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Research Article/Araştırma Makalesi

The effect of some fibers and lecithin on phase separation and storage stability of tahini

Bazı liflerin ve lesitinin tahinde faz ayrımı ve depolama stabilitesi üzerine etkisi

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ABSTRACT

The high percentage of oil in the structure of tahini tends to separate from other components during storage. Although oil separation is structurally a natural occurrence, it is undesirable for consumers, leading to the perception of the product as low-quality. Throughout the storage period, a significant portion of the oil accumulates on the surface, and the remaining part below solidifies, making consumption more challenging. The tahini used in the study was brought to the laboratory immediately after production and stored in glass jars with 100 mL each. No additives were added to the sample used as the control group. Three types of additives (sesame fiber, sugar beet fiber, and lecithin) were added to tahini in different proportions (0.5%, 1%, 2%, 3% w/v). The effect of the type of additive, level of addition, and time on preventing the separation of the oil phase from tahini was investigated at different storage times (0th, 30th, 60th, and 90th days). Additionally, its effect on pH, free fatty acidity (FFA), TBA, and conjugated diene values was also studied. According to the results of the analysis of variance, it was determined that the types of additives, levels of addition, and storage time had very significant effects on all parameters ($p<0.01$). According to the results obtained from the study, as the additive levels increased, the amount of oil separated from tahini decreased compared to the control, but no difference was determined between the levels. The main goal of this study is to reduce the percentage of separated oil from tahini, and according to the results of the additives used at different levels, it was determined that the separation of the oil phase was prevented up to 30% compared to the control sample.

Keywords: Tahini, phase separation, fiber, storage stability

ÖZ

Tahinin yapısında bulunan yüksek oranındaki yağ, depolama sırasında diğer yapılardan ayrılmaktadır. Yağ ayrışması yapısal olarak doğal bir olay olmasına rağmen tüketiciler tarafından arzu edilmemekte ve ürün kalitesiz olarak nitelendirilmektedir. Depolama süresi boyunca yağın büyük bir kısmı yüzeyde birikmekte ve altta kalan kısım katılaşarak tüketimi zorlaştırmaktadır. Çalışmada kullanılan sıvı tahin üretimden hemen sonra laboratuvara getirilerek cam kavanozlara 100'er mL dökülerek depolanmıştır. Kontrol grubu olarak kullandığımız örneğin içerisine hiçbir katkı maddesi ilave edilmemiştir. Tahin içerisine üç çeşit katkı (susam lifi, şeker pancarı lifi ve lesitin) ve bu katkılar da farklı oranlarda (% 0.5, % 1, % 2, % 3 (w/v)) ilave edilmiştir. Depolamanın farklı zamanlarında (0., 30., 60. ve 90. gün) katkı çeşidinin, katkı seviyesinin ve zamanın sıvı tahinden yağ fazının ayrılmasının engellenmesi üzerine etkisi araştırılmıştır. Ayrıca pH, serbest yağ asitliği (SYA), TBA ve konjuge dien değerleri üzerine olan etkisi de çalışılmıştır. Varyans analiz sonuçlarına göre bütün parametrelerin üzerine katkı çeşitlerinin, katkı oranlarının ve depolama zamanının

çok önemli etkileri belirlenmiştir ($p<0.01$). Çalışmadan elde edilen sonuçlara göre, katkı seviyeleri arttıkça tahinden ayrılan yağ miktarı kontrole göre azalmış ancak seviyeler arasında fark belirlenmiştir. Çalışmanın en temel amacı sıvı tahinden % ayrılan yağ miktarını azaltmak olup farklı seviyelerde kullanılan katkıların sonuçlarına göre, yağ fazın yapıdan ayrılmasının kontrol örneğine göre % 30 düzeylerine kadar engellediği belirlenmiştir.

Anahtar Kelimeler: Tahin, faz ayrılması, lif, depolama stabilitesi

Introduction

Tahini production from sesame involves several stages, including hulling, drying, roasting, and grinding. By taking advantage of the density difference with salty water, sesame husks are separated, leaving sesame kernels behind. The sesame kernels are then washed to remove the salt, roasted, and sieved before being ground. The ground and roasted sesame kernels constitute the final product, tahini. Throughout the production stages from sesame to the final product, tahini, changes occur in the physical, chemical, and antioxidant properties (Özcan, 1993; Güven et al., 2007).

