $\text{kg m}^{-2}$ )

### Model Description and Input data

Aqua-Crop crop was developed by Food and Agriculture Organization. The aim of the model was to predict water use efficiency, water requirement and crop productivity under water-stressed conditions (Hsiao *et al.*, 2009; Raes *et al.*, 2009). Aqua-Crop originates from the Doorenbos and Kassam (1979) method using crop yield response factor ( $K_y$ ) by separating evapotranspiration ( $ET$ ) into crop transpiration ( $Tr$ ), soil evaporation ( $E$ ) and the final yield ( $Y$ ) into the harvest index ( $HI$ ) and biomass ( $B$ ). This led to equation 7 as the basis for the Aqua-Crop growth engine. The biomass can also be divided into the yield ( $Y$ ), while the ratio of yield to biomass is known as harvest index (HI), thus, the yield can be obtained using Equation 8.

$$B = WP \times \Sigma Tr \quad (7)$$

$$Y = B \times HI \quad (8)$$

Where:  $WP$  = water productivity ( $\text{kgm}^{-3}$ ),  $Tr$  = Transpiration (mm) and  $B$  = Biomass ( $\text{t ha}^{-1}$ )

The aqua-Crop model input parameters include crop with its growth, soil with its water balance, development, and yield. Others include the atmosphere with its thermal conditions, evaporative demand, rainfall and  $\text{CO}_2$  concentration (Hsiao *et al.*, 2009; Raes *et al.*, 2009; Steduto *et al.*, 2012). The climate components involves daily weather data on maximum and minimum rainfall, air temperature,  $\text{CO}_2$  concentration and  $ET_o$ . These data were obtained from an agrometeorological station at the Maiduguri International Airport. While the daily  $ET_o$  was calculated using the FAO Penman-Monteith equation installed in the model. The soil file involves soil characteristics including field capacity, permanent wilting point, volumetric water content at saturation and saturated hydraulic conductivity of the different soil profile depths. These parameters were obtained following standard international procedures.

The crop input component of Aqua-Crop contains both conservative and user-specific parameters. Some of the user-specific ones involve emergence time, plant density, maturity time, canopy senescence, yield formation duration, flowering period rooting depth, and reference HI. Some of the conservative parameters include canopy growth, soil water extraction pattern, crop coefficient for transpiration at full canopy; and water stress response coefficients for canopy expansion, water productivity for biomass; stomatal closure, and early canopy senescence. Groundwater effects due to capillary were not simulated because in the study location the watertable is below effective root zone (typically  $> 7$  m). The input data also include information relating to field



management and irrigation. These input variables are management and location-specific.

### **Model Calibration and Validation**

The results obtained from the field experimental and meteorological data from Maiduguri International Airport were used to calibrate the Aqua-Crop model. The purpose of the calibration is to adjust some model parameters to make the model match the measured data at the specific location ([Farahani \*et al.\*, 2009](#)). Calibration was performed with the four different irrigation regimes and mulch materials treatments by first matching the ability of the parameters for the fully irrigated treatment with both no mulch and mulched experimental plots. Then, the water stress parameters were changed manually around the default value to reproduce the measured values, the process involved the comparison of simulated and observed values for canopy cover, biomass, actual evapotranspiration, water productivity and yield of Onion. The procedure is an iterative approach from which sensitive parameters are adjusted, mainly non-conservative parameters, and assessing both the absolute and relative differences. For each change in input parameters, simulations were run using the calibrated crop file and the corresponding irrigation file. Thus, continuous iterations of the parameters were done until satisfactory results for all the irrigation treatments in the calibrated experiment were achieved.

The Aqua-Crop model was calibrated using the measured data from the experimental field at the Dala Irrigation site during the 2020/2021 experimental season. Calibration was performed with the four irrigation regimes and two selected mulch materials for each irrigation treatment by first matching the ability of the fully irrigated treatment under no mulch condition in terms of the canopy cover ( $CC$ ), yield ( $Y$ ), biomass ( $B$ ), actual crop evapotranspiration and the evapotranspiration water productivity. While the remaining two mulch materials with the four irrigation regimes were used for the validation of the Aqua-Crop model. The conservative and non-conservative parameters used to calibrate the Aqua-Crop model for simulating the Onion growth, yield and water used efficiency parameters are presented in Table 1.

**Table 1.** Parameters used to calibrate the Aqua-Crop model for simulating onion crop.

S/No	Description	Values				Units
		NM	SM	WM	RM	
1	Base Temperature	10	10	10	10	°C
2	Cut- off Temp	30	30	30	30	°C
3	Initial canopy cover	1.65	1.65	1.65	1.65	%
4	Canopy size seedling	5.00	5.00	5.00	5.00	cm <sup>2</sup> plant <sup>-1</sup>
5	Canopy growth co-eff.	0.94	1.02	0.99	0.98	% GDD
6	Canopy decline co-eff.	0.37	0.64	0.49	0.49	%GDD
7	Maximum canopy cover	62.8	69.5	64.8	63.9	%
8	Water productivity ( <i>WP</i> )	18.5	18.0	18.0	17.5	g m <sup>-2</sup>
9	Canopy expansion growth threshold $P_{upper}$	0.20	0.25	0.20	0.20	
10	Canopy expansion growth threshold $P_{lower}$	0.55	0.50	0.45	0.45	
11	Effect of canopy shelter on soil <i>ET</i> in late season ( <i>Ke</i> )	60	65	65	65	%
12	Effect of crop transpiration ( <i>KcTr</i> )	1.15	1.15	1.00	1.00	
13	Saturation	5.0	5.0	5.0	5.0	%
14	Early canopy senescence stress coefficient ( $P_{upper}$ )	0.55	0.50	0.45	0.45	
15	Shape factor for soil-water stress	3.0	3.0	3.0	3.0	
16	Stomata closure threshold ( $P_{upper}$ )	0.55	0.50	0.45	0.45	
17	Reference harvest index ( <i>HIO</i> )	65	68	63	63	%
18	Time of transplanting to recover	18	16	17	17	Days
19	Time of transplanting to max. cc	77	75	72	72	Days
20	Time of transplanting to senescence	89	84	88	88	Days
21	Time of transplanting to harvest	100	100	100	100	Days
22	Irrigation regimes (100%)	403.8	348.9	378.2	383.6	mm
23	Irrigation regimes (85%)	376.0	334.0	354.3	362.2	mm
23	Irrigation regimes (70%)	344.5	315.5	331.5	332.8	mm
24	Irrigation regimes (55%)	284.6	252.6	269.7	275.7	mm

Aqua-Crop model performance was evaluated based on how simulated data are close to the observed data and was determined using the following statistical indicators; Root Mean Square Error (Heng et al., 2009), Nash and Sutcliffe Efficiency Coefficient (Nash and Sutcliffe, 1970) and Coefficient of Residual Mass (Kahimba et al., 2009) as expressed in Equations 9, 10 and 11 respectively.

**Root Mean Square Error (RMSE):** This is the measure of the average magnitude of the difference between simulated (*S*) and observed (*O*) data. It ranges from 0 to positive infinity with the former showing good and the latter indicating poor performance.

$$RMSE = \sqrt{\frac{\sum_{i=1}^N (O_i - S_i)^2}{N}} \quad (9)$$

**Nash-Sutcliffe Efficiency Coefficient (NSE):** The aim of computing NSE was to determine how well the observed and simulated data are match. The NSE is calculated as one minus the ratio of the error variance of the modeled time-series divided by the variance of the observed time-series. In the situation of a perfect model with an estimation error variance equal to zero, the resulting Nash–Sutcliffe Efficiency equals 1 (NSE = 1). Thus, an NSE of 1 indicates an excellent match between the simulated and observed data.

$$EF = 1 - \frac{\sum_{i=0}^N (O_i - S_i)^2}{\sum_{i=0}^N (O_i - O_{av})^2} \quad (10)$$

**Coefficient of residual mass (CRM):** This describes the tendency of the crop model to either under-predict or over-predict, a positive indicates a tendency of under-prediction, whereas a negative value shows over-prediction as expressed in Equation 7 ([Igbadun \*et al.\*, 2012](#); [Kahimba \*et al.\*, 2009](#)).

$$CRM = \frac{\sum S_i - \sum O_i}{S_i} \quad (11)$$

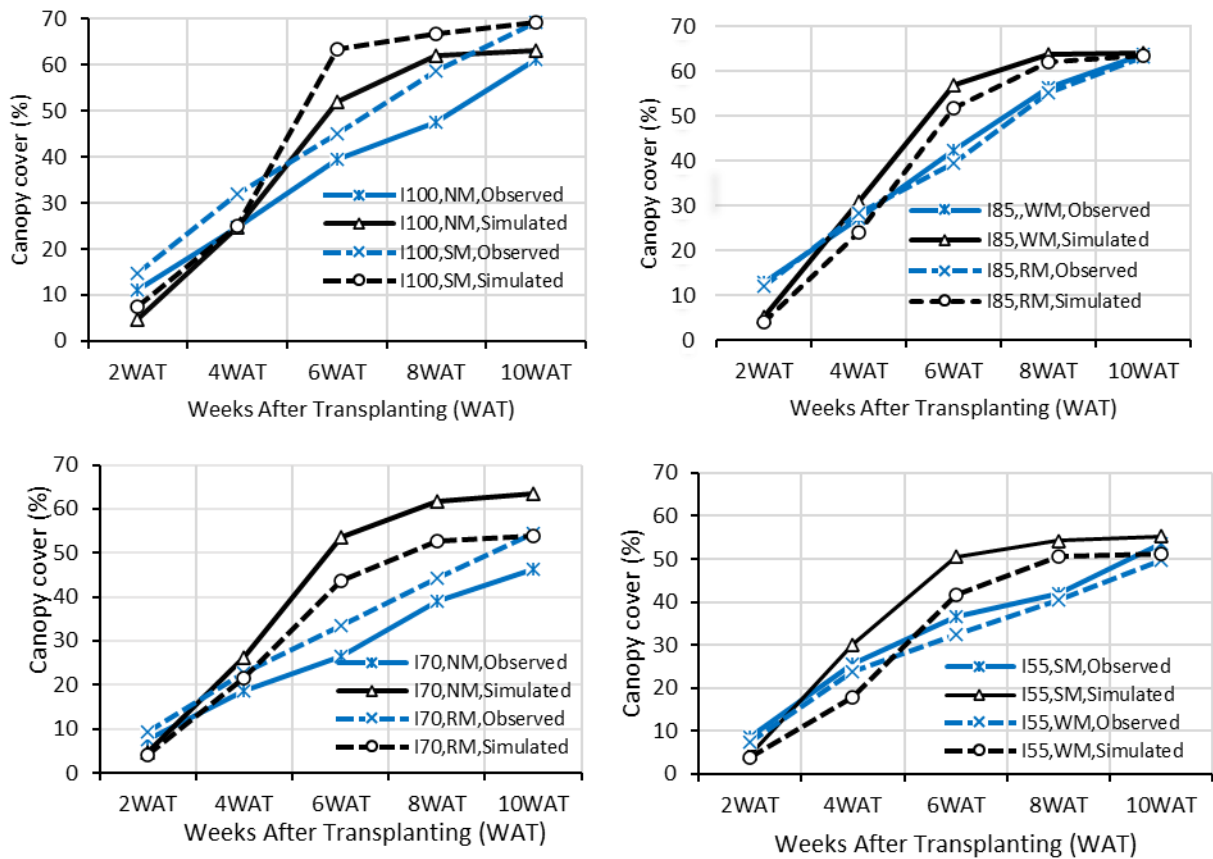
Where;  $S_i$  and  $O_i$  = simulated and observed values,  $N$  = number of observations and  $O_{av}$  = mean of the observed values

## RESULTS AND DISCUSSION

### Calibration for Canopy Cover (CC)

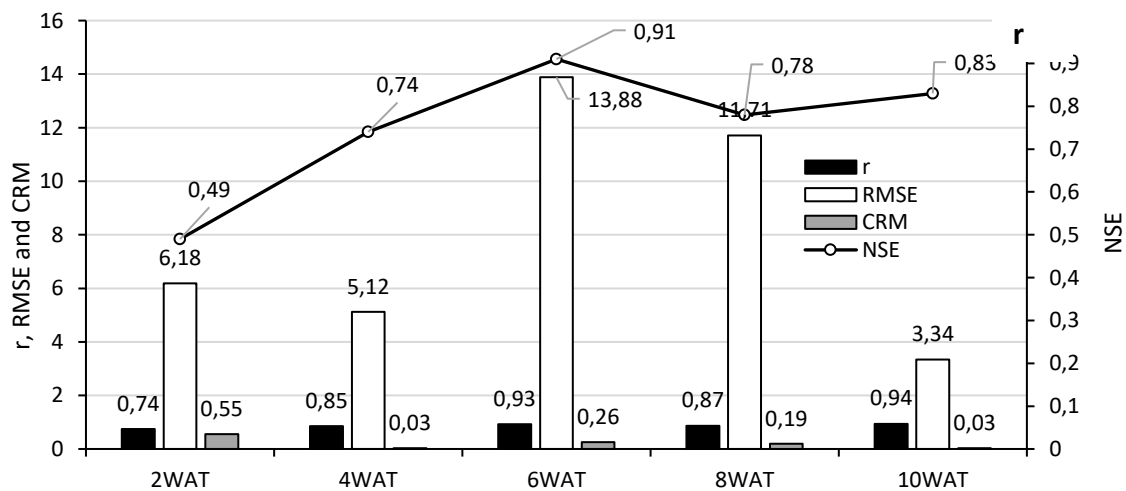
The development of a green canopy cover for the Onion under different irrigation and mulch materials treatment for 10 weeks after transplanting (WAT) is presented in Figure 1. The figure revealed how CC of Onion was underestimated by the Aqua-Crop model at the early growth stage (2WAT) throughout the treatments irrespective of irrigation levels or mulch materials. Whereas, at 4WAT, the simulated CC values at plots with white synthetic mulch (SM) and full irrigation slightly overestimated the observed values. The simulated model values recorded at 6WAT and 8WAT has greatly overestimated the observed field values throughout the experiment as shown. However, the model simulated values recorded during the experiment were very close to the observed field values at 10WAT. This result shows that as the crop approaches the maturity stage, CC enters a declining phase due to leaf senescence as observed. Observation data confirmed that the treatments with extreme water stress condition have a shorter CC than those with no stress. Also, the observed and simulated CC growths were well fitted for treatment with 100 and 85% irrigation under both no-mulch and mulch conditions. This assertion has also been observed by [Farahani \*et al.\* \(2009\)](#) for cotton, [Geertz and Raes \(2010\)](#) for quinoa and [Zelege \*et al.\* \(2011\)](#) for canola. Generally, the result indicates that Onion crop CC increases with an increase in the number of weeks after transplanting while decreasing with an increase in deficit irrigation irrespective of mulch conditions.

Figure 2 shows the result obtained from statistical tests and a strong agreement between the observed and simulated CC values for all treatments. The correlation coefficient values ( $r$ ) ranging from 0.74 to 0.94 with the maximum value of  $r = 0.94$  was recorded at 10WAT while the minimum value of 0.74 at 2WAT. The co-efficient values per treatment were closer to 1, indicating a positive linear relationship between observed and simulated CC development and similar findings were reported by [Kiptum \*et al.\* \(2013\)](#) with a good relationship of  $r = 0.95$  between observed and simulated CC.



**Figure 1.** Simulated and observed canopy cover under different irrigation and mulch treatments.

While the EF and CRM values were recorded throughout the growing period ranged from -1.58 to 0.99 and 0.03 to 0.19 respectively. The model efficiency was better predicted at 6WAT and 10WAT with the efficiency values of 0.99 and 0.83 respectively. The CRM values indicated that the model has slightly underestimated the observed values. However, a significant difference in RMSE was observed with the increase in deficit irrigation levels in both no-mulched and mulched fields. The maximum and minimum values of RMSE obtained during CC development were 13.88 and 3.34% at 6WAT and 10WAT respectively.



**Figure 2.** Statistical testing for results simulated and observed values of canopy cover.

### Calibration for onion bulb yield and biomass

The comparison between the simulated and observed values of Onion bulb yield and biomass and their percentage deviation are presented in Table 2. The result revealed that irrespective of the level of irrigation and mulch, the observed values for both Onion bulb yield and biomass recorded were underestimated by the Aqua-Crop model except at 100% irrigation with white synthetic mulch with a % deviation of 0.7. The result was in line with a report by [Agbemabiese et al. \(2017\)](#) which suggested that crop yields were overestimated at 10% irrigation level and underestimated at deficit levels. The highest model values of 6.09 ton ha<sup>-1</sup> and 8.24 ton ha<sup>-1</sup> of bulb yield and biomass respectively were recorded at 100% irrigation under white synthetic mulch. While the corresponding minimum values of 3.77 ton ha<sup>-1</sup> and 5.94 ton ha<sup>-1</sup> were obtained at 55% irrigation with wood shave mulch. The % deviation of the model simulated values from the observed values in these fields were ranged between -14.6 to 0.7% for Onion bulb yield and -17.6 to -7.4% for biomass which shows a satisfactory prediction. The deviations recorded in this study were also in line with work by [Nazeer and Hussein \(2012\)](#) who reported that the performance of the model to estimate Onion biomass and bulb yield was satisfactory.

**Table 2.** Observed and simulated Onion bulb yield and total biomass and their % deviations.

Treatment		Yield (ton ha <sup>-1</sup> )			Biomass (ton ha <sup>-1</sup> )		
Irrigation	Mulch	Observed	Simulated	% dev.	Observed	Simulated	% dev
100%	NM	5.06	5.02	-0.8%	8.87	7.46	-15.9%
	SM	6.05	6.09	0.7%	10.00	8.24	-17.6%
85%	WM	5.52	4.94	-10.5%	9.24	8.24	-10.8%
	RM	5.37	4.81	-10.4%	8.29	7.41	-10.6%
70%	NM	4.72	4.13	-12.5%	7.71	6.78	-12.1%
	RM	4.58	4.19	-8.5%	7.18	6.65	-7.4%
55%	SM	4.71	4.02	-14.6%	7.84	6.49	-17.2%
	WM	3.77	3.58	-5.0%	6.62	5.94	-10.3%

Table 3 presents the validation results of Aqua-Crop model performance for onion biomass, bulb yield, water productivity and actual crop evapotranspiration under different irrigation levels and mulch materials. From the table, the Aqua-Crop model has perfectly predicted both the bulb yield and biomass at both full and deficit irrigation irrespective of the mulching conditions. This was proved by the correlation coefficient (r) values between simulated and observed Onion bulb yield and biomass of 0.91 and 0.94 respectively. The average value of RMSE obtained was 0.59 ton ha<sup>-1</sup> and 0.10 ton ha<sup>-1</sup> for bulb yield and biomass respectively. The EF and CRM values for the yield were respectively recorded as 0.35 and 0.04, thus the model has slightly underestimated the observed values. The corresponding values of EF and CRM values of biomass obtained were 0.27 and 0.10 respectively which also indicates a slight underestimation of the observed values. Generally, the RMSE, EF and correlation (r) values obtained indicated that the Aqua-Crop simulation model has satisfactorily simulated Onion yields in the study area. This result contradicts the finding by [Hussain \(2012\)](#) that performance indicators of RMSE and Nash Coefficient of efficiency on simulated onion biomass and yield under deficit irrigation gave overestimated results and declared the model's performance as unsatisfactory.

**Table 3.** Statistical index validation for simulated and observed values of onion yield and biomass.

Parameters	Corrrelation (r)	RMSE	EF	CRM
Onion Crop Biomass (B)	0.94	0.91	0.27	0.10
Onion Bulb Yield (Y)	0.91	0.59	0.35	0.04

**Actual crop evapotranspiration (ET<sub>a</sub>) and water productivity (ET<sub>wp</sub>)**

The differences in the seasonal ET<sub>a</sub> and ET<sub>wp</sub> between the simulated and observed values for different irrigation levels and mulch are presented in Table 4. The simulated ET<sub>a</sub> has generally achieved an acceptable performance under different irrigation levels and mulch conditions. However, the Aqua-Crop model underestimated the observed ET<sub>a</sub> at 100% irrigation level under white synthetic mulch materials. Aqua-Crop model was able to predict ET<sub>a</sub> for all the treatments with acceptable % deviations ranging between -7.3 to 23.5%, the highest % deviation was recorded at 55% irrigation under wood mulch and the lowest deviation was observed at experimental plots with white synthetic mulch. However, a larger % deviation was noted under severe water stress treatments. Thus, the performance of the Aqua-Crop model reduces as water-stress increases. For the ET<sub>wp</sub>, the simulated values underestimated the observed values in almost all the treatments except at 100% irrigation under white synthetic mulch. This could be as a result of a larger % deviation of ET<sub>a</sub> that was observed at water-stressed plots. This result was in line with the findings by [Agbemabiase et al. \(2017\)](#) which states that the Aqua-Crop model underestimated ET<sub>wp</sub>. The % deviation observed between the observed and simulated ET<sub>wp</sub> values were fairly estimated for most of the treatments as shown in Table 4. Results indicate that the % deviations in ET<sub>wp</sub> values are a function of the level of plant water stress. However, both simulated and observed ET<sub>wp</sub> was seemingly better at irrigation treatments with white synthetic mulch materials, indicating a potential for water saving.

**Table 4.** Comparison between simulated and observed ET<sub>a</sub> and ET<sub>wp</sub> and their % deviation.

Treatment		ET water productivity, ET <sub>wp</sub> (kg m <sup>-1</sup> )			Actual crop ET,		ET <sub>a</sub> (mm)
Irrigation	Mulch	Observed	Simulated	% dev	Observed	Simulated	% dev
100%	NM	1.25	1.22	-2.4%	403.80	415.90	0.20
	SM	1.73	2.01	16.2%	348.90	303.20	-4.40
85%	WM	1.56	1.29	-17.3%	354.30	384.20	8.40
	RM	1.48	1.32	-10.8%	362.20	365.30	0.90
70%	NM	1.37	1.05	-23.4%	344.50	394.10	14.40
	RM	1.38	1.12	-18.8%	332.80	374.90	2.70
55%	SM	1.86	1.59	-14.5%	252.60	252.60	0.00
	WM	1.38	1.07	-22.5%	269.70	333.20	23.50

The validation of Aqua-Crop model performance for seasonal ET<sub>a</sub> and ET<sub>wp</sub> of onion under different irrigation levels and mulching materials is presented in Table 5. From the table, the model performance of seasonal ET<sub>a</sub> under different irrigation and mulching were satisfactory with the correlation coefficient value (r) of 0.74. The RMSE value was 33.23, while the EF and CRM values were 0.29 and -0.05 respectively. The negative CRM value is an indication that the simulated values slightly overestimates

the observed values at ET<sub>a</sub>. However, the model performance at seasonal ET<sub>wp</sub> of Onion was excellent with the correlation coefficient (*r*) as 0.90. The RMSE value was low 0.32 kg m<sup>-3</sup>. Similarly, the CRM and EF values recorded were 0.08 and -3.32 respectively, which shows that the model underestimates the observed values. However, the negative EF values indicate that the average observed field values is a better prediction of the model. The result obtained from this research is in line with a report by [Atefeh and Ali \(2013\)](#) which suggested that the amount of water required by crop and water use efficiency simulated by the Aqua-Crop model had well adapted and correlated with field measures.

