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Phytochemical Profile and Antioxidant Activities of *Zingiber officinale* (Ginger) and *Curcuma longa* L.(Turmeric) Rhizomes

Ümit Erdoğan[1*], Sabri Erbaş[2]

Abstract: The aim of our study was to evaluate and collate the chemical constituents and antioxidant properties of dry rhizomes of Ginger and dry rhizomes of Turmeric. The assay for quantification of the phenolic compounds in the samples was carried out using the reversed phase-high performance liquid chromatography (RP-HPLC). To determine mineral components in samples inductively coupled plasma optical out flow spectroscopy (ICP-OES) procedure was applied. The most abundant phenolic components in turmeric rhizomes (mg componenet 100 g^{-1} dried rhizome) are ferulic acid (93.59 mg), benzoic acid (40.09 mg), vanillin (26.69 mg) and p-coumaric acid (23.25mg) respectively. On the other hand, the most common phenolic components in ginger rhizomes are Benzoic acid (33.31mg), Ferulic acid (11.41 mg) and vanillin (11.83 mg). In addition, ethanolic extract ginger (EEG) and ethanolic extract turmeric (EET) had an effective DPPH• scavenging, hydrogen peroxide scavenging, ferric ions (Fe^{3+}) reducing power activities. According to ICP-OES analysis results of rhizomes and extracts, the potassium was, quantitatively, the most abundant mineral in samples. Subsequently, sodium, magnesium, phosphorus and calcium were identified, respectively.

Keywords: Antioxidant, mineral content, ginger, turmeric, phenolic compound.

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1. INTRODUCTION

Reactive oxygen species (ROS) is a common term that contains all reactive forms of oxygen, involving both radical and non-radical species that participate in the initiation and/or propagation of chain reaction (Kumar et al., 2011). ROS are superoxide radical ($O_2 \cdot -$), hydrogen peroxide (H_2O_2) and hydroxyl radical (OH•), which are formed in small amounts during normal oxygen metabolism ROS are continuously produced during normal physiologic events and can easily initiate the peroxidation of membrane lipids, leading to the accumulation of lipid peroxides. ROS can also damage important biomolecules such as nucleic acids, lipids, proteins, and carbohydrates, and cause DNA damage that can lead to mutations (Ak and Gulçin, 2008). This damage causes various diseases such as cancer, atherosclerosis, amyloidosis, age-related immune deficiency, senile dementia and hypertension and is known to play a role in the biological aging process (Kopáni et al., 2006). There are many defense mechanisms to prevent the formation of ROS and their damage. These mechanisms are known as "antioxidant defense systems" or simply "antioxidants." Antioxidant components can sweep free radicals and prolong shelf life by delaying the lipid peroxidation process, which causes food and pharmaceutical products to deteriorate (Halliwell, 1996). Recent research has shown that antioxidants with herbal-derived radical scavenging activity are crucial in curing free radical-borne diseases during the aging process (Mohan et al., 2015). An inquiry of normally happening antioxidant ingredients from plant sources may give prompts the advancement of novel medicines, which may diminish the danger of long-term infections brought about by free radicals (Abuja and Albertini, 2001).

Turmeric (*Curcuma longa* L.), a perennial plant that belongs to the Zingiberaceae family, is widely cultivated in Asian countries. The rhizomes of this plant are the most useful and are used for culinary and traditional medicinal purposes (Bagchi, 2012). Curcumin is the most important bioactive component of turmeric, which is also used as a spice. Turmeric powder, curcumin and its derivatives and

many other extracts from the rhizomes were found. Investigations of turmeric have uncovered various pharmacological properties (Wichitnitha et al., 2009). However, Ginger, whose Latin name is *Zingiber officinale*, is a plant of the Zingiberaceae family, which can grow up to one meter in length, with long leaves and yellow-red flowers. Ginger is a well-known herb to contain several bioactive compounds, anti-inflammatory, carminative, antiseptic properties and antioxidants that possesses health-promoting properties (Mushtaq et al., 2019).

The aim of our study was to evaluate and collate the chemical constituents and antioxidant properties dry rhizomes of Ginger and dry rhizomes of Turmeric. In the current study we have made an assay to determine the dietary advantages of these the two rhizomes.

2. MATERIAL AND METHODS

2.1. Chemicals

Gallic acid was purchased from Merck. BHA, BHT, L-Ascorbic Acid, DPPH•,the folin-ciocalteu reagent, Potassium ferricyanide, Disodium hydrogen phosphate, Potassium dihydrogen phosphate, Ferric chloride, Sodium Carbonate and trichloroacetic acid (TCA) were obtained from Sigma (Sigma–Aldrich GmbH, Sternheim, Germany). All other chemicals used were analytical grade and obtained from either Sigma–Aldrich or Merck.

2.2. Plant material

The turmeric and ginger were obtained from the Faculty of Agriculture- Isparta University of Applied Sciences. Plant specimens were also identified by Prof. Hasan Baydar and deposited at the herbarium of Faculty of Agriculture, Isparta University of Applied Sciences, with voucher specimen numbers TP32-2020 and GP32-2020. The preferred materials were cleaned to get rid from the dirt and other foreign particles, the cloves of turmeric ginger were peeled subsequently, for the preparation of ginger and turmeric extract and powders in the same way.

2.3. Preparation of ethanolic extract of turmeric and ginger powder

Ginger (EEG) and turmeric extracts (EET) were prepared using 96% ethanol. 100 grams dry powdered samples were soaked 500 mL in ethanol (96%) in a sealed 1 liter container for 24 hours at room tempurature, with intermittent shaking. Extracts were further washed with fresh ethanol (250 ml and 125 ml) and were filtered through Whatman No. 41 filter paper. Then the filtrates were concentrated under vacuum using a rotary evaporator under reduced pressure at 50 ^{o}C. From 100 grams of turmeric and ginger powder samples, 3.71 and 5.58 grams of extract were obtained, respectively (Table 1). EEG and EET were utilized for the discovery of their mineral profile and antioxidant capacity. To assess the anti- oxidative point of view, total phenols (TPC), DPPH radical scavenging potantial (1, 1-diphenyl-2-picrylhydrazyl), hydrogen peroxide scavenging and FRAP (Ferric reducing antioxidant power) assays were conducted and kept in in obscurity at +4 ^{o}C until use.

2.4. Elemental analysis

To determine components in samples inductively coupled plasma optical outflow spectroscopy (ICP-OES) procedure was applied. Sample preparation process was done by using wet burning method by adding 8 mL HNO_3 + 2 mL H_2O_2 to 0.2-0.3 gram sample using Milestone ETHOS ONE model microwave sample preparation unit according to EPA 3015 a method. The final volume was completed to 20 mL with distilled water. ICP OES measurements were made in accordance with EPA 6010 method by using Perkin Elmer OPTIMA 5300 DV device. The accompanying 16 substance components were determined: Al, Ca Cd, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, P, Pb, Se and Zn. The acquired outcomes are displayed in Table 3. Results were calculated as mg element g^{-1} dried rhizome using external calibration curves, constructed for each mineral standard. Investigation of each sample was done in triplicate. All the findings were indicated as the mean of triplicate measurements.

2.5. Determination of total phenolics content (TPC)

The quantity of total phenolic substance in the turmeric and ginger extracts was figured out with Folin-Ciocalteu reagent as indicated by the technique for Slinkard and Singleton (1977). Folin-Ciocalteu is a procedure used for the estimation of total phenolic compounds. Gallic acid was used as a standard phenolic compound. Briefly, 40 µl sample (1 mL of extract solution contains 1 mg extracts), a gallic acid calibration standard, or blank (deionized or distilled water) was put into a 15 ml falcon tube. Then, 3.16 ml water, followed by 200 µl FC reagent was added and mixed thoroughly by pipetting or inverting and incubate 1 to 8 min. After 5 min, 600 µL of Na_2CO_3 (20%) was added and afterward the mix was permitted to represent 2 h with discontinuous shaking. The absorbance was measured at 765 nm in a spectrophotometer (Perkin Elmer Lambda 20 UV VIS Spectrophotometer). A calibration curve was made by getting ready 40 µl aliquots of 31.25, 62.5, 125, 250, 500 and 1000 µg mL^{-1} arrangements of Gallic acid and the outcomes were stated as gallic acid equivalents in milligram per gram [mg GAE/g] of the sample. Absorbance=0:0011 x Total phenols [Gallic Acid Equivalent (mg)] +0.0174 (R^2: 0.9997). The assessment was performed in triplicate. The measure of phenolic content in extracts was determined by the accompanying equation:

$$T = C1 \times V/M$$

Where, T = Total phenolic content mg g^{-1} of extracts in GAE [Gallic acid equivalent]; C1 = The Concentration of Gallic acid established from the calibration curve mg mL^{-1}; V = The Volume of extract solution [mL] M = The Weight of the extract [g].

2.6. Analysis of Phenolic Components

The assay for quantification of the phenolic compounds has previously been described by Caponio et al., (1999). The reversed phase-high performance liquid chromatography (RP-HPLC) was used. The analytical HPLC system

employed consisted of a a SCL-10 Avp System controller, a SIL–10AD vp Autosampler, a LC-10AD vp pump, a DGU-14a degasser, a CTO-10 A vp column heater and a diode array detector set at 278 nm. The separation was achieved on a Agilent Eclipse 5µm XDB 250 x 4,6 mm column at 30°C. The flow rate was 0.8 ml/min, injection volume was 20 µl. Gradient elution of two solvents was used: Solvent A consisted of acetic acid–water (3:97, v/v), solvent B: methanol and the gradient program used. The analytical data were evaluated using a Shimadzu Class-VP Chromatography Laboratory Automated Software System (Chiyoda-ku, Tokyo, Japan). The gradient used was similar to that used for the determination of phenolics in sage and rosemary (Baydar et al., 2007) with some modifications. The amount of phenolic compounds in the extract was calculated as mg 100 g^{-1} dried rhizome using external calibration curves, constructed for each phenolic standard.

2.7. 1,1-diphenyl-2-picryl-hydrazyl (DPPH•), free radical scavenging activity

The free-radical-scavenging capacity of the extracts was evaluated, using the DPPH stable radical and following the methodology described by Gulçin (2006). Briefly, 0.1mM solution of DPPH• in ethanol was prepared and 1ml of this solution was added to 3 mL of EEG or EET solution in ethanol at different concentrations (10-20 µg/mL). After 30 min, the absorbance was measured at 517 nm against ethanol as a blank in a spectrophotometer (Perkin Elmer Lambda 20 UV VIS Spectrophotometer). The lower the measured absorbance value of the reaction mixture, the higher the free radical scavenging potential.

The ability to sweep the DPPH• radical was counted up using the following equation:

DPPH• scavenging effect (%) = [(AControl − ASample / AControl) × 100]

where AControl is the absorbance of the control reaction (ethanol solution containing 0.1 mM DPPH) and ASample is the absorbance in the presence of ginger and turmeric extracts and standarts (BHT and BHA) (Erdoğan and Gökçe, 2021).