Sesame seeds undergo a separation process in tahini production, where approximately 15-18% of the seed is separated as husk and bran. The remaining portion is ground to produce tahini. Thus, from 100 kg of sesame seeds, around 82-85 kg of tahini can be obtained (Görgüç, 2018; Yüzer and Gençcelep, 2023). During tahini production, the hulling process leads to a loss of mineral content, particularly calcium. The husk and bran portion has a high level of oxalic acid (> 3%), resulting in an acrid taste due to its calcium and selenium content. Additionally, it forms complexes with metals such as phthalates (present at 1.44% in dry matter) and phytic acid (> 2%), including zinc, iron, and calcium. As a result, these minerals cannot be absorbed in the digestive system. For these reasons, sesame seeds used in tahini production are stripped of their husk (Lokumcu, 2000; Bandyopadhyay et al., 2008; Liu and Chiang, 2008; Cano-Medina et al., 2011).

Tahini is considered a valuable food due to its composition, containing approximately 60% fat, 26% protein, and rich B-complex vitamins, including high-quality protein (such as the essential amino acid methionine). Its raw material, sesame, contributes to its richness in minerals such as calcium, magnesium, iron, zinc, phosphorus, and dietary fiber. Additionally, sesame contains compounds like sesamin and sesamolin, which belong to the lignan group of plant-derived compounds with a polyphenolic structure, making them powerful antioxidants (Özcan, 1993; Karakahya and Yılmaz, 2006; Batu and Elyıldırım, 2009). Sesame, with its phytosterol content resembling the chemical structure of cholesterol, is also rich in phytosterols. It is known that phytosterols obtained through the diet can lower blood cholesterol levels, strengthen the immune system, and reduce the risk of cancer (Güven et al., 2007).

Tahini, high levels of protein and fat, forms a colloidal structure. Essentially, tahini consists of sesame oil and hydrophilic solids dispersed within this oil, creating a suspension. Suspensions are heterogeneous systems with two phases. The external phase (continuous phase) can be a liquid or a semi-solid. The internal phase (dispersed phase) is composed of solid particles that are insoluble in the external phase. The sizes of droplets in the dispersed phase are crucial and typically range from 1 to 10 microns. A dispersion containing droplets below 0.1 micron in size is referred to as a colloidal solution. The small size of particles, especially below 0.1 micron, contributes to the continuous stability of the dispersion, aided by molecular movements in the continuous phase. The merging of droplets in the dispersed phase is also hindered by the increasing viscosity of the liquid in the continuous phase. The close densities of the liquids further reduce the likelihood of gravitational separation. Stabil suspensions ensure the homogeneity of a mixture by keeping the dispersed droplets in the continuous suspension. The tendency of droplets in the continuous phase to merge and coalesce (coalescence, flocculation) contributes to the instability of the suspension. Thickeners and stabilizers are substances that

increase the viscosity of the continuous phase, ensuring the stability of the dispersed phase. Suspension agents are used to slow down settling and increase viscosity. The usage amounts of suspension agents vary between 0.1-10%, depending on the type (Acartürk, 2009).

Tahini, containing 55-60% oil in its structure, undergoes separation from other structures during storage. Although oil separation is a natural occurrence, it is generally undesired by consumers, who may perceive the product as spoiled or of low quality. Throughout the storage period, a significant portion of the oil tends to separate, leaving a solidified bottom layer that makes consumption challenging. The phase separation of the oil, occurring due to particle sedimentation and density differences, is the most prominent characteristic of tahini during prolonged storage (Al-Mahasneh et al., 2017; Evlogimenou et al., 2017; Yüzer and Gençcelep, 2024). Despite its chemical resistance to spoilage reactions, the main challenge during tahini storage is colloidal instability (Çiftçi et al., 2008).

Tahins' storage stability is a primary concern for both producers and consumers. Firstly, during storage, particles in tahini tend to settle, leading to oil separation and sediment formation, adversely affecting consumer acceptability. Secondly, lipid oxidation is one of the most common issues that can develop during storage, resulting in bitterness and an unpleasant taste (Hou et al., 2020). The pH value of the sugar fibers was chosen because it was close to the pH value of tahini. Because, otherwise, the structural differences that may occur due to the acidity difference of the additive added could change the results. Lecithin is one of the most important emulsifiers used in foods. We added it because we thought that oil separation could be reduced by adding an emulsifier. In this study, the aim is to prevent or minimize the phase separation of oil that occurs during the room temperature storage of tahini. This is achieved by increasing the viscosity of the suspension through the addition of certain fibers to hinder the separation of oil from the structure. Additionally, lecithin is introduced to act as an emulsifier, and various changes occurring in the product during this period are examined.

Material and Method

Material

In this study, tahini, sugar beet fiber, sesame fiber, and soy lecithin were used as materials. Tahini was immediately obtained from PROGIDA/SAMSUN, a company based in Samsun, following its production, and it was brought to the laboratory to commence the study. Sugar beet fiber (SBF, Fibrex 600) was purchased from Nordic Sugar in Denmark. Soy lecithin (powdered soy lecithin, Tito Gıda, ISTANBUL) was procured by purchasing it. Sesame fiber was obtained by taking the residue of tahini waste, purchased as bran from PROGIDA, washing it repeatedly with water, and then drying the remaining part in an oven (40°C).

