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The Investigation of Possible Use of Olive Oil Production Wastes in Wool Dyeing

Zeytinyağı Üretim Atıklarının Yün Boyamacılığında Kullanım Olanaklarının Araştırılması

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Abstract

In this research, the dyeability of wool fabrics by using olive oil production waste was investigated. In this study, olive oil wastes were separated into two components as olive oil wastewater and pomace by filtration method. 100 % wool fabrics were dyed with conventional method with olive oil wastewater. In addition, the pre-mordanting method was preferred as the mordanting method. Alum, ferrous sulphate, tannic acid and tartaric acid were used as mordant materials, and various mordant concentrations and different mordant durations were studied. In dyeing, 100 % olive oil wastewater was used as dyeing liquor and all dyes were dyed in 1:20 liquor ratio. Color analyses of dyed fabrics were made, CIEL*a*b* and K/S values were examined. In addition, the coloring agents in olive oil wastewater were investigated by HPLC analysis. Light fastness, washing fastness, wet and dry rubbing fastness, acidic and alkaline perspiration fastness and water stain fastness were investigated.
Keywords: *Olive oil wastewater, Mordanting, Natural dye*

Öz

Bu araştırmada, yünlü kumaşların zeytinyağı üretim atığı kullanılarak boyanabilirliği araştırılmıştır. Bu çalışmada zeytinyağı atıkları filtrasyon yöntemiyle karasu ve pirina olmak üzere iki bileşene ayrıştırılmıştır. % 100 yün kumaşlar geleneksel yöntemle karasu ile boyanmıştır. Ayrıca mordanlama yöntemi olarak ön mordanlama yöntemi tercih edilmiştir. Mordan maddesi olarak alum, demir (II) sülfat, tannik asit ve tartarik asit kullanılmış, çeşitli mordan konsantrasyonları ve farklı mordan süreleri ile çalışılmıştır. Boyamada boyama banyosu olarak % 100 karasu kullanılmış ve tüm boyalar 1:20 flotte oranında boyanmıştır. Boyanan kumaşların renk analizleri yapılmış, CIEL*a*b* ve K/S değerleri incelenmiştir. Ayrıca HPLC analizleri ile de zeytinyağı atık suyunda bulunan renklendirici maddeler araştırılmıştır. Işık haslığı, yıkama haslığı, yaş ve kuru sürtme haslığı, asidik ve alkali terleme haslığı ve su lekesi haslığı incelenmiştir.
Anahtar Kelimeler: *Karasu, Mordanlama, Doğal boyama*

1. Introduction

Olive oil is the most important product of the olive plant in Turkey as well as all over the world [1]. It occurs in the by-products that are formed together with the main product during the processing of olive oil, and these by-products that pollute the environment are evaluated negatively in terms of the environment. By-products obtained from olive oil production are olive plant leaves, olive oil wastewater (black water) and pomace [2]. Pollution problems caused by the waste water generated during olive oil production are frequently encountered in our country and in the countries with a coast to the Mediterranean Sea, and approximately 5.4 × 10^6 m^3 of waste water is generated annually in parallel with the oil produced [3, 4]. A large amount of olive oil production waste occurs during the olive oil production phase. Olive oil factories in Turkey are generally small enterprises and can use solid waste (pomace) as fuel, but cannot recycle black water (olive oil waste water) [5, 6]. When olive oil wastewater is discharged to the environment without treatment, it creates color changes in the discharge area, prevents the passage of sunlight into the water and causes an increase in the need for dissolved oxygen. Due to its high pollution content, olive oil wastewater is among the important environmental problems [3]. Blackwater is a dark brown-black colored, malodorous, acidic pH, high suspended solids content, high turbidity and high organic matter wastewater produced during olive oil extraction [7, 8]. Olive oil wastewater is generally characterized by a high organic load, including biodegradable resistant phenolic compounds, sugars, tannins, pectin, lipids, polyphenols and polyalcohols [9]. The fact that olive black water contains high Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Suspended Solids, oil and grease, and various phenol and polyphenol compounds with phytotoxic properties shows that it has a significant pollution potential [10]. In addition, this olive black water, whose pH values vary between 4.6-5.8, suspended solids varies between 6.000-69.000 mg/l, COD values between 37,000-318,000 mg/l, and BOD values between 15.000-135.000 mg/l [11].