2.8. Hydrogen peroxide scavenging activity

The hydrogen peroxide scavenging test was carried out following the procedure of Ruch (1989). The fundamental of this assay that there is a reduce in absorbance of H_2O_2 upon oxidation of H_2O_2. 43 mM hydrogen peroxide solution was prepared in phosphate buffer (pH: 7.4). EEG or EET at different concentrations (10- 20 µg /mL) in 3.4mL phosphate buffer was added to 0.6mL of H_2O_2 solution (43mM) and absorbance of the reaction mixture was recorded at 230 nm. The blank solution contained phosphate buffer solution free hydrogen peroxide.

The percentage of H_2O_2 scavenging by EEG, EET and standard compounds was calculated using the following equation:

H_2O_2 scavenging effect (%) = [(AControl − ASample / AControl) × 100]

where Ac is the absorbance of the control and As is the absorbance in the presence of EEG, EET or other scavengers (BHA and BHT) (Benkeblia, 2005).

2.9. Ferric cyanide (Fe^{3+}) reducing antioxidant power assay

The reducing capacity (RP) of the extracts was assessed as described by Oyaizu (1986). The FRAP method is based on the reduction of (Fe^{3+}) ferricyanide in stoichiometric excess relative to the antioxidants (Benzie and Strain, 1996). 1 mL of EEG or EET solution in ethanol at different concentrations (250-1000 µg/mL) was added to 2.5 mL of 0.2 M sodium phosphate buffer (pH 6.6) and 2.5 mL of 1% potassium ferricyanide [K_3 Fe $(CN)_6$] solution. The reaction mixture was thoroughly mixed and the mixture was then left in an ultrasonic water bath for 20 min at 50 ºC. At the end of the incubation, 2.5 mL of 10% trichloroacetic acid was added to the mixture and centrifuged at 2000 rpm for 10 min. The supernatant (2.5 mL) was mixed with 2.5 mL of distilled water and 0.5 mL of 0.1% ferric chloride. The colored solution was read at 700 nm against the blank with reference to standard using UV Spectrophotometer. Ascorbic acid was used as a reference standard. It was noteworthy that the iron reducing power of the samples was comparable to that of standard reference. Higher absorbance indicated greater reducing capacity.

2.10. Statistical analysis

The presented data (mean ±standard deviation) resulted from at least three independent experiments and analyzed by SPSS (version 17 for Windows 10 pro, SPSS Inc.). One-way analysis of variance (ANOVA) was performed by standard methodology and $p < 0.05$ was considered significant and $p < 0.01$ was very significant.

3. RESULTS AND DISCUSSION

3.1. Total phenolic content and ethanolic extraction yield

Extraction efficiency of turmeric and ginger powders and the mean values of total phenolics (TPC) in EET and EEG investigated in this study are presented in (Table 1). The total phenolic content of EET and EEG was found to be 82.47±2.70 and 48.56±1.64 respectively (mg GAE/g). As per the ongoing reports, a profoundly positive connection between total phenols and antioxidant capacity was found in many plant species (Velioglu et al., 1998). As it can be seen in Table 1, the extraction efficiency of GP (5.58 %) is higher than TP (3.71 %).

Table 1. Extraction efficiency of dry rhizomes of turmeric and ginger and total phenolic content (TPC) in EET and EEG.

	Extraction yield (% in 100 g)	Total phenolic contents (mg GAE/g)
TP	3.71	-
GP	5.58	-
EET	-	82.47 ± 2.70
EEG	-	48.56 ± 1.64

TP: Turmeric powder, GP: Ginger powder, EET: ethanolic extract turmeric, EEG: ethanolic extract ginger

3.3 HPLC analysis of phenolic compounds

The amounts of phenolic compounds detected in the samples are presented in Table 2. Results are expressed in mg 100 g^{-1} of dry sample. The most abundant phenolic components in turmeric rhizomes are ferulic acid (93.59 mg 100 g^{-1} dry sample), benzoic acid (40.09 mg 100 g^{-1} dry sample), vanillin (26.69 mg 100 g^{-1} dry sample dry sample) and p-coumaric acid (23.25mg 100 g^{-1} dry sample) respectively. Other phenolic components were identified as Caffeic acid (6.75 mg 100 g^{-1} dry sample) and Chlorogenic acid (4.37 mg 100 g^{-1} dry sample) respectively. On the other hand, the most common phenolic components in ginger rhizomes are Benzoic acid (33.31 mg 100 g^{-1} dry sample), Ferulic acid (11.41 mg 100 g^{-1} dry sample) and vanillin (11.83 mg 100 g^{-1} dry sample) respectively. Other phenolic components were identified as Caffeic acid (6.71 mg 100 g^{-1} dry sample), Chlorogenic acid (3.99 mg 100 g^{-1} dry sample), Cinnamic acid (3.46 mg 100 g^{-1} dry sample) and Syringic acid (2.12 mg 100 g^{-1} dry sample). From the results, it was observed that the phenolic component contents of turmeric and ginger rhizomes are partially similar.

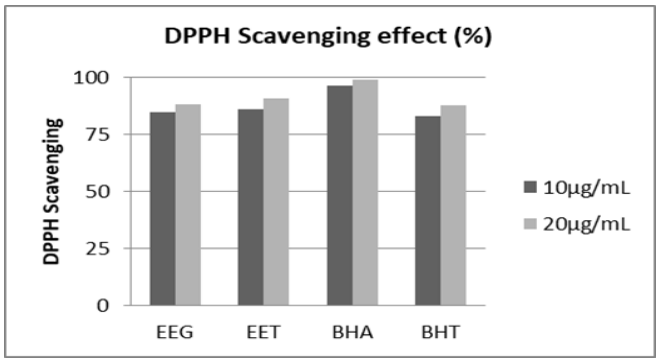
Table 2. Phenolic compounds of Ginger and Turmeric

Phenolic Compound	HPLC Retention Time (min)	Ginger (mg $100g^{-1}$of dry sample)	Turmeric (mg $100g^{-1}$of dry sample)
Chlorogenic acid	14.6	3.99	4.37
Caffeic acid	17.3	6.71	6.75
Syringic acid	19.9	2.12	-
Vanillin	21.0	11.83	26.69
p-Coumaric acid	24.5	-	23.25
Ferulic acid	28.1	11.41	93.59
Benzoic acid	34.8	33.31	40.09
Cinnamic acid	66.7	3.46	-

3.4. DPPH radical scavenging activity

In this study, free radical scavenging activities of EEG, EET and standards such as BHA and BHT were determined using a DPPH method. DPPH is often used to evaluate the free radical scavenging effects of different antioxidant substances (Ozcelik *et al.*, 2003). In the radical formation, this molecule had an absorbance at 517 nm which vanished after receipt of an electron or hydrogen radical from an antioxidant compound to become a stable diamagnetic molecule (Matthäus, 2002) Figure 1 displayed a significant decrease ($p < 0.01$) in the concentration of DPPH radical due to the scavenging ability of EET, EEG and standards. BHA and BHT were used as standarts for free radical scavengers. The scavenging effect of EET, EEG and standards on the DPPH radical decreased in the order of BHA > EET >EEG> BHT which were 98.8%, 90.9%, 88.3% and 87.8%, at the concentration of 20µg/mL, respectively. Free radical scavenging activity of these samples also increased with an increasing concentration.

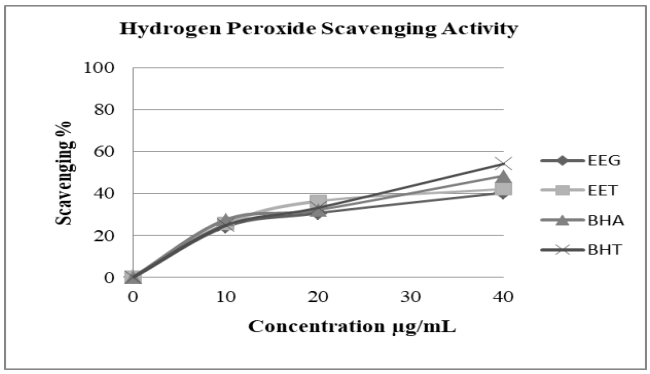
Figure 1. Scavenging effect of EET, EEG, BHA and BHT, on the stable DPPH• at different concentrations (10–20 µg/mL) (EET: ethanolic extract turmeric, EEG: ethanolic extract ginger, DPPH•: 1,1-diphenyl-2-picryl-hydrazyl free radicals, BHA: butylated hydroxyanisole, BHT: butylated hydroxytoluene, Data expressed as mean± S.D (n=3).



3.5. Hydrogen peroxide scavenging effects

The scavenging ability of EEG and EET on H_2O_2 is shown in Figure 2 and compared with BHA and BHT as standards. 40 µg/mL of EEG and EET exhibited 40.28 ± 0.73 and 42.04 ± 0.37 % scavenging activity ($p < 0.05$) on H_2O_2, respectively. However, BHA and BHT showed 48.32 ± 0.08 and 54.03 ± 1.39 % H_2O_2 scavenging activity at the same concentration. These findings demonstrated that EEG and EET had powerful hydrogen peroxide scavenging activity. The H_2O_2 scavenging effects 40 µg/mL concentration of EEG and EET and standards decreased in the order of BHT > BHA > EET > EEG. However, as the concentration of EEG and EET increases, radical scavenging activity in hydrogen peroxide increases.

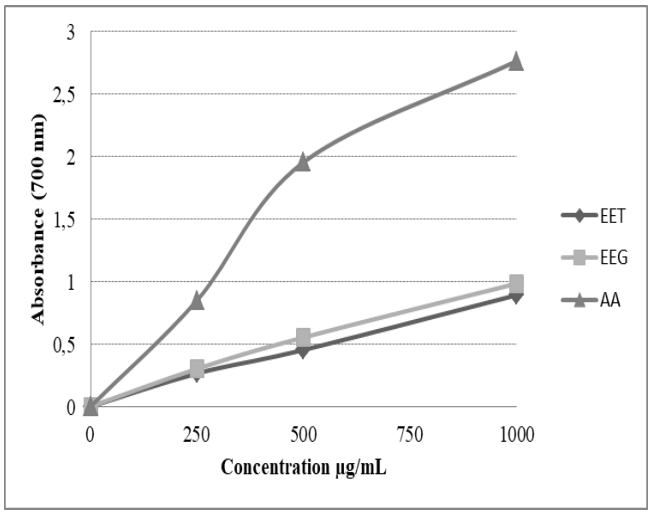
Figure 2. Scavenging effect of EET, EEG, BHA and BHT on H_2O_2, at different concentrations (10–40 µg/mL) (EET: ethanolic extract turmeric, EEG: ethanolic extract ginger, BHA: butylated hydroxyanisole, BHT: butylated hydroxytoluene, H_2O_2: Hydrogen Peroxide, The Scevenging (%) was the mean ± SD (n =3) $p < 0.05$).