Method

Tahini preparation processes

After receiving freshly produced tahini at the factory, it was transported to the laboratory and poured into glass jars in 100 ml increments. The sample used as the control group had no additives. Additives were introduced into the jars after the samples were placed (% 0.5, % 1, % 2, and % 3 ratios w/v) and mixed at 1000 rpm for 5 minutes using an ultra turrax (IKA Werk Tp 18-10, UpM, Staufen, Germany). Subsequently, the samples were kept at room temperature, and targeted analyses were conducted at specified intervals (0th, 30th, 60th, and 90th days).

Composition analyses

The dry matter content of tahini samples was determined using the drying method. The protein content of the samples was determined based on the Kjeldahl method. The fat content of the samples was determined using the Soxhlet extraction method. The ash content of the samples was determined through incineration (Anonymous, 2000). The surface color of tahini samples was determined using the Minolta Chrometer CR-300 (Japanese). The CIE L* (brightness), a* (redness), and b* (yellowness)

values of the samples were obtained from three randomly selected points on the sample surface.

Determination of water holding capacity

Additives' water-holding capacity was determined according to the method reported by Vioque et al. (2000). For water-holding capacity, 0.5 g of additive samples was mixed with 5 ml of distilled water. The prepared solution was left at room temperature and centrifuged at 3000 g for 30 minutes. The difference between the initially added volume of distilled water and the supernatant volume for the additive samples was determined, and the results were calculated as mL of absorbed water per gram of additive.

Determination of oil holding capacity

The oil-holding capacity of additives was measured according to the method reported by Vioque et al. (2000). For oil-holding capacity, 0.5 g of additive samples was mixed with 5 ml of corn oil for 30 minutes, and then centrifuged at 3000 g for 30 minutes. The volume of separated oil from the additives was measured, and the results were calculated as mL of absorbed oil per gram of additive.

Determination of inflatable capacity

Water binding/swelling capacity analysis was determined according to the method applied by Lecumberri et al. (2007). Initially, 1 g of additive (M) was placed in a graduated cylinder, and its volume (V1) was measured. Then, 10 ml of distilled water was added, and the mixture was shaken until a homogeneous dispersion was formed. The obtained dispersion was left at room temperature (25°C) for 24 hours to allow the powder to fully absorb the water. After 24 hours, the volume of the swollen additive (V2) was measured and recorded. The water absorption capacity (WAC) (ml g-1) was calculated using the formula WAC=(V2-V1)/M.

Water solubility ındex (WSI)

The analysis of water solubility index was conducted according to the method described by Nadeem et al. (2011). A 1% aqueous solution of contributions was prepared and agitated at a constant speed for 1 hour in a shaking water bath (Nüve, Istanbul). The study was conducted at room temperature. The obtained mixture was centrifuged at 3000 g for 10 minutes. The accumulated supernatant on the surface was collected in a Petri dish, and the samples were dried at 105℃ for 18 hours and weighed (S3). The water solubility index (WSI) (%) was calculated using the formula S3/S1×100, where (S1) represents the sample amount.

pH value

Samples were diluted with distilled water at a ratio of 1:10 and homogenized, after which the pH values were measured using a pH meter (Starter 2100, OHAUS). The pH meter was calibrated with buffer solutions of pH 4.00 and 7.00 before conducting the measurements.

Determination of the separated oil ratio

For determining the amount of separated fat in stored jars (100 g), the accumulated fat on the surface was drawn with a syringe, weighed (in grams), and recorded. The weighed amount of fat was then calculated as a percentage relative to the weight of the total tahini.

Free fatty acid (FFA) analysis

The analysis of free fatty acids was determined according to the method of Nas et al. (2001). This analysis was performed on the separated fat that emerged on the surface of the tahini. Five grams of the separated fat from 250 mL of stirred tahini were weighed, and then 50 mL of diethyl ether:ethanol (1:1, v/v) mixture was added. The mixture was shaken for 1 minute to dissolve the fat and fatty acids. Next, 3-4 drops of phenolphthalein were added, and titration was carried out with 0.1 N NaOH in a burette until a permanent light pink color was obtained (at least 15 seconds). The volume of NaOH consumed was recorded. The percentage of free fatty acids was calculated in terms of oleic acid.