**Table 5.** Statistical index validation for simulated and observed values of ET<sub>a</sub> and ET<sub>wp</sub>.

Parameters	Corrrelation ( <i>r</i> )	RMSE	EF	CRM
Actual Crop ET (ET <sub>a</sub> )	0.73	33.23	0.29	-0.05
Water Productivity (ET <sub>wp</sub> )	0.90	0.32	-3.32	0.08

## CONCLUSION

Aqua-Crop model was calibrated and validated for its ability to predict canopy cover development, biomass, yield, actual ET and ET water productivity of Onion grown under different irrigation levels and mulch conditions in semi-arid Nigeria. The model tends to underestimate canopy cover during early growth stages irrespective of irrigation levels and mulch conditions. However, at the developmental and middle crop growth stage, the model has greatly overestimated the observed values. Nevertheless, the model simulated values were close to the observed field values at the late growing stage. Therefore, treatments with severe water stress have a shorter crop canopy cover than treatments with no stress. The statistical indicators used (RMSE, EF and CRM) indicates that Aqua-Crop was able to simulate canopy cover development with a high degree of accuracy, although the model's performance decreases as deficit irrigation intensifies. Generally, the model underestimated the observed field values for both Onion bulb yield, biomass, ET<sub>a</sub> and E<sub>wp</sub> recorded at the deficit irrigation levels, except the observed values of Onion bulb yield recorded at 100% irrigation with white synthetic mulch. The correlation coefficient recorded for both bulb yield, biomass, ET<sub>a</sub> and E<sub>wp</sub> was very good with a coefficient of correlation (*r*) as 0.91, 0.94, 0.73 and 0.90 respectively. Hence, the results of this study suggest that the Aqua-Crop model can be used to predict the Onion growth and yield parameters with a high degree of reliability under different irrigation and mulch management strategies in the semi-arid region, but it is important to note that the prediction accuracy reduces as water-stress conditions increases.

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## DECLARATION OF COMPETING INTEREST

The authors declare that there have no conflict of interest.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

**Nura Jafar Shanono:** Investigation, methodology, conceptualization, formal analysis, data curation, writing - original draft, review, and editing, visualization.

**Baba Saleh Abba:** Investigation, methodology, formal analysis, formal analysis, validation, review, and editing, visualization.

**Nuraddeen Mukhtar Nasidi:** Investigation, validation, review, and editing, visualization.

## ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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## Production of Diabetic Pumpkin (*Cucurbita moschata* Duch.) Marmalade

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### ABSTRACT

Pumpkin (*Cucurbita moschata* Duch.) is a widely cultivated plant in some parts of the world. It is rich in dietary fiber, carotenoids, phenolic compounds, vitamins, and minerals. The aim was this study to produce pumpkin-based diabetic marmalade with enhanced functional properties. Marmalade production, based on pumpkin fruit, it was prepared with four different recipes by adding spices, stevia, and granulated sugar at different rates. All samples of marmalades were analyzed to determine, pH, titration acidity, water activity, total phenolic and antioxidant capacity, color, consistency and sensory analyzes. The addition of stevia and spices increased the total phenolic and antioxidant capacity of marmalades. The water activity values of the samples were in the range of 0.924-0.932. The addition of spices to marmalade caused a decrease in color L\* value and darkened its color. In sensory evaluation, marmalades with stevia were more appreciated in terms of smell, taste, and flavor. With this study, it has been seen that an alternative product can be produced for diabetes patients that is accepted, nutritious, functional and has high added value can be produced from pumpkin.

#### RESEARCH ARTICLE

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## Diyabetik Bal Kabağı (*Cucurbita moschata* Duch.) Marmelatı Üretimi

### ÖZET

Balkabağı (*Cucurbita moschata* Duch.) dünyanın bazı bölgelerinde yaygın olarak yetiştirilen bir bitkidir. Diyet lifi, karotenoidler, fenolik bileşikler, vitamin ve mineraller bakımından zengindir. Bu çalışmada bal kabağı bazı fonksiyonel özellikleri artırılmış diyabetik marmelat üretimi amaçlanmıştır. Marmelat üretimi balkabağı meyvesi esas alınarak farklı oranlarda baharat, stevia ve toz şeker ilavesi yapılarak dört farklı reçete ile gerçekleştirilmiştir. Üretilen marmelat örneklerinde pH, titrasyon asitliği, su aktivitesi, toplam fenolik ve antioksidan kapasite, renk, konsistens ve duyu analizleri yapılmıştır. Stevia ve baharat ilavesi marmelatların toplam fenolik madde ve antioksidan kapasite değerlerini arttırmıştır. Örneklerin su aktivitesi değerleri 0.924-0.932 aralığında saptanmıştır. Marmelatlara baharat ilavesi renk L\* değerinde azalmaya neden olarak rengini koyulaştırmıştır. Duyusal değerlendirmede stevia ilaveli marmelatlar koku, tat ve lezzet bakımında daha çok beğenilmiştir. Bu çalışma ile bal kabağından diyabet hastaları için kabul gören, besleyici, fonksiyonel ve katma değeri yüksek alternatif bir ürün üretilebileceği belirlenmiştir.

#### ARAŞTIRMA MAKALESİ

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#### Anahtar Kelimeler:

- Bal Kabağı,
- Diyabet,
- Marmelat,
- Stevia,
- Tarçın

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### GİRİŞ

Kabakgiller (Cucurbitaceae) familyasının bir üyesi olan balkabağı (*Cucurbita moschata* Duch.) dünya çapında yaygın olarak tüketilen bir meyvedir. Bal kabağı mineral maddeler, çoklu doymamış yağ asitleri, antioksidan maddeler ve diyet lifi bakımından zengindir (Seymen ve ark., 2020). Bal kabağının ortalama bileşimi Çizelge 1'de gösterilmektedir.

**Çizelge 1.** Balkabağının genel bileşimi (Kaya ve ark., 2016).

**Table 1.** General composition of pumpkin.

Besin Öğeleri	Miktar %
Su	91.60
Karbonhidrat	6.50
Protein	1.00
Toplam yağ	0.10
Lif	0.5
Kül	0.80

Yapılan çalışmalar balkabağının, görme bozukluğunun önlenmesinde, bazı kanser türlerine ait riskin azaltılmasıyla, hipertansiyon ve yüksek kolesterol risklerinin azaltılmasında da etkisi olduğunu göstermektedir (Seo ve ark., 2005; Xanthopoulou ve ark., 2009; Noelia ve ark., 2011; Tarwaca ve ark., 2021). Tüketicilerin doğal ve fonksiyonel ürünlere yönelimi daha sağlıklı beslenmeye verilen önemi arttırmaktadır. Bal kabağı meyvesi kurabiye ve bisküvi (Aydın, 2014), kek (Gülseren, 2019), meyve cipsi (Cavuş, 2021), dondurma (İlter, 2019), ekmek

(Polat, 2007), yoğurt (Çağlayan, 2019), reçel (Seymen, 2019) ve bebek maması (Gündoğdu, 2020) gibi ürünlerde kullanılmaktadır. Ülkemizde ise en çok reçel ve tatlı olarak tüketilmektedir (Karaağaç, 2013).

Türk gıda kodeksi reçel, jöle, marmelat ve tatlandırılmış kestane püresi tebliğine göre; geleneksel marmelat, meyve pulpu, püre, meyve suyu ve sulu ekstraktlarının veya bitkilerin kök, yaprak, çiçek gibi yenilebilen kısımlarının gerektiğinde şekerler ve su ilave edilerek sürülme kıvamına getirilmiş karışımı şeklinde tanımlanmaktadır. Ayrıca geleneksel marmelatta çözünebilir kuru madde içeriği %55'den az olamaz. Diğer bir tanıma göre marmelat; meyve ezmesine (pulp) şeker ilavesi ile hazırlanan kıvamlı bir üründür (Cemeroğlu ve ark., 2013). Ülkemizde gerek geleneksel gerekse de teknolojik olarak [kuşburnu (Sengül ve ark., 2018), alıç (Vatansever, 2016), kızılıçık (Sengül ve ark., 2018), Trabzon hurması (Kaya ve ark., 2016), çakal eriği (Sezer ve ark., 2016), armut (Yangılar, 2021), yonoz eriği (Sezer ve ark., 2016), yenedünya (Topuz, 1998), balkabağı (Seymen ve ark., 2020) ve hünnap (Kaplan ve Okçu, 2020) gibi birçok meyveden] marmelat üretilmektedir.

Yapılan literatür taramalarında kuşburnu, kızılıçık, çakal eriği ve Ahlat armudu meyvelerinden geleneksel olarak üretilen marmelatların fiziksel ve kimyasal özellikleri ile antioksidan aktivite ve fenolik bileşenlerinin belirlendiği (Sengül ve ark., 2018), alıç marmelatının duyuşal, mikrobiyolojik ve fonksiyonel özelliklerinin belirlendiği (Vatansever, 2016), Trabzon hurması bazlı karışık meyveli geleneksel marmelat üretiminin yapıldığı (Kaya ve ark., 2016), farklı erik türlerinden (çakal eriği ve yonoz eriği) elde edilen marmelatların HMF, antosiyanin ve renk değerlerinin belirlendiği (Tamer ve ark., 2010; Sezer ve ark., 2016) çalışmalar tespit edilmiştir. Ayrıca balkabağından reçel ve marmelat üretimi ve üretilen marmelat ve reçellerin fizikokimyasal ve fonksiyonel özelliklerinin karşılaştırıldığı bir çalışmaya rastlanmış ancak geleneksel ya da ticari olarak üretilen diyabetik bal kabağı marmelatı üzerine yapılmış herhangi bir çalışmaya rastlanmamıştır. Bu çalışmada ülkemizde tarımı yapılan ve genellikle tatlı ve reçel üretiminde kullanılan, sağlık açısından faydaları bilenen balkabağından stevia ilaveli diyabetik marmelat üretimi gerçekleştirilmiş ve üretilen marmelatların kimyasal, fonksiyonel ve duyuşal özellikleri belirlenmiştir.

## MATERYAL ve YÖNTEM

### Materyal

Çalışmada ana materyal olarak Tokat ilindeki semt pazarlarından temin edilen tamamen olgunlaşmamış turuncu renkli bal kabakları (*Cucurbita moschata*) kullanılmıştır. Bal kabakları temin edildikten sonra marmelat üretimi gerçekleştirilinceye kadar +4°C'de depolanmıştır.

### Yöntem

#### Marmelat Üretimi

Marmelat üretimi amacıyla ayıklama, kabuk soyma ve yıkama işlemine tabi tutulan bal kabağı örnekleri rendelenmiş ve Çizelge 2'de verilen reçetelere göre dört farklı karışım hazırlanarak marmelat üretimi gerçekleştirilmiştir. Pişirme işlemi marmelatlar 58 brikse gelince sonlandırılmıştır. Üretim sonrası örnekler 94°C'de sıcak dolmuş tekniğine uygun olarak cam kavanozlara alınmıştır.

**Çizelge 2.** Bal kabağı marmelatı üretim reçeteleri.

**Table 2.** Formula for pumpkin marmalade production.

Hammaddeler	A	B	C	D
Balkabağı	3.5 kg	3.5 kg	3.5 kg	3.5 kg
Toz şeker	1400 g	1400 g	-	-
Stevia şekeri	-	-	350 g	350 g
Su	1 L	1 L	1.4 L	1.4 L
Tarçın	0	12.5 g	0	10.5 g
Zencefil	0	12.5 g	0	10.5 g

### Analiz Metotları

Çalışmada kullanılan bal kabağına ve üretilen marmelatlar aşağıda belirtilen analizler uygulanmıştır.

### pH ve titrasyon asitliği

20°C'deki marmelatların pH değerleri; WTW-Inolab marka ve level-1 model pH metre kullanılarak belirlenmiştir (AOAC, 1990). Titrasyon asitliği ise AOAC (1990)'de belirtilen potansiyometrik yöntemle gerçekleştirilmiştir. Sonuçlar sitrik asit cinsinden g 100 g<sup>-1</sup> olarak hesaplanmıştır (Cemeroğlu ve ark., 2013).

### Renk tayini

Chroma meter, CR-300 (Japan) model renk ölçüm cihazı kullanılarak gerçekleştirilmiştir. Marmelatların L\*, a\* ve b\* değerleri ölçülerek, renk değerlerindeki değişimler (L\*, a\*, b\*, ΔE, ΔC, Hue açısı) belirlenmiştir (Cemeroğlu, 2007). ΔE ve ΔC ve Hue açısı değerleri aşağıdaki eşitlikler kullanılarak hesaplanmıştır.

$$\Delta E = [(L^*)^2 + (a^*)^2 + (b^*)^2]^{1/2} \quad (1)$$

$$\Delta C = [(a^*)^2 + (b^*)^2]^{1/2} \quad (2)$$

$$\text{Hue açısı} = \tan^{-1} (b^*/a^*) \quad (3)$$

### Toplam fenolik madde ve antioksidan kapasite analizleri

Örnek hazırlama amacıyla 2 g örnek ve 10 ml %50'lik metanol içerisinde 10 dk ultrasonik su banyosunda bekletilmiştir. Ardından 15 dk mekanik çalkalayıcıda çalkalanmış ve 8500 rpm'de de 20 dk santrifüj edilmiştir. Berrak kısım alınarak analiz edilinceye kadar -18°C'de depolanmıştır (Singleton ve Esau, 1969).

**Toplam fenolik madde tayini:** Analiz 2 N Folin-Ciocalteu fenol ayırıcı kullanılarak Singleton ve Esau (1969) tarafından tanımlanan yöntemle gerçekleştirilmiştir. 2 N 100 µL Folin-Ciocalteu ayırıcı, 100 µL ekstrakt veya 100 µL standart gallik asit çözeltileri 2.3 mL saf su ve 1 mL %7 sulu sodyum karbonat çözeltisi karıştırılmış oda sıcaklığında 2 saat bekletilmiş ve 750 nm dalga boyunda absorbansları ölçülmüş ve sonuçlar "gallik asit eş değeri" olarak hesaplanmıştır (Singleton ve Esau, 1969).

**DPPH radikal süpürme aktivitesi:** DPPH (2,2 diphenyl-1-picrylhydrazyl) yöntemiyle antioksidan kapasite tayini Brand-Williams ve ark. (1995) tarafından belirtilen yöntemle yapılmıştır. 50 µL ekstrakt veya trolox standart çözeltileri (50 µL) üzerine 1.95 mL 100 µM DPPH ilave edilmiş ve karıştırıldıktan sonra 10 dk bekletilmiştir.

Ardından 517 nm’de absorbans değerleri okunmuş ve sonuçlar “trolox eşdeğeri” cinsinden verilmiştir ([Brand-Williams ve ark., 1995](#)).

**FRAP yöntemiyle antioksidan kapasite tayini:** [Benzie ve Strain \(1996\)](#) tarafından tanımlanan yöntemle göre yapılmıştır. Örnekler (100 µL) veya uygun konsantrasyondaki standart çözeltisi (100 µL) ve 2900 µL çalışma solüsyonu karıştırılmıştır. Karışım 30 dk karanlık oda koşullarında bekletilmiştir. Süre sonunda 593 nm’de spektrofotometrede absorbans değerleri kaydedilmiştir ve sonuçlar “trolox eşdeğeri” cinsinden hesaplanmıştır ([Benzie ve Strain, 1996](#)).

#### **Su aktivitesi ( $a_w$ )**

Su aktivitesi 20°C’ye ayarlanmış AquaLab (Model Series 3TE) su aktivitesi cihazı kullanılarak ölçülmüştür ([Hughes ve ark., 2002](#)).

#### **Konsistens tayini**

Bostwick konsistometresi kullanılarak belirlenmiştir. Marmelatın akış hücresi üzerinde 30, 60, 90 ve 120 saniyelerde kat ettiği mesafe ölçülmüştür.

#### **Duyusal analiz**

Marmelat örneklerinde duyuşal değerlendirme 30-50 yaş aralığında 20 panelist ile gerçekleştirilmiştir. Duyusal değerlendirme de renk, kıvam, koku, tat ve lezzet ve genel izlenim özellikleri bakımından 1-5 puan aralığında hedonik skala kullanılarak değerlendirilmiştir ([Gould 1977; Watts ve ark., 1989](#)).

#### **İstatistiksel analizler**

Dört farklı reçete üretimi gerçekleştirilmiş ve üretilen reçetelere farklı analizler uygulanmıştır. Elde edilen verileri analiz etmek için SPSS istatistiksel programı kullanılmış ve (SPSS, Inc., Chicago, IL, USA) sonuçlar tanımlayıcı istatistikler kullanılarak verilmiştir. Deneylerde bulunan değerler, DUNCAN çoklu karşılaştırma testi ile değerlendirilmiştir. Gruplar arasındaki farklılık  $\alpha=0.05$  önem düzeyinde belirlenmiştir.

## **BULGULAR ve TARTIŞMA**

Çalışmada materyal olarak kullanılan bal kabağına ait analiz sonuçları Çizelge 3’te gösterilmektedir. Bal kabağından marmelat üretilmeden önce yeşil kabuk kısımları ve varsa ezilmiş, darbe yemiş ve çürüme belirtisi olan kısımları uzaklaştırılmıştır. Bal kabağı turuncu renkli ve sulu bir görüntüye sahiptir. Yapılan çalışmalar bal kabağıнын karotenoidler ve tokoferoller gibi antioksidan maddeler bakımından zengin olduğunu göstermektedir ([Xanthopoulou ve ark., 2009](#)). Ayrıca bal kabağıında toplam kuru madde %7-10 ile arasında değiştiğı bildirilmektedir ([Guine ve ark., 2011](#)).

**Çizelge 3.** Balkabağı analiz sonuçları.  
*Table 3. Raw pumpkin analysis results.*

Balkabağı	Değer
L*	51.47±1.16
a*	9.72±0.79
b*	42.61±2.37
aw	0.982±0.04
pH	7.24
Titrasyon Asitliği (%)	0.59±0.05
Toplam Fenolik Madde (mg 100 g <sup>-1</sup> )	16.65±1.46
FRAP (µM trolox eşdeğer 100 g <sup>-1</sup> )	1.90±0.01
DPPH (µM trolox eşdeğer 100 g <sup>-1</sup> )	3.84±0.01

Dört farklı reçete ile üretilen marmelatların renk değerleri tespit edilmiştir (Çizelge 4). Renk değeri, bir gıdanın tüketiciler tarafından tercih edilmesinde önemli rol oynamaktadır. Uygulanan ısıl işlem ile oluşabilecek maillard reaksiyonun ve ilave edilen tarçın gibi baharatların etkisi ile renkte esmerleşme meydana gelebilmektedir. Marmelat örnekleri arasında L\* değerinin (koyuluk/açıklık) istatistiksel olarak önemli düzeyde farklılık gösterdiği ( $\alpha=0.05$ ) ve 37.56-41.66 arasında değiştiği belirlenmiştir. Marmelatların baharat ilavesinin rengi koyulaştırdığı ve toz şeker ile yapılan marmelatların daha koyu renkte olduğu tespit edilmiştir. Kırmızılık/yeşillik değeri olarak bilinen a\* değerlerinin istatistik olarak farklı olduğu ( $\alpha=0.05$ ) belirlenmiş olup stevia ilaveli örneklerde bu değerler daha da yükseldiği tespit edilmiştir. Stevia ilavesinin marmelatların kırmızılık değerini yükselttiği görülmektedir. Benzer artışlar literatürde tespit edilmiştir (Alencar ve ark., 2015; Salar ve ark., 2020). Marmelatlarda en yüksek b\* (sarılık/mavilik) değeri stevia ilaveli örneklerde tespit edilmiştir. Marmelat örneklerinin toplam renk farkı ( $\Delta E$ ), kroma değeri ( $\Delta C$ ) ve hue açısı değerlerinin en yüksek baharatsız stevia ilaveli marmelatda olduğu saptanmıştır. Ayrıca marmelatların  $\Delta E$ ,  $\Delta C$  ve hue açısı değerleri arasında istatistiksel olarak önemli farklılık bulunmuştur ( $\alpha=0.05$ ).

**Çizelge 4.** Bal kabağı marmelatı renk değerleri.

*Table 4. Color values for pumpkin marmalade.*

Reçete	L*	a*	b*	$\Delta E$	$\Delta C$	Hue Açısı
A	37.56±0.20 <sup>c</sup>	3.54±0.22 <sup>d</sup>	26.11±0.34 <sup>c</sup>	-	-	-
B	34.68±0.21 <sup>d</sup>	3.71±0.13 <sup>c</sup>	20.91±0.26 <sup>d</sup>	5.88	26.29	-1.40
C	41.66±0.17 <sup>a</sup>	5.11±0.16 <sup>b</sup>	31.55±0.37 <sup>a</sup>	6.99	32.12	-9.57
D	40.06±0.24 <sup>b</sup>	5.97±0.15 <sup>a</sup>	29.08±0.54 <sup>b</sup>	4.58	14.74	-0.16

a, b, c, d aynı sütunda farklı harfle işaretlenmiş ortalamalar istatistiki olarak Duncan testine göre birbirinden farklıdır ( $\alpha=0.05$ )

Marmelat örneklerinin bazı önemli kalite özelliklerine ait analiz sonuçları Çizelge 5'te gösterilmektedir. Reçel, marmelat gibi ürünlerde iyi jel oluşumu ve uzun raf ömrü için pH seviyesi önem arz etmektedir. Türk Gıda Kodeksi 2006/55 nolu tebliğe göre marmelatların pH 2.8-3.5 arasında olmalıdır. Çalışmamızda üretilen marmelatların herhangi bir asit ilavesi yapılmamış olup pH değerlerinin 7.28-7.55 arasında değiştiği saptanmıştır. Bu değerler Seymen ve ark. (2020) yaptıkları bal kabağı marmelatının pH değerinden (4.77) ve tebliğde istenen pH değerinden yüksektir. Marmelat üretiminde reçeteye baharat ilavesi pH değerinde düşmeye neden olmaktadır. Üretilen



tüm marmelat örneklerinin pH değerleri istatistiksel olarak farklılık göstermektedir ( $\alpha=0.05$ ). Örneklerin titrasyon asitliği değerleri sitrik asit cinsinden hesaplanmış olup titrasyon asitliği değerleri arasında istatistiki bir fark bulunmamıştır. Örneklerin titrasyon asitliği %0.24-0.29 arasında tespit edilmiştir. Titrasyon asitliği değerleri literatür ile benzerlik göstermektedir (Seymen ve ark., 2020). Gıdaların bozulmadan saklanabilmesinde önemli parametrelerden birisi de su aktivitesi değeridir. Marmelat örneklerinin su aktivitesi değerlerinin 0.924-0.932 arasında değişmiş olup, toz şeker ilaveli marmelatların su aktivitesi değerlerinin daha düşük olduğu saptanmıştır. Literatürde farklı meyvelerden elde edilen marmelatların su aktivitesi değerlerinin 0.810-0.980 arasında değiştiği (Sengül ve ark., 2018) ve çalışmamızda elde edilen sonuçların literatür ile benzerlik gösterdiği tespit edilmiştir.