3.6. Total reductive capability using the potassium ferricyanide reduction method

In this method, the reducing capacity of the extracts (EET and EEG) were accomplished using Fe^{3+} to Fe^{2+} reduction assay. In this analysis, yellow color turned pale green and blue color depending on the concentration of antioxidant capacity in the samples. The ability of a constituent to reduce iron may serve as a significant indicator of its potential antioxidant activity. As can be seen from Figure 3, EEG and EET had effective reducing power using the potassium ferricyanide reduction method when compared to the standard. At different concentrations (250–1000 µg/mL), EEG and EET demonstrated powerful reducing ability (r^2: 0.9973 and r^2: 0.9935, respectively) and these differences were statistically very significant ($p < 0.01$). The reducing power of EEG, EET and AA were increased with increase of sample concentrations. Reducing power ability of EEG, EET and standard compound exhibited the following order: AA >EET> EEG.

Figure 3. Total reductive potential of different concentrations (250–1000µg/mL) of EEG (r^2: 0.9973), EET (r^2: 0.9935), and reference antioxidant: ascorbic acid using spectrophotometric detection of the Fe^{3+}–Fe^{2+} transformations. In the presence of reductants, Fe^{3+}/ferricyanide complex reduces to the ferrous form (EET: ethanolic extract turmeric, EEG: ethanolic extract ginger, AA; ascorbic acid, Absorbance was the mean ± *SD* (n =3) $p < 0.01$).



3.7. Mineral content

Mineral content of the studied samples is given in Table 3. Elemental analysis of both ethanol extracts and powder of ginger and turmeric rhizomes were performed. The potassium was, quantitatively, the most abundant mineral in samples. Subsequently, sodium, magnesium phosphorus and calcium were identified, respectively. While the amount of these elements is higher in ginger and turmeric rhizomes, it decreased in EEG and EET.

Table 3. Mineral analysis of samples (Al, Ca, Cd, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, P, Pb, Se and Zn) (mg element g^{-1})

Samples mg element g^{-1}				
Element	GP	TP	EEG	EET
Al	0.102 ± 0.0020	0.159 ± 0.0015	<0.012	<0.012
Ca	0.635 ± 0.0081	0.666 ± 0.0097	< 0.025	< 0.025
Cd	<0.005	<0.005	<0.005	<0.005
Cr	<0.004	<0.004	<0.004	<0.004
Cu	0.003 ± 0.0001	0.002 ± 0.0001	0.005 ± 0.0000	0.001 ± 0.0000
Fe	0.081 ± 0.0001	0.175 ± 0.0015	0.001 ± 0.0000	<0.010
Hg	<0.051	<0.051	<0.051	<0.051
K	13.84 ± 0.189	24.73 ± 0.173	1.388 ± 0.0129	3.782 ± 0.0240
Mg	1.686 ± 0.0115	1.582 ± 0.0181	0.002 ± 0.0001	0.018 ± 0.0000
Mn	0.244 ± 0.0015	0.019 ± 0.0002	0.001 ± 0.0000	<0.002
Mo	<0.003	<0.003	<0.003	<0.003
Na	3.069 ± 0.0336	0.406 ± 0.0091	0.889 ± 0.0103	0.315 ± 0.0038
P	0.996 ± 0.0074	1.584 ± 0.0133	0.304 ± 0.0019	0.090 ± 0.0003
Pb	< 0.030	< 0.030	< 0.030	< 0.030
Se	< 0.021	< 0.021	0.001 ± 0.0001	< 0.021
Zn	0.010 ± 0.0001	0.003 ± 0.0001	0.002 ± 0.0000	0.001 ± 0.0000

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4. CONLUSION

As indicated by the consequences of this investigation, it is obviously show that EEG and EET have high antioxidant capacity, high mineral profile and free radical scavenging potantial against different antioxidant systems in vitro. EEG and EET were discovered to be a powerful antioxidant in different in vitro assays including: RP, DPPH•, and hydrogen peroxide scavenging capacity when compared to standard antioxidant compounds such as BHA, BHT and ascorbic acid. Both the powders of turmeric and ginger rhizomes and their extracts have been found to be rich in bioactive components and mineral content. These essays have significant applications for the food and pharmaceutical industry. It should be encouraged to produce and novel products containing turmeric and ginger extracts, which have high antioxidant activities and are rich in bioactive substances.

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Effects of Activated Carbon on Medium Density Fiber Board Properties

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Abstract: The negative effects of formaldehyde emission in MDF production on human health are known, and therefore many scientists are working to reduce formaldehyde emissions. In this study, the effects of MDF plates on formaldehyde emission were investigated by adding different amounts of activated carbon into urea formaldehyde resin during the synthesis phase. First, the gelation time behavior of the resin was studied by observing the pH, gelation time, solid content, flow time and viscosity of the modified resin in comparison to the standard reference resin which has no activated carbon inside. The dosing of the activated charcoal in the dry resin was kept at 1wt%, 3wt% and 5wt%. After that modified resin was used in the production of 40x40 cm^2 MDF samples by using laboratory scale press line with full automation system. İnternal bonding strength, surface soundness, screw holding resistance, water absorption and thickness swelling were also measured in addition to the main interested parameter formaldehyde emission level which is determined via spectrometric technique following an extraction procedure. Threshold values for activated carbon were determined to be 1wt%. Formaldehyde emission level was observed where addition of 1wt% activated carbon into the urea formaldehyde adhesive decreased the formaldehyde emission 52% comparison to reference whereas addition of activated carbon at above its threshold level provided 47% decreasing.

Keywords: activated carbon, formaldehyde emission, MDF, adsorbent.

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1. INTRODUCTION

The increase in diseases in the world has increased the awareness of the chemical hazards that will come from the products used. For this reason, it has become an important issue to reduce the chemicals released over time from the products that are constantly used in furniture industry. Medium density fiber boards (MDF) that are used different areas school, home, etc. is an important wood panel industry composite material consisting urea formaldehyde. Urea formaldehyde resin has an important use in wood panel industry. This resin is preferred because it is cheap and transparent but ıt has disadvantages. These disadvantages are low water resistance and high formaldehyde emission that is measured from MDF. As it known, formaldehyde has many negative effects on human health. One important among these is the increasing risk of cancer. In 2004 the Internal Agency for Research on Cancer (IARC) classified formaldehyde as harmful chemical for human body (Pizzi 1994; Beram and Yasar, 2020).

Formaldehyde is used in the production of urea formaldehyde resins used in the MDF industry and depending on the reaction conditions between urea and formaldehyde, some amount formaldehyde can remain in the environment without reacting. In addition, some formaldehyde is released due to bond formation in the condensation stage of the resin, which develops during the pressing stage of the MDF production. Due to these reasons, some formaldehyde which is called free formaldehyde remains in the fiber board plate produced (Pizzi 1989).

Consequently, the use of formaldehyde on wood panels currently has been reduced to particular levels, regarded as not harmful to human health. Formaldehyde emission can be lowered by several methods. Several methods for producing low formaldehyde emission MDF panels have been studied, such as reducing formaldehyde to urea mol ratio and addition of formaldehyde scavengers in to resin. However mostly mechanical and physical properties of wood based panels have been affected badly. In addition to this, decreasing formaldehyde mol ratio causes to spread of curing time at MDF production. In this situation requires more energy and time. In the 21st century, where energy and time are important, this is an undesirable situation. The other factors affecting formaldehyde emission are wood type, resin type, type of hardeners, press conditions, amount

of resin used in MDF production and storage time. Moreover, modifications of the resin with different amine containing chemicals are also important to reduce formaldehyde emission.

Activated carbons have been used as adsorbents in various fields, for instance, solvent recovery, gas separation, and deodorization. The activated carbon is characterized by a strong adsorption capacity which is attributed to its large internal surface area, porosity and high degree of surface reactivity. In related to this the use of activated carbon is one of the possible methods to reduce formaldehyde emission (Kumar et al., 2013).

The use of activated carbon as formaldehyde absorbent has been analyzed by many researchers rayon based activated carbon as formaldehyde absorbent and activated charcoal have been used as bio-scavenger for decreasing formaldehyde emission from melamine formaldehyde resin.

As the relevance, in this work aimed to investigate the effect of activated carbon addition in to urea formaldehyde resin properties, formaldehyde emission values of MDF, mechanical and physical properties of MDF.

2. MATERIALS AND METHODS

2.1. Materials

Urea and formaldehyde to be used in the resin synthesis was provided from AGT AĞAÇ SAN.TİC.A.Ş. Mixed wood fibers which contain of soft and hardwood fibers that were be used in the MDF productions were provided by AGT. The activated carbon powders that have 200 mesh particle size, 900-950 m^2/g surface area, iodine number greater than 900 and pH 8-10 were procured from ECS KİMYA.

2.2. Synthesis of urea formaldehyde resin

Urea formaldehyde resin synthesis basically; it is dived into two stages: an alkaline condensation stage in which mono-, di- and trimethylolureas forms are formed, and a condensation stage of the formed methylolureas in acid environment. Table 1 shows the basic steps of the resin manufacturing parameters.

In the synthesis process, 45% industrial type aqueous formaldehyde solution and powdered urea have been used. The mol ration of formaldehyde/urea was taken as 1.04/1.00. Pure water was added by weighing powder urea in the appropriate mole ratio into three-necked glass reaction ballon flask assembly, then placed in heated magnetic stirrer unit and set to heat at 40 °C. At this stage, the appropriate molar ratio of formaldehyde was added gradually and the pH of the reaction media was adjusted to 8.20 with 20% NaOH solution by weight. Reaction was continued at 40°C for 30 minutes. Then, for polycondensation, the pH was arranged with ~4.5 with formic acid. The reaction was continued at 90 °C for 100 – 120 minutes by controlling flow time of resin with DIN Cup 4. Finally, while the resin was cooled to 70 °C, its pH was adjusted to 8.5 and the reaction was continued for a while. Finally, vacuum drying was applied to the solution and resin was cooled to 40 °C, and the solid content of resin was reduced to 58% from %60 by weight.

In order for the synthesized 1.04 mol urea formaldehyde resin to cure sufficiently in the plate press stage, it must be used with a hardener. As the hardener 20% by weight aqueous ammonium chloride was used, constituting 4% by weight based on the resin solid content.

Table 1. Resin manufacturing parameters

Resin manufacturing parameters	
Parameters	**Values**
pH	8.20 ±10
Viscosity (cP@ 25°C)	160 cP±10 at 30 rpm
Flow time (second @ 25°C)	25±5
Gelation time (second)	60±5
Solid content (%)	58±1

2.3. Mixing of activated carbon with urea formaldehyde resin

To obtain a uniform dispersion of activated carbon powder in the urea formaldehyde resin, mechanical stirring with YOKEŞ VBR-600 high shear disperser mixer was done for 30 min at 1200 rpm by using cowls type blade. Activated carbon was added to the urea formaldehyde resin at 1%, 3% and 5% by weight according to the resin solid weight. The modified resin was named based on percent added as AC1, AC3 and AC5. AC0 indicates reference resin. AC0 shows that absence of activated carbon powder in the resin.