The use of natural dyes for textile dyeing has drastically declined after the discovery of synthetic dyes in 1856 [12]. The environmental awareness towards the protection of nature, which started in the USA in the 1960s and became widespread all over the world especially after the 1980s and intensified in the 1990s, also affected the textile industry. As a result of this environmental movement, the concept of textile ecology has emerged, and eco textiles (environmentally friendly textiles) have come to the fore. Textile ecology covers ecology, human ecology and waste ecology in textile production, and it is aimed at not harming the environment and people in all production stages from fiber production to obtaining clothes [13]. Recently, the increase in interest in natural textile products and the development of organic cotton production in our country have increased the interest in natural colorants [14]. Plant-based natural sources of dyestuffs, plants containing dyes obtained through agriculture (root dye, indigo, budgerigar, etc.), wastes and by-products obtained from agricultural and forestry products (timber waste, bark, vegetable parts/bark left after agricultural harvest) /stalks etc.), wastes and by-products obtained from the food and beverage industry (vegetable and fruit pulp from canned food and fruit juice production, seeds, pulp and waste water from olive oil production, etc.) [15].

When the natural dyestuff components contained in plants used in different fields as dyes and pigments are examined, it has been observed that there are organic compounds such as flavonoids (quarcetin, epicatechin, etc.), quinones (alizarin, juglone, etc.) and indigotin in their structures [16, 17]. When the organic components of black water, which is olive oil waste material, are examined, phenolic alcohols (trisol, hydroxytrisol), phenolic acids (cinnamic acid derivatives such as caffeic acid, ferulic acid, coumaric acid, benzoic acid derivatives such as vanilic, gallic, veratric, protocatechin and hydroxybenzoic acids, and hydroxyphenylacetic acids), secoroid derivatives (verbascoside, dialdehyde form of dicarboxymethyl oleuropen aglycone, ligstroside), flavonoids (luteolin-7-glucoside, luteolin) have also been identified [18, 19, 20, 21]. The phenolic components contained in olive oil waste materials show that they can be used as natural dyestuffs, but this natural source is not used in terms of natural dyeing.

There are limited number of studies in the literature using olive oil waste material. In a study conducted in the literature, researchers studied the dyeability of polyamide fabrics with the extraction obtained from the olive plant juice that emerged during the olive oil production stage. In the dyeings, the conventional method and the ultrasonic dyeing method were studied and the dyeing methods were compared. In addition, the natural components of the extraction obtained from olive plant juice were analyzed by HPLC. Washing, rubbing, sweat and light fastness of the dyeings were examined. As a result of the studies, it has been observed that as the power level increases in ultrasonic dyeing, the K/S values increase, there is more dye absorption, and the K/S values decrease as the pH of the dyeing bath increases. By HPLC analysis, it was found that hydroxytyrosol, tyrosol, vanillic acid, 3-hydroxybenzoic acid, M-coumaric acid, luteolin, apigenin, cinnamic acid organic components in olive plant juice. Good fastness values were obtained in both techniques [22]. In another study, olive wastewater was used as a dyestuff and the dyeability of acrylic fibers was investigated. In the study, the effect of dyeing conditions on the dyeing process, fastness properties, optimum dyeing conditions using ANOVA and Pareto diagrams, as well as environmental effects were examined. It was observed that the optimum conditions were 100 °C, 105 min, pH 3, 1:50 liquor ratio. Fastness properties were found to be moderate. In addition, an increase in the amount of phenol and COD values removed were observed in consecutive dyeings with olive wastewater [23]. In another study, wool fabric was dyed with diluted and boiled pomace. Ferrous sulfate, alum, tin two chloride, copper sulfate, potassium dichromate were used as mordant material in the dyeing. Mordanting was carried out according to pre-mordanting, meta-mordanting and post-mordanting methods. The color values, light and washing fastness of the dyeings were examined. As a result of the study, different color values were found according to the mordant type and mordanting method. Washing and light fastness values were also found to be high [24]. In another current study, the researchers used olive wastewater, which is a potential natural dye source, for dyeing wool. The effect of dyebath pH, temperature, time and salt addition on the dyeability and fastness properties of wool were investigated [25].