**Çizelge 5.** Bal kabağı marmelatı kimyasal analiz sonuçları.

**Table 5.** Chemical analysis results for pumpkin marmalade.

Reçete	Su Aktivitesi ( $a_w$ )	pH	Titrasyon Asitliği (%)
A	0.924±0.001 <sup>b</sup>	7.55	0.25±0.002 <sup>a</sup>
B	0.927±0.003 <sup>b</sup>	7.30	0.24±0.005 <sup>a</sup>
C	0.931±0.002 <sup>a</sup>	7.51	0.29±0.004 <sup>a</sup>
D	0.932±0.003 <sup>a</sup>	7.28	0.24±0.004 <sup>a</sup>

a,b,c,d aynı sütunda farklı harfle işaretlenmiş ortalamalar istatistiki olarak Duncan testine göre birbirinden farklıdır ( $\alpha=0.05$ )

Marmelat örneklerine ait toplam fenolik madde ( $\text{mg } 100\text{g}^{-1}$ ) miktarları Çizelge 6'da gösterilmektedir. Bal kabağı meyvesinde  $16.65$  ( $\text{mg } 100\text{g}^{-1}$ ) olan toplam fenolik madde miktarı marmelatlarda  $17.90$ - $45.65$  ( $\text{mg } 100\text{g}^{-1}$ ) aralığına yükselmiştir. Örneklerin toplam fenolik madde miktarları istatistiki olarak farklılık göstermektedir ( $\alpha=0.05$ ). Marmelat üretiminde yüksek fenolik madde içeriğine sahip baharat kullanımının fenolik madde miktarını yükselttiği belirlenmiştir. Ayrıca yapısında fenolik madde içeren stevia ilavesinin toz şeker ilavesine göre fenolik madde miktarında daha fazla artışa neden olduğu görülmektedir. Seymen ve ark. (2020) yaptıkları çalışmada bal kabağı marmelatının toplam fenolik madde değerini  $71.92$   $\text{mg GAE } 100 \text{ g}^{-1}$  arasında bildirmişlerdir. Sezer ve ark. (2016) erik marmelatlarının toplam fenolik madde miktarını  $36$ - $47.75$   $\text{mg } 100 \text{ g}^{-1}$ , Esin ve Kaya (2021) frenk üzümü marmelatında  $70.77$   $\text{mg GAE } 100 \text{ g}^{-1}$ , Türkmen ve ark. (2019) erik bazlı geleneksel marmelatlarda  $1.71$ - $2.12$   $\text{mg GAE } 100 \text{ g}^{-1}$  arasında rapor etmişlerdir. Çalışmamızda elde edilen toplam fenolik madde miktarı literatür ile kıyaslandığından düşük bulunmuştur.

**Çizelge 6.** Bal kabağı marmelatı fonksiyonel analiz sonuçları.

**Table 6.** Functional analysis results for pumpkin marmalade.

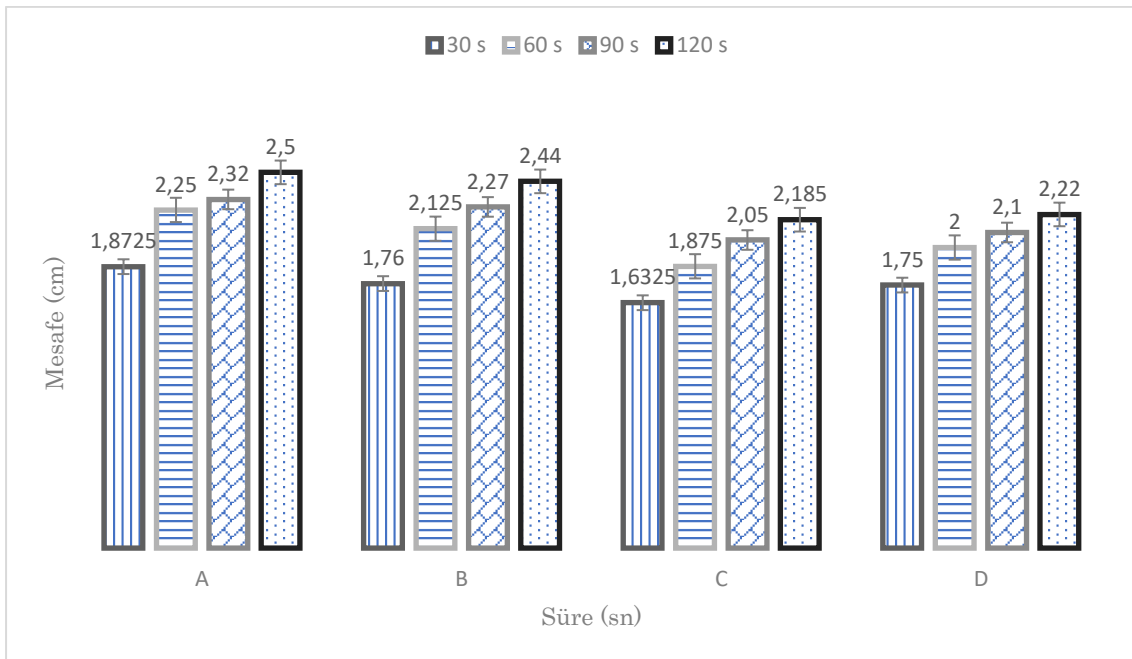
Reçete	Toplam Fenolik Madde ( $\text{mg } 100 \text{ g}^{-1}$ )	FRAP ( $\mu\text{M trolox eşdeğer } 100 \text{ g}^{-1}$ )	DPPH ( $\mu\text{M trolox eşdeğer } 100 \text{ g}^{-1}$ )
A	17.90±1.24 <sup>d</sup>	2.48±0.03 <sup>b</sup>	3.72±0.01 <sup>d</sup>
B	40.40±1.52 <sup>b</sup>	2.74±0.02 <sup>a</sup>	3.90±0.01 <sup>b</sup>
C	20.15±1.06 <sup>c</sup>	2.51±0.01 <sup>b</sup>	3.85±0.01 <sup>c</sup>
D	45.65±0.78 <sup>a</sup>	2.79±0.05 <sup>a</sup>	4.07±0.05 <sup>a</sup>

a,b,c,d aynı sütunda farklı harfle işaretlenmiş ortalamalar istatistiki olarak Duncan testine göre birbirinden farklıdır ( $\alpha=0.05$ )

Marmelat örneklerinin antioksidan kapasite değerleri iki farklı yöntemle ölçülmüş olup sonuçlar Çizelge 6'da gösterilmektedir. Örneklerin antioksidan kapasite

değerlerinin baharat ilavesi ile artış gösterdiği belirlenmiştir. Bu durumun antioksidan aktivitesinin yüksek olduğu bilinen tarçın ilavesinden kaynaklandığı düşünülmektedir. DPPH ve FRAP değerleri arasında istatistik olarak önemli düzeyde farklılık mevcuttur ( $\alpha=0.05$ ). Marmelatların DPPH değerleri  $3.72 \mu\text{M TE } 100\text{g}^{-1}$ -  $4.07 \mu\text{M TE } 100\text{g}^{-1}$  arasında, FRAP değerleri ise  $2.48$ - $2.79 \mu\text{M TE } 100\text{g}^{-1}$  arasında belirlenmiştir. Literatür ile karşılaştırıldığında antioksidan değerlerinin daha düşük olduğu görülmüştür. Toplam fenolik madde ve antioksidan aktivite değerlerinin literatürden daha düşük çıkmasının bal kabağının olgunluk düzeyi, yetiştiği iklim ve toprak yapısı ve bal kabağı çeşitlerindeki farklılıklar olduğu düşünülmektedir. Yapılan çalışmalarda farklı meyvelerden üretilen marmelatların antioksidan madde içerikleri; [Seymen ve ark. \(2020\)](#)  $24.99 \mu\text{M TE g}^{-1}$ , [Türkmen ve ark. \(2019\)](#)  $58.98$ - $79.49 \mu\text{M TE g}^{-1}$  arasında, [Sezer ve ark. \(2016\)](#)  $454.03$ - $658.06 \text{ mg } 100 \text{ g}^{-1}$  olarak rapor edilmiştir. Marmelat örneklerinin antioksidan kapasitelerinde oluşan artışın üretim esnasında oluşan maillard reaksiyon ürünlerinden ve ilave edilen baharatlardan kaynaklandığı düşünülmektedir.

Marmelat ve reçellerde kıvam arzu edilen bir durumdur. Çalışmamızda örneklerin kıvamı Bostwick konsistometresi kullanılarak belirlenmiştir. Bu kapsamda marmelatın akış hücresi üzerinde 30, 60, 90 ve 120 saniyelerde kat ettiği mesafe cm olarak ölçülmüştür. Örneklerin konsistometre değerleri  $1.75$ - $2.50$  cm aralığında ölçülmüş olup sonuçlar Şekil 1'de gösterilmektedir. Şekil 1 irdelendiğinde toz şeker ilavesinin stevia ilavesine göre kıvamı azalttığı ve marmelatların daha akışkan yapıya dönüşmeye başladığı görülmektedir. Stevia şekerinin yapıdaki suyu daha iyi tutmasının buna neden olduğu düşünülmektedir. Her iki üretim tipi içinde baharat ilavesinin kıvamda önemli bir değişikliğe neden olmadığı tespit edilmiştir. Literatürde vizkozite değerlerinin ölçüldüğü çalışmalar mevcuttur. Yoğunluk/kıvam değerleri ise Bostwick konsistometresi ile ölçülmektedir. Bundan dolayı vizkozite değerlerinin kıvam değerleri ile karşılaştırılması yapılmamıştır.



Şekil 1. Bal kabağı marmelatı kıvam sonuçları.

Figure 1. Pumpkin marmalade consistency results.

Duyusal değerlendirme 20 panelist ile gerçekleştirilmiştir. Duyusal değerlendirmede örneklerin almış olduğu puanlar dikkate alındığında baharatsız toz şeker ilaveli örneğin en yüksek puanı aldığı ardından en yüksek puanları stevia ilaveli örneklerin aldığı belirlenmiştir. Örneklerin renk, kıvam, koku, tat ve lezzet ve genel beğeni olarak değerlendirilmesi istenmiştir. Renk ve kıvam açısından en beğenilen örnek A reçetesi iken, koku ve tat/lezzet değerinde en beğenilen örnek stevia ilaveli örnek reçeteleri olmuştur. Stevia ilavesi ile üretilen marmelatların genel olarak beğenildiği ve tüketilebileceği düşünülmektedir.

## SONUÇ

Taze meyve ve sebzeler sağlık açısından önemli bileşikleri bünyelerinde içermektedirler. Özellikle taze bal kabağının bulunmadığı dönemlerde reçel ve marmelatlar bu hammaddelere ulaşmada alternatif birer ürün durumundadır. Reçel ve marmelatlar; çekici renkli ve aromalı ürünler sunmaktadırlar. Bu araştırmada diyabet hastaları için alternatif bir ürün üretmek amacıyla ülkemizde genellikle reçel ve tatlısı yapılan bal kabağından stevia ilaveli marmelat üretilmiştir. Stevia ilavesinin üretilen marmelatların fiziksel ve duyusal özelliklerini etkilemediği, fonksiyonel özelliklerini ise iyileştirdiği görülmektedir. Araştırmadan elde edilecek bulgular ülkemizde kısmi olarak değerlendirilen bal kabağının işlenip, alternatif tüketime katkı sağlayacağını, diyabet hastaları için fonksiyonel bir ürün meydana getirilebileceğini göstermektedir.

## ÇIKAR ÇATIŞMASI

Yazarlar arasında herhangi bir çıkar çatışması yoktur.

## YAZAR KATKISI

**Ali Cingöz:** Araştırma fikrini belirledi, geliştirdi, analiz yaptı, sonuçları analiz etti, yorumladı ve yazdı.

**Aslıhan Demirdöven:** Araştırma yöntemlerini önerdi, analiz sonuçlarını kontrol etti, makale düzenlemesi ve kontrolünü yaptı.

## ETİK KURUL KARARI

Bu makale Etik Kurul Kararı gerektirmemektedir.

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## Motorbike Powered Trailer for Transporting Poultry Birds

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### ABSTRACT

The demand for poultry products has made it necessary for farmers to transport poultry birds from farmhouses to where they could be processed for the consumers. However, transporting poultry birds over a long distance, and under unfavourable conditions, could cause a shift in their behaviours, and biochemical reactions, resulting in an increase in birds' traumatic injuries, weight loss, and poor meat quality. Therefore, a motorbike powered trailer was developed, having an average loading capacity of 50 to 54 broiler chickens, with a live body weight of 1.5 kg, for small-scale farmers. The climatic conditions (temperature and relative humidity) within the trailer were evaluated when the trailer was tested on the motion for 35 minutes during the hot period of the day (1:30 pm to 2:05 pm). The result showed that the indoor temperatures ranged between 29.3°C and 31.6°C and the outdoor temperatures ranged from 31.0°C and 33.3°C. Similarly, the indoor relative humidity was between 61 and 69% while that of outdoor relative humidity was between 56% and 64%. The mean apparent equivalent temperature (AET) of the trailer was estimated as 30.45 ± 0.54°C. This implies that the thermal zone within the trailer could be considered safe for poultry birds during hot weather periods in the humid tropical climate. The total production cost of the trailer was two hundred and forty-three US dollars, eighty cents (\$243.80).

#### RESEARCH ARTICLE

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- DHT sensors

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## INTRODUCTION

Livestock, according to [Thornton, \(2010\)](#) occupies about 30% of the planet's ice-free terrestrial surface area, serves as a source of animal-based protein ([Jongbo, 2020](#)), and their production has contributed immensely to the economic growth of any nation especially in ensuring food security ([Nabarro and Wannous, 2014](#)). They are the source of employment for over 1.3 billion people worldwide, contribute about 40% of the global income from agricultural products and also support the livelihoods of over 600 million poor small-scale farmers in developing countries ([Nabarro and Wannous, 2014](#); [Thornton, 2010](#)). Poultry is one of the sectors of livestock production which has greatly contributed to the animal-based protein source for humans. Large numbers of poultry birds have been slaughtered for consumption compared to other livestock ([Weeks, 2014](#)). To meet up with the increased demand for poultry globally, there has been an increase in the poultry production intensification and transportation of livestock ([Minka and Ayo, 2007](#)).

The transportation system is an important component of livestock production ([Schwartzkopf-Genswein et al., 2012](#)) and its efficiency depends on the economic, environmental perspectives and animal welfare ([Frisk et al., 2018](#)). Livestock could be transported from the farm to other places outside the farm for veterinary treatment, marketing and slaughtering purposes ([Broom, 2019](#); [Frisk et al., 2018](#)). During transportation, animals could be subjected to situations that could negatively affect their welfare which could be assessed using welfare indicators such as animal behaviours, animal physiological response and carcass quality measures ([Broom, 2019](#)). Some of the factors affecting the welfare of animals during transportation include human attitudes to livestock, journey planning, a mixture of animals from different social groups, method of driving and road conditions, and floor space per animal on the vehicle ([Broom, 2019](#); [Frisk et al., 2018](#)). Others include journey distance, animal inspection during the journey, time that the journey starts, number of pick-ups and stops along the journey and the climatic condition of the vehicle during the journey ([Broom, 2019](#); [Frisk et al., 2018](#)). Although subjecting animals to the transport of long journeys may not negatively affect the animal welfare, it is appropriate to ensure that the health, feed and water intake, rest and the thermal condition of the animal environment are critically considered ([Frisk et al., 2018](#); [Nielsen et al., 2011](#)). Similarly, subjecting animals to short-distance journeys could reduce the cost of transportation, animals' injury and environmental pollution ([Frisk et al., 2018](#)).

Transportation in poultry production could cause various degrees of stress, varying from discomfort to death of birds ([Schwartzkopf-Genswein et al., 2012](#)). For broiler chickens, thermal stress is the major problem confronting them during transportation and its directly related to their poor welfare signs shown when in transit ([Strawford et al., 2011](#)). In a study conducted by [Vosmerova et al. \(2010\)](#), they indicated that pre-transport handling procedures of broilers such as catching, crating and loading and low ambient temperature during transportation caused more stress to broilers than high temperature (35°C) during transportation. The higher percentage of dead on arrival (DoA) of broiler, reported by [Chauvin et al. \(2011\)](#) and [Schwartzkopf-Genswein et al. \(2012\)](#), was shown to be caused by the climatic conditions broiler were subjected to before the slaughtering process. While [Arikan et al. \(2017\)](#) and [Voslarova et al. \(2007\)](#) reported that long transportation distances caused more broiler

losses than short transportation distances. This shows that potential stressors such as extreme temperatures, variable vehicle speeds, noise pollution, water and feed deprivation, vibration due to poor road and overcrowding that could cause fear, discomfort and high mortality during transportation ([Mitchell and Kettlewell, 2014](#); [Vosmerova et al., 2010](#)) are more pronounced when subjecting broilers to long-distance under extreme weather conditions.

The thermal condition of the microclimate within the vehicle is a major concern as regards the problems associated with the stress birds are subjected to and that the problem could only be minimised by improving vehicle designs to provide the environmental conditions required by the on-board birds ([Mitchell and Kettlewell, 2014](#)). There are a few studies on different designs and development of road transportation systems for poultry. [Hui \(2013\)](#) developed and evaluated an actively heated and ventilated transport vehicle for broiler chickens transported under harsh Canadian Prairies winter conditions. He reported that the integration of active ventilation and heating system in poultry transportation vehicles improved the climatic conditions of the microclimate of broilers during harsh winter conditions. In a study carried out by [Aldridge et al. \(2019\)](#), they reported that dual boarding of the external part of the transport vehicle maintained the indoor temperature at about 8.0°C when the outdoor temperature was as cold as -16°C during winter. However, during the summer conditions, introducing double boarding to the exterior part of the transport trailer resulted in a temperature gain of 2.0°C within the trailer. [Norton et al. \(2013\)](#) used computational fluid dynamics (CFD) to simulate the environmental heterogeneity in a dual-mode ventilated (naturally and mechanically) ferry transportation. They indicated that the naturally ventilated deck of the ferry transport was hotter and highly humid compared to that of the mechanically ventilated deck. Most of the studies in the literature are either for commercial purposes or the developed countries and less or no consideration was given to the small-scale farmers in the developing countries who could not afford the available trailers for transporting their animals. Therefore, there is a need for the development of a less expensive trailer that an average farmer in developing countries could afford and used for transporting their poultry birds.

Therefore, the specific objectives of this study were to (i) design and develop a motorbike powered trailer for transporting small animals such as poultry birds and (ii) evaluate the indoor environmental conditions and (iii) evaluate the thermal zone (AET) of the trailer using internet of things (IoT) based instrumentation.

## MATERIALS AND METHODS

The design and development of the trailer were considered necessary after a feasibility study was carried out across some places in Ondo State, Nigeria to understand the transportation system used by the small-scale farmers for transporting their animals. Figure 1 shows the way an average farmer used to convey their poultry birds using a crate or cage fastened to the back of a motorbike. Figure 1a shows a farmer trying to take a ride with some birds in a crate. Figure 1b shows a bike man conveying matured broiler chickens with a cage that was not properly fastened to the bike. Figure 1c shows a bike carrying a well-sited broiler cage but with a small sitting space for the bike man to sit on. Figure 1d shows a half stand motorbike carrying a broiler crate at an angle



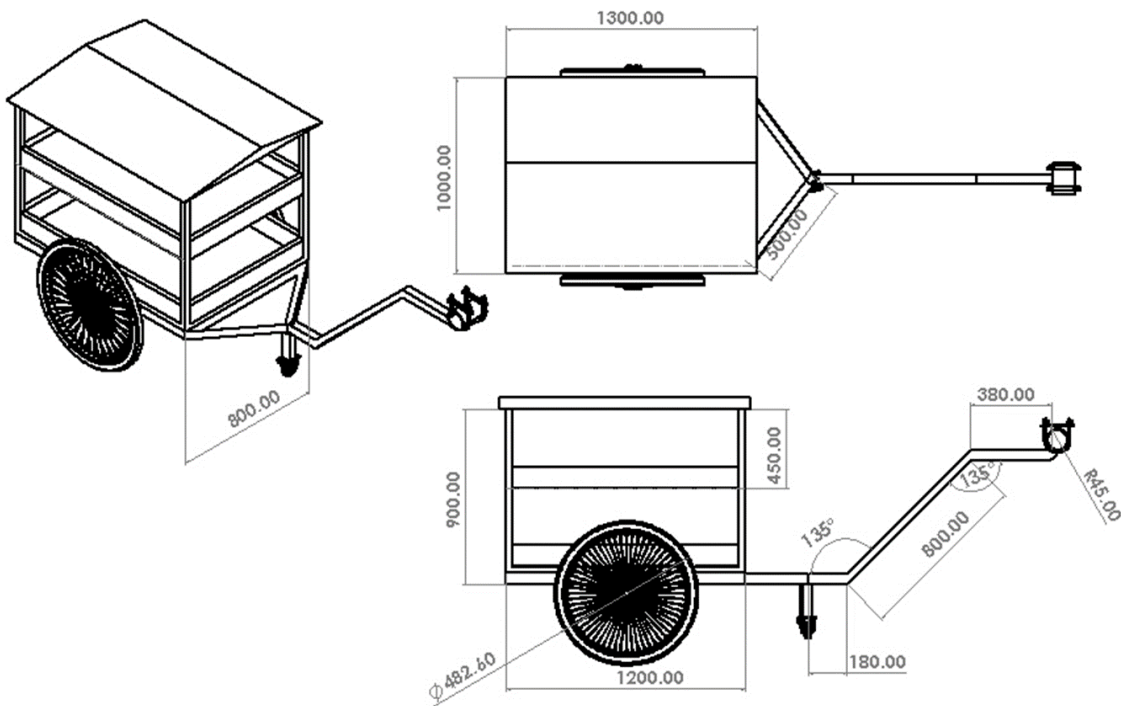
that could cause discomfort for the birds. Considering the figures, it could be observed that transporting poultry birds in crates/cages, fastened to the back of the motorbike, could cause discomfort for both the farmers and the birds. In addition, it could result in poor welfare and thermal stress for birds during transportation.