2.4. Characterization of physical properties of activated carbon containing urea formaldehyde resins

Viscosity measurements were done by Brookfield LV DV2T viscometer by using spindle no 1 at 30 rpm 25°C. Flow time measurements were done by DIN cup 4mm. Gelation time tests were done by using water bath at 100 °C with stirring according to related standard test method.

2.5. Preparation of medium density fiberboard and physical and mechanical testing

Table 2 shows the basic production parameters of MDF boards containing different amounts of activated carbon. The resin free wood fibers (a mixture of 15% beech wood fiber + 85% pine wood fiber) with an average moisture content of 30% were dried in an ındustrial oven for approximately 6 hours until 2% - 4% humidity was achieved. Theoretically the amount of dry fiber was calculated and activated carbon added urea formaldehyde resin was weighed as 12% according to dry fiber amount. Activated carbon added resin was sprayed onto the wood fibers with the help of a mixer with a nozzle system, and a homogenous glue fiber mixture was tried to be obtained. After a 3 g of fiber sample taken from the resinated wood fiber mixture and analyzed in a moisture analyzer and, it was determined that it had an average moisture content of 9% - 10%, and this value was appropriate for pressing. The glued fibers that activated carbon added resin were transferred into a 40x40 cm^2 mold with the help of a

vacuum suction unit and the preform was formed before the press. Then, it was transferred to the IMAL PAL laboratory press unit and pressed with a pressure of 120 N/cm^2 for 326 seconds. Table 3 shows all the details of the MDF that contains activated carbon added resin and for reference MDF. The boards were then conditioned to attain uniform moisture content in panels. After that, the boards were cutted and tested according to related standard test method for determining of internal bond strength (EN 319), Edge screw holding resistance (EN 320), and surface soundness (EN 311). Physical tests of samples as thickness swelling and water absorption (EN 317), moisture content determination were done (EN 322). The mechanical properties of MDF panels were evaluated according to TS EN 622-5. Internal bonding tests and other mechanical tests were done with universal testing machine (IMAL IB800 Board property tester)

Table 2. MDF manufacturing parameters with different loading activated carbon

MDF manufacturing parameters	
Parameters	**Values**
Size	400*400 mm
Thickness	17 mm ± 1
Target density	740±20 kg/m^3
Press Pressure	120 N/cm^2
Pressing Time	326 seconds
Press temperature (for both top and bottom plate)	190 °C
UF resin wt % of dry wood fibers	12wt%
Activated carbon wt % of solid resin content	1%, 3% and 5%
Number of boards for each type of concentrations	4

2.6. Formaldehyde emission testing

The formaldehyde emissions from MDF panels were evaluated using the EN-120 (perforator method). 100 g sample were put in a round bottomed flask that contain the 600 mL of toluene. The 1000 mL of distilled water was poured into the perforator attachment. The samples were boiled with the toluene for 2 hours. In this test method the distilled water absorbs the formaldehyde and the volatile organic compounds captured by the boiling toluene. Formaldehyde trapped by the water is then quantitatively determined using UV spectrophotometer.

3. Results

3.1. Effect of activated carbon on the resin physical properties

As shown in the table 3, increasing with amount of activated carbon increases the viscosity of urea formaldehyde resin and extended the gelation time period.

Table 3. Resin properties with addition activated carbon

Sample	**pH**	**Flow Time (second @ 25°C)**	**Gelation Time (second)**	**Viscosity (cP@ 25°C)**	**Solid Content (%)**
Reference	8.15	20.00	53	164	58,76
AC1	8.30	20.12	66	174	59,12
AC3	8.52	23.91	88	197	59,69
AC5	8.63	25.13	96	227	60,15

The reactivity of the UF resin depends on the amount of free formaldehyde which produces more acidic during the curing process when the hardener is added (Moslemi 2020). The pH values in Table 3 are the values measured only with the activated carbon, without the addition of hardener. Gelation time test were done with adding the hardener ammonium chloride solution. Since the pH of the resin medium is high, we expect the gel time to be extended. This situation was parallel to the literature. The high pH value of the activated carbon increased the pH value of the resin and extended the gel time period even after the addition of hardener, since the environment was not acidic enough with the increasing concentration of activated carbon.

Resin flow time, viscosity and solid content increased with the addition of activated carbon. Increasing the resin viscosity and flow time will decrease the resin fluidity and cause a decrease in the adhesive property. This situation may cause weakening of the mechanical strength of MDF (Anjum 2020).

3.2. Physical and mechanical properties of MDF panels

Physical tests were done as water absorption and thickness swelling for 24h. For the water absorption tests, the test results for the MDF samples coded as AC0, AC1, AC3 and AC5 are respectively; it is 48.13%, 49.19%, 45.92% and 53.1%. The results are shown that in figure1.

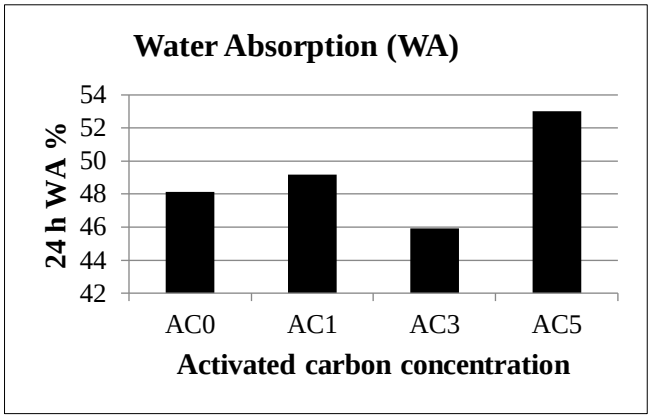

Figure 1. Water Absorption results of MDF panels

For the thickness swelling tests, the results for the MDF samples coded as AC0, AC1, AC3 and AC5 are respectively; as shown in figure 2, it is 19.48, 19.66%, 19.46% and 22.01%.

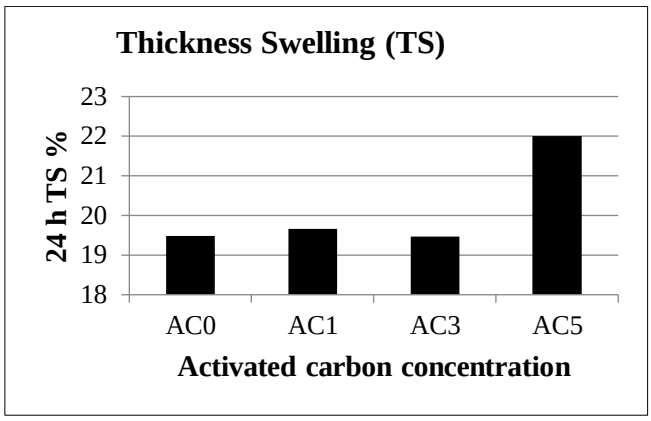

Figure 2. Thickness swelling results of MDF panel

When the results of MDF samples were evaluated, there was an increase of 0.91% in swelling value compared to the reference at 24 hours swelling tests with the addition of 1% activated carbon. There was a 10% decrease and 13% increase for the 3% and 5% concentrations respectively. Based on the results, it was observed that the addition of 1% activated carbon did not cause a significant increase in swelling value. Addition of 3% active carbon caused a decrease in swelling value. When all concentrations are evaluated ıt can be stated that the threshold value is 3% for the swelling test because the addition of 5% activated carbon negatively affected the swelling value of the system within increase of 13%. With the addition of activated carbon, the change in water absorption values at increasing values at increasing rates compared to the reference is an increase of 2.2%, a decrease of 4.6% and an increase of 10.4% respectively.

Table 4. Thickness swelling and water absorption values of MDF panels

Sample	Density kg/m^3	Moisture %	24 h TS %	24 h WA %
Reference	756.64	4.93	19.48	48.13
AC1	753.48	4.99	19.66	49.19
AC3	758.37	4.63	19.46	45.92
AC5	755.34	4.90	22.01	53.01

According to table 4 thickness swelling (TS) and water absorption (WA), activated carbon particle addition did not affect the moisture and density values of the MDF panels, and did not cause a significant change in TS and WA values. This was also observed in the study of in a literature (Kumar et al, 2013).

Mechanical tests were done as internal bonding resistance, surface soundness and screw holding resistance. For the internal bonding tests, the results for MDF samples coded as AC0, AC1, AC3 and AC5 are respectively it is 0.32 N/mm2, 0.34 N/mm2, 0.32 N/mm2 and 0.30 N/mm2. The results are shown in figure 3. The results for surface soundness tests are respectively 0.80 N/mm^2, 0.69 N/mm^2, 0.75 N/mm^2, and 0.84 N/mm^2. The results are shown in figure 4. For the screw holding resistance tests, the results for MDF samples coded as AC0, AC1, AC3 and AC5 are respectively it is 690.00 N, 737.50 N, 668.50 N and 660.75 N. The results are shown in figure 5.