Determination of thiobarbituric acid reactive substance (TBARS)

For the determination of TBARS number, 10 g of tahini sample was weighed into a beaker, and then 25 mL of 20% trichloroacetic acid (TCA) and 20 mL of distilled water were added. The mixture was homogenized for 2 minutes using an Ultra Turrax (10,000 rpm). The resulting mixture was filtered through Whatman No:1 filter paper, and 5 mL of the filtrate was transferred to screw-capped tubes. Then, 5 mL of 0.02 M TBA (2-thiobarbituric acid) solution was added, the cap was closed, and the tubes were shaken. After shaking, the tubes were kept in a boiling water bath at 93°C for 30-35 minutes, then cooled for 10 minutes in tap water, and the absorbance value against a blank at 532 nm wavelength was read in a spectrophotometer. The read absorbance values were multiplied by a factor of 7.8 to determine the TBARS number as mg malondialdehyde (MDA) per kg of the sample, following the method described by Lemon (1975).

Determination of conjugated dienes

The conjugated diene numbers of tahini samples were determined according to Juntachote et al. (2007). For this purpose, 3 g of tahini sample was mixed with 30 mL distilled water to create a solution. Then, 0.5 mL of this mixture was taken and mixed with 5 mL hexane: isopropanol (3:1) and centrifuged at 2000 g for 5 minutes. After centrifugation, the absorbance of the upper phase at 233 nm wavelength was measured. The read absorbance was expressed as the conjugated diene value (Juntachote et al., 2007).

Statistical Analysis

The experiments were set up and conducted as two replicates according to a completely randomized experimental design. Some analyses were only performed on fresh products, and the storage factor was not considered in these analyses. Research data were subjected to analysis of variance using a statistical software package, and sources of variation deemed statistically significant were compared with the Duncan multiple comparison test (SPSS, 2020).

Results and Discussion

The results of the composition analyses of tahini and additives used in the study are presented in Table 1 and 2.

Table 1. Tahini composition analysis results (Mean ± standard deviation)

Properties	*Results*
(%) Moisture)	0.14±0.028
(%) Dry matter	99.86±0.028
(%) Oil	51.14±0.89
(%) Protein	24.24±1.28
(%) Ash	2.69±0.026
TBARS Values (*malondialdehyde/Kg)*	5.748±1.32
Conjugated diene	0.765±0.030
pH	5.63±0.084
*L**	27.37±0.001
*a**	1.74±0.002
*b**	9.38±0.004

According to the Tahini Regulation, tahini should have a maximum of 3.2% ash, a minimum of 50% mass fraction of sesame oil, at least 20% protein, a maximum of 1.5% moisture, a maximum of 2.4% acidity (as oleic acid), and a negative value for bitterness (Kreis); additionally, it should not contain foreign substances except for starch (Anonymous, 2015). For example, the obtained tahini in this study has a mass fraction of oil at 51.14%, protein content at 24.24%, dry matter at 99.86%, and ash at 2.69%. Upon examining the data, it is observed that the tahini acquired for the study complies with the regulations. Numerous studies have indicated that tahini typically contains 50-60% fat, 16-28% high-value protein, and B vitamins (Lokumcu Altay and Ak, 2005; Akbulut and Çoklar, 2008; Çiftçi et al., 2008; Hou, 2017; Hou et al., 2018; Tounsi et al., 2019; Yüzer and

Gençcelep, 2024).

In numerous studies, it is stated that sesame seeds have an average protein content ranging from 18% to 25%, while tahini can have protein content reaching up to the 28% range. The protein content of tahini in this study was found to be 24.7%, and these results are consistent with the mentioned values. According to the Tahini Regulation (Anonymous, 2015), the ash content should not exceed 3.2%. In our study, the ash content was determined to be 2.69%, which is in compliance with the values specified in the tahini regulation. Various studies have reported ash content in tahini ranging from 3.00% to 4.05% (Sawaya et al., 1985; Kömez, 2002). Factors such as incomplete hull removal during tahini production, insufficient washing to remove the salt used in hull separation, excessive water use during sesame cultivation, and the variety of sesame can contribute to an increase in ash content in the final tahini product, along with potential adverse effects during processing steps (Özcan, 1993; Güneşer, 2009).

Lokumcu Altay and Ak (2005) found the moisture, protein, fat, and ash contents of tahini to be 0.63%, 26%, 58.8%, and 2.55%, respectively. Akbulut and Çoklar (2008) determined the moisture content of tahini to be 1.86%, protein content at 23.77%, fat content at 55.42%, fiber content at 3.11%, and ash content at 2.78%. Hou (2017) reported that tahini contains 0.12% moisture, 59.71% crude fat, 17% protein, 5.01% ash, 3.78% crude fiber, and 7.70% total carbohydrates. Hou et al. (2018) found that the moisture content of tahini ranged from 0.12% to 1.10%, fat content from 51.80% to 61.56%, protein content from 16.08% to 20.10%, crude fiber content from 2.53% to 3.78%, total ash content from 4.48% to 5.70%, and total carbohydrate content ranged from 6.23% to 18.57%.