**Figure 1.** Transportation of poultry birds using the motorbike.

#### **Design details and the description of the trailer**

A motorcycle powered trailer was designed using SOLIDWORKS 2021 and fabricated in the Department of Agricultural and Environmental Engineering workshop, Federal University of Technology, Akure, Nigeria (Figure 2). The trailer was designed to aid the transportation of poultry birds from one location to another considering the stress involved in the existing poultry transportation system earlier discussed. This trailer would, in the process, reduce the stress undergone by the farmer and the poultry birds being transported.



**Figure 2.** Trailer for transporting poultry birds. All dimensions are in mm.

**Design of the trailer’s shaft (axle)**

The shaft (axle) of the trailer with a total length of 1.10 m, carrying a uniformly distributed cage of an average weight of 1.03 kN m<sup>-1</sup> and a total estimated poultry weight of 1.155 kN m<sup>-1</sup>, was designed and calculated. The total uniformly distributed load, carried by the trailer was estimated as 2.731 kN m<sup>-1</sup>. To estimate the total shear force and the bending moment of the axle of the trailer, the weights of the axle’s components were mathematically estimated. The maximum bending moment ( $M_b$ ) was estimated as 0.392 kN m<sup>-1</sup> while the torsional moment ( $M_t$ ) of the axle was calculated as 0.011 kN m<sup>-1</sup> using;

$$M_t = \frac{60 \times P}{2\pi N} = \frac{9550 \times P}{N} \text{ Nm} \tag{1}$$

where  $P$  is the average power (5.0 kW) of the motorbike pulling the trailer and  $N$ , the number of revolutions (1300 rpm) of the bike’s wheel.

The diameter ( $d$ ) of the trailer’s axle was estimated as 34.11 mm using;

$$d^3 = \frac{16}{\pi S_s} \sqrt{(k_b M_b)^2 + (k_t M_t)^2} \tag{2}$$

where  $S_s$  is the allowable stress (55.0 MPa),  $K_b$  is the bending stress factor (1.5),  $K_t$  is the torsional stress factor (1.0),  $M_b$  is the maximum bending moment, and  $M_t$  is the torsional moment. The factor of safety considered suitable for the shaft diameter was 0.09. In this study, a standard shaft diameter of 35 mm was selected for the trailer since a 34.11 mm diameter shaft was not readily available for the study.

### Description of the trailer

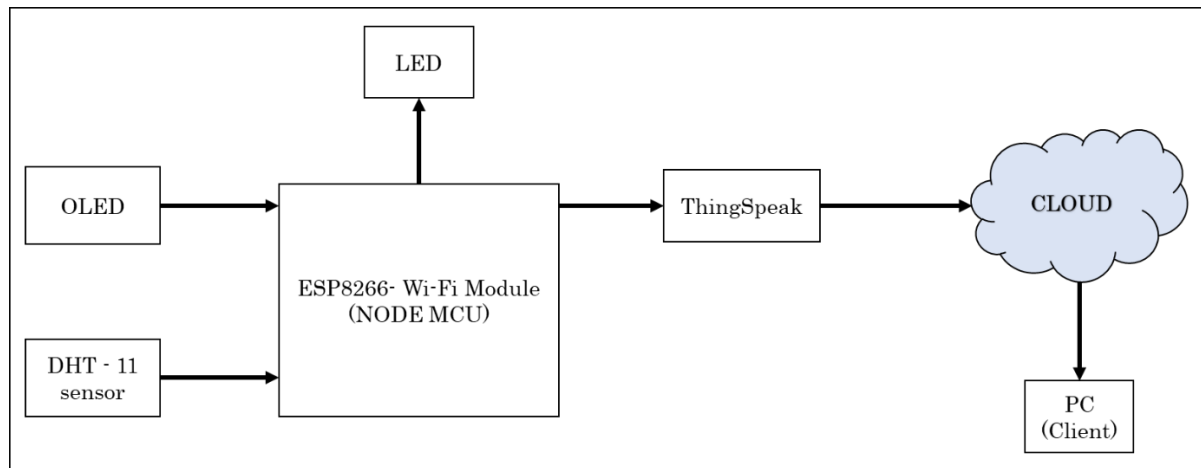
The trailer, as shown in Figure 3, consists of a frame, two wheels, a hitching point, wire gauze as a side covering, two floors (upper and lower), a supporting stand, openings and a top cover. With the hitching point, the trailer could easily be attached to the motorcycle. The frame with a length of 1200 mm, a height of 900 mm and a width of 800 mm, was fabricated using 45 x 45 mm angle iron. The frame was set on two wheels with a rim diameter of 482.6 mm. The trailer has two floors/ layers (upper and lower) which serve as compartments for poultry birds. The floors were made of wood of 20 mm thickness. A supporting stand, made of a universal swivel caster, was attached to the frame at the front to ensure that the trailer could stand when not hauled and to make the attachment of the trailer to the motorcycle very easy. The sidewalls of the trailer were covered with 20 x 30 mm wire gauze. The average loading capacity of the trailer was estimated as 50 to 54 broiler chickens, with a live body weight of 1.5 kg. This was achieved based on the stocking density of 42 kg m<sup>-2</sup> reported by [Giersberg et al. \(2016\)](#). The total production cost of the trailer was two hundred and forty-three US dollars, eighty cents (\$243.80).



**Figure 3.** Developed motorbike powered trailer for transporting poultry birds.

### Instrumentation and data collection

For this study, an environmental monitoring device was developed to monitor the temperature and relative humidity within and outside of the trailer. The device comprised DHT-11 sensors, an ESP8266 Wi-Fi module (NodeMCU), an OLED and a LED display. The block diagram of the system is shown in Figure 4. The NodeMCU, a Wi-Fi communication module ESP8266, is an IoT platform open-source ([Pasika and Gandla, 2020](#)). It collects the temperature and relative humidity from the DHT-11, processed the data and uploads the data to the ThingSpeak server ([Pasika and Gandla, 2020](#)). The ThingSpeak is an open-source application. It is an IoT data collection application, capable of analysing and transmitting the data in real-time ([Pasika and Gandla, 2020](#)). The system was designed in an Embedded-C and simulated using Arduino IDE. On the ThingSpeak server, the authorised users could gain access to the uploaded data by signing into their account using the user ID and password.



**Figure 4.** The block diagram of the system for the environmental monitoring.

The technical specifications of the DHT-11 sensors used in this study are shown in Table 1. The DHT-11 has already been calibrated by the manufacturing industry. Therefore, there was no need for further calibration before the sensors were deployed for environmental parameter monitoring. However, the reliability of the sensors was tested by comparing its data with that of the data obtained from HTC-1, a temperature and humidity sensor. The result of the test showed that there was no significant difference between the data of DHT-11 and that of HTC-1. Therefore, the DHT-11 was deployed directly without further calibration.

**Table 1.** Technical specifications of the DHT-11 sensor

Technical specifications	DHT 11 sensor					
	Temperature			Relative humidity		
	Minimum	Typical	Maximum	Minimum	Typical	Maximum
Measurement range	0°C	-	50°C	20%	-	90%
Accuracy	±1°C	-	±2 °C	-	±4 %	±5 %
Resolution	1°C, 8 Bit	1°C, 8 Bit	1°C, 8 Bit	1%, 8 Bit	1%, 8 Bit	1%, 8 Bit
Response time	6 s	-	30 s	6 s	10 s	15 s
Sampling interval	60 s					

To evaluate the thermal conditions of an empty motorbike powered trailer, as shown in Figure 5, before using it for poultry transportation, the environmental monitoring device was installed inside and outside the trailer to monitor the air temperature and air relative humidity of the trailer. The outdoor DHT-11 sensor was placed 0.10 m above the cover of the trailer while the inside DHT-11 sensor was placed inside the upper floor of the trailer. The motorbike-powered trailer was driven on an off-road track, a typical Nigeria road, for thirty-five (35) minutes from 1:30 pm to 2:05 pm during which the environmental condition was monitored and recorded. The period of the study was considered appropriate because the weather in the humid tropical climate starts to get warm, according to [Jongbo \(2020\)](#), from midday (noon) to around 4:00 pm and the warm condition was considered to negatively affect the microclimate of the animals. It is necessary to clearly state that the trailer was tested without poultry birds inside. Only the environmental parameters (air temperature and air relative humidity) were evaluated to understand the climatic condition within the trailer. Likewise, the effect of motorbike speed on the indoor environmental parameter was not evaluated since there was no animal in the trailer. Consequently, varying the motorbike speed was not considered necessary and the speed was maintained at a speed range of 30 to 40 km h<sup>-1</sup> during the period of the road test.

The data from the sensors was immediately monitored by an assistant through a ThingSpeak chart on an android phone. The application enables the user to easily visualise the data in the ThingSpeak channels. The data was later downloaded in the form of a Google spreadsheet or CSV file format on a laptop (HP ProBook 4540s) for further processing.



**Figure 5.** Road testing of the motorbike powered trailer and data acquisition.

### Data analysis

The data acquired from the trailer was processed on Microsoft Excel Professional Plus 2019 and analysed using JMP® Pro 13.0.0 (SAS Institute Inc. USA). A t-test analysis was conducted to determine the significance level between the mean indoor and outdoor climatic conditions of the trailer when not occupied by poultry birds. To determine the thermal zones for the trailer during transportation, an Apparent Equivalent Temperature (AET), a physiological stress response model, expressed by [Mitchell \(2006\)](#) and used by [Aldridge et al. \(2019\)](#), was adopted and used. The AET (Equation 3) was estimated from the air temperature and the relative humidity obtained from within the trailer. The AET within the trailer could assist in understanding the level of physiological stress, and the changes in the deep body temperature of broiler chickens during transportation ([Mitchell, 2006](#)). It is clear that as the AET reaches 45°C, mild thermal stress could set in during transportation. However, with the AET reaching 65°C and above, severe thermal stress and high mortality could occur ([Mitchell, 2006](#)).

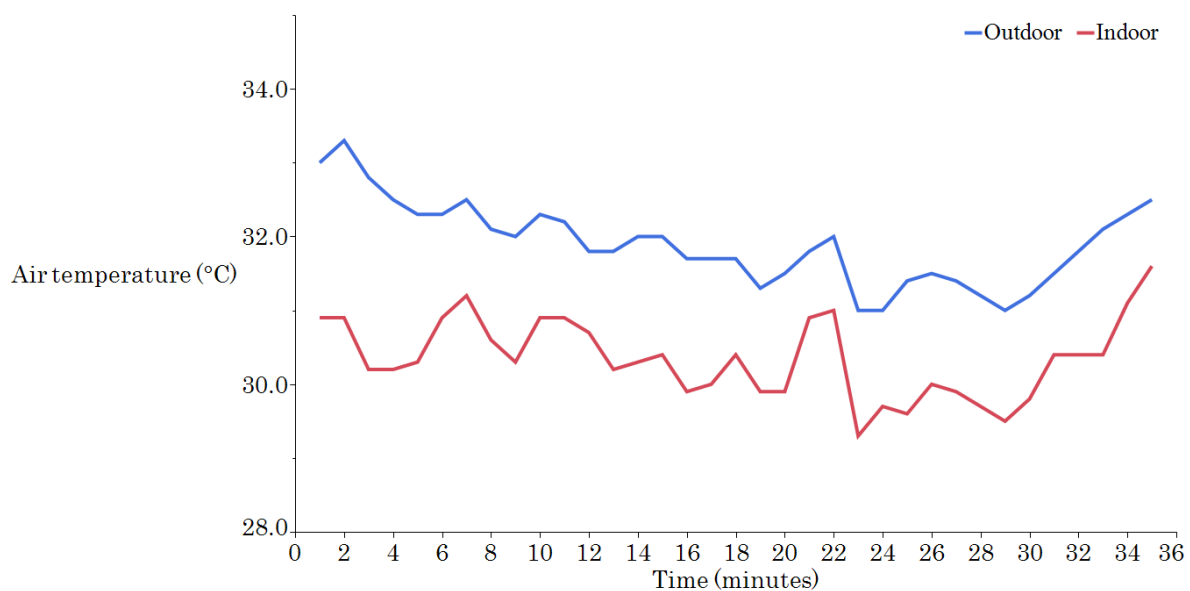
$$AET = T_i + \frac{10^{\left(30.5905 - 8.2 \times \log_{10}(K) - \frac{3142.31}{K}\right)} \times \left(\frac{rH}{100}\right)}{0.93 \times (0.0006363601K + 0.472)} \quad (3)$$

where  $T_i$  is the measured air temperature (°C),  $K$  is the air temperature corrected to Kelvin (°C + 273.15), and  $rH$  is the measured relative humidity (%).

## RESULTS AND DISCUSSION

The suitability of the trailer for poultry bird transportation was tested and evaluated based on the climatic conditions (air temperatures and air relative humidity) within the trailer on a road similar to a typical farm road in Nigeria. The air temperatures (within

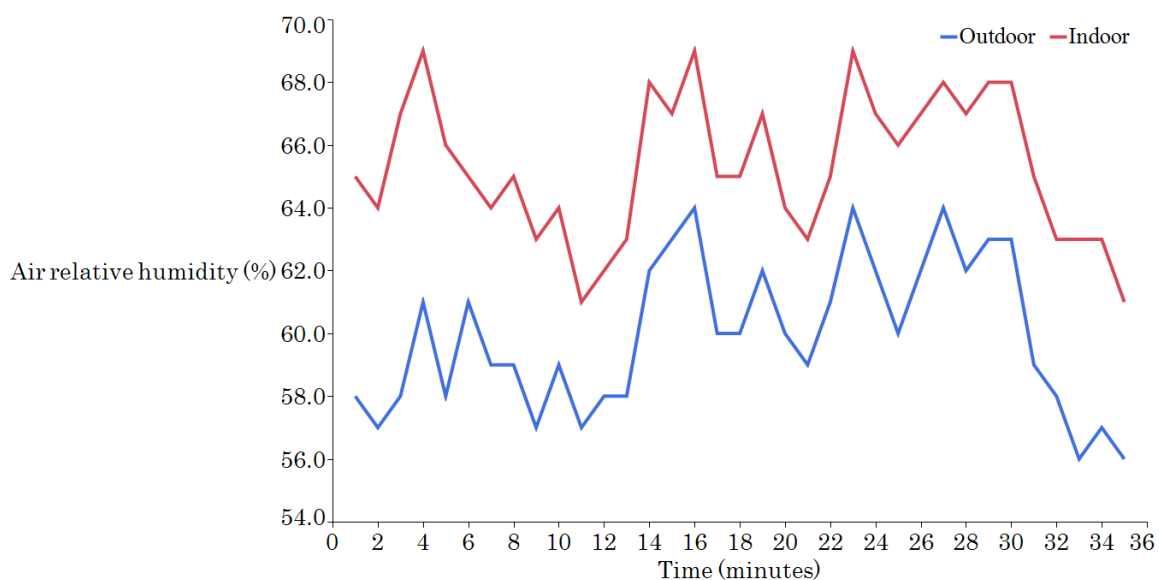
and outside) of the trailer are illustrated in Figure 6. It could be observed in the figure that the air temperatures of both the outdoor and the indoor had a similar pattern. From Figure 6, as the outside air temperature varied between 31°C and 33.3°C, the indoor air temperature was observed to vary between 29.3°C and 31.6°C. This indicates that the trailer was able to minimise the indoor temperature by about 2°C. The mean air temperatures observed both within and outside the trailer were  $30.4 \pm 0.54^\circ\text{C}$  and  $31.9 \pm 0.56^\circ\text{C}$  respectively. The result of the t-test carried out to determine the level of significance between the indoor temperature and that of the outside air temperatures showed that there was a significant difference ( $p < 0.0001$ ) between the mean indoor air temperature and that of the outside air temperature. According to [Schwartzkopf-Genswein et al. \(2012\)](#), the indoor temperature of the trailer for transporting small animals should not exceed 30°C to prevent heat stress. However, in the humid tropical climate, achieving such thermal conditions inside the vehicle could be difficult since air temperature is usually above 30°C ([Jongbo, 2020](#)). Therefore, in this study, an average air temperature of 30.4°C was observed at an average motorbike speed of 30 to 40 km h<sup>-1</sup> within the trailer. This thermal condition could be considered safe for the poultry birds when transported on Nigerian roads under humid tropical climatic conditions. Higher live shrinkage and higher core body temperature of broiler during transportation should be prevented by not exposing broiler chickens to higher indoor temperatures ([Schwartzkopf-Genswein et al., 2012](#)). In the reviewed work of [Broom \(2019\)](#), transporting small animals such as turkeys under an indoor air temperature of 35°C could negatively affect the behaviour, physiology and carcass quality of the birds. Therefore, the air temperature (high or low), to which small animals are subjected during transportation, could result in suffering, poor welfare and sudden death of the animals ([Nielsen et al., 2011](#)).



**Figure 6.** Air temperature variation within and outside the trailer.

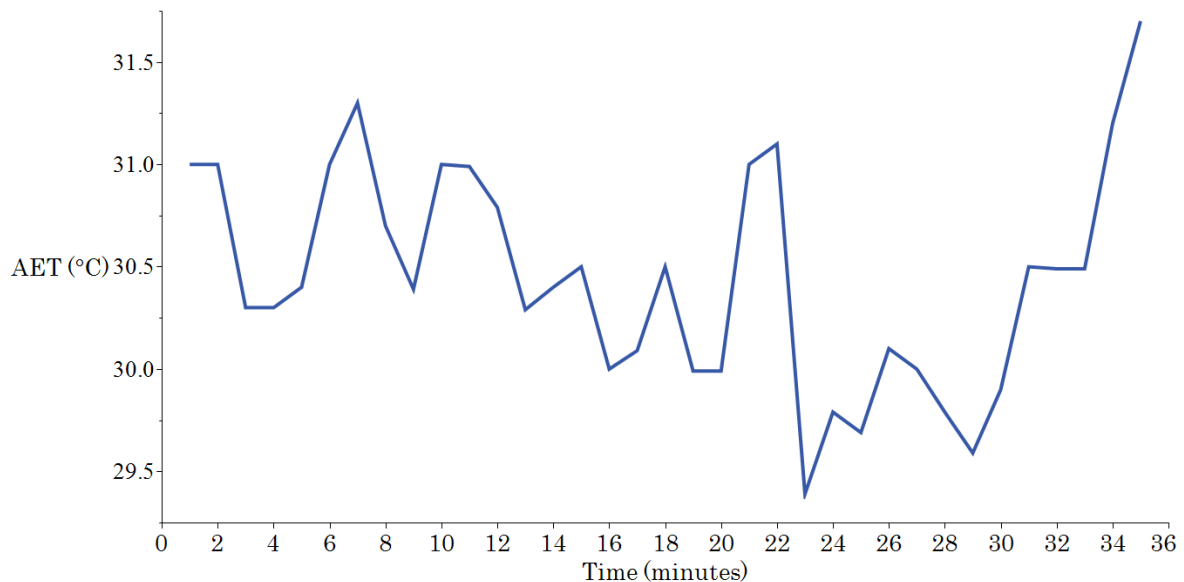
The air relative humidity, within and outside the trailer, was evaluated. The results of the assessment, as shown in Figure 7, indicated that the value of the outdoor air relative humidity ranged from 56% to 64% while that of indoor varied between 61% and 69%. The mean air relative humidity, inside and outside of the trailer, was  $65.3 \pm 2.27\%$

and  $59.9 \pm 2.39\%$  respectively. This indicates that the indoor relative humidity was about 5% higher than the outdoor air relative humidity compared to the air temperature which was reduced by  $2^{\circ}\text{C}$ . The t-test analysis conducted showed that there was a significant difference ( $p < 0.0001$ ) between the outdoor air relative humidity and the indoor air relative humidity. The increase in air relative humidity, as the air temperature increased, observed in this study, has previously been reported by [Aldridge et al. \(2019\)](#) and [Jongbo \(2020\)](#). The mean air relative humidity obtained for 35 minutes, that the trailer in the study, was tested is similar to that obtained by [Aldridge et al. \(2019\)](#) for 15 to 45 minutes. The hot period of the humid tropical climate is generally characterised by high air temperature and high relative humidity of about  $30^{\circ}\text{C}$  and 80% respectively ([Kiki et al., 2020](#)), which could be detrimental to animals' welfare and health. Therefore, for poultry birds, raised in the humid tropical climate, subjecting them to an average air relative humidity of less than 70%, as reported in this study, might help to alleviate the negative effect of the high temperature (above  $30^{\circ}\text{C}$ ) on the welfare and health of poultry birds during transportation.



**Figure 7.** Air relative humidity variation within and outside the trailer.

To understand the thermal zone (safe, alert and dangerous) within the trailer, the apparent equivalent temperature (AET), of the empty trailer was evaluated as shown in Figure 8. The result of the study shows that the AET varied from  $29.39^{\circ}\text{C}$  to  $31.70^{\circ}\text{C}$  with a mean of  $30.45 \pm 0.54^{\circ}\text{C}$ . According to [Mitchell \(2006\)](#), AET of  $40$  to  $45^{\circ}\text{C}$  could cause moderate thermal stress for broiler while severe and high mortality could occur as the AET reaches  $65^{\circ}\text{C}$  and above. In this study, it is clear that even at high outdoor temperatures during the summer period, the AET within the trailer could be maintained at a safe zone since the estimated AET was  $30.45 \pm 0.54^{\circ}\text{C}$  compared to the AET of  $80.5^{\circ}\text{C}$  obtained by [Aldridge et al. \(2019\)](#) which was severe and detrimental. The high AET obtained by [Aldridge et al. \(2019\)](#) could have occurred as a result of high relative humidity within the trailer. Exposure of broiler to high air temperature and high relative humidity is highly detrimental to the birds' welfare, health, production and economic growth of farmers ([Jongbo, 2018](#)).



**Figure 8.** The apparent equivalent temperature of the trailer.

## CONCLUSION

A motorbike powered trailer suitable for small scale farmers for the transportation of poultry birds was developed. The empty trailer was tested to understand the thermal conditions within it when operated on the typical Nigeria road which is almost like off-road tracks. The results of the tests have shown that the air temperature and air relative humidity of the trailer were 2°C and 5% respectively lesser than the outdoor thermal condition during the summer periods. The thermal zone within the trailer indicated that the trailer could provide a safe zone for poultry birds, most especially the broiler chickens, in the tropical humid climate which is generally above the thermal comfort zone (24°C) of broiler chickens. Since the trailer has not been evaluated when occupied with broiler chickens, it is difficult to appropriately quantify the AET, the effect of motorbike speed on both poultry birds and the environmental parameters, the behavioural and physiological responses of broiler chickens, the performance of broiler chickens and the effect of broiler chickens on the thermal conditions of the trailer. Therefore, further study would involve evaluating the effect of motorbike speed on the indoor thermal conditions, surface temperatures of broiler chickens, sensible heat transfer of broiler chickens, the live body weight of broiler chickens and the AET of broiler chickens when subjected to different road terrace and motorbike speeds.