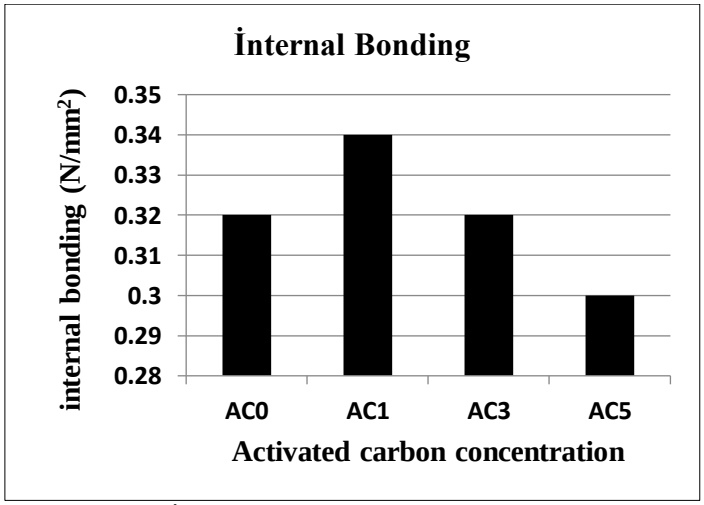

Figure 3. İnternal bonding results of MDF panels

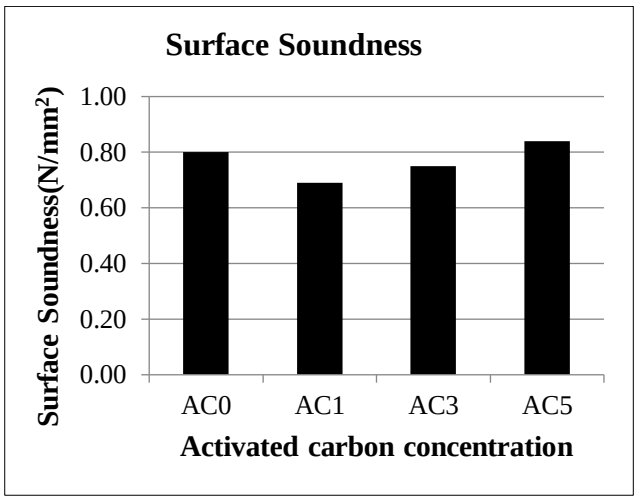

Figure 4. Surface soundness results of MDF panels

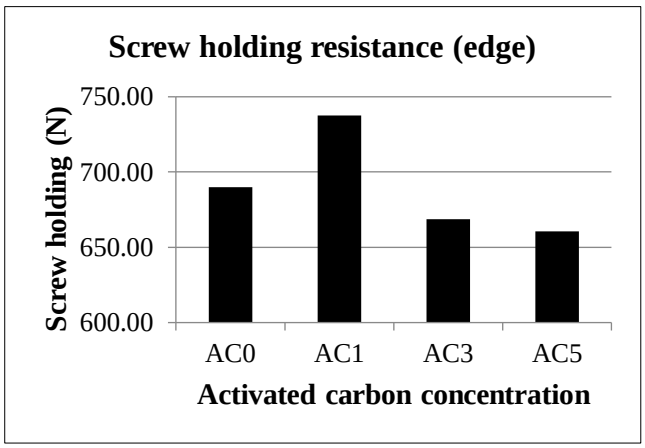

Figure 5. Screw holding resistance results of MDF panels

Mechanical tests were examined; there was a 6.25% increase in internal bonding values for the 1% concentration. With the addition of activated carbon at increasing concentrations for the 3% and 5% ratios, there was a 5.9% decrease and a 12.5% decrease in the internal bonding values, respectively. The screw holding resistance values are analyzed, an increase of 6.4%, a decrease of 3.1% and a decrease of 4.2% were observed, respectively compared to the reference at increasing concentrations. When the surface soundness test results are examined, it is 12.5% decrease, 6.25% decrease, 5% increase compared to reference at increasing rates respectively.

Based on the test results of MDF internal bonding strength, it can be deduced that MDF with less active carbon added mostly exhibits higher strength than control MDF. This can be explained by the fact that the incorporation of activated carbon in MDF fills the space between the fibers in the MDF, thereby intensifying the close contact of the fiber-carbon-fiber system, thereby strengthening the hydrogen bond and van der Waals forces. (Darmawan et al., 2010)

By holding the formaldehyde by the activated carbon, the free formaldehyde in the resin is prevented from escaping from the reaction medium during the curing of the formaldehyde. This strengthens cross-linking. However, the higher activated carbon loading (above 1%), results in less effective retention of formaldehyde due to the agglomeration of the activated carbon particles, the internal bonding strength is reduced. (Resmi et al., 2017)

3.3. Formaldehyde emission tests of MDF panels

Figure 6 shows the formaldehyde emission testing results by the perforator method. The formaldehyde emission tests were done with samples that having 5% moisture content. The value of formaldehyde emission of samples that are named as AC0, AC1, AC3 and AC5 are respectively 22.33, 10.60, 10.67 and 11.78 mg/100g board.

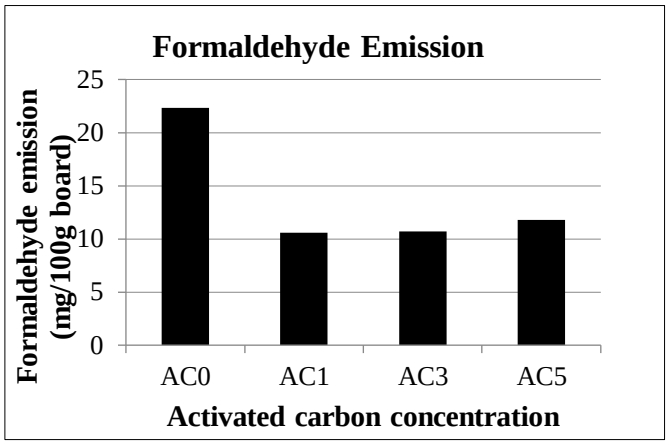

Figure 6. Formaldehyde emission results of MDF panels

According to formaldehyde emission test results, with the addition of activated carbon, a decrease of 52.5%, 52.2% and 47.2% was observed in the emission values, respectively.

That the addition of activated carbon into the resin system reduces the formaldehyde emission values for all concentrations was detected. Such lowering was caused by the capability of the microstructure of the activated charcoal to adsorb formaldehyde in the MDF (Rong et al., 2002; Pari et al., 2006; Beram et al., 2021). Further, the porous structure afforded greater surface area of the adsorbent (activated carbon) and the holding of the adsorbate (formaldehyde) by activated charcoal through the secondary force of hydrogen bonding as well as van der Waals type. This enhanced the intimate take-up of adsorbate on the surface of adsorbent, thereby intensifying the adsorption of formaldehyde by the activated carbon incorporated MDF.

Since the activated carbon used has an iodine number value of over 900 and surface area high that its adsorption capacity to be high based on the information in the literature was expected situation (Medek 2006)

4. Discussion and Conclusions

The main purpose of this study is to produce MDF panels that are sensitive to the environment and human health by reducing formaldehyde emission. For this reason, activated carbon, which is a good adsorbent due to its surface area

and porous structure, was used as filler in the urea formaldehyde resin system. The study also aimed to protect the mechanical and physical strength values while reducing the emission values. For this reason, the threshold value for the addition of activated carbon into the resin system has been tried to be determined.

In addition to the emission tests, when all mechanical and physical strength tests were examined, it was decided that the optimum activated carbon concentration was 1%. With the addition of 1% active carbon, the emission value was reduced, while the internal bonding strength, screw holding resistance were increased and a value close to the reference was obtained in surface soundness tests. For thickness swelling and water absorption values, we see that the addition of activated carbon does not cause a significant change compared to the reference.

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Farklı Elektrot Türlerinin Kullanıldığı Elektrokimyasal Prosesler ile Organik Atıksulardan Renk Giderim Veriminin Belirlenmesi ve İşletme Parametrelerinin Optimizasyonu

Oğuz Şahiner [1*], Murat Solak

Özet: Avrupa Birliği üyesi ülkeler endüstriyel atıksularda renk parametresinin analizinde EN ISO 7887'de belirlenen standartlar çerçevesinde renklilik sayısı (RES) yöntemini kullanmaktadır. Çalışmada, maya üreten işletmenin deşarj atıksuyunun RES birimi olarak titanyum ve paslanmaz çelik elektrotlar kullanılarak elektrokimyasal prosesle renk giderim verimi araştırılmıştır. Akım yoğunluğu, pH ve elektroliz süresi gibi işletme parametrelerinin RES436, RES525 ve RES620 renk parametrelerinin giderimi üzerindeki etkileri Yanıt Yüzey Metodu (YYM) ile optimize edilmiştir.

Keywords: Maya endüstrisi deşarj atıksuyu, elektrokimyasal arıtma, titanyum elektrot, paslanmaz çelik elektrot, optimizasyon, yanıt yüzey metodolojisi.

Determination of Color Removal Efficiency from Organic Wastewaters with an Electrochemical Processes Using Different Electrode Types and Optimization of Operational Parameters

Abstract: European Union member countries use the spectral absorption coefficient (SAC) method within the framework of the standards set in EN ISO 7887 in the analysis of the color parameter in industrial wastewaters. In the study, the color removal efficiency of the discharge wastewater of the yeast production industry was investigated as SAC unit by electrochemical process using titanium and stainless steel electrodes. The effects of operating parameters such as pH, current density and electrolysis time on the removal of SAC436, SAC525 and SAC620 color parameters were optimized by the Response Surface Method (RSM).

Keywords: Yeast industry discharge wastewater, electrochemical treatment, titanium electrode, stainless steel electrode, optimization, response surface methodology.

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1. GİRİŞ

Gıda endüstrisi, artan temel ihtiyaçların karşılanması için stratejik öneme sahip, üretimin her aşamasında yüksek miktarlarda su ihtiyacı olan ve buna bağlı olarak da atıksu üretimi oldukça yüksek olan endüstriyel üretim alanlarından biridir. Gıda endüstrisi alanında faaliyet gösteren maya üretim tesisleri dünyada ve ülkemizde önemli bir yere sahiptir (Balcıoğlu, 2013). Ekmek mayası üretiminde, şeker fabrikalarının yan ürünü olan melas, endüstriyel sürdürülebilirlik yaklaşımlarından biri olan endüstriyel simbiyoz örneği olarak maya endüstrisinin hammaddesi olarak kullanılmaktadır.

Maya endüstrisi atıksuları, kimyasal oksijen ihtiyacı (KOİ), biyokimyasal oksijen ihtiyacı (BOİ), toplam organik karbon

(TOK), azot, fosfor, renk gibi yüksek konsantrasyonlarda kirletici içeriğe sahiptir (Balcıoğlu, 2013). Maya üretimi süreçlerinde ortaya çıkan atıksulardaki yoğun renk (koyu kahverengi) içeriği, biyopolimer kompleksi olan, biyokimyasal reaksiyonlar sonucu oluşan melanoidden kaynaklanmaktadır. Bu içeriklerin parçalanması oldukça zordur (Alkan, 2010). Bu tür atıksuların arıtımında biyolojik, kimyasal arıtma teknikleri kullanılmaktadır. Biyolojik prosesler, atıksudaki organik maddeleri parçalayan mikroorganizma yumaklarının çöktürme havuzunda çöktürerek giderimi üzerine tasarlanmıştır. Konvansiyonel biyolojik prosesler; Aktif çamur prosesleri, damlatmalı filtreler ve döner biyolojik disklerdir (Aydın, 2020). Ayrıca kimyasal koagülasyon, kimyasal çöktürme, elektrokoagülasyon ve fenton prosesi gibi prosesler de organik içerikli kirleticilerden renk gideriminde kullanılan proseslerdir (Haksevenler vd. 2014). Elektrokimyasal prosesler, kimyasal proseslere ve çeşitli kirleticilerin gideriminde kullanılan proseslere alternatif olabilecek ve son dönemlerde önemi artmış olan bir arıtma tekniğidir (Cansu, 2018). Elektrokimyasal arıtım prosesleri içerdikleri mekanizma anlamında koagülasyon, adsorbsiyon, absorbsiyon, çöktürme flotasyon, oksidasyon gibi proseslerin bir ya da bir kaçını kapsayabilmektedir (Ihara vd, 2004; İlhan vd. 2007). Elektrooksidasyon (EO) prosesi çözünmeyen bir anot malzeme kullanılarak organik maddelerin oksitlenmesini sağlamaktadır (Fil, 2004; Kul, 2005). EO prosesinde yaygın şekilde kullanılan elektrotlar grafit (Kannan vd. 1995), titanyum (Xion vd. 2003), paslanmaz çelik (Bejankiwar vd. 2005), bor kaplı elmas (Martínez-Huitle vd. 2008) gibi çözünmeyen elektrotlardır. EO prosesi temel olarak doğrudan veya dolaylı oksidasyon olmak üzere 2 farklı proses olarak gerçekleşebilir. Doğrudan oksidasyon (Anodik oksidasyon) prosesinde; kirleticiler anot yüzeyinde adsorbe edilir ve daha sonra anodik elektron transfer reaksiyonu ile ayrıştırılır. Dolaylı oksidasyon prosesinde reaksiyon hipoklorit / klor, ozon ve hidrojen peroksit gibi güçlü oksidantlar ile gerçekleşir (Alfredo vd. 2014).