In a study on the color values of imported tahini, Kömez (2002) found the a* value to be in the range of 3.50-11.19 and the b* value to be in the range of 23.44-32.04. For domestic tahini, the color values were found to be a* in the range of 3.57-9.42 and b* in the range of 22.53-31.19. Karaman et al. (2017) reported a* and b* values for tahini as 3.12 and 13.88, respectively. When comparing the results obtained in this study, it is observed that the a* value of the tahini used in our study is lower, and the b* value is higher. This difference is presumed to be related to the variety of sesame used.

Tahini is a food substance rich in oil, one of its main components. Tahini is particularly rich in oleic fatty acid, and the second-highest fatty acid present is linoleic acid. Considering the fatty acid composition of the product, it can be said that it contains a high proportion of unsaturated fatty acids. Consequently, the product is considered to be highly susceptible to oxidation. In this regard, the determined TBARS and conjugated diene values in tahini were found to be high.

Three different additives and four different ratios were used in the study, and the physical properties of the additives used in the study are provided in Table 2. The results of the analyses conducted on these additives are believed to be beneficial in interpreting the prevention of oil separation in tahini during storage. Knowing some properties of the additives used in the product is also important in determining the changes they will induce in the product's structure.

In such studies, it is crucial that the additives used do not cause significant differences in certain physical, chemical, and sensory properties of the product. Tahini has a very low water content (Table 1), making it an oil-based product. To prevent possible oil separation, lecithin has been added to the formulation as an emulsifier and stabilizer.

Table 2. Composition analysis results of additives (Mean ± standard deviation)

Properties	*Sugar beet fiber*	*Sesame fiber*	*Lecithin*
(%) Moisture	6.19±0.38	2.64±0.07	2.35±0.01
(%) Ash	5.52±0.01	12.04±0.04	11.13±0.06
(%) Swelling capacity	3.43±0.11	2.46±0.11	0.50±0.00
(%) Water holding capacity	5.37±0.19	6.94±0.21	2.70±0.33
(%) Oil holding capacity	2.88±0.26	3.82±0.25	2.19±0.49
Water solubility index	16.20±1.04	6.16±2.15	43.60±1.15
pH	4.86±0.01	7.38±0.01	7.48±0.02
*L**	74.02±0.95	45.40±1.04	80.63±2.29
*a**	3.08±0.12	4.93±0.14	4.34±0.32
*b**	26.81±0.32	19.09±0.17	40.96±0.95
Dimension	<250µm	<250µm	----

Lesitin has been recognized as "GRAS" (generally recognized as safe) by the Food and Agriculture Organization (FAO), meaning there are no restrictions on the amount of lecithin that can be used in food (Garti, 2001). Lecithins are multifunctional products with broad applications at low levels (0.5-3%), often performing multiple functions in a food product. Their primary function is emulsification, which involves holding two different liquids together to form emulsions of oil in water or water in oil. With their amphoteric properties, lecithins play an indispensable role in food systems (Hui, 1992; Garti, 2001).

Dietary fibers are classified into two groups based on their solubility: soluble and insoluble fibers. Soluble dietary fiber forms a gel and a dense structure by binding water. Insoluble dietary fiber, on the other hand, can absorb up to 20 times its weight in water but does not form a viscous structure (Thebaudin et al., 1997). The nutritional fiber content of sugar beet pulp is 67% insoluble and 21% soluble in dry matter, making up a total of 88%. Insoluble fibers can retain up to 5 times their weight in fat, a feature crucial for preserving lost fat during food processing, as commonly occurs in the production of food items. This characteristic is significant for enhancing the technological properties of food. The high fat-binding capacity of dietary fiber is important for stabilizing fat and water emulsions (Grigelmo-Miguel et al., 1999). It has been determined that the fat-binding capacity of dietary fiber varies with particle size, with larger particles binding more fat. Wheat bran and sugar beet fiber, characterized by their large particles, are noted for their high fat-binding capacities (Thebaudin et al., 1997).

Sesame meal is obtained at the end of sesame oil production. Sesame meal typically contains approximately 40% crude protein and 24% mineral substances (P, K, Ca), as reported by Uğurluay (2002) and Sreedevi and Sivasankar (2009). The main disadvantage of using sesame meal as a food additive is its very low solubility in water, but this drawback can be overcome by modifying the protein (Escamilla-Silva et al., 2003; Radha et al., 2008). Solubility and swelling properties are interconnected. The initial solubility of polysaccharides is associated with swelling. Water moves towards the solid structure, and macromolecules swell until they are completely dispersed, expressing solubility. In contrast, some polysaccharides like cellulose cannot disperse due to their structural characteristics (Thebaudin et al., 1997).