## DECLARATION OF COMPETING INTEREST

The authors would like to declare that there is no conflict of interest.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

The authors would like to declare their contributions to the manuscript as follow:

**Ayoola Olawole Jongbo:** Investigation, methodology, conceptualization, formal analysis, data collection, writing- original draft, review and editing.



**Timothy Oluwatimilehin Adelaja:** Investigation, methodology, data collection writing-original draft.

## ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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## The Effect of Drying Processes on Model and Quantitative Color Quality Characteristics of Cauliflower (*Brassica oleracea* L.)

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### ABSTRACT

Drying is the process of removing the moisture in the product by evaporation up to a certain threshold value. The moisture content of the cauliflower vegetable is 90.76%, causing microbiological and enzymatic deterioration. For this reason, cauliflower should be preserved by drying in order to prolong its consumption life. In this study, drying process was carried out using different drying methods (oven, vacuum oven and convective dryer at 40, 50, 60 and 70°C drying temperatures and in the shade). The effects of different drying temperatures applied to the cauliflower vegetable on the color properties and drying kinetics were investigated. It was determined that the lowest drying time was 3.75 hours with the convective drying method at 70°C, and the greatest drying time was 275 hours with the shade drying method. In the mathematical modeling, Lewis, Wang Sing and Page equations were used. Among all drying models, the best drying data was found in the convective drying of the Page model equation at 70°C and the oven drying method at 40°C, R<sup>2</sup> with value of 0.9998 in Wang and Singh model equation. In terms of color values, it has been determined that the method that best preserves fresh cauliflower is drying in the shade.

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# Karnabaharın (*Brassica oleracea* L.) Model ve Kantitatif Renk Kalite Özelliklerine Kurutma İşlemlerinin Etkisi

## ÖZET

Kurutma üründeki nemin belirli bir eşik değerine kadar buharlaştırılarak uzaklaştırılması işlemidir. Karnabahar sebzesinin nem içeriğinin %90.6 olması sebebiyle mikrobiyolojik ve enzimatik bozulmalar meydana getirir. Bu nedenle karnabahar sebzesinin tüketim süresini uzatmak için kurutarak muhafaza edilmesi gerekmektedir. Bu çalışmada, farklı kurutma yöntemleri (etüv, vakumlu etüv ve konvektif kurutucuda 40, 50, 60 ve 70°C kurutma sıcaklığında ve gölgede) kullanılarak kurutma işlemi gerçekleştirilmiştir. Karnabahar sebzesine uygulanan farklı kurutma sıcaklıklarının renk ve kuruma kinetiğine etkisi araştırılmıştır. Elde edilen kuruma performans değerleri incelendiğinde; en kısa kuruma süresi konvektif kurutucuda 70°C kurutma yöntemi ile 3.75 saat, en uzun kuruma süresi ise gölgede kurutma yöntemi ile 275 saat olduğu tespit edilmiştir. Yapılan matematiksel modellemede, Lewis, Wang Sing ve Page eşitlikleri kullanılmıştır. Tüm kurutma modeller arasında kuruma verilerini en iyi Page model eşitliğinin konvektif kurutma 70°C ve Wang and Singh model eşitliğinin etüv kurutma 40°C yönteminde R<sup>2</sup> değeri 0.9998 olarak tespit edilmiştir. Karnabaharın renk özelliklerini en iyi koruyan yöntemin ise gölgede kurutma olduğu belirlenmiştir.

### ARAŞTIRMA MAKALESİ

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### Anahtar Kelimeler:

- Kurutma,
- Modelleme,
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- Karnabahar

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## GİRİŞ

Karnabahar, Brassicaceae familyasında yer alan Brassica cinsine ait bir sebzedir. Brassica sebzeleri genel olarak insanları akciğer, gastrointestinal sistem ve prostat kanserine karşı korur. DNA onarımını artıran ve östrojen antagonisti olarak işlev gören bir kimyasal olan indol-3-karbinol, kanser hücrelerinin büyümesini yavaşlatmaktadır (Rahman ve Gani, 2019). Ayrıca C ve A vitamini, folik asit, fosfor, kalsiyum ve yağ asitleri açısından da zengindir. Tüm dünyada önemli miktarda üretimi yapılan bu sebze doğrudan taze olarak tüketilebilirken turşu, salata ve çorba gibi malzemelerde çok geniş bir kullanım alanına sahiptir (Alibas ve Koksall, 2015; Sahin ve Doymaz, 2017).

Çin ve Hindistan karnabahar yetiştiren ülkelerin başında gelmektedir. Türkiye ise karnabahar yetiştiren ülkeler arasında 7. sıradadır. Karnabahar üretimi olarak dünyada ve Türkiye' de yıldan yıla giderek artış göstermektedir. Türkiye'de son beş yılda karnabahar üretimi yaklaşık 35 007 ton artış göstermiş ve 2021 yılında 234 717 ton karnabahar üretilmiştir (TÜİK, 2021). Bu kadar üretimi yapılan bir meyvenin hasat sonrası muhafazası da oldukça önemlidir. Yüksek nem içeriğine sahip olan karnabahar sebzesi mikrobiyal bozulmaya karşı hassastır. Bu nedenle uygun bir muhafaza yöntemi gerekmektedir.

Gıdaların muhafaza edilmesi için geçici veya kalıcı yöntemler vardır. Geçici yöntemler, gıdayı soğukta muhafaza ederek hava ile temasını kesmek ve nem alımını önlemek gibi özetlenebilir. Daha uzun veya kalıcı muhafaza için önerilen en iyi yöntem

ise kurutmazdır. Kurutma, sebze ve meyvelerin içerdikleri %80-95 oranlarındaki suyun %10-13 düşürülerek tüketim ömrünün uzatılmasını sağlama işlemidir. Bu sebeple kurutma, ürün içindeki nem seviyesini bozulmayı önleyecek seviyeye düşürdüğü için nem miktarındaki azalma olarak da tanımlanabilmektedir (Gülçimen, 2008).

Gıdaların kurutulmasındaki en önemli amaç, dayanma süresi kısa olan ürünlerin raf ömrünü artırmaktır. Nem içerikleri belirli bir orana kadar düşürülmüş olan gıdalar, normal atmosfer koşullarında, kimyasal, mikrobiyolojik ve enzimatik bozulmalara karşı daha dayanıklıdır. Kurutulan ürünlerin hacimleri de önemli oranda azaldığından taşıma ve depolamada da kolaylık sağlanır (Şahin, 2014).

Kurutma işlemi esnasında kütle ve ısı transferi sebebiyle kuruma sonrası ürünlerin aroma, besin değeri, yapı ve renk gibi kalite özelliklerini etkileyen birçok kimyasal, biyokimyasal ve fiziksel değişimler oluşmaktadır (Di Scala ve Crapiste, 2008; Vega-Galvez ve ark., 2009; Yıldız Turgut ve Topuz, 2020). Birçok endüstriyel kurutma yöntemi daha yüksek kaliteli ürün elde etmek için geliştirilmiştir. Geliştirilen metotlar arasında etüvde, mikrodalga, vakum, dondurarak ve sıcak hava kurutma yöntemleri en yaygın kullanılan yöntemlerdir (Krokida ve Maroulis, 2000; Marques ve ark., 2006; Sagar ve Kumar, 2010; Yıldız-Turgut ve Topuz, 2020).

Bu çalışmada etüvde, vakumlu etüvde, laboratuvar tipi konvektif kurutucuda ve gölgede kurutma yöntemleri kullanarak karnabahar sebzesinin kuruma kinetiği ve renk kriteri açısından en uygun kurutma yönteminin belirlenmesi amaçlanmıştır.

## MATERYAL ve YÖNTEM

### Materyal

Çalışmada kullanılan materyal karnabahar sebzesidir. Materyaller Tokat ilindeki yerel bir marketten satın alınmıştır. Nem tayini ve kurutma işlemleri için Tokat Gaziosmanpaşa Üniversitesi Biyosistem Mühendisliği Kurutma Laboratuvarına getirilmiştir. İşlemler sonlanana kadar ürünler  $+4\pm 0.5^{\circ}\text{C}$  sıcaklıkta muhafaza edilmiştir. Çalışmada kullanılan taze karnabahar Şekil 1'de verilmiştir.



Şekil 1. Kurutma materyali taze karnabahar.

*Figure 1. Drying material fresh cauliflower.*

### Nem tayini

Kurutma işlemi öncesinde taze karnabahar sebzesi nem içeriğinin belirlenmesi için ortalama 50 g yaş meyve kullanılarak  $70^{\circ}\text{C}$  sıcaklıkta ağırlık değişimi sabitlenene kadar kurutulmuştur (Yağcıoğlu, 1999). İlk ve son ağırlıkları kaydedilen ürünün yaş baza nem içeriği 1 ve 2 numaralı eşitliklere göre hesaplanmıştır.

$$N_y = \frac{W_i - W_s}{W_i} \times 100 \quad (1)$$

$$N_k = \frac{W_i - W_s}{W_s} \times 100 \quad (2)$$

Burada;  $N_y$ : Yaş baza göre nem (%),  $N_k$ : Kuru baza göre nem (%),  $W_i$ : Yaş örneğin ağırlığı  $W_s$ : Kuru örneğin ağırlığı (g).

### Kurutma yöntemi

Karnabaharlar  $\pm 4^\circ\text{C}$  sıcaklıkta muhafaza edildikten sonra denemede kullanılmaya başlanmadan önce ortam sıcaklığına gelene kadar bir süre dış ortamda bekletilmiştir. Karnabaharlar küçük ağaç şeklinde koparılmıştır. Daha sonra etüv, vakumlu etüv ve konvektif kurutucuda 40, 50, 60 ve  $70^\circ\text{C}$  sıcaklıklarda ve gölgede kurutma yapılmıştır. Kurutma işlemleri üçer paralel halinde yapılmıştır. Kurutucu içerisindeki ürünler ilk önce 3 kere yarım saat, 3 kere 1 saat, 3 kere 2 saat, 4 kere 3 saat, 4 kere 4 saat ve sonrasında ürünler son nem değerine ulaşıncaya kadar 5 saatte bir ölçüm alınmıştır. Ölçümler %1 g hassasiyete sahip bir terazi ile tartılarak ürün nemi yaş baza göre %10-13 seviyesine kadar kurutulmuştur.

### Renk ölçümü

Renk, gıda ve tarımsal ürünlerinin en önemli kalite değerleri kriterlerden biridir. Ürünlerde meydana gelen olumsuz renk değişimleri, ürünlerin market değerini önemli seviyede olumsuz etkilemektedir ([Krokida ve ark., 2000](#); [Adiletta ve ark., 2014](#); [Polatci ve ark., 2020](#)).

Taze ve kurumuş karnabahar sebzelerinin renk ölçümleri L, a ve b olarak ve hue değerleri olarak, Minolta marka CR300 model renk ölçer ile yapılmıştır. Bu değerler; "L" meyvenin parlaklık değerini ifade etmekte olup 0-100 arasında bir değer almaktadır. "a" kırmızı-yeşil, "b" ise sarı-mavi renkleri ifade etmektedir. Bu değerler + işaretli olursa "a" kırmızıyı "b" sarı renkte olduğunu – işaretli değerler alırsa "a" yeşil ve "b" mavi renkte olduğunu ifade etmektedir ([McGuire, 1992](#)). Ölçülen L, a ve b değerleri ürün hakkında tek başına bir anlam ifade etmezken bu değerler kullanılarak ticari renk değeri açısından önemli olan hesaplanarak belirlenen kroma, kırmızılık indeksi, hue açısı, toplam renk değişimi ve kahverengileşme indeks değerleri belirlenmiştir. Bu değerler bazı renk ölçüm cihazlarında doğrudan ölçülebilmektedir, ancak çalışmada kullanılan renk ölçüm cihazı bu özellikte değildir.

Hesaplanan bu değerler;

**Kroma değeri**, rengin doygunluğunu göstermektedir. Canlı renklere yüksek değerler hesaplanırken solgun renklere düşük değerler hesaplanmaktadır. Kroma değeri 3 numaralı eşitlik kullanılarak hesaplanmıştır.

**Hue değeri**, ölçülen kırmızılık ve sarılık değerleri kullanılarak hesaplanan bir renk radyantını ifade etmektedir. Hue değeri 4 numaralı eşitlik kullanılarak hesaplanmıştır.

$$C = (a^2 + b^2)^{1/2} \quad (3)$$

$$h^\circ = \tan^{-1} \left( \frac{b^*}{a^*} \right) \quad (4)$$

**Toplam renk değışim değeri ( $\Delta E$ ):** Taze karnabaharın renk değerlerini kurutma işlemleriyle ne kadar değıştiğini belirlemektir. Toplam renk değışim değeri 8 numaralı eşitlik kullanılarak hesaplanmıştır (Celen ve ark., 2015).

$$\Delta L = L_{taze} - L^2 \quad (5)$$

$$\Delta a = a_{taze} - a \quad (6)$$

$$\Delta b = b_{taze} - b \quad (7)$$

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2} \quad (8)$$

**Kahverengileşme indeks değeri (BI):** Karnabahar kurutma işlemi sonunda gerçekleşen kahverengilik değeri belirtmektedir. Kahverengileşme indeksi 9 ve 10 numaralı eşitlik kullanılarak hesaplanmıştır (Plou ve ark., 1999).

$$X = \frac{a+(1,75 \times L)}{[(5,645 \times L)+(a-(3,012 \times b))]} \quad (9)$$

$$BI = \frac{[100(x-0,31)]}{0,17} \quad (10)$$

### Meyve ağırlık ölçümü

Belirli aralıklarda bitki materyalinin ağırlıkları ölçülmüştür. Ağırlıklar 0.01g hassasiyetindeki ANDGF300 model terazi ile ölçülmüştür.

### Kurutma verilerinin matematiksel modellemesi

Tarımsal ürünler kurutulurken nem değışimini modellemek, gerçek değerler ile tahmini değerler arasındaki ilişkiyi ifade etmek için bazı model eşitliklerinden yararlanılır. Bu çalışmada oluşturulan kurutma eğrilerinin katsayıları ile eğrilerin "R<sup>2</sup>" ve "p" değeri SigmaPlot 10.0 programı kullanılarak belirlenmiştir. Kurutma için en uygun üç model eşitliği seçilmiş ve aralarında karşılaştırma yapılmıştır. Bu modelleme eşitlikleri Lewis, Page ve Wang Singh 'dır. Çizelge 1'de bu eşitlikler verilmiştir.

### Çizelge 1. Kurutma model eşitlikleri.

*Table 1. Drying model equations.*

No	Model ismi	Eşitlik	İlgili Kaynak
1	Lewis	$f=\exp(-k.t)$	<a href="#">Lewis (1921)</a>
2	Page	$f=\exp(-kt^h)$	<a href="#">Page (1949)</a>
3	Wang Singh	$f=1+k.t+h.t^2$	<a href="#">Wang ve Singh (1978)</a>

f: Modelin fonksiyonu, k-h: Modele ait sabit katsayılar, t: Süre

### İstatistiksel analiz

Tüm kurutulmuş örneklere ait renk değerlerinin istatistiksel açıdan tazelerin özelliklerini ne kadar muhafaza edebildiğini belirleyebilmek için veriler SPSS 26.0 programında işlenerek çoklu karşılaştıma testi (DUNCAN) yapılmıştır.

## BULGULAR ve TARTIŞMA

Deneme kapsamında elde edilen sonuçlar aşağıda verilmiştir.

### Kuruma verileri

Karnabahar sebzelerinin nem içeriği %90.76 olarak belirlenmiştir. [Baloch ve ark. \(2015\)](#), Pakistan da yetiştirilen karnabahara yapmış oldukları kurutma sonrası mineral içeriklerini inceledikleri çalışmada karnabaharın nem içeriğini %90.62 bulmuşlardır. Araştırmacılar, mevcut çalışmadaki nem içeriğine yakın bir değer tespit etmişlerdir. Bu değer ürünlerde hasat sonrasında bozulmalara sebep olacak ve uzun süre tüketim için uygun olmayacaktır. Bu nedenle ürün içerisindeki nem değerini %10-13'e kadar düşürerek depolanabilir toplam su seviyesine kadar kurutulması gerekmektedir. Denemeler sonucunda ürünlerin kuruma süreleri ve ortalama son nem değerleri Çizelge 2'de verilmiştir.

**Çizelge 2.** Karnabahar sebzelerine ait kuruma performans değerleri.

**Table 2.** *Drying performance values of cauliflower vegetable.*

Kurutma Yöntemleri	Ortalama Son Nem Değerleri (%yb)	Kurutma Süreleri (saat)
Etüv	40°C	83.5
	50°C	30.5
	60°C	26.5
	70°C	16.5
Vakumlu Etüv	40°C	134.5
	50°C	76.5
	60°C	38.5
	70°C	28.5
Konvektif Kurutucu	40°C	43.5
	50°C	10.5
	60°C	6.5
	70°C	3.75
Gölgede Kur.	-	291

Çizelge 2 incelendiğinde kuruma süresi kuruma koşullarına göre değişiklik göstermiştir. Yöntemler arasında sıcaklık kuruma süresini etkilemiştir. Kuruma süresi sıcaklık ile doğru orantılıdır. Yani sıcaklık arttıkça kuruma süresi azalmıştır. Buna göre en kısa kuruma süresi konvektif kurutma 70°C yöntemi ile 3.75 saat, en uzun kuruma süresi ise gölgede kurutma yöntemi ile 291 saat olduğu tespit edilmiştir. [Vargas ve ark. \(2022\)](#), brokoli, lahanaya ve ıspanağın fitokimyasal içeriği ve fiziksel özelliklerinin değerlendirilmesi için farklı kurutma yöntemleri kullandıkları çalışmada brokoli ve kara lahanayı 14 saat ağırlık değişimi önemsiz hale gelene kadar kurutmuşlardır. Çalışmada aynı familyadan olan karnabahar sebzelerini benzer yöntemde 16.5 saatte kurutulmuştur. Kuruma süresindeki farkın sebebi lahanaya ve brokolinin nem içeriğinin karnabahardan daha düşük olmasından kaynaklı olduğu düşünülmektedir.

### Renk değerleri

Karnabahar sebzelerinin taze ve kurutulmuş örneklerine ait ölçülen ve hesaplanan renk değerleri Çizelge 3 ve Çizelge 4'te verilmiştir.

Çizelge 3 incelendiğinde L, a ve b değerleri %5 önem seviyesinde tazeye kıyasla istatistiksel açıdan ( $P < 0.05$ ) farklı bulunmuştur. Ancak L değerlerine bakıldığında etüv 50°C ile vakumlu etüv 50°C-60°C-70°C, etüv 70°C ile konvektif kurutucu 50°C-70°C ve



vakumlu etüv 40°C ile konvektif kurutucu 60°C istatistiki açıdan ( $P<0.05$ ) benzer olduğu görülmüştür. Çalışmada, a değerleri incelendiğinde etüv 40°C-50°C ile 70°C ve konvektif kurutucu 40°C ile 60°C yöntemleri arasında istatistiki açıdan ( $P<0.05$ ) bir farkın olmadığı tespit edilmiştir. b değerinde ise vakumlu etüv 40°C-50°C ile konvektif kurutucu 60°C istatistiki açıdan ( $P<0.05$ ) önemli bulunmuştur.

Çizelge 4 incelendiğinde taze ürünün kroma (renk doygunluğu) değeri 13.70 olarak bulunmuştur. En düşük kroma değeri gölgede kurutma yöntemi ile 17.45 bulunurken en yüksek değer ise etüv 60°C ile 27.39 olarak belirlenmiştir.

Taze ürünlerin hue açısı 80.63 olarak belirlenmiştir. Yapılan denemelerde en düşük hue açısı vakumlu etüv 70°C ile 64.38 olarak tespit edilirken en yüksek hue açısı ise konvektif kurutucu 60°C yöntemi ile 78.80 olarak bulunmuştur.

BI (kahverengileşme indeksi) yapılan denemelerde en düşük değeri konvektif kurutucu 70°C yöntemi ile 42.31 bulunurken en yüksek değer ise vakumlu etüv 70°C yöntemi ile 76.23 olarak bulunmuştur.

Toplam renk farklılık değeri en fazla etüv 40°C kurutma yönteminde 44.55 olarak belirlenirken en az ise vakumlu etüv 70°C kurutma yönteminde 32.69 olarak tespit edilmiştir.

### Çizelge 3. Karnabahar sebzesine ait ölçülen renk değerleri.

Table 3. Measured color values of cauliflower vegetable.

		L	a	b
<b>Taze Karnabahar</b>		2.74±79.55 <sup>a</sup>	1.44±2.23 <sup>i</sup>	4.29±13.52 <sup>h</sup>
<b>Etüv</b>	40°C	5.72±62.11 <sup>b</sup>	0.67±6.55 <sup>e</sup>	3.37±21.65 <sup>cde</sup>
	50°C	6.52±55.08 <sup>de</sup>	0.90±6.86 <sup>e</sup>	5.43±22.96 <sup>abc</sup>
	60°C	2.06±57.12 <sup>cde</sup>	1.17±8.17 <sup>cd</sup>	3.47±26.14 <sup>a</sup>
	70°C	3.71±58.36 <sup>bcd</sup>	0.81±7.09 <sup>e</sup>	3.33±18.54 <sup>efg</sup>
<b>Vakumlu etüv</b>	40°C	4.20±60.85 <sup>bc</sup>	1.09±9.04 <sup>bc</sup>	1.91±25.48 <sup>ab</sup>
	50°C	5.99±54.77 <sup>de</sup>	1.32±9.34 <sup>b</sup>	3.85±25.27 <sup>ab</sup>
	60°C	4.20±53.89 <sup>de</sup>	1.76±8.00 <sup>c</sup>	4.72±20.71 <sup>cdef</sup>
	70°C	7.31±48.10 <sup>de</sup>	1.47±10.59 <sup>a</sup>	5.82±22.09 <sup>bcd</sup>
<b>Konvektif kurutucu</b>	40°C	5.15±49.46 <sup>f</sup>	1.24±4.82 <sup>fg</sup>	5.00±19.20 <sup>defg</sup>
	50°C	7.55±58.32 <sup>bcd</sup>	1.23±4.23 <sup>gh</sup>	5.93±18.45 <sup>efg</sup>
	60°C	6.83±61.52 <sup>bc</sup>	0.96±5.03 <sup>fg</sup>	3.98±25.41 <sup>ab</sup>
	70°C	5.54±58.37 <sup>bcd</sup>	0.78±5.35 <sup>f</sup>	2.84±17.72 <sup>fg</sup>
<b>Gölgede Kur.</b>	-	6.91±53.66 <sup>e</sup>	1.73±3.53 <sup>h</sup>	3.95±17.09 <sup>g</sup>

$P<0.05$  önem seviyesinde

**Çizelge 4.** Karnabahar sebzesine ait hesaplanan renk değerleri.

**Table 4.** Calculated color values of cauliflower vegetable.

		<b>C</b>	<b>Hue</b>	<b><math>\Delta E</math></b>	<b>BI</b>
<b>Taze Karnabahar</b>		13.70	80.63	67.06	20.30
<b>Etüv</b>	40°C	22.62	73.16	44.55	49.86
	50°C	23.96	73.37	38.09	61.92
	60°C	27.39	72.64	39.08	70.18
	70°C	19.85	69.06	42.46	46.55
<b>Vakum Etüv</b>	40°C	27.03	70.46	42.07	64.05
	50°C	26.94	69.72	37.38	72.90
	60°C	22.20	68.87	37.60	58.53
	70°C	24.50	64.38	32.69	76.23
<b>Konvektif Kurutucu</b>	40°C	19.79	75.91	34.65	55.27
	50°C	18.93	77.09	42.94	42.66
	60°C	25.90	78.80	42.97	58.02
	70°C	18.51	73.19	43.16	42.31
<b>Gölgede Kur.</b>		17.45	78.34	39.50	42.43

#### Modelleme verileri

Matematiksel modelleme sonucu ile elde edilen “R<sup>2</sup>” ve “p” değerleri Çizelge 5’de verilmiştir.