In recent years, the interest in natural dyes has increased due to the orientation to sustainable resources. Black water and olive pomace, which are formed during olive oil production, constitute an important pollution potential in terms of environment due to their composition and the odor they spread. Wastewater, which is described as black water, is wastewater that is difficult to treat due to the high amount of oils it contains, the high amount of COD and low molecular weight phenolic substances. The treatment methods applied are insufficient in terms of cost, efficiency and practicality. Treatment and disposal of olive pomace and black water without polluting the environment is an important environmental problem for Turkey as well as for other olive oil producing countries.

In this study, the use of textile dyeing as an alternative method in the evaluation of wastes generated during olive oil production was investigated. 100% wool fabric was dyed with olive oil mill wastewater in a liquor ratio of 1:20. Ferrous sulphate, alum, tannic acid, tartaric acid were used as mordant and they were applied by at different concentrations and mordanting times. HPLC analyzes of the black water used and/or the dyed fabrics were made and the organic components they contained were determined. Colorimetric color evaluation of the dyed fabrics was made and fastness tests were applied to washing, wet/dry rubbing, acidic/basic sweat, light and water drop. In this study, the use of olive oil production wastes as natural dyestuffs in textiles was investigated.

2. Material and Method

2.1. Material

In the study, commercially produced 100% merino wool fabric (plain weave, 240 g/m^2, warp yarns per centimeter 29 yarns/cm and weft yarns per centimeter 25 yarns/cm) and that was ready for dyeing was used. Ferrous sulphate, alum, tannic acid and tartaric acid were used as mordant agent (Sigma-Aldrich). Olive oil production waste was taken from TARIŞ olive oil mill, which is located in Bozdoğan/Aydın/Turkey (37°40′26″N; 28°20′47″E). Olive oil wastewater was filtrated and it was used directly for dye bath.

2.2. Method

2.2.1. Mordanting procedure

The wool fabrics was mordanted pre-mordanting method. Mordanting methods were performed at different concentrations (2%, 4%, 6%, 8%, 10%, owf). At pre-mordanting method, mordanting times were 2, 4, 16 and 24 hours for wool fabrics. All mordanting procedures were done at liquor ratio of 1:20. After mordanting, mordant liquor was drained and the fabrics were rinsed with the pure water. Pre-mordanting procedure for wool fabric was given in Figure 1.