The characteristics of the additives used in the study should be examined to ensure that they do not cause changes in the moisture and ash values of tahini. This is crucial because these values are regulated by regulations. Additionally, the additives should be soluble in the suspension and exhibit good fat-binding properties. It is important that the color values, which significantly influence consumers' purchasing decisions and preferences, do not undergo substantial changes due to the additives.

Protein interactions with water and/or fat are crucial in food systems as they influence properties such as taste and texture. A critical range for water-holding capacity values for viscous foods like soups and sauces, determined by Aletor et al. (2002), is between 1.49 and 4.72 (g/g). Therefore, sesame meal protein hydrolysates, due to their

high water-holding capacity, find applications in the food industry to prevent water loss in bread and cakes, as well as to enhance the utilization of cooked and frozen foods (Vioque et al., 2000; Onsaard et al., 2010). The water-holding capacity of sesame meal hydrolysate has been found to be 3.43 g water/g protein, and the fat-holding capacity is 2.21 ml fat/g protein. Upon examining fat absorption and water-holding capacity, it has been determined that sesame protein exhibits low fat absorption and high water-holding capacity (Demirhan Yılmaz, 2012).

The most crucial feature influencing consumer preferences and decisions is color, and it is one of the essential appearance characteristics of food items (Maskan, 2001). Color is a parameter used in the process control during roasting because as browning and caramelization reactions progress, brown pigments increase (Moss and Otten, 1989).

At the beginning of storage (day 0), after the addition of additives to tahini, the color values for untreated tahini were determined as follows: L* (brightness) value was 27.37, a* (redness) value was 1.74, and b* (yellowness) value was 9.38. In tahini with sugar beet fiber additives, there was no significant change in L* (brightness) and a* (redness) values compared to untreated tahini. However, it was observed that the b* (yellowness) value decreased as the amount of fiber added increased, mainly due to the high b* value of sugar beet fiber (26.81). In tahini with sesame fiber additives, there was no significant change in L* values, while there was a slight decrease in both a* and b* values, especially as the level of additives increased. In tahini with lecithin additives, there was no significant change in L*, a*, and b* values, as the levels of additives were introduced at percentages such as 0.5%, 1%, 2%, and 3%. In general, regardless of the type of additive, the study concluded that the additives used did not cause an excessive color change in tahini, making them suitable for use.

Table 3. Additive addition to tahini, addition rate, storage analysis and analysis results of interactions (Mean ± standard deviation)

	pH	*% Amount of separated oil*	*FFA (% oleic acid)*	*TBA Values* malondialdehyde MA/kg	*Conjugated diene*
Additives (K)					
Sugar beet fiber	5.37 c	4.51 b	0.32 b	7.75 a	0.89 c
Sesame fiber	5.92 b	4.78 a	0.34 a	7.36 c	1.10 b
Lecithin	6.04 a	4.46 b	0.29 c	7.65 b	1.23 a
P<0.01	**	**	**	**	**
Level (S)					
0.0	5.69 c	5.54 a	0.26 d	7.02 e	1.25 a
0.5	5.76 b	4.19 b	0.30 c	7.50 d	0.89 e
1.0	5.80 a	4.42 b	0.32 b	7.61 c	0.93 d
2.0	5.82 a	4.30 b	0.33 a	7.80 b	1.09 c
3.0	5.80 a	4.48 b	0.36 a	8.02 a	1.21 b
P<0.01	**	**	**	**	**
Storage (days) (G)					
0	5.71 d	--	--	5.74 d	0.76 b
30	5.78 b	2.73 c	0.55 a	7.79 c	1.18 a
60	5.75 c	6.49 b	0.35 c	8.05 b	1.18 a
90	5.85 a	9.12 a	0.38 b	8.78 a	1.18 a
P<0.01	**	**	**	**	**
KxS	**	**	**	**	**
KxG	**	**	**	**	**
SxG	**	**	**	**	**
KxSxG	**	**	**	*	**

**P<0.01: There is a very significant difference, *P<0.05: There is a significant difference
a-d: Numbers followed by different letters in a column are significantly different (P<0.05)

The increase in free fatty acidity and pH changes in tahini can occur as a result of microbiological or enzymatic reactions. Osmophilic yeasts are microorganisms capable of producing acid by

utilizing the sugar in the product. However, due to the low water content in the product, the probability of microbiological developments is low, leading to the consideration that this change may be related to enzymatic reactions. In a study conducted by Gamli and Hayoglu (2007) on peanut butter, it was reported that the total acidity value increased with temperature and duration for products stored at 4°C and 20°C, while the pH decreased. In their study, they suggested that the increase in acidity could be attributed to microbiological or enzymatic reactions (Gamli and Hayoglu, 2007).