Çizelge 5. Modelleme eşitliklerine ait hesaplanan değerler.

Table 5. Calculated values of modeling equations.

Model Eşitlikleri	Kurutma yöntemi	R <sup>2</sup>	p	k	h		
Page	Etüv	40°C	0.9965	0.0001	0.0039	1.4572	
		50°C	0.9955	0.0001	0.0104	1.5970	
		60°C	0.9942	0.0001	0.0252	1.5008	
		70°C	0.9884	0.0001	0.0368	1.4965	
	Vakumlu Etüv	40°C	0.9981	0.0001	0.0051	1.3006	
		50°C	0.9980	0.0001	0.0068	1.4049	
		60°C	0.9986	0.0001	0.0241	1.3250	
		70°C	0.9981	0.0001	0.0193	1.5265	
	Konvektif Kurutucu	40°C	0.9995	0.0001	0.2099	0.9219	
		50°C	0.9997	0.0001	0.3569	1.0314	
		60°C	0.9993	0.0001	0.6335	1.0587	
		70°C	0.9998	0.0001	0.9279	1.1026	
	Gölgede Kur.	-	0.9976	0.0001	0.0040	1.1512	
	Wang Singh	Etüv	40°C	0.9998	0.0001	-0.0147	3.2086
			50°C	0.9992	0.0001	-0.0340	2.9244
			60°C	0.9942	0.0001	-0.0615	0.0008
70°C			0.9948	0.0001	-0.0697	0.0005	
Vakumlu Etüv		40°C	0.9996	0.0001	-0.0128	3.9663	
		50°C	0.9992	0.0001	-0.0207	9.7668	
		60°C	0.9993	0.0001	-0.0444	0.0005	
		70°C	0.9974	0.0001	-0.0523	0.0006	
Konvektif Kurutucu		40°C	0.8991	0.0001	-0.0833	0.0015	
		50°C	0.9826	0.0001	-0.2568	0.0160	
		60°C	0.9706	0.0001	-0.4289	0.0442	
		70°C	0.9950	0.0001	-0.6718	0.1110	
Gölgede Kur.		-	0.9991	0.0001	-0.0062	9.9414	
Lewis		Etüv	40°C	0.9803	0.0001	0.0215	-
			50°C	0.9701	0.0001	0.0535	-
			60°C	0.9758	0.0001	0.0841	-
	70°C		0.9681	0.0001	0.1040	-	
	Vakumlu Etüv	40°C	0.9899	0.0001	0.0170	-	
		50°C	0.9853	0.0001	0.0284	-	
		60°C	0.9890	0.0001	0.0591	-	
		70°C	0.9781	0.0001	0.0736	-	
	Konvektif Kurutucu	40°C	0.9989	0.0001	0.1848	-	
		50°C	0.9995	0.0001	0.3681	-	
		60°C	0.9987	0.0001	0.6518	-	
		70°C	0.9990	0.0001	0.9437	-	
	Gölgede Kur.	-	0.9949	0.0001	0.0081	-	

Çizelge 5 incelendiğinde R<sup>2</sup> değeri en yüksek Page model eşitliğinin konvektif kurutma 70°C yönteminde ve Wang and Singh model eşitliğinin etüv 40°C 0.9998 bulunurken, R<sup>2</sup> değeri en düşük ise Wang Sing model eşitliğinin konvektif kurutma 40°C yönteminde 0.8991 olarak tespit edilmiştir. R<sup>2</sup> değerinin yüksek olması deneysel

olarak hesaplanan verilerle modellerin tahmin ettiği verilerin birbirine yakınlığının yüksek olduğunu, düşük olması ise modelin tahmin ettiği verilerin birbirleri arasındaki ilişkinin zayıf olduğunu göstermektedir. [Sahin \(2014\)](#), karnabahar ve brokolinin kurutma karakteristiklerine ön işlem süresinin ve sıcaklığının etkisi çalışmasında Henderson ve Pabis, Page, Logaritmik, Midilli vd., Wang ve Singh Newton ve Two-term model eşitliklerini kullanmış ve en iyi tahmin eden kuruma modeli Midilli vd. olmuştur. Bu farklılığın nedenin ön işleme kuruma sürelerindeki azalmanın etkisi olacağı düşünülmektedir.

## SONUÇ

Üretimi dünyada ve Türkiye de oldukça fazla olan karnabaharın muhafaza koşullarının iyileştirilmesi oldukça önemlidir. İçerdiği yüksek nem sebebiyle çabuk bozulan karnabahar sebzesine uygun bir kurutma yöntemi belirlenmelidir. Bu çalışmada, karnabahar sebzesinin farklı kurutma yöntemlerinde renk kriteri ve kuruma kinetiğince en uygun yöntem belirlenmiştir. Etüvde 40, 50, 60 ve 70°C'de, vakumlu etüv 40, 50, 60 ve 70°C'de, konvektif kurutucu 40, 50, 60 ve 70°C'de ve gölgede kurutma yöntemleri kullanılmıştır.

Karnabahar sebzesine ait kuruma performans değerleri incelendiğinde; en kısa kuruma süresi konvektif kurutucu 70 °C kurutma yöntemi ile 3.75 saat olarak tespit edilmiştir. Belirlenen R<sup>2</sup> değerleri arasında en yüksek eşitlik Page model eşitliğinin konvektif kurutma 70°C yönteminde ve Wang and Singh model eşitliğinin etüv 40°C 0.9998 olarak tespit edilmiştir. Renk değerleri açısından da karnabaharın özelliklerini en iyi koruyan yöntemin gölgede kurutma yöntemi olduğu belirlenmiştir.

## ÇIKAR ÇATIŞMASI

Yazarlar herhangi bir çıkar çatışması olmadığını beyan ederler.

## YAZAR KATKISI

**Burcu Aksüt:** Çalışmanın yazım aşamasında, kurutma denemelerinin yapılmasında, kuruma ve modelleme verilerinin işlenmesi aşamasında katkı sağlamıştır.

**Hakan Polatçı:** Çalışma materyalinin temininde, planlama, kontrol ve analiz aşamasında katkı sağlamıştır.

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Bu makale Etik Kurul Kararı gerektirmemektedir.




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## Effect of Fermentation on Drying Characteristics of Three Varieties of Trifoliate Yam

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### ABSTRACT

This study determined the effect of fermentation on drying characteristics of trifoliate yam varieties (white, yellow and deep-yellow trifoliate yam). Thin layer drying method was adopted using oven dry method at constant drying temperature of 70°C with air velocity of 2.35 m s<sup>-1</sup> and relative humidity 35%. The unfermented samples showed lower values of drying constant than the fermented samples. The drying constant of unfermented samples of *Trifoliate Yam* A, B and C (A= Deep-Yellow, B= White, C= Yellow) were -0.729, 1.3972 and 0.2787, respectively. While, the drying constant of fermented samples of *Trifoliate Yam* D, E and F (D= Deep-Yellow, E= White, F= Yellow) were -0.776, -0.763 and 1.5815, respectively. The drying rate of the samples solely dependent on the magnitude of drying constants sequel to this, fermented samples with larger magnitude of drying constant will dry faster than the unfermented samples with lower drying constant. Best fit equations and relationship between moisture content and drying time were developed with correlation coefficient (R<sup>2</sup>) higher than 0.94. The sample A, B, C, D, E and F reached a constant moisture ratio of 0.021, 0.015, 0.021, 0.015, 0.014 and 0.016 at drying time of 540, 600, 600, 480, 540 and 540 minutes, respectively. Sample B and C had highest drying time followed by sample A, E and F while sample D had the lowest value of drying time. The fermentation had significant effect on the drying characteristics of trifoliate yam slices and drying of trifoliate yam samples occurred solely in the falling rate period which showed that internal moisture diffusion phenomenon is dominant and controlled the drying process.

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## INTRODUCTION

*Dioscorea dumetorum* (Trifoliolate Yam) has proven to be the most nutritious among the eight species of yam basically grown and consumed in the West and Central Africa (Sefa-Dedeh and Afoaka, 2002). According to Lape and Treche (1994), Trifoliolate Yam demonstrated to be rich in protein, primarily balanced in essential amino acids with easily digested starch. According to Christian *et al.* (2018) and Lyonga and Ayu-Takem (1982), Trifoliolate Yam species is a very high yielding yam species which requires no staking like other yam species, consequently saving much labour during pre-harvest and post-harvest operations. Compared to other yam species, it has other common names such as African bitter yam, Wild Yellow and White-Colored trifoliolate (three-leaved) yam and cluster yam (Ugwuanyi Nnadi *et al.*, 2020). In Ibo tribe mostly South-East Nigeria, bitter Trifoliolate Yam is usually referred to as *ji una or jona* and food for the adult (Anthony *et al.*, 2016). It cannot be processed into fufu like other yam species due to its soft texture which favors old people with poor teeth structure as a result of old age, but they can be used as vegetable. It serves as raw material for formulations of new bio products upon it was found to be the cheapest and high yielding crop. Breweries and other similar companies explore the benefits of this underutilized yam species in preparation of beer (Anthony *et al.*, 2016). This underutilized yam species, when properly processed like other yam species can be used in making of yam flakes, instant flour for the bakery sector or starch in diverse pharmaceutical applications (Ukpabi and Ndimele, 2014). It has lots of medicine relevance as it has been proven to be a direct remedy for treatments of diabetes, malaria and other numerous ailments mostly in the South-East Nigeria, yet it remains an underutilized tropical tuber and probably it may be driven into extinction in no distant (Clifford *et al.*, 2013). In the modern world, handling and processing of agro based products are the most important aspect in food and nutritional security. Due to urbanization most food products are required to be produce in different forms in order to cater for daily nutritional requirements of a common man and also cater for the alarming pollution in the world (Nkhata *et al.*, 2018 and Omemu, 2011). Mostly, processing of agricultural products is done to improve consumer acceptability, palatability and transportability. They can also have adverse effect on the nutrient profile of food products by retaining its nutritional value Chaves-Lopez *et al.* (2014) and Omemu, (2011). There are some techniques implored in processing of Trifoliolate Yam of which most of the methods are localized to certain areas while others are practiced globally. This research intends to adopt the most commonly used processing techniques (fermentation). Fermentations of food products are broadly used in processing of food products for production of numerous varieties of dishes in Africa. It is a prerequisite for development of acceptable texture, flavor food products. According to Buta and Emire (2015) and Chaves-Lopez *et al.* (2014) it improves the nutritional quality, digestibility and safety of foods. Fermentation can be used to minimize the antinutritional content in a food products and improve nutrient availability (Hotz and Gibson, 2007; Omemu, 2011; Nkhata *et al.*, 2018).

Drying characteristics is the commonest food process employed in improving food stability and security, as far as it noticeably declines the negative effect of water in the material, deterioration, microbiological activity, physical and chemical changes during its processing and storage ([Mujumdar and Law 2010](#)). It also, causes colour change, weight reduction, and enhances aesthetic and sensory effects of food ([Brennan, 2006](#)). Therefore, the basic goal is to limit moisture content to levels that halt or slow down the growth of spoilage microorganisms and incident of chemical reactions in order to extend the shelf-life of food ([Oduro \*et al.\*, 2007](#)). According to [Maskan \(2001\)](#) the high quality fast-dried foods have become necessary in the recent times which aggravated a renewed interest in drying operations. Furthermore, there is a high demand for convenient foods more especially ready to eat and instant products, which are desired to contain the less contents of additives and preservatives ([Mujumdar and Law 2010](#)). Therefore, the interest of the research was to determine the effect of fermentation on the drying characteristics of three varieties of Trifoliate Yam.

## MATERIALS and METHODS

### Source of sample

The Deep-Yellow, Yellow and White Trifoliate Yam (*Dioscorea dumentorum*) varieties used for this research work were harvested from Enugu State agricultural development programme, Enugu State, Nigeria at physiological maturity. They were immediately transported in a heap and stored in temperature and relative humidity rate of  $28^{\circ}\text{C}\pm 3^{\circ}\text{C}$  and  $82\pm 5\%$  respectively.

### Preparation of the Sample

The sourced Trifoliate Yam tubers from each variety (White Trifoliate Yam, Deep-Yellow Trifoliate Yam and Yellow Trifoliate Yam) were divided into two equal batches. The first batch which represent the raw (unfermented sample) were peeled, washed and then sliced into a rectangular shape of about  $30\times 20\times 10$  mm thickness using stainless kitchen knife and vernier caliper. The second batch were peeled, washed and soaked in a distilled water, the traditional fermentation method as reported by [Oladele and Oshidi \(2008\)](#) was used to ferment the sample. Both the fermented and unfermented processed samples were classified as A= Unfermented Deep-Yellow Trifoliate Yam, B= Unfermented White Trifoliate Yam, C= Unfermented Yellow Trifoliate Yam, D= Fermented Deep-Yellow Trifoliate Yam, E= Fermented White Trifoliate Yam and F= Fermented Yellow Trifoliate Yam. The micro-organisms involved in the traditional fermentation were naturel inoculants from the air. The samples were collected after 72 hours.





**Figure 1.** Pictorial showing procedures in preparing Trifoliate Yam flour sample.

#### **Determination of Drying Characteristics of Trifoliate Yam**

The samples were harvested and the research was carried out between November and December 2021 at Bioprocess laboratory, in Enugu State University of Science and Technology, Enugu State, Nigeria. The two prepared batches of Trifoliate Yam were introduced into the hot air oven dryer. The oven dryer was allowed to operate for 60 minutes without the sample in order to obtain the experimental design condition. The Samples were weighed before they were loaded in a drier and removed at interval of 60 minutes to record moisture loss until three consecutive constant weight and moisture content were obtain indicating equilibrium condition (John *et al.*, 2020). Thin layer drying method was adopted using oven dry method at constant drying temperature of 70°C (the temperature that retains the nutritive value of a biomaterial under drying process) with air velocity of 2.35 m s<sup>-1</sup> and relative humidity 35%.

#### **Moisture content at any time of drying**

The moisture content of the sample at any given time and condition were determined using the Equation (1) reported by (Okeke *et al.*, 2020)

$$M_{ct} = \frac{W_t - W_d}{W_t} \quad (1)$$

$M_{ct}$  = Moisture content (%wt) at time t;

$W_t$  = Initial weight of the sample at any time

$W_d$  = Weight of the dried sample

**Drying rate at any time of drying**

The drying rate of the sample were determined using the Equation (1) reported by (Dai *et al.*, 2017) with little modification.

$$D_R = \frac{M_{t_1} - M_{t_2}}{t_2 - t_1} \quad (2)$$

$D_R$  = Drying rate (%/mins)

$M_{t_1}$  = Moisture content at  $t_1$ , (g g<sup>-1</sup>)

$M_{t_2}$  = Moisture content at  $t_2$  (g g<sup>-1</sup>)

$t_2$  = Time of drying at  $M_{t_2}$

$t_1$  = Time of drying at  $M_{t_1}$

**Moisture ratio of Trifoliolate Yam**

Moisture ration of the samples were determined using the Equation (3) reported by (Dai *et al.*, 2017) with little modification.

$$M_R = \frac{M_{t_1}}{M_0} \quad (3)$$

$M_R$  = Moisture ratio

$M_{t_1}$  = Moisture content (dry basis) at any time

$M_0$  = Initial moisture content (dry basis) of the sample.

**Multiple Regression analysis**

Regression analysis is a statistical technique that can test hypothesis that a variable is dependent upon one or more other variables. Version 2 was used to analyze data generated from the drying characteristics of Trifoliolate Yam varieties.

**RESULTS AND DISCUSSION****Drying curve and drying rate moisture relationship.**

Drying curves may be represented graphically as averaged moisture content versus time, drying rate versus time, moisture ratio versus time or drying rate versus averaged moisture content (Coumans, 2000; Saeed *et al.*, 2008).

**Table 1.** Drying characteristics of fermented and unfermented Trifoliolate Yam varieties dried at 700°C using oven drying method.

Drying time (mins)	Sample A		Sample B		Sample C		Sample D		Sample E		Sample F	
	Drying rate (mins)	MC (%)	Drying rate (mins)	MC (%)	Drying rate (mins)	MC (%)	Drying rate (mins)	MC (%)	Drying rate (mins)	MC (%)	Drying rate (mins)	MC (%)
0	-	49.80	-	62.61	-	49.42	-	65.05	-	76.42	-	63.74
60	0.46	22.0	0.62	25.30	0.25	34.51	0.67	24.82	0.75	31.56	0.63	26.14
120	0.28	15.91	0.34	19.70	0.21	24.53	0.41	16.27	0.48	19.24	0.42	13.04
180	0.22	10.30	0.30	13.20	0.19	15.50	0.32	8.12	0.38	7.67	0.32	5.6
240	0.18	6.38	0.22	8.94	0.16	9.96	0.25	4.82	0.30	3.56	0.26	2.05
300	0.16	2.56	0.19	4.45	0.14	6.26	0.21	1.26	0.25	1.31	0.21	1.06
360	0.14	1.18	0.17	2.00	0.13	2.52	0.18	1.04	0.21	1.15	0.17	1.05
420	0.11	1.07	0.15	1.56	0.11	1.29	0.15	1.04	0.18	1.13	0.15	1.05
480	0.10	1.07	0.13	1.25	0.10	1.05	0.13	1.04	0.16	1.13	0.13	1.05
540	0.09	1.07	0.11	1.14	0.09	1.04	0.00	0.00	0.14	1.13	0.00	0.00
600	0.00	0.00	0.10	1.14	0.08	1.04	0.00	0.00	0.00	0.00	0.00	0.00

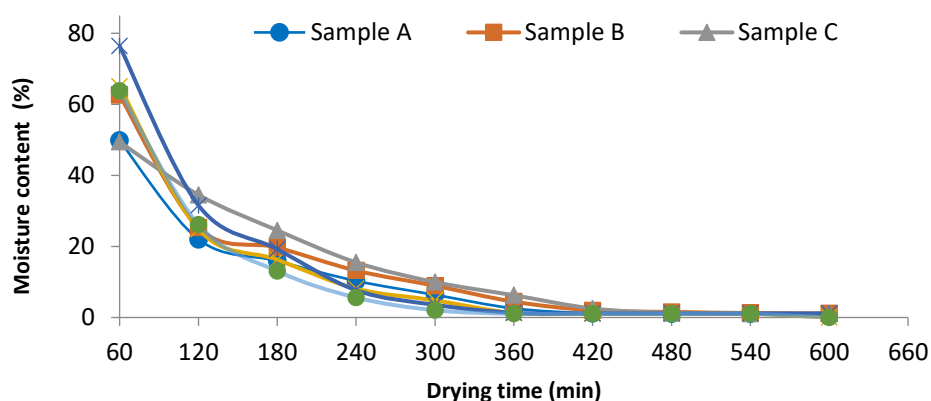
Note: A= Unfermented Deep-Yellow Trifoliolate Yam, B=Unfermented White Trifoliolate Yam, C=Unfermented Yellow Trifoliolate Yam, D= Fermented Deep-Yellow Trifoliolate Yam E=Fermented White Trifoliolate Yam and F=Fermented Yellow Trifoliolate Yam.

It was observed from the Table 1 and from Figure 2 that the unfermented samples displayed a better drying curve properties with longer drying time while fermented samples had short drying time and this could be as a result of hardened surface of the unfermented samples which prevented free migration of water from the sample during drying (Minkah, 2007). The drying rate also indicates the quantity of moisture evaporated per unit time. It was found that at the beginning of drying, there was a higher rate of moisture loss in all the samples and this rate decreased as the drying time increased and this might be as a result of the nature of water present in the sample (Akpınar et al., 2003) or due to internal pressure generated that forces the moisture in vapor form outside the Trifoliolate Yam varieties (Nguyen and Price, 2007).

The drying rate and moisture content decreased steadily with drying time at all properties considered. The results revealed that the drying of Trifoliolate Yam samples occurred solely in the falling rate period which showed that internal moisture diffusion phenomenon is dominant and controlled the drying process. The results were in agreement with Falada and Abbo (2007); Saeed et al. (2008); Singh et al. (2008); Doymaz (2011); Ju et al. (2016) reported for drying characteristics of different yam specie and potato. The average required drying time for fermented and unfermented Trifoliolate Yam varieties were 500 mins and 580 mins respectively. It was observed that fermented samples had lower drying time due to that 72 hour fermentation has altered the internal structures of the samples loosen the sample pores which hastened the free movement of water both on the surface and internal portion in the sample. This result also indicated that the fermented sample had faster drying rate which reduces the risk of spoilage and improves quality of the biomaterials. The effect of moisture content and drying time were properly described by best fitting regression equation for unfermented and fermented samples as  $MC=0.0002t^2- 0.2428t+61.317$ ,  $R^2 = 0.9951$  and  $MC = 696469t^{-2.136}$ ,  $R^2 = 0.9438$  respectively while the effect of drying time on the drying rate were described with  $Dr = 4E-07t^2 - 0.0006t + 0.2787$ ,  $R^2 = 0.9958$  and

$Dr = 16.911t^{0.77}$ ,  $R^2 = 0.9917$  for unfermented and fermented samples respectively. These equations can also be used to model the drying characteristics of fermented and unfermented Trifoliolate Yam samples. Generally, from Table 1, it was observed that sample E had the highest drying rate ranged from 0.75–1.13 mins with average drying rate and time of 0.32 and 260 mins respectively followed by sample D (0.67–0.13 mins with average drying rate and time of 0.29 and 225 mins respectively. The sample F which recorded 0.63–0.13 mins drying rate with average value of its drying rate and time as 0.28 and 225 mins respectively had the same average drying time with sample D. Sample B had 0.62–0.10 mins range of drying rate with mean value of 0.23 and 294 mins drying rate and time respectively while 0.46–0.09 range of drying rate with average drying rate and time of 0.19 and 260 mins respectively for sample A and drying rate range of 0.25-0.08 mins with mean value of 0.15 and average time of 294 mins for sample C respectively.