Bu çalışmada paslanmaz çelik ve titanyum elektrotlar kullanılmıştır. Bu elektrotların kullanıldığı EO prosesi ile maya üretimi süreçlerinde ortaya çıkan, işletmenin arıtma tesislerinde arıtılarak deşarj standartlarına getirdiği ve alıcı ortama deşarj edilen atıksu alınarak, renk parametresi açısından tekrar kazanılabilirliğinin belirlenmesi için RES parametresi olarak renk giderim verimleri incelenmiştir.

2. MATERYAL VE YÖNTEM

2.1. Atıksu Karakterizasyonu

Deneysel çalışmalarda kullanılan atıksu maya üretim fabrikasının arıtma tesisi çıkışından alınmıştır (deşarj suyu). Ham atıksu karakterizasyonu Çizelge 1'de görülmektedir.

Çizelge 1. Ham atıksu karakterizasyonu

Parametre	Değer/ Konsantrasyon	Parametre	Değer/ Konsantrasyon
pH	7,66±0,2	Renk (m^{-1})	
İletkenlik (mS/cm)	6,43	RES436 (m^{-1})	6,81
TDS (mg/L)	3,97	RES525 (m^{-1})	5,21
KOİ (mg/L)	300±10	RES620 (m^{-1})	4,14

2.2. Optimizasyon Çalışmaları

Çalışmada model reaktör ile maya üretimi yapan ve SKKY kapsamındaki Yönetmelik değerlerini sağlayan bir işletmenin deşarj noktasından atıksu numunesi alınmıştır. Alınan atıksu numunesinden renk giderimi üzerine elektroliz süresi, pH ve akım yoğunluğu gibi işletme parametrelerinin etkisi araştırılmıştır. Araştırmalarda parametre aralıkları literatür taraması ve ön deneysel çalışmalar ile belirlenmiştir. Parametrelerin etkin giderim aralıklarını belirlemek amacıyla, pH 4,5-10,5, akım yoğunluğu 90-150 A/m^2 ve elektroliz süresi 30-60 dk. aralığında olacak şekilde istatistiksel analize göre hazırlanan deney serisi uygulanmıştır (Çizelge 2). Optimizasyon çalışmalarında Yanıt Yüzey Metodu kullanılmıştır. 3D grafikler ve ANOVA analizi Design Expert programı ile hazırlanmıştır.

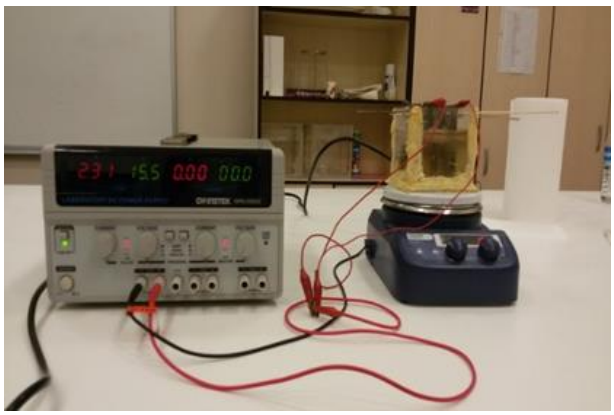
Çizelge 2. Farklı elektrot türleri için aralıklar

Faktörler	Titanyum	Paslanmaz Çelik
pH	4,5-9,5	4,5-9,5
Akım Yoğunluğu (A/m^2)	80-140	60-120
Elektroliz Süresi (dk.)	30-60	15-75

2.3. Deney Düzeneği

Renk gideriminin elektrokimyasal proses ile yapıldığı deneysel çalışmalarda, voltaj ve akım kontrolü DC güç kaynağı ile sağlanmıştır. Deneylerde kullanılan model reaktörün hacmi 500 ml'dir. Elektrotların boyutları 50*80*0.5 mm olup, su içerisinde elektroliz işleminin gerçekleştiği bölümün boyutları 50*55 mm'dir (165 cm^2 aktif yüzey alanına sahip elektrotlar için 90, 120 ve 150 A/m^2 akım yoğunlukları için hesaplanan ve sisteme verilen akım sırasıyla, 1,5A, 2A ve 2,5 A'dir).

Elektrokimyasal proses ile maya endüstrisi deşarj atıksularından renk giderimi çalışmasında kullanılan prosesin şematik gösterimi Şekil 1'de verilmiştir.



Şekil 1. EO prosesinin şematik gösterimi

2.4. Metot

2.4.1. Renk tayini

Maya endüstrisi arıtma sonrası deşarj sularından renk giderim veriminin belirlenmesi amacıyla RES metodu kullanılmıştır. Bu metot ile ölçümde 3 farklı renk analizi gerçekleştirilmektedir (Remazol Yellow RR gran için 436 nm, Remazol Red RR gran için 525 nm, Remazol Blue RR gran için 620 nm dalga boylarında ölçüm yapılır). Sonuçlar m^{-1} birimi olarak RES-436, RES-525 ve RES-620 şeklinde belirlenmektedir ((EN ISO 7887) (EPA 2009)).

Hach Lange DR6000 model spektrofotometrede 436, 525, 620 nm dalga boylarında numunenin absorbans değerleri okunmuş, bu absorbans değerleri Denklem 1'de yerine konularak, RES 436, RES 525, RES 636 değerleri hesaplanmıştır.

$$RES = \frac{A}{d}.f \qquad (1)$$

A: λ dalga boyunda çözeltinin absorbansı (cm^{-1})
d : Küvet kalınlığı (mm)
f: Spektral absorbans değeri m^{-1} birimi için faktör, f=1000
RES(λ): λ dalga boyundaki renklilik sayısı (RES) değeri (m^{-1})

2.4.2. KOİ, pH, iletkenlik tayini

KOİ analizi SM 5220-D metoduna göre Hach DR6000 model spektrofotometre kullanılarak, pH ve iletkenlik ölçümleri elektrometrik metoda (Standard Metod 4500-H^+) göre Hanna model cihaz ile belirlenmiştir (APHA, 2005).

3. BULGULAR

Titanyum Elektrot bağlı Elektrokimyasal Proses için optimizasyon çalışmalarında elde edilen RES436, RES525 ve RES620 renk parametreleri için ANOVA analizi sonuçları Çizelge 3'te verilmiştir.

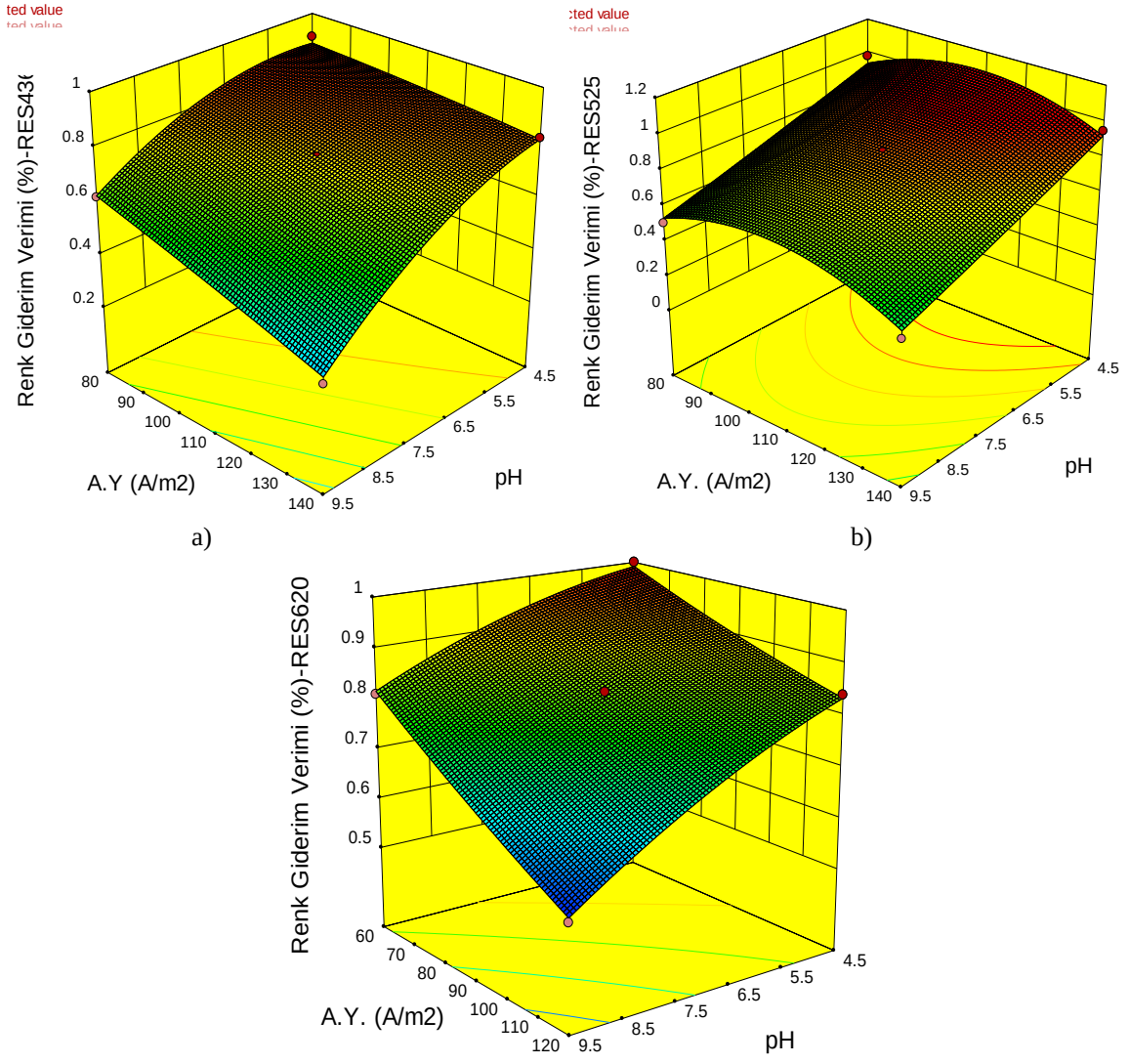
ANOVA analizinde, p değerlerine bakıldığında, RES436 renk değeri için pH ve akım yoğunluğu parametrelerinin elektroliz süresine göre daha etkin olduğu söylenebilir ($p<0,05$). RES525 renk değeri için sadece pH değerinin etkin olduğu, RES620 renk değeri için de pH ve akım yoğunluğu parametrelerinin prosesi etkileyen parametreler olduğu belirlenmiştir. 3D grafiklerde de bu tespitler görülmektedir.

Quadratik modele uyumlu olduğu belirlenen istatistiksel analiz sonucunda R^2 değerleri RES436 için 0.99, RES525 için 0.99 ve RES620 için 0.98'dir.