The addition of sugar beet has led to a decrease in the pH value of tahini. The reason for this is that the pH value of the added additive (4.86, Table 2) is lower than the pH value of tahini. On the other hand, the addition of other additives (sesame fiber and lecithin) resulted in an increase in pH value. This is because the pH values of these additives (7.38 and 7.48, Table 2) are higher than the pH value of tahini. It has been observed that there is not much research on the pH of sesame and its products. As the ratio of added additives to tahini increased, a general increase in pH value was observed, but there was no increase at levels of 1%, 2%, and 3%. It is believed that this difference is the result of the difference in the pH values of the added additives (Table 2). The pH value of sugar beet, sesame fiber, and lecithin (4.86, 7.38, and 7.48, respectively) has resulted in a balance as the levels increase, and the increase has not been statistically determined as different.

In a study conducted by Al-Nabulsi et al. (2014), the pH of tahini was found to be 6.76. In another study on mixtures of tahini, honey, and grape molasses by Karaman et al. (2017), the pH of tahini was determined to be 6.50. The values determined for tahini in this study are lower than those determined in previous studies. It is believed that this difference is attributed to factors such as variety, type, processing method, processing temperature, and compositional variations. Upon examining Table 3, an increase in pH values is observed with the increase in storage time. This increase can be attributed to the breakdown of fatty acids present in the environment, the decomposition of proteins influenced by environmental conditions, and the breakdown of carbohydrates, among other effects.

Tahini has a colloidal structure containing high levels of protein and fat. It is essentially a suspension consisting of sesame oil and hydrophilic solids dispersed in this oil. The fat, constituting about 55-60% of tahini's structure, separates from other structures during storage. One of the most distinctive physical characteristics of tahini is the phase separation of the oil and particle sedimentation due to density differences, which occurs after prolonged storage (Evlogimenou et al., 2017).

One of the physical problems encountered in high-fat foods is fat separation. Fat separation also forms the basis for chemical deterioration because the exposed oil comes into more contact with oxygen and undergoes oxidation more rapidly. The separation of the fat phase relies on the different densities of the components in the product. When the product contains components with both water and fat phases, the use of emulsifiers such as lecithin or stabilizers becomes crucial, and effective mixing processes can also prevent this issue (Muego-Gnanasekharan and Resurreccion, 1992).

As seen in Table 3, lower levels of accumulated surface oil were observed in tahini with added additives at varying ratios compared to the control. However, as the ratio increased, there was no significant change in the amount of separated oil. During storage, oil separation increased, and these values continued to double at the measured time intervals.

Free fatty acidity is a result of the hydrolysis of fat from triglyceride structures due to various factors. While known antioxidants can prevent disruptions caused by oxidative rancidity and oxy-polymerization events, they are not effective for hydrolysis and reversion (Çakmakçı and Gökalp, 1992). Changes in free fatty acidity during storage provide information about the degree of bitterness in the product. It helps determine how far the hydrolysis mechanism has progressed

(Hamilton, 1989).

Evlogimenou et al. (2017) have predicted that oil-rich raw materials' aqueous extraction residues can be included to enhance the stability of tahini against oil separation and particle sedimentation. In the current research, three aqueous extraction residues obtained from hazelnut, corn seed, and sesame seed were converted into powder form and compared as effective physical stabilizers for tahini. As a result, when these powders were included in tahini and stored for an extended period, they increased the stability of tahini against oil separation up to a certain level. The improvement in stability against oil release was reported to be associated with an increase in the number of solid particle interactions within the tahini structure and the strength of these interactions.

In a study investigating the effect of temperature and particle size on tahini to enhance storage durability and address the issue of oil separation, it was reported that in tahinis stored at 20 °C, the oil separation increased as the particle size decreased. However, at a temperature of 30 °C, the storage durability became independent of particle size (Çiftçi et al., 2008). In the same study, statistical analyses revealed that tahini's storage stability was dependent on both temperature and particle size, but the researchers noted that the influence of temperature on stability was more significant than that of particle size. The researchers determined a critical particle size of 5 µm for tahini at all three storage temperature levels (20 °C, 30 °C, and 40 °C).

In concentrated suspensions, the particle size distribution is crucial. In a conducted study, it was determined that 76% of tahini consists of particles smaller than 10 µm, while 14% of the particles are in the size range of 100-500 µm. In the context of unwanted oil separation in tahini, it has been reported that the fraction with smaller particle size is more effective (Lindner and Kinsella, 1991). Although tahini is resistant to chemical degradation reactions in terms of shelf life, colloidal instability during storage is the main problem (Çiftçi et al., 2008).