It can be concluded that the fermented sample had a better drying characteristic than unfermented samples and therefore, for the interest of processing of Trifoliolate Yam species into various bio food products the 72 hours fermentation process should be adopted as it improves the mineral compositions, the health benefits and reduces the risk of spoilage and also improves quality of the products. This study was performed to facilitate the understanding of design, modeling and operation of a continuously oven dryer.



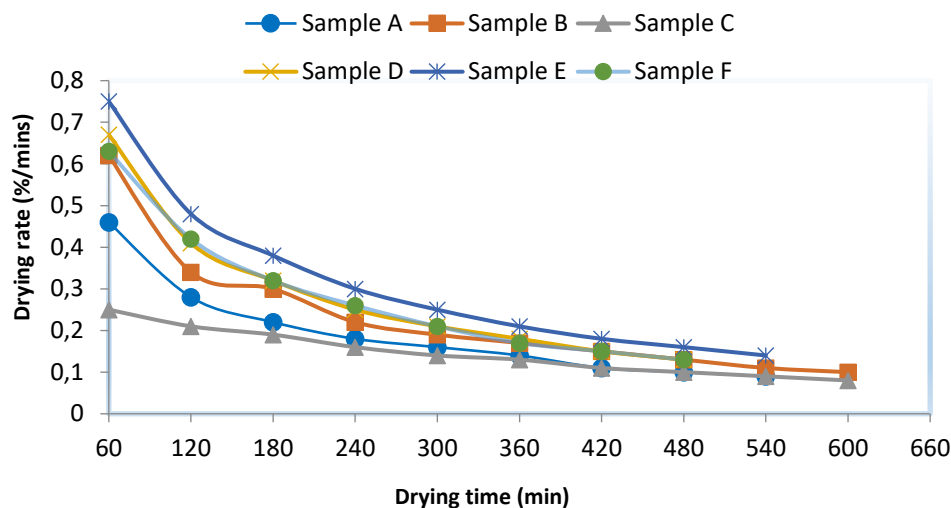
**Figure 2.** Drying curve of unfermented and fermented Trifoliolate Yam varieties dried at 70°C using oven temperature.

**Table 2.** Best fit equations and relationship between moisture content and drying time of unfermented and fermented Trifoliolate Yam varieties.

Samples	Best fitting regression equation	R <sup>2</sup> Values	(k)
A	MC = 71.777e <sup>-0.009t</sup>	0.9647	4.273
B	MC = 77.368e <sup>-0.008t</sup>	0.9712	4.348
C	MC = 0.0002t <sup>2</sup> - 0.2428t + 61.317	0.9951	-8.517
D	MC = 0.0004t <sup>2</sup> - 0.3451t + 71.851	0.8847	-7.824
E	MC = 696469t <sup>-2.136</sup>	0.9438	13.453
F	MC = 0.0004t <sup>2</sup> - 0.3543t + 71.469	0.8825	-7.824

Note: A= Unfermented Deep-Yellow Trifoliolate Yam, B=Unfermented White Trifoliolate Yam, C=Unfermented Yellow Trifoliolate Yam, D= Fermented Deep-Yellow Trifoliolate Yam E=Fermented White Trifoliolate Yam and F=Fermented Yellow Trifoliolate Yam, MC = Moisture content, t= drying time, k = Drying constant.

From Figure 3 and Table 2 the relationship between change in moisture content and with time are presented. The correlation coefficient ( $R^2$ ) of the samples which measures the relationship and variation between variables were 0.9647, 0.9712, 0.9951, 0.8847, 0.9438 and 0.8825 for sample A, B, C, D, E and F respectively. The best fit equations for sample A, B, C, D, E and F were  $MC= 71.777e^{-0.009t}$ ,  $MC= 77.368e^{-0.008t}$ ,  $MC=0.0002t^2 - 0.2428t + 61.317$ ,  $MC = 696469t^{-2.136}$  and  $MC = 0.0004t^2 - 0.3543t + 71.469$ . These values of mathematical equation and correlation coefficient are good prediction of the drying basis of moisture value at any time in the drying process and indicated that the mathematical equation fits the drying processes since their values are very close to 1.



**Figure 3.** Drying rate of unfermented and fermented Trifoliolate Yam varieties dried at 70°C using oven temperature.

**Table 3.** Best fits equation and relationship between drying rate and drying time of unfermented and fermented Trifoliolate Yam varieties.

Samples	Best fitting regression equation	R <sup>2</sup> Values	(k)
A	$Dr = 9.4432t^{-0.729}$	0.9883	2.245
B	$Dr = -0.208\ln(t) + 1.3972$	0.9399	1.570
C	$Dr = 4E-07t^2 - 0.0006t + 0.2787$	0.9958	17.504
D	$Dr = 16.911t^{-0.776}$	0.9917	2.827
E	$Dr = 18.478t^{-0.763}$	0.9887	2.916
F	$Dr = -0.239\ln(t) + 1.5815$	0.988	1.431

Note: A= Unfermented Deep-Yellow Trifoliolate Yam, B=Unfermented White Trifoliolate Yam, C=Unfermented Yellow Trifoliolate Yam, D= Fermented Deep-Yellow Trifoliolate Yam E= Fermented White Trifoliolate Yam and F= Fermented Yellow Trifoliolate Yam, Dr = Drying rate, t= drying time, k = Drying constant.

From Figure 4, the drying rate of unfermented and fermented Trifoliolate Yam varieties was presented, and Table 3 presented the best fit equations and relationship between drying rate and drying time of unfermented and fermented Trifoliolate Yam varieties. The drying constant of sample A, B, C, D, E and F were -0.729, 1.3972, 0.2787, -0.776, -0.763 and 1.5815 respectively. It was observed that fermented sample (D, E, F) had -0.776, -0.763 and 1.5815 while unfermented samples (A, B, C) had -0.729, 1.3972 and 0.2787 respectively. The larger the magnitude of drying constants the faster the water is removed, therefore, fermented samples with larger magnitude of drying

constant will dry faster than the unfermented samples with lower drying constant. The correlation coefficient ( $R^2$ ) of the samples which measures the relationship and variation between variables were 0.9883, 0.9399, 0.9958, 0.9917, 0.9887 and 0.988 for sample A, B, C, D, E and F respectively. This indicated that the mathematical equation of the drying process suite the prediction extremely well since their  $R^2$  values were very close to 1.00. It showed that, the drying model is extremely good prediction of the drying basis of moisture value at any time (t) in the drying process. The Figure 4, also showed that the first falling rate period of the samples ranged from 60 minutes to 120 minutes, which is the region where moisture on the surface of the sample are removed, the second falling rate period ranged from 180 minute to 360 minutes, this displayed the region where the internal moisture content of the sample are removed while the critical falling rate period ranged from 420 minutes to 600 minutes, it is the region where the drying rate falls to constant and this indicated the end of the drying process.

### Moisture ratio and moisture loss relationship

The Table 3 presented the moisture ratio of fermented and unfermented Trifoliolate Yam varieties dried at 70°C using oven dry method. The average moisture ratio and drying time of sample A, B, C, D, E and F are 0.135, 0.121, 0.190, 0.110, 0.098, 0.099 and 300 mins, 330 mins, 330 mins, 270 mins, 300 mins, 270 mins respectively. It was observed that sample C had the highest moisture ratio of 0.698 followed by sample A, E, F, B and D with values of 0.440, 0.412, 0.410, 0.404 and 0.381 respectively.

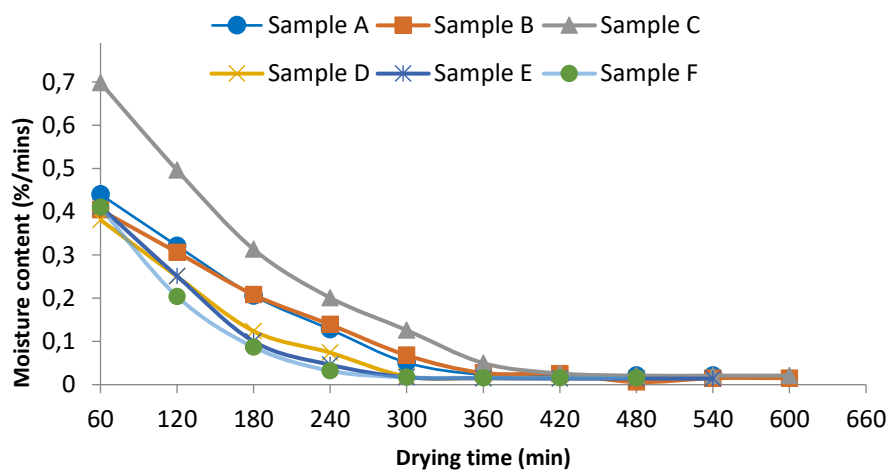
**Table 4.** Moisture ratio of fermented and unfermented Trifoliolate Yam varieties dried at 70°C using oven drying method.

Drying time (mins)	Sample A	Sample B	Sample C	Sample D	Sample E	Sample F
0	0.00	0.00	0.00	0.00	0.00	0.00
60	0.440	0.404	0.698	0.381	0.412	0.410
120	0.321	0.306	0.496	0.250	0.251	0.204
180	0.206	0.208	0.313	0.124	0.100	0.087
240	0.128	0.139	0.201	0.074	0.046	0.032
300	0.051	0.068	0.126	0.019	0.017	0.017
360	0.023	0.027	0.050	0.015	0.015	0.016
420	0.021	0.025	0.026	0.015	0.014	0.016
480	0.021	0.006	0.021	0.015	0.014	0.016
540	0.021	0.015	0.021	0.000	0.014	0.000
600	0.000	0.015	0.021	0.000	0.000	0.000

Note: A= Unfermented Deep-Yellow Trifoliolate Yam, B=Unfermented White Trifoliolate Yam, C= Unfermented Yellow Trifoliolate Yam,, D= Fermented Deep-Yellow Trifoliolate Yam E=Fermented White Trifoliolate Yam and F=Fermented Yellow Trifoliolate Yam.

From the Table 4 and Figure 5, it was observed that from the theoretical moisture ratio plots, the moisture ratio was found to be zero at t=0, this is because the initial moisture content from which the moisture ratio at t=0 was calculated is as starting point of the drying. From the Figure 5, it was found that the unfermented samples (A, B and C) had highest average moisture ratio values. The moisture ratio of fermented and unfermented sample can be described with best fitting regression equation

$Mr = 3E-06t^2 - 0.0026t + 0.5875$  with  $R^2$  value of 0.9949 and  $Mr = -0.189 \ln(t) + 1.1348$  with  $R^2$  value of 0.9293, respectively. In comparison of Table 1 and 3 with Figure 3 and Figure 4, it was observed that as the drying time progresses the drying rate decreases with increase in moisture ratio. It is therefore believed that as the drying rate decreases the moisture ratio increases. The drying rate and the moisture ratio of trifoliolate varieties are correlated with and solely dependent on the drying temperature. Moisture ratio with highest  $R^2$  value indicated that the equation can be used to model the drying process. From all indications, it was observed that the sample A had highest value of correlation coefficient of 0.996 with best fits quadratic equation of  $Mr = 3E-06t^2 - 0.0026t + 0.5875$ . From the Table 3 above, it took sample A, B, C, D, E and F 540 mins, 600 mins, 600 mins, 480 mins, 540 mins and 540 mins to attain a constant moisture ratio of 0.021, 0.015, 0.021, 0.015, 0.014 and 0.016 respectively. Sample B and C had highest drying time followed by sample A, E and F while sample D had the lowest value of drying time.



**Figure 4.** Moisture ratio of unfermented and fermented Trifoliolate Yam varieties dried at 70°C using oven temperature.

From Figure 1, the effect of adopted process parameters (72 hours fermentation and 70°C drying temperature) on the physical/sensory properties of the Trifoliolate Yam varieties. It was observed that drying temperature and 72 hours fermentation altered the colour of the samples. Both white, deep-yellow and yellow trifoliolate yam had similar colour (brown) of different intensities as finished products. Therefore, the process method adopted can not retain the colour of the Trifoliolate Yam flour varieties.

**Table 5.** Drying kinetic equation and relationship between moisture ratio and drying time of unfermented and fermented Trifoliolate Yam varieties.

Samples	Best fitting regression equations	R <sup>2</sup> Values	(k)
A	$Mr = 3E-06t^2 - 0.0026t + 0.5875$	0.996	14.914
B	$Mr = 2E-06t^2 - 0.0021t + 0.5248$	0.9959	14.508
C	$Mr = 4E-06t^2 - 0.0037t + 0.8824$	0.9928	15.201
D	$Mr = -0.189\ln(t) + 1.1348$	0.9504	1.666
E	$Mr = -0.185\ln(t) + 1.1206$	0.8922	1.687
F	$Mr = 773.5t^{-1.8}$	0.9453	6.650

Note: A= Unfermented Deep-Yellow Trifoliolate Yam, B=Unfermented White Trifoliolate Yam, C=Unfermented Yellow Trifoliolate Yam, D= Fermented Deep-Yellow Trifoliolate Yam E= Fermented White Trifoliolate Yam and F= Fermented Yellow Trifoliolate Yam, Mr = Moisture ratio, t= drying time, k = Drying constant.

## CONCLUSION

The Trifoliolate Yam slices were dried in a laboratory oven dryer at 70°C. The study reviewed that the moisture loss was subdued by diffusion mechanism. The drying temperature had significant effect on the drying characteristics of Trifoliolate Yam slices. It was found that at the beginning of drying, there was a higher rate of moisture loss in all the samples and this rate decreased as the drying time increased. The drying of Trifoliolate Yam samples occurred solely in the falling rate period which showed that internal moisture diffusion phenomenon are dominant and controlled the drying process. The average required drying time for fermented and unfermented Trifoliolate Yam varieties were 500 mins and 580 mins respectively and this can be useful in designing drying equipment. The first falling rate period of the samples ranged from 60 minutes to 120 minutes, which is the region where moisture on the surface of the sample is removed, the second falling rate period ranged from 180 minutes to 360 minutes, this displayed the region where the internal moisture content of the sample is removed while the critical falling rate period ranged from 420 minutes to 600 minutes, it is the region where the drying rate falls to constant and this indicated the end of the drying process. It can be concluded that the fermented sample had a better drying characteristic than unfermented samples. Sample A, B, C, D, E and F attained constant moisture ratio of 0.021, 0.015, 0.021, 0.015, 0.014 and 0.016 at 540 mins, 600 mins, 600 mins, 480 mins, 540 mins and 540 mins respectively. Sample B and C had the highest drying time followed by sample A, E and F while sample D had the lowest value of drying time.

## DECLARATION OF COMPETING INTEREST

The authors declared that there is no conflict of interest during and after this research.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

**Obiageli Ugwuanyi-Nnadi:** Investigation, writing-original draft, data curation,

**Brendan Ekeke Eje:** Methodology, validation and review, and editing.

**Patrick Ejike Ide:** Investigation, formal analysis, writing-original draft, methodology, writing - original draft.



## ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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## Improving the Nutritional and Functional Properties of Pearl Millet Pasta: A Review

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### ABSTRACT

Pasta, generally prepared from durum wheat is extensively consumed worldwide. Apart from being healthy and convenient food, the other outstanding characteristics of pasta include, low glycemic index, low cost, ease of preparation, extended stability during storage with relatively easier preparation. For functional pasta, care must be given to ensure that the added ingredient should enhance the nutritional profile, have minimal impact on pasta quality, palatability and consumer preferences. This review paper presents an overview of the various processing aspects of pearl millet. This comprises recent information about the improvement in the storage period of pearl millet flour (PMF), development of pearl millet pasta and addition of functional ingredients to enhance its nutritional quality. It is observed that the keeping quality of PMF can be enhanced by adopting germination, roasting, fermentation, microwave treatment, hydrothermal treatment, and refrigeration. Development of complete pearl millet pasta is not possible; functional pearl millet-based pasta can be designed using composite flour, the addition of pulses, legumes, fruit and vegetable powder to increase the demand for pearl millet.

#### RESEARCH ARTICLE

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- Anatomy,
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## INTRODUCTION

Pasta is popular ready to cook cereal food among young as well as old generation across the world. In recent times, the popularity of pasta has amplified drastically, with higher than 30 different brands in the Indian market having worth of around 250-300 crore ([Anonymous, 2016a](#); [Anonymous, 2016b](#)). The global pasta market was having a worth of 22.3 billion US\$ in 2021 with major dominance from the European companies. The market of North America, Asia Pacific, Europe, Latin America, Middle East and Africa are also having significant market share. Pasta products from companies like Barilla Holding, Grupo Ebro Puleva, Nestle, De Cecco, Makfa, etc. are more prevalent and preferred by the consumers ([Anonymous, 2021](#)). Conventionally, pasta products are prepared using an appropriate and homogeneous blend of durum wheat semolina and water. This mixture in dough form is subjected to extrusion to attain a required shape at ambient temperature and pressure or sometimes under vacuum followed by drying ([Aktan and Khan, 1992](#)). It has been reported that few human beings cannot tolerate wheat gluten, which is associated with celiac disease, wheat allergy, and non-celiac gluten sensitivity a specific disorder of intestinal absorption ([Kowlessar, 1972](#)). As per the reports, about 6-8 million people in India are affected from the celiac disease ([Anonymous, 2017](#)).

The primary purpose for cultivating pearl millet is for its utilization as food and forage in the semi-arid tropics and drought-prone regions of Asia and Africa ([Satyavathi \*et al.\*, 2021](#)). On the contrary, it is being used as a feed for poultry and livestock in USA ([Serba \*et al.\*, 2020](#)). The incorporation of pearl millet flour for preparing pasta-like products would be beneficial, owing to its in carbohydrate (67.5 g per 100 g), fat (5 g per 100 g), protein (11.6 g per 100 g), fiber (1.2 g per 100 g), mineral (2.3 g per 100 g), iron (8 mg per 100 g), calcium (42 mg per 100 g), zinc (3.1 g per 100 g) and vitamin B, especially niacin, B6 and folic acid. Pearl millet is a gluten-free grain having abundant energy, i.e. 361 kcal per 100 g, which is comparatively higher than rice (345 kcal per 100 g), wheat (346 kcal per 100 g), sorghum (349 kcal per 100 g) and maize (125 kcal per 100 g) ([Gopalan \*et al.\*, 2004](#); [Leder, 2004](#)). Consequent to cooking, pearl millet can retain its alkaline properties, making it suitable people with wheat allergies ([Satyavathi \*et al.\*, 2021](#)).

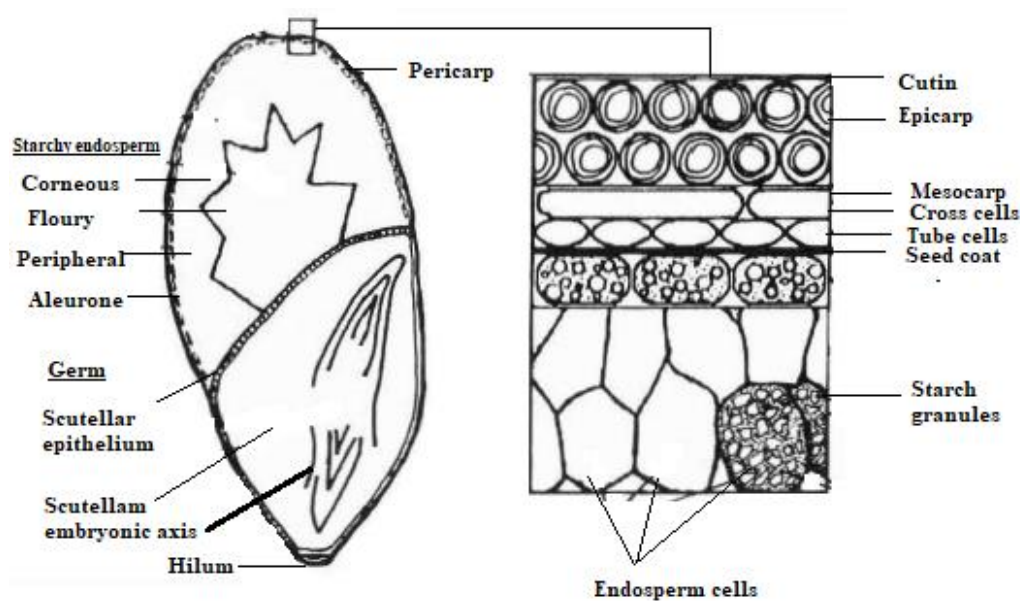
Pearl millet contains phytic acid, trypsin inhibitors, tannins, goitrogens and oxalic acid, which act as antinutritional factors and hence reduce the bioavailability of the nutrients present in the grain. Bitterness and rancidity become crucial for maintaining the pearl millet flour (PMF) quality while storage at moist conditions and with higher exposure of oxygen ([Nantanga \*et al.\*, 2008](#)). Hence, the processing of pearl millet demands the inactivation of antinutritional factors to avail full benefits of its nutritional qualities.

Owing to the increasing awareness about having a healthy lifestyle, many people are preferring high fiber and low-fat foods in their diet. Many nutritive and fortified products from pasta are emerging with added proteins, minerals, vitamins from pulses, legumes and soybean etc. Therefore, the development and adoption of pearl millet-based pasta with functional ingredients provides a great opportunity for value addition and improving the nutritional status of the consumers. Hence, this review paper was intended to systematically compile and review the nutritional properties of pearl millet,

milling and storage of PMF, the addition of functional constituents and storage conditions on the quality aspects of pearl millet pasta.

## ANATOMY AND NUTRITIONAL PROPERTIES

The structure of pearl millet grain is shown in Figure 1. The pericarp is composed of epicarp, mesocarp and endocarp. The seed coat present below the endocarp has 0.4  $\mu\text{m}$  of thickness. The aleurone layer is underneath the seed coat having a thickness of one cell layer. Starchy endosperm is composed of peripheral, corneous and floury areas. The aleurone, pericarp and endosperm portions of pearl millet have the presence of polyphenolic pigments. The taste and the grain's unattractive grey colour are takes place due to these pigments ([McDonough and Rooney, 1989](#)).