Şekil 2'de RES436, RES525 ve RES620 renk giderim verimleri için pH ve akım yoğunluğu parametrelerinin etkisi görülmektedir. Buna göre, pH değerinin tüm renk parametreleri gideriminde etkin olduğu görülmüştür. Akım yoğunluğunun artması bir noktaya kadar RES525 renk giderim verim artışı ile paralel hareket etmiş yaklaşık 100 A/m^2 akımdan sonra giderim verimi duraklamıştır. RES436 ve RES620 renk gideriminde akım yoğunluğunun artması ANOVA analizinde de görüldüğü üzere giderim verimini olumsuz yönde etkilemiştir. Titanyum elektrot ile tekstil endüstrisi atıksuyunun elektrokimyasal yöntem ile arıtılmasının incelenmiş olduğu bir çalışmada, 18 dakikalık elektroliz süresinden sonra KOİ, BOİ ve renk parametreleri için giderim verimi %80'in üzerinde olduğu belirlenmiştir (Kocaer vd. 2002; Vlyssides vd. 2000).

Çizelge 3. Titanyum için ANOVA analizi sonuçları

Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**p-value**
RES 436					
Model	0.59	9	0.066	49.66	0.0002
A-pH	0.25	1	0.25	189.51	<
B-A.Y	0.054	1	0.054	40.94	0.0014
C-E.S.	2.000E-004	1	2.000E-004	0.15	0.7141
AB	9.025E-003	1	9.025E-003	6.79	0.0480
AC	2.500E-005	1	2.500E-005	0.019	0.8963
BC	0.021	1	0.021	15.81	0.0106
A^2	0.024	1	0.024	18.33	0.0079
B^2	1.442E-004	1	1.442E-004	0.11	0.7553
C^2	0.24	1	0.24	182.29	<
Residual	6.650E-003	5	1.330E-003		
Lack of Fit	6.650E-003	3	2.217E-003		
Pure Error	0.000	2	0.000		
Cor Total	0.60	14			
R^2	0.99				
Adj R^2	0.97				
RES 525					
Model	1.11	9	0.12	53.39	0.0002
A-pH	0.37	1	0.37	159.86	<
B-A.Y	4.050E-003	1	4.050E-003	1.75	0.2431
C-E.S.	0.011	1	0.011	4.86	0.0786
AB	9.000E-004	1	9.000E-004	0.39	0.5601
AC	3.600E-003	1	3.600E-003	1.56	0.2675
BC	0.096	1	0.096	41.54	0.0013
A^2	1.641E-004	1	1.641E-004	0.071	0.8006
B^2	0.093	1	0.093	40.01	0.0015
C^2	0.56	1	0.56	240.70	<
Residual	0.012	5	2.313E-003		
Lack of Fit	0.011	3	3.833E-003	115.00	0.0086
Pure Error	6.667E-005	2	3.333E-005		
Cor Total	1.12	14			
R^2	0.99				
Adj R^2	0.97				
RES 620					
Model	0.66	9	0.073	33.10	0.0006
A-pH	0.44	1	0.44	197.34	<
B-A.Y	0.017	1	0.017	7.73	0.0389
C-E.S.	2.000E-004	1	2.000E-004	0.090	0.7759
AB	0.026	1	0.026	11.56	0.0193
AC	2.500E-005	1	2.500E-005	0.011	0.9195
BC	6.250E-004	1	6.250E-004	0.28	0.6180
A^2	0.17	1	0.17	78.86	0.0003
B^2	1.131E-003	1	1.131E-003	0.51	0.5069
C^2	3.692E-004	1	3.692E-004	0.17	0.7000
Residual	0.011	5	2.215E-003		
Lack of Fit	0.011	3	3.692E-003		
Pure Error	0.000	2	0.000		
Cor Total	0.67	14			
R^2	0.98				
Adj R^2	0.95				



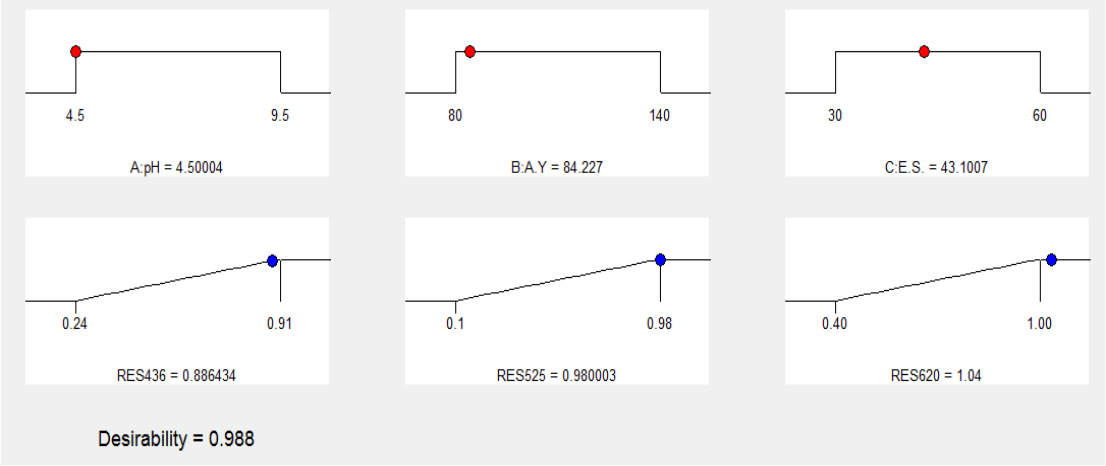
Şekil 2. Titanyum elektrotlar kullanılarak yapılan EO prosesinde akım yoğunluğu ve pH parametrelerinin renk giderim verimine etkisi a) RES 436 b) RES 525 c) RES 620 (Elektroliz Süresi: 45 dk.)

Yapılan çalışma sonucunda elde edilen denklemler
Çizelge 4'te verilmiştir.

Çizelge 4. Farklı renk değerleri için belirlenen eşitlikler

RES436		**RES525**		**RES620**	
+0.76		+0.87		+0.98	
-0.18	* A	-0.21	* A	-0.23	* A
-0.083	* B	-0.023	* B	+0.046	* B
-5.000E-003	* C	+0.037	* C	+5.000E-003	* C
-0.047	* AB	-0.015	* AB	+0.080	* AB
+2.500E-003	* AC	-0.030	* AC	-2.500E-003	* AC
+0.072	* BC	+0.16	* BC	-0.013	* BC
-0.081	* A^2	+6.667E-003	* A^2	-0.22	* A^2
-6.250E-003	* B^2	-0.16	* B^2	+0.018	* B^2
-0.26	* C^2	-0.39	* C^2	-0.010	* C^2

Şekil 3'te maksimum RES436, RES525 ve RES620 renk giderim verimi için optimum pH değeri 4.57, akım yoğunluğu 139.84 A/m^2, elektroliz süresi 58 dk. olarak tespit edilmiştir. Optimum koşullarda RES436 giderim verimi yaklaşık % 97 olarak, RES525 ve RES620 renk giderim verimleri ise % 99'un üzerinde olduğu tespit edilmiştir.



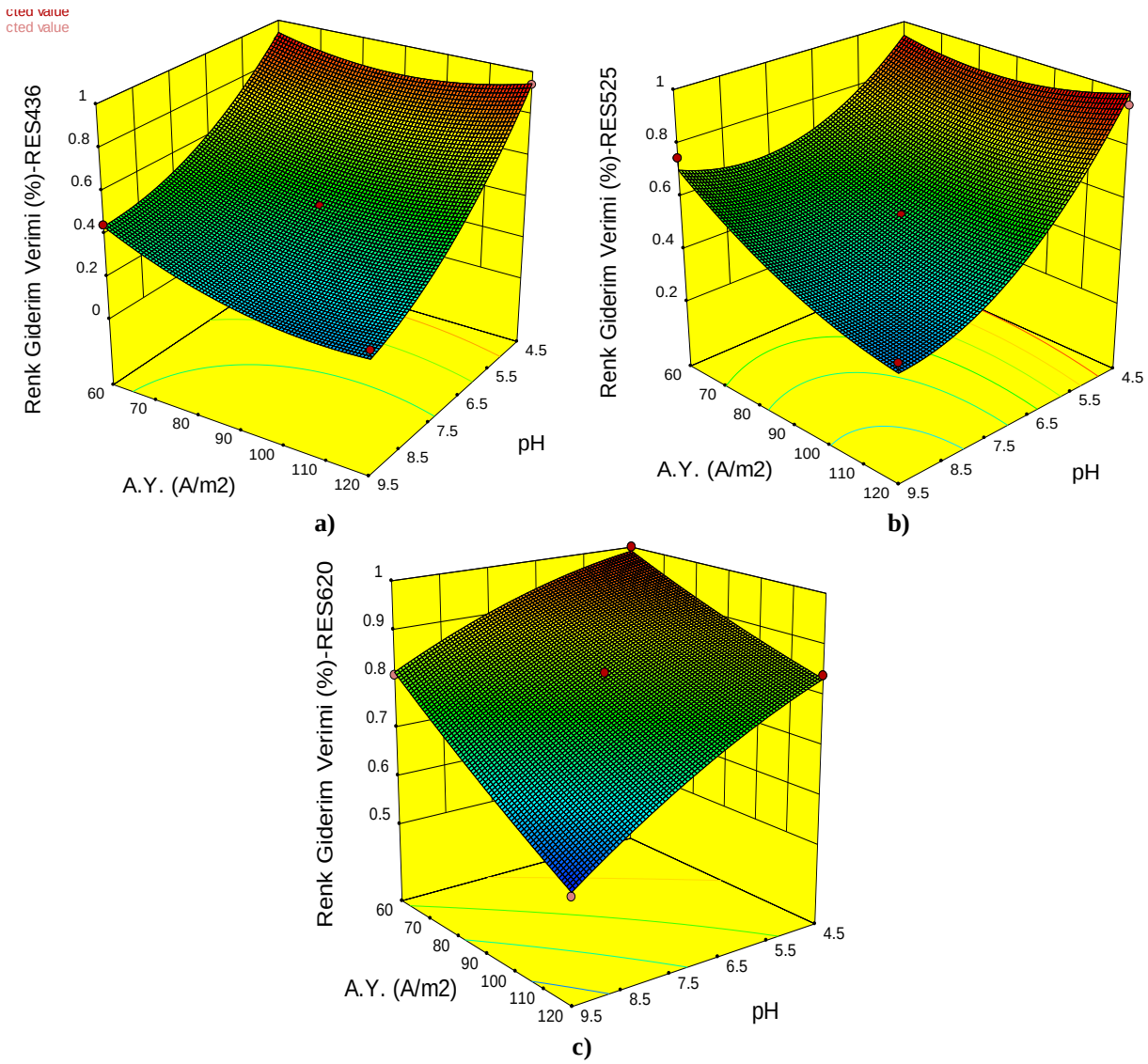
Şekil 3. Renk giderim verimlerini maksimize eden optimum değerler

Paslanmaz çelik elektrot bağlı elektrokimyasal proses için deneysel çalışma sonucunda elde edilen RES436, RES525 ve RES620 renk parametreleri için ANOVA analizi sonuçları Çizelge 5'te görülmektedir. Quadratik modele uyumlu olarak belirlenen istatistiksel analiz sonucunda R^2 değerleri RES436 için 0.99, RES525 için 0.97 ve RES620 için 0.99 olarak tespit edilmiştir.