In their research to prevent phase separation in tahini using natural waxes, Ögütcü et al. (2018) added sunflower (%1 and %3) and beeswax (1.3 and %5) to commercially obtained tahini at specific concentrations. Samples were stored at 25 and 35°C for 21 days. Centrifugation stability, oil leakage, textural properties, viscosity, and consumer tests were analyzed. Samples with added beeswax and sunflower wax exhibited lower oil leakage values compared to plain tahini (control). Viscosity measurement showed that control and samples with added beeswax (%1 and %3) exhibited pseudo-plastic rheological behavior. Textural measurements indicated that tahini prepared with %3 sunflower wax was denser and more adhesive than tahini prepared with %1 sunflower wax and %5 beeswax. Additionally, the textural properties of the samples were significantly influenced by storage temperatures. Furthermore, samples with %1 and %3 sunflower wax and %5 beeswax could be spreadable, while those with %1 and %3 beeswax added became fluid. Consequently, the addition of sunflower and beeswax not only restricted phase separation in tahini but also transformed it into a spreadable form depending on the beeswax concentration.

In the research conducted by Yüzer and Gençcelep (2024), additives were added in order to prevent or minimize oil phase separation, which occurs in sesame paste stored at room temperature and is not desired by consumers. As additives, nanofibers containing sesame proteins produced by the electrospinning method and sesame protein isolates (SPIs) were added. Added additives acted by preventing the separation of the oil phase from the structure, and some changes in the product during storage (0, 30, 60, and 90 days) were investigated. The values obtained showed that the additives reduced oil separation up to 63% compared to the control group. As a result of the study, it was determined that the separation of the oil phase from the structure could be prevented up to 24.73% with the addition of SPI and up to 63.02% with the addition of sesame protein nanofiber (SPINL) compared to the control groups. It has been determined that the addition of SPI to

sesame pastes does not completely prevent the problem of oil separation in sesame paste, but is effective in delaying and reducing it.

According to the results of free fatty acidity analyzed by extracting the accumulated oil on the surface of tahini with the help of an injector (Table 3), the amount of free fatty acids (FFA) increased as the additive levels increased. The analysis results on the 30th day, as shown in Table 3, were higher compared to the 60th and 90th days. This phenomenon can be explained by the further breakdown of fatty acids into advanced decomposition products. In the same samples, the increase in TBARS (Thiobarbituric Acid Reactive Substances) and conjugated diene results on the 30th day (Table 3) was proportionally higher compared to other days.

One of the oldest and most widely used methods for determining lipid oxidation in foods and other biological systems is the 2-thiobarbituric acid (TBA) test. Malondialdehyde is a minor product in the oxidation of polyunsaturated fatty acids (Logani and Davies, 1979; Shahidi et al., 2002).

Upon examining Table 3, it can be observed that the TBARS levels increased with the increasing additive levels. However, it can be noted that these increases were relatively low at low levels of additives. Additionally, for all types of additives at the same level, an increase in TBARS values was observed as the storage time increased. The results suggest that, over time, in addition to oil separation, auto-oxidation within tahini may also increase.

The delay of oxidation is crucial for everyone involved in the food chain, from food producers to consumers. Various techniques can be employed to prevent oxidation, including preventing food contact with oxygen, applying low-temperature processes, inactivating enzymes catalyzing oxidation, reducing oxygen pressure, and using appropriate packaging. The most effective way to protect foods against oxidation is the use of substances with antioxidant properties (Pokorny et al., 2001).

Under normal conditions, lipids are not in radical form but can form radical forms due to the influence of heat, metals, or light (Choe and Min, 2006). The oxidative breakdown of lipids in foods results in the formation of potentially toxic secondary substances, leading to a loss of nutritional quality, rancid odor, and flavor (Choe and Min, 2006). The average conjugated diene values of stored tahini with different levels of additives are provided in Table 3. As shown in Table 3, an increase in both level and storage time led to an increase in conjugated diene values, except for the unsupplemented tahini group (level 0), similar to the TBARS results. It is anticipated that hydroperoxides formed from fatty acids play a role in the gradual conjugation formation and the increase in conjugated diene values. The amount of oil separated increased during the storage period. TBA values increased during the storage period, but the Conjugated diene values did not change after the 30th day.

In conclusion, it was observed that the three types of additives used reduced the separated fat phase from the structure by an average of 25-30%. Moreover, especially for reducing fat separation, it is crucial for the selected additives to mix better with the product and increase phase viscosity, so the particle size should be lower. The presence of protein-based additives in the content of the additives is also essential for holding the fat in the structure and thus preventing flocculation.

Attachments

This study is derived from the master's thesis prepared by Ercan YETKİN and accepted by the Graduate School of Ondokuz Mayıs University.

Conflict of Interest Statement

The authors declare no conflict of interest.

Author contributions

Ercan YETKİN: Investigation, Methodology, Writing original draft, Review & editing, Hüseyin GENÇCELEP: Review & editing, Supervision, Resources.

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