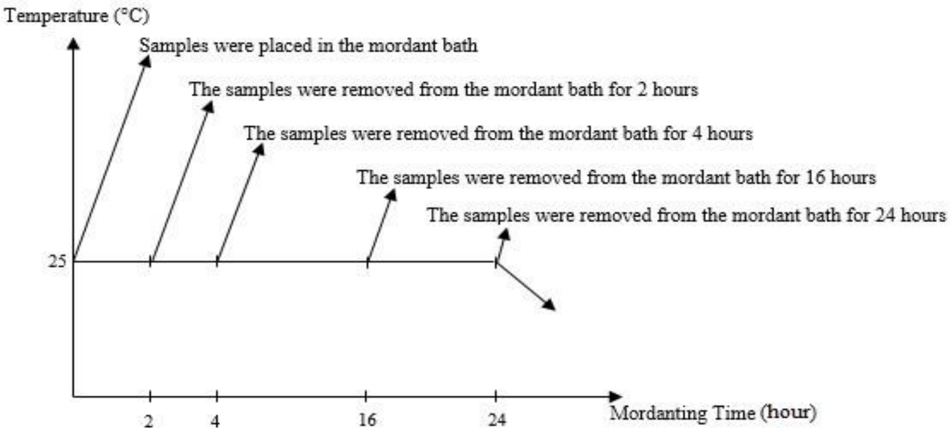

Figure 1. Pre-mordanting procedure for wool fabric

2.2.2. Dyeing procedure

Dyeing procedure was carried out at laboratory type dyeing machine (ATAÇ). Each dyeing procedure was performed in a liquor ratio of 1:20. The samples of wool fabrics were stained according to Figure 2.

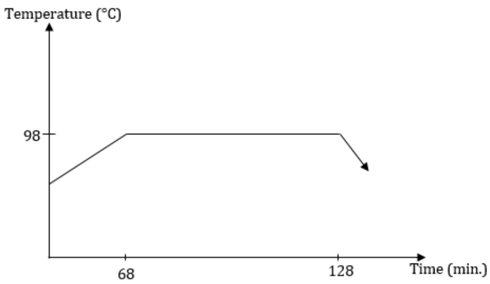

Figure 2. Dyeing procedure for wool fabric

2.2.3. Color evulation

All color measurements were performed using a Minolta 3600D (D65, specular inclusion, 10°) spectrophotometer. The spectrophotometer was equipped with software able to automatically calculate CIEL*a*b* and color strength (K/S) values from the reflectance values at the appropriate wavelength for each dyeing.

2.2.4. Fastness Testing

Color fastness to washing (TS EN ISO 105 C06/A1S), to rubbing (TS EN ISO 105 X12), to perspiration (TS EN ISO 105 E04), to water spotting (AATCC Test Method 104-2004) and to artificial light (Xenon Arc lamp - TS EN ISO 105 B02) were performed.

2.2.5. HPLC-DAD analysis

Chromatographic experiments were carried out using an Agilent 1200 series system (Agilent Technologies, Hewlett-Packard, Germany) including a G1329A ALS autosampler and a G1315A diode-array detector. Chromatograms were obtained by scanning the sample from 191 to 799 nm with a resolution of 2 nm and the chromatographic peaks were monitored at 255, 268, 276, 350, 491, 520, 580 and 620 nm. A G1322A vacuum degasser and a G1316A thermostated column compartment were used. The data were evaluated using Agilent Chemstation. A Nova-Pak C18 analytical column (3.9 mm x 150 mm, 4 mm, Part No WAT 086344, Waters) protected by a guard column filled with the same material was used. Analytical and guard columns were maintained at 30 C^{o}. The HPLC gradient elution was performed using the previously reported method in references [26] and [27]. Chromatographic separations of the hydrolyzed samples were performed using a gradient elution program that utilizes two solvents: solvent A: H_2O - 0.1% TFA (trifluoro-acetic acid) and solvent B: CH_3CN (acetonitrile) - 0.1% TFA. The solvent selection originated from previous publications. The flow rate was 0.5μL/min and the applied elution program is described in Table 1.

Table 1. Gradient Elution Program for HPLC

Time (min)	*H_2O + 0.1 % TFA (%)*	*CH_3CN + 0.1 % TFA (%)*
0.0	95.0	5.0
1.0	95.0	5.0
20.0	70.0	30.0
25.0	40.0	60.0
28.0	40.0	60.0
33.0	5.0	95.0
35.0	5.0	95.0
45.0	95.0	5.0

Samples were weighted and they were hydrolyzed in 400 mL of a solution mixture of 37% HCl:MeOH:H_2O (2:1:1, v/v/v) in conical glass tubes exactly 8 min in a water-bath at 100 C° to extract organic dyestuffs. After rapid cooling under running cold water, the solution was evaporated just to dryness in a water-bath at 55–65 C° under a gentle stream of nitrogen. Subsequently, the dry residues were dissolved in 400 µL of the mixture MeOH:H_2O (2:1, v/v) was centrifuged at 4000 rpm for 10 min; 100 µL of the supernatant was injected into the HPLC equipment.

3. Results

3.1. Color analysis

The color yield (K/S) and the CIEL*a*b* coordinates for dyed wool fabrics were reported for ferrous sulphate and alum in Table 2, for tannic acid and tartaric acid Table 3. In addition, the color chart of the dyed samples was given in Figure 3.

When L* values of wool fabrics were observed that the highest value was in ferrous sulphate mordant agent. According to L* values, darkness of fabrics was changed to ferrous sulphate > tannic acid > tartaric acid > alum. Color analysis results of the samples showed that mordant agents were increased a* and b* values. The highest a* and b* values were in tartaric acid and alum mordant agents. When K/S values were examined unmordanted sample had the lowest K/S value. K/S values had seen a rise with using a mordant agent. When compared the yield of dyeing it was found to be ferrous sulphate > tartaric acid > alum > tannic acid.

3.2. Fastness properties

Fastness properties of wool fabrics were reported in Table (4-7).

According to Table (4-7), when looked at washing fastness properties were increased with using mordant agent. All results of dyed wool fabrics washing fastness values were 5. Acidic perspiration fastness of dyed wool fabrics were generally 4/5, alkaline perspiration fastness of dyed wool fabrics generally 4/5 in ferrous sulphate and alum mordant agents. In tannic acid and tartaric acid mordant agents, alkaline perspiration fastness were usually 4. When considering the light fastness ferrous sulphate mordant agent was increased the fastness properties. Tartaric acid and alum mordant agents were decreased the light fastness properties. Mordanting process was increased the rubbing fastness properties, these values were changed to range 4/5 to 5. Water spotting fastness results were very good at all types of mordant agents.

3.3. HPLC-DAD studies

Figure 4 for wool fabric depicts the HPLC chromatograms of samples, which were taken from dye bath at different dyeing time at the dyeing temperature.

After HPLC analysis of wool fabric has found that: at 4.260 min retention time (λmax: 256nm) was p-hydroxybenzoic acid, at 7.320 min retention time (λmax: 215 - 280 nm) was epicatechin derivative, at 9.56 min retention time (λmax: 255 nm) was p-hydroxybenzoic acid derivative and 22.787 min retention time (λmax: 276 nm) was cinnamic acid (Figure 5).

When the HPLC analyses were examined, it was seen that the dyestuff was taken up at the first moment when the wool fabrics reached the dyeing temperature. When the HPLC analyses of the samples taken from the fabric in 10-minute periods after reaching the dyeing temperature were examined, it was observed that the p-hydroxybenzoic acid derivative at a retention time of 9.56 minutes adhered to the fibers as the dyeing progressed, while the other substances formed more bonds with the fibers in the first

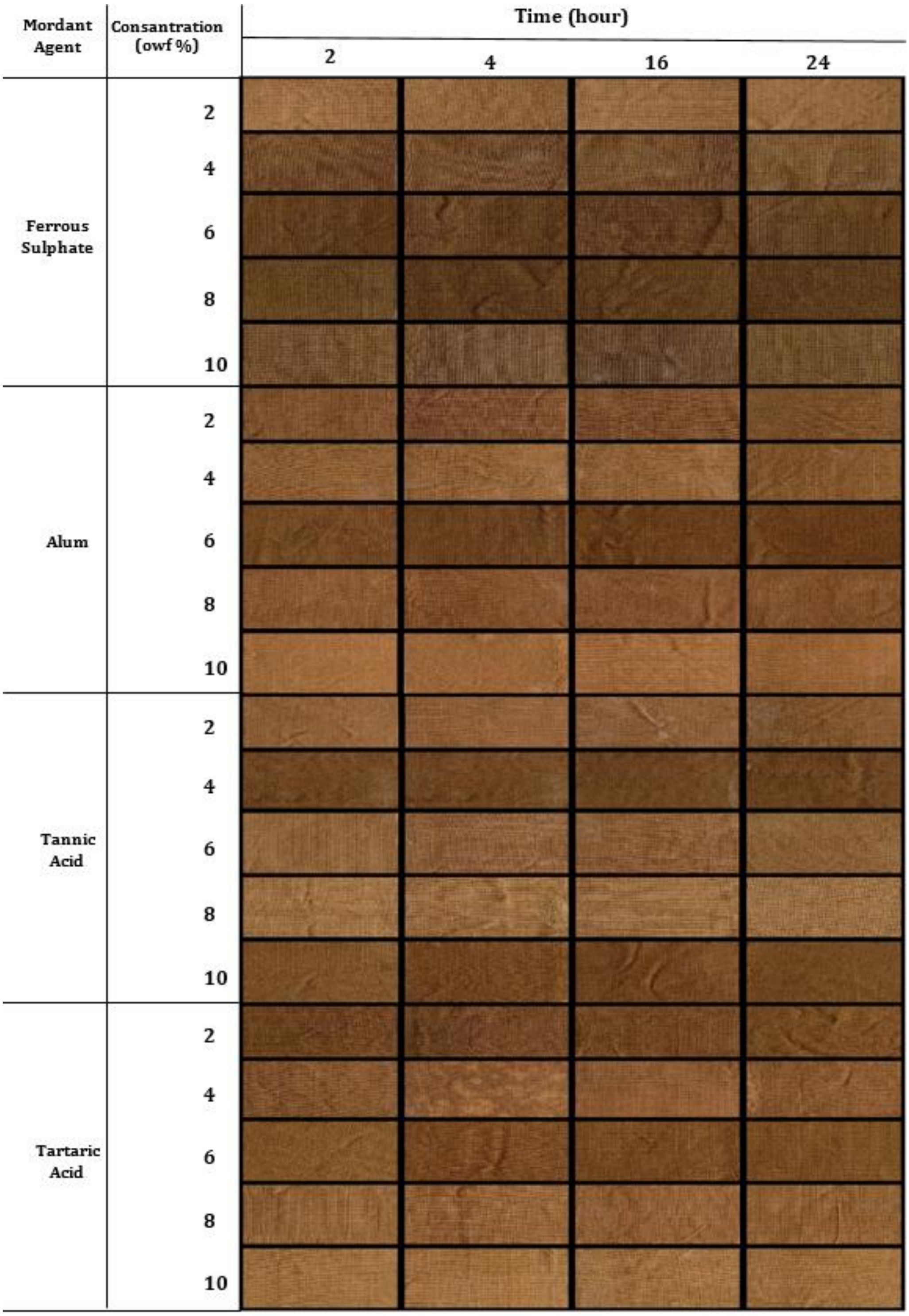

Figure 3. The color chart of the dyed samples

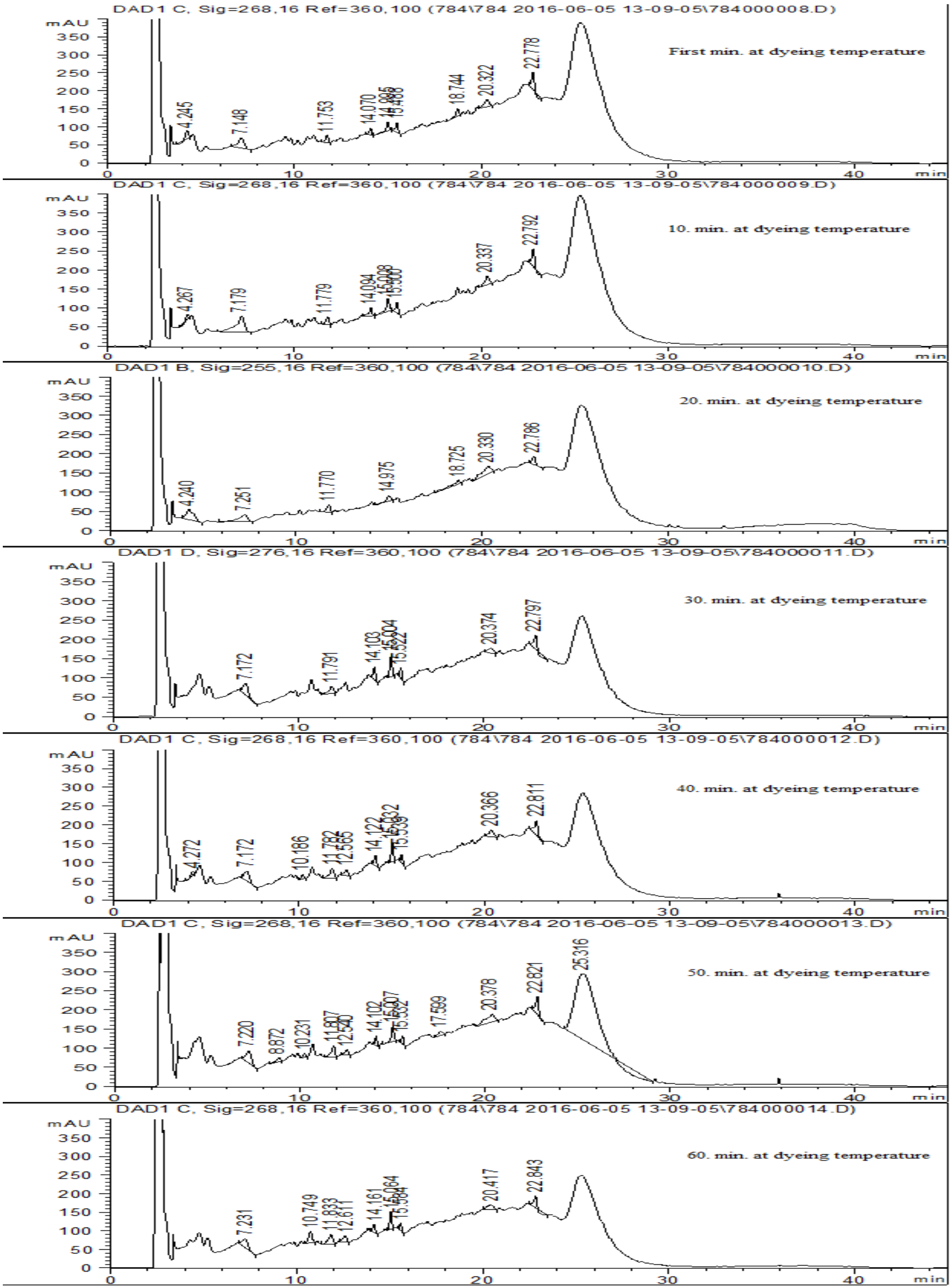

Figure 4. HPLC-DAD chromatograms at different dyeing times at the dyeing temperature for wool fabric

minute at the dyeing temperature. In the HPLC analysis made on the wool fabric, it was observed that p-hydroxybenzoic acid in the 4.26 minute retention time also bonded with the fiber at the dyeing temperature, then did not give a peak in the samples taken at the 50th and 60th minutes of dyeing. It is thought that it causes the bonds with benzoic acid to break and move away from the fiber.

When the HPLC analyzes were examined; it had been observed that the first time the wool fabric reaches the dyeing temperature, it was take more dyestuff. When the HPLC analyzes of the samples taken from the fabric in 10-minute periods after reaching the dyeing temperature were examined, it was seen that p-hydroxybenzoic acid at a retention time of 4.26 minutes bonded with the fiber at the dyeing temperature, then it did not give a peak in the samples taken at the 50th and 60th minutes of dyeing. It was thought that after a certain period of time, it bonds with other substances and causes the bonds with p-benzoic acid to break and move away from the fiber.