ANOVA analizinde, RES436 renk değeri için akım yoğunluğu, pH, ve elektroliz süresi parametrelerinin p değerlerine bakıldığında, pH ve akım yoğunluğu parametrelerinin elektroliz süresine göre daha etkin olduğu görülmektedir ($p<0,05$). RES525 renk değeri için sadece pH değerinin etkin olduğu, RES620 renk değeri için de pH ve akım yoğunluğu parametrelerinin prosesi etkileyen parametreler olduğu belirlenmiştir. 3D grafiklerde de bu tespitler görülmektedir.

Şekil 4'te RES436, RES525 ve RES620 renk giderim verimleri için pH ve akım yoğunluğu parametrelerinin etkisi görülmektedir. Buna göre, pH değerinin tüm renk parametreleri gideriminde etkin olduğu, özellikle pH <5 olması durumunda renk giderim verimleri >%80 olarak tespit edilmiştir. RES436, RES525 ve RES620 renk giderim verimleri için akım yoğunluğu parametrelerinin artması giderim verimini olumsuz etkilemiştir. Çeliğin katot olarak, alüminyum ve demirin anot olarak kullanıldığı bir

elektrokimyasal proseste tekstil endüstrisinde kullanılan boyar madde bulunan numuneden renk giderimi incelenmiş olup; Elektroliz süresi, pH ve akım yoğunluğu gibi değişken parametrelerin renk giderim verimi üzerine etkileri araştırılmıştır. Akım yoğunluğu 2,5 mA/cm^2 olduğunda renk giderimi %20 iken, akım yoğunluğu 12.5 mA/cm^2 olduğunda renk giderimi %98 olarak gerçekleşmiştir. Bu proses için optimum akım yoğunluğu 11,25 mA/cm^2 olarak belirlenmiştir. pH'ın <2 olması durumunda en düşük giderim verimi elde edilmiştir. 5-9 arası renk giderim veriminde bir değişiklik olmazken, ph 9 dan sonra giderim verimi artmıştır (Daneshvar vd. 2007).



Şekil 4. Paslanmaz çelik elektrotlar kullanılarak yapılan EO prosesinde akım yoğunluğu ve pH parametrelerinin renk giderim verimine etkisi. a) RES 436 b) RES 525 c) RES 620 (Elektroliz Süresi: 30 dk.)

Çizelge 5. Paslanmaz Çelik için ANOVA analizi sonuçları

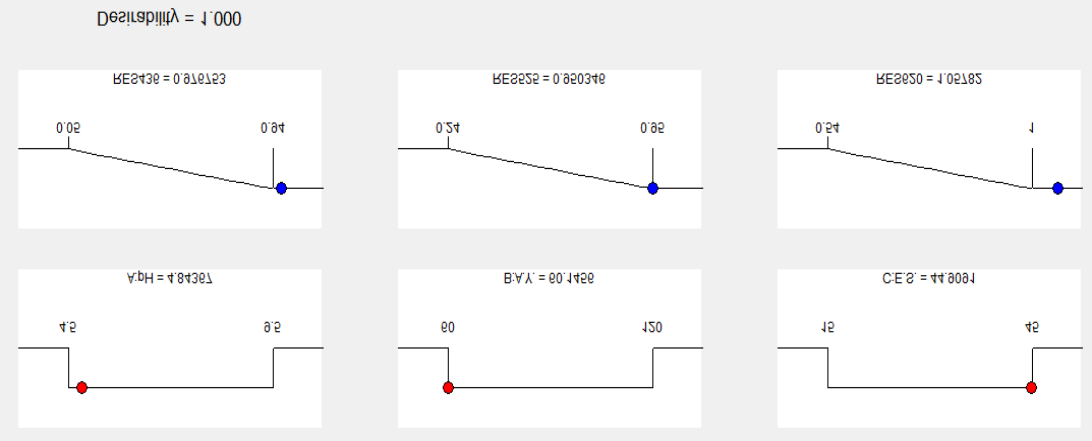
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob
RES 436					
Model	0.59	9	0.066	49.66	0.0002
A-pH	0.25	1	0.25	189.51	<
B-A.Y	0.054	1	0.054	40.94	0.0014
C-E.S.	2.000E-004	1	2.000E-	0.15	0.7141
AB	9.025E-003	1	9.025E-	6.79	0.0480
AC	2.500E-005	1	2.500E-	0.019	0.8963
BC	0.021	1	0.021	15.81	0.0106
A^2	0.024	1	0.024	18.33	0.0079
B^2	1.442E-004	1	1.442E-	0.11	0.7553
C^2	0.24	1	0.24	182.29	<
Residual	6.650E-003	5	1.330E-		
Lack of Fit	6.650E-003	3	2.217E-		
Pure Error	0.000	2	0.000		
Cor Total	0.60	14			
R^2	0.99				
Adj R^2	0.97				
RES 525					
Model	1.11	9	0.12	53.39	0.0002
A-pH	0.37	1	0.37	159.86	<
B-A.Y	4.050E-003	1	4.050E-	1.75	0.2431
C-E.S.	0.011	1	0.011	4.86	0.0786
AB	9.000E-004	1	9.000E-	0.39	0.5601
AC	3.600E-003	1	3.600E-	1.56	0.2675
BC	0.096	1	0.096	41.54	0.0013
A^2	1.641E-004	1	1.641E-	0.071	0.8006
B^2	0.093	1	0.093	40.01	0.0015
C^2	0.56	1	0.56	240.70	<
Residual	0.012	5	2.313E-		
Lack of Fit	0.011	3	3.833E-	115.00	0.0086
Pure Error	6.667E-005	2	3.333E-		
Cor Total	1.12	14			
R^2	0.99				
Adj R^2	0.97				
RES 620					
Model	0.66	9	0.073	33.10	0.0006
A-pH	0.44	1	0.44	197.34	<
B-A.Y	0.017	1	0.017	7.73	0.0389
C-E.S.	2.000E-004	1	2.000E-	0.090	0.7759
AB	0.026	1	0.026	11.56	0.0193
AC	2.500E-005	1	2.500E-	0.011	0.9195
BC	6.250E-004	1	6.250E-	0.28	0.6180
A^2	0.17	1	0.17	78.86	0.0003
B^2	1.131E-003	1	1.131E-	0.51	0.5069
C^2	3.692E-004	1	3.692E-	0.17	0.7000
Residual	0.011	5	2.215E-		
Lack of Fit	0.011	3	3.692E-		
Pure Error	0.000	2	0.000		
Cor Total	0.67	14			
R^2	0.98				
Adj R^2	0.95				

Yapılan çalışma sonucunda elde edilen denklemler Çizelge 6'da görülmektedir

Çizelge 6. Farklı renk değerleri için belirlenen eşitlikler

RES436		**RES525**		**RES620**	
+0.44		+0.53		+0.81	
-0.31	* A	-0.22	* A	-0.11	* A
-0.058	* B	-0.079	* B	-0.11	* B
+0.12	* C	+0.12	* C	+0.034	* C
-0.060	* AB	-0.10	* AB	-0.028	* AB
+0.035	* AC	+0.073	* AC	-7.500E-003	* AC
-0.035	* BC	-0.030	* BC	+0.015	* BC
+0.11	* A^2	+0.16	* A^2	-0.024	* A^2
+0.084	* B^2	+0.062	* B^2	+8.333E-003	* B^2
-0.026	* C^2	-0.023	* C^2	+0.048	* C^2

Şekil 5'te maksimum RES436, RES525 ve RES620 renk giderim verimi için optimum pH değeri 4,84, akım yoğunluğu 60,15 A/m^2, elektroliz süresi 44 dk. olarak tespit edilmiştir. Optimum koşullarda RES436 giderim verimi yaklaşık %97, RES525 giderim verimi yaklaşık % 95 ve RES620 renk giderim verimi > %99.99 olarak belirlenmiştir.



Şekil 5. Renk giderim verimlerini maksimize eden optimum değerler.

Optimize edilen koşullarda maya endüstrisi arıtım sonrası deşarj suyunun (deneysel çalışmalarda kullanılan ham atıksu) ve EO prosesi sonrası atıksudaki renk değişimi Şekil 6'da görülmektedir.

Şekil 6. İşletmenin deşarj atıksuyu ve EO prosesi ile arıtılmış s

4. TARTIŞMA VE SONUÇLAR

Yanıt yüzey metodu ile yapılan elektrokimyasal proseslerin optimizasyon çalışmalarında titanyum elektrot için akım yoğunluğunun 84.23 A/m^2, pH değerinin 4.55 ve elektroliz süresinin 43 dk. olduğu optimum şartlarda RES436, RES525, RES620 renk giderim verimleri sırasıyla % 89, % 98, % 99,99 olarak tespit edilmiştir. Paslanmaz çelik elektrot için pH değerinin 4.84, akım yoğunluğu 60.15 A/m^2, elektroliz süresi 45 dk. olduğu optimum şartlarda RES436, RES525, RES620 renk giderim verimleri sırasıyla % 98, % 95, % 99,99 olarak tespit edilmiştir.

İstatistiksel analiz sonucunda quadratik modele uyumlu olduğu belirlenen modelin, R^2 değerleri titanyum elektrotta RES436 için 0.99, RES525 için 0.99 ve RES620 için 0.98 olarak, paslanmaz çelik elektrotta RES436 için 0.99, RES525 için 0.97 ve RES620 için 0.99 olarak bulunmuştur. Akım yoğunluklarına bağlı olarak prosesin enerji tüketimleri ise sırasıyla, 34,2-122,2 kWsa/m^3 ve 4,64-42,86 kWsa/m^3 aralığında değişim göstermiştir. Aynı giderim verimleri için paslanmaz çelik elektrodunun kullanımının, titanyum elektrot kullanımına göre enerji maliyeti açısından daha uygun olacağı düşünülmektedir.

Titanyum ve paslanmaz çelik elektrotların kullanıldığı elektrokimyasal proseslerin maya endüstrisi deşarj sularındaki rengi oluşturan ve giderimi oldukça kompleks olan melanoidlerin parçalanarak giderilmesinde oldukça etkin olduğu belirlenmiştir. Özellikle, deşarj suyunun tüm renk parametreleri için giderim verimi > %89'dur. Bu durumda işletme tarafından arıtıldıktan sonra deşarj edilen suyun elektrokimyasal prosesler ile tekrar kullanım açısından arıtılabileceği ve arıtılan suyun işletmede tekrar kullanımı için uygulanan prosesler üzerine renk haricinde farklı parametreler ile de değerlendirmeler yapılması gerektiği düşünülmektedir.

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