**Figure 1.** Overall structure of pearl millet grain ([McDonough and Rooney, 1989](#)).

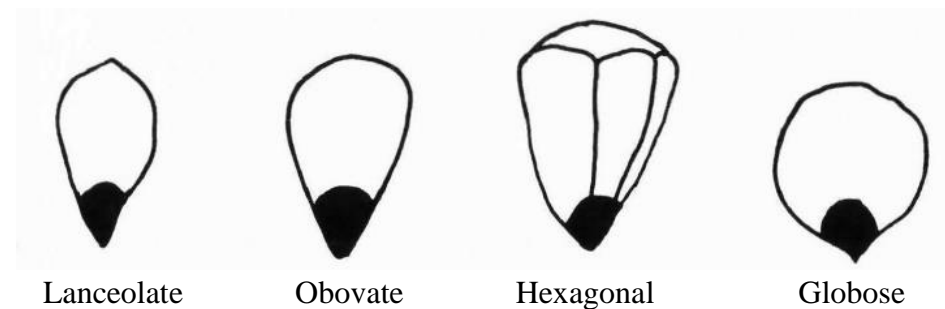
The presence of germ percentage in pearl millet grain is more than other cereal grains except maize ([Abdelrahman \*et al.\*, 1984](#)). Pearl millet grains were found in three different sizes such as large size kernel (>2920  $\mu\text{m}$ ), medium-size kernel (<2920 but >2240  $\mu\text{m}$ ), and small size kernel (<2240  $\mu\text{m}$ ). Reduction in the seed size is related to the increased percentage of bran and decrease in the proportion of endosperm (Table 1).

The protein content (%) in whole grain, endosperm, germ and bran was reported to be 13.27, 10.88, 24.52 and 17.07, respectively. The corresponding fat content in whole grain (6.26%), endosperm (0.53%), germ (32.18%) and bran (5.04%), whereas the ash percentage were whole grain (1.68%), endosperm (0.32%), germ (7.18%) and bran (3.20%), respectively ([Abdelrahman \*et al.\*, 1984](#)).

**Table 1.** Proportions of different parts of pearl millet.

Size	Weight (mg)	Endosperm (%)	Germ (%)	Bran (%)
Large	18.94	76.21	16.62	7.17
Medium	13.74	75.08	17.40	7.52
Small	10.39	73.89	15.47	10.64

[McDonough and Rooney \(1989\)](#) reported that pearl millet exhibited different shapes (obovate, lanceolate, hexagonal and globose) (Figure 2) and colour (white, yellow, brown, purple and grey).

**Figure 2.** Shapes of pearl millet grain ([McDonough and Rooney, 1989](#)).

[Tiwari \*et al.\* \(2014\)](#) reported the composition of pearl millet including moisture (10.4%), protein (12.2%), ash (1.5%), crude fibre (1.74%), fat (1.5%), carbohydrate (70.34%), iron (6.86 mg per 100 g), zinc (3.059 mg per 100 g), antioxidant activity (0.353 mmolTrolox per g), phytic acid (728 mg per 100 g), total polyphenols (352.6 mg per 100 g) and tannin content (0.14% CE).

[Jalgaonkar and Jha \(2016\)](#) emphasized the significance of reduced particle diameter (780, 600, 500, 425, 313.5 and 241  $\mu\text{m}$ ), which has resulted in increasing the nutritional profile of PMF. Results indicated that reduction in the particle size from 241 to 780  $\mu\text{m}$  has yielded variation in the protein (9.83-13.43%), fat (1.49-6.15%), ash (0.81-2.02%), iron (8.79-9.07 mg per 100 g), and zinc (2.72-3.13 mg per 100 g) content. This trend could be attributed to the size reduction of bran and aleurone layers into finer particles; however, the endosperm was pulverized into coarser particles.

### Pre-treatment effect on the storage of PMF

PMF has a tendency to become bitter and rancid within a short storage period. The reason is the conversion of glycerides and following with the increase of free fatty acids owing to the presence of an active lipase enzyme in PMF ([Pruthi 1981](#); [Arora \*et al.\*, 2002](#)).

Such undesirable chemical reactions are generally responsible for the development of off-flavour compounds predominantly under the presence of higher moisture and oxygen during storage ([Nantanga \*et al.\*, 2008](#)). Germination, roasting, fermentation, microwave treatment, hydrothermal treatment, and refrigeration are reported by researchers to enhance the keeping quality of PMF.

Effect of malting (16 h with germination of 48 or 72 h, followed by kilning for 24 h at 50°C), and hot water blanching (98°C for 30 s) on pearl millet was studied by [Archana \*et al.\* \(1998\)](#). Results indicated a significant decrease in grain's antinutritional

factors (polyphenols and phytic acid) due to malting and blanching treatment. The decrease in polyphenols was 28%, 38% and 40% in blanching, malting 48 h and malting 72 h treatments, respectively. The corresponding value for phytic acid was 38%, 46% and 53%, respectively. Malting of pearl millet grain showed higher destruction of antinutritional factor. Among the three treatments, the malting of grain for 72 h was superior in minimizing the polyphenols and phytic acid. Similar findings were reported by [Osman \(2009\)](#) that germination of pearl millet grain for 5 days of pearl millet caused a reduction in phytic acid and protein digestibility. Also, germination treatments coupled with other indigenous treatments such as fermentation will reduce tannin content and improve the nutritional content.

[Olamiti \*et al.\* \(2020\)](#) concluded that malting (MT) was done by keeping the grains in distilled water for 6 h followed by washing with formaldehyde, germination for 24 h, 48 h, and 72 h at 25°C, drying at 50°C for 10 h and packing in airtight polyethylene resealable bags) and fermentation (FT) (using lactic acid bacteria for 24-72 h at 25°C followed by draining, washing and drying at 50°C for 10 h) treatment to two pearl millet cultivars had a prominence effect on crystallinity as well as functional groups of the treated flour. The optimal processing time for MT and FT of colour were reported as 54.40 and 63.30 h for Babala pearl millet cultivar (BPM) and 50.69 and 39.38 h for Agrigreen pearl millet cultivar (APM). The optimum time for MT and FT of thermal properties were 40.94 and 29.07 h for BPM, and 45.78 and 42.60 h for APM, respectively.

[Arora \*et al.\* \(2002\)](#) highlighted the effect of dry heat treatment (100 ± 2°C for 120 min) applied to pearl millet grains before milling has substantially improved the storage life of PMF without affecting its acceptability. The study corroborated that increasing fat acidity is among the promising indicators reflecting PMF deterioration during storage. Fat acidity values of the heat-treated flour raised from 30.3-123.7 mg KOH per 100 g and 28-50.5 mg KOH per 100 g, respectively, during storage study of 28 days.

[Rathi \*et al.\* \(2004\)](#) reported that inactivation of lipase activity by depigmentation method had improved the colour of pearl millet. The depigmentation method included dipping grain in 0.2 N HCL solution for 18 h and then blanching (98°C for 30 s). The method improved the colour of grain and sensory quality parameters, soluble dietary fibre, in-vitro protein digestibility, and in-vitro starch digestibility of pearl millet pasta by preserving the nutritional content. The flour prepared from lighter colour of grain was successfully utilized for the making pasta.

[Nantanga \*et al.\* \(2008\)](#) reported that toasting treatment (120°C for 16 h) was more efficient than boiling treatment (100°C for 15 min) to the pearl millet. The fat acidity observed of flour samples, i.e. untreated (0.11 to 3.73 g per kg), toasted (0.01 to 0.68 g per kg), and boiled (0.00 to 0.04 g per kg) increased during three months of storage.

[Mohamed \*et al.\* \(2011\)](#) reported storage of flour (whole and dehulled pearl millet flour) under refrigeration (4±1°C) condition had a non-significant effect on the increase in antinutritional parameters (polyphenols and phytic acid). However, refrigeration in combination with cooking treatment (20 min in water bath followed by drying, grinding and pass through 0.4 mm screen) and then storing for one and two months at ambient temperature (25°C) or refrigeration (4±1°C) controlled the increase in antinutritional factors during refrigerated storage.

[Yadav \*et al.\* \(2012a\)](#) highlighted that microwave treatment (900 W, 2450 MHz for 80 s) to pearl millet grain (18% moisture) was significantly reduced the lipase activity with the increase in storage quality of treated flour up to one month at room condition.

[Yadav \*et al.\* \(2012b\)](#) reported that hydrothermal treatment (steaming at 1.05 kg per m<sup>2</sup>) before (20 min) and after (15 min) pearling ceased the lipase activity. Treated flour samples kept in polyethylene pouches (75  $\mu$ ) were found in good condition for up to 50 days when stored at room condition (15-35°C).

[Tiwari \*et al.\* \(2014\)](#) reported that fermentation (36 h) and dry heat treatment (100°C for 60 s) to pearl millet grain caused a reduction in phytic acid (45.32% and 43.88%), total polyphenols (20.96% and 0.69%), iron (2.19% and 1.02%) and zinc content (8.14% and 2.42%), respectively. Percentage reduction in iron and zinc content was minimum in heat-treated grain compared to pearled (25 min) and fermented grain. The storage life of PMF at ambient conditions for pearled, fermented and heat-treated flour was 2, 4 and 6 days, respectively.

[Jalgaonkar \*et al.\* \(2016\)](#) found that untreated, roasted (110°C for 60 s), and hydrothermally treated (boiling water for 15 min, followed by drying at 60°C for 2 h) PMF can be stored up to 2, 2 and 30 days at ambient conditions and 3, 3 and 45 days, respectively, at controlled condition (30 $\pm$ 1°C at 50 $\pm$ 2% RH) without undue deterioration in quality.

### **Effect of functional ingredients on quality of the pasta**

The presence of gluten protein in durum wheat (*Triticum durum*) semolina is associated with specific disorders like celiac disease, wheat allergy, and non-celiac gluten sensitivity. The inadequacy of micronutrients and excess of toxic ions are of concern for the consumption of wheat ([Abecassis \*et al.\*, 2000](#)). The usage of pearl millet having good sources of nutrients, especially micronutrient as well as gluten-free grain for preparation of pasta provides great opportunity for value-addition. Incorporating millet by 100% or substituting wheat to maximum percentage in pasta improves nutritional profile and makes them superior to wheat-based pasta.

Pearl millet grain possesses undesirable grey colour due to the presence of polyphenolic pigments, affecting its sensorial properties. Hence, to improve its colour and nutritional properties, [Rathi \*et al.\* \(2004\)](#) carried out depigmentation of pearl millet flour. The authors prepared pasta from raw and depigmented PMF along with chickpea flour (4:1) and reported that depigmentation significantly improved the pasta colour. The protein, dietary fibre, fat, ash percentage of raw and depigmented pearl millet pasta was higher than pasta from complete semolina. In vitro starch and protein, digestibilities improved significantly by 16.9% and 6.56%, respectively, after depigmentation. A decrease in protein and total dietary fibre content by 6.74 and 4.01% was observed in the pasta obtained from depigmented flour. The depigmentation of pearl millet was an effective technique for the development of pasta with better quality and digestibility.

[Yadav \*et al.\* \(2014\)](#) optimized the formulation of wheat: PMF (9:1) with different vegetable paste (2 % dry solids of tomato, carrot, spinach, turnip) for the development of vegetable blended composite pasta. Pasta prepared in this experiment had iron (2.7-4.3 mg per 100 g), calcium (23.5-40.9 mg per 100 g), phosphorous (121-244 mg per 100 g), sodium (8.9-21.1 mg per 100 g), and potassium



(130-190 mg per 100 g). The addition of vegetable paste resulted in an increase in the firmness, with minimum stickiness and grueling loss, shelf-life of 3 months without adding preservative was achieved. The consumers accepted the product in terms of colour, aroma, texture, taste, mouthfeel under ambient storage conditions.

[Devi \*et al.\* \(2015\)](#) utilized the cold extrusion technique and prepared gluten-free sweet vermicelli with different constituents *viz.* pearl millet (30%-80%), roasted green gram (20 %-23.5%), sorghum (15%-30%), guar gum (1%-2%), and sugar (12%-20%). The optimized composition adjudged was pearl millet (48%): sorghum (15%): green gram (23.5%): guar gum (1.5%): sugar (12%). The obtained vermicelli prepared was found to be superior based on the nutritional, sensorial characteristics along with the shelf life of 3 months.

[Gull \*et al.\* \(2015\)](#) used PMF (10-50 %), finger millet flour (FMF) (10-50 %), and carrot pomace powder (CPP) (2-10 %) as a substitution of semolina for pasta preparation. Cooking loss in durum wheat semolina pasta was found as 7.66% but gradually increased from 13.60 to 17.20 % and 15.20 to 24.40 % and 10 to 16.40% with an increased percentage of PMF (10-50%), FMF (10-50%) and CPP (2-10%), respectively. The control pasta showed the highest firmness (5.94 N) and cooked weight (33.93 g per 10 g), and consequent to the incorporation of CPP, FMF and PMF, the firmness and cooked weight decreased from 4.24 to 2.14 N, and 32.34 to 25.07 g per 10 g, respectively. The optimized incorporation of 4% CPP, 20% FMF and 30% PMF in durum wheat semolina was found better by keeping cooking weight, cooking loss, colour, and firmness of pasta in consideration.

[Jalgaonkar and Jha \(2016\)](#) determined the influence of particle size (241 to 780  $\mu\text{m}$ ) of PMF on the quality characteristics of pasta and mentioned 450  $\mu\text{m}$  particle size as the best.

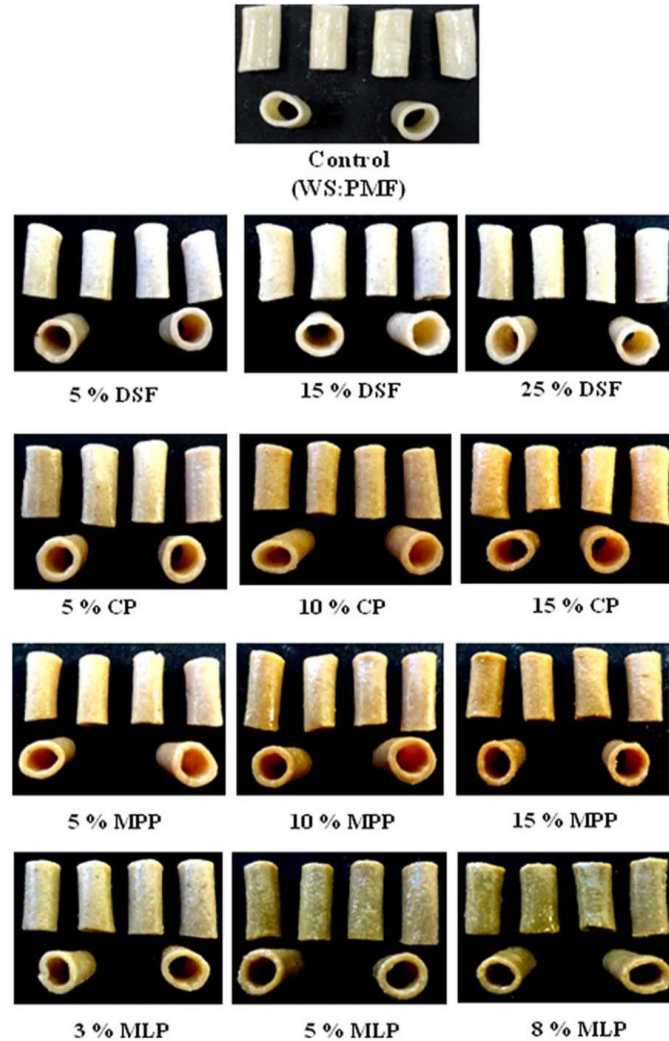
They emphasized that the development of pasta using 100% PMF irrespective of its particle size can't be achieved due to the drawbacks of retaining physical integrity/stability after cooking. The blend composition (wheat semolina: pearl millet) as 70:30 was better. The addition of pearl millet resulted in increased protein, zinc and iron content of pasta; however, the textural properties (hardness, gumminess, chewiness, and springiness) were decreased. The optimized blend composition had cooking losses (<8%), protein content (>10%), and ash content (<0.7%). It was also recommended that maximum PMF incorporation in the blend can be 50:50 for obtaining pasta with acceptable quality and enhanced nutritional benefits.

Pearl millet-based pasta was prepared and stored at ambient conditions for 6 months in 100  $\mu\text{m}$  thick biaxially oriented polypropylene (BOPP) films. The analysis revealed proportionate increase in moisture content (from 8.87 to 11.90%), water activity (from 0.51 to 0.66), free fatty acid (from 0.48 to 0.82 %), peroxide value (from 2.10 to 5.79 meq per kg of oil), fat acidity (from 20.54 to 37.77 mg per 100 g) during the entire storage. However, no microbial growth (total plate count, yeast, mould, *E. Coli*, *Salmonella*, and *Shigella*), as well as presence of rancidity or bitter taste as evaluated by sensory evaluation report was detected ([Jalgaonkar \*et al.\*, 2017](#)).

[Jalgaonkar \*et al.\* \(2017\)](#) developed functional pearl millet pasta by incorporating defatted soy flour (DSF), carrot powder, (CP) mango peel powder (MPP), and moringa leaves powder (MLP) (Fig. 3). The authors concluded that 15% DSF, 10% CP, 5% MPP, and 3% MLP can be incorporated for obtaining better pasta which was also suitable in terms of colour, cooking loss (<8%), hardness and sensory attributes. A percentage

reduction of about 5-7% in the nutritional composition of functional pasta was observed while cooking.

Jalgaonkar *et al.* (2019) highlighted that optimum extrusion operating conditions for the development of pasta from pearl millet (wheat semolina: PMF as 50:50) was obtained at 70°C (barrel temperature), 30% (w.b.) (feed moisture content), 12 rpm (feeder speed) and 1:10 (screw speed: feeder speed) with lesser cooking time ( $\leq 5.25$  min), cooking loss ( $\leq 7.45\%$ ) and high hydration capacity ( $\geq 2.30$  g per g), SC ( $\geq 3.14$  ml per g), good hardness ( $\geq 11.11$  N), SP ( $\geq 1.24$  N) and CH ( $\geq 6.09$  N mm).



**Figure 3.** Cooked pearl millet-based pasta incorporated with different levels of DSF, CP, MPP, MLP (Jalgaonkar *et al.*, 2018).

## CONCLUSION

The information presented in this review will encourage the overall utilization of pearl millet, highlighting its nutritional and therapeutic benefits. The development and adoption of pearl millet pasta with the incorporation of functional ingredients provide a great opportunity for value addition, thereby improving the nutritional status of the consumers with protection from degenerative diseases.

## DECLARATION OF COMPETING INTEREST

The authors declare that they don't have any conflict of interest.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

**Kirti Jalgaonkar:** Conceptualization, formal analysis, methodology, supervision, writing-original draft.

**Manoj Kumar Mahawar:** Supervision, validation, visualization, writing-review & editing.

**Sharmila Patil:** Conceptualization, methodology, supervision, writing-original draft

**Jyoti Dhakane-Lad:** Formal analysis, visualization, writing-review & editing.

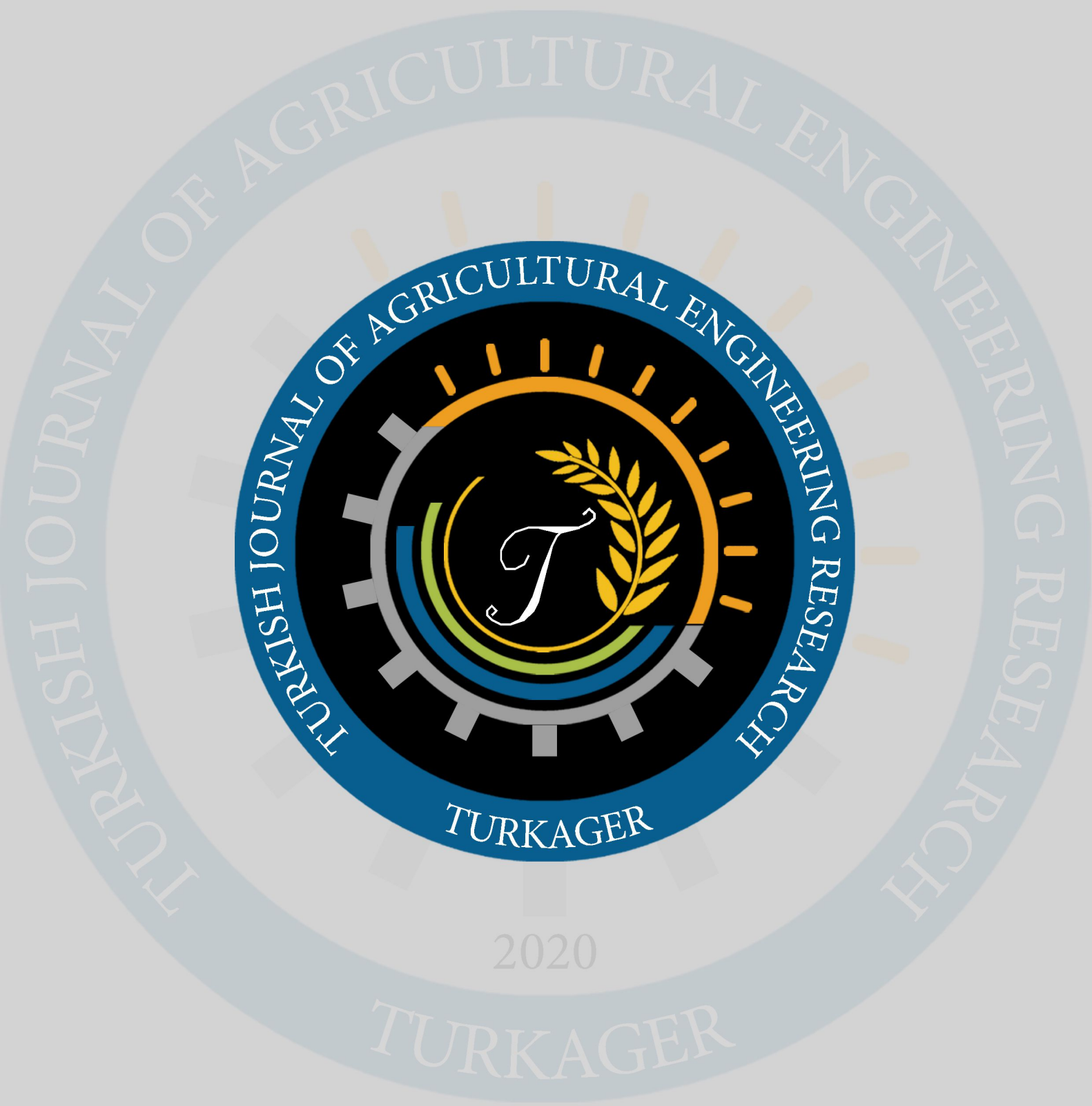
## ETHICS COMMITTEE DECISION

This article does not require any ethical committee decision.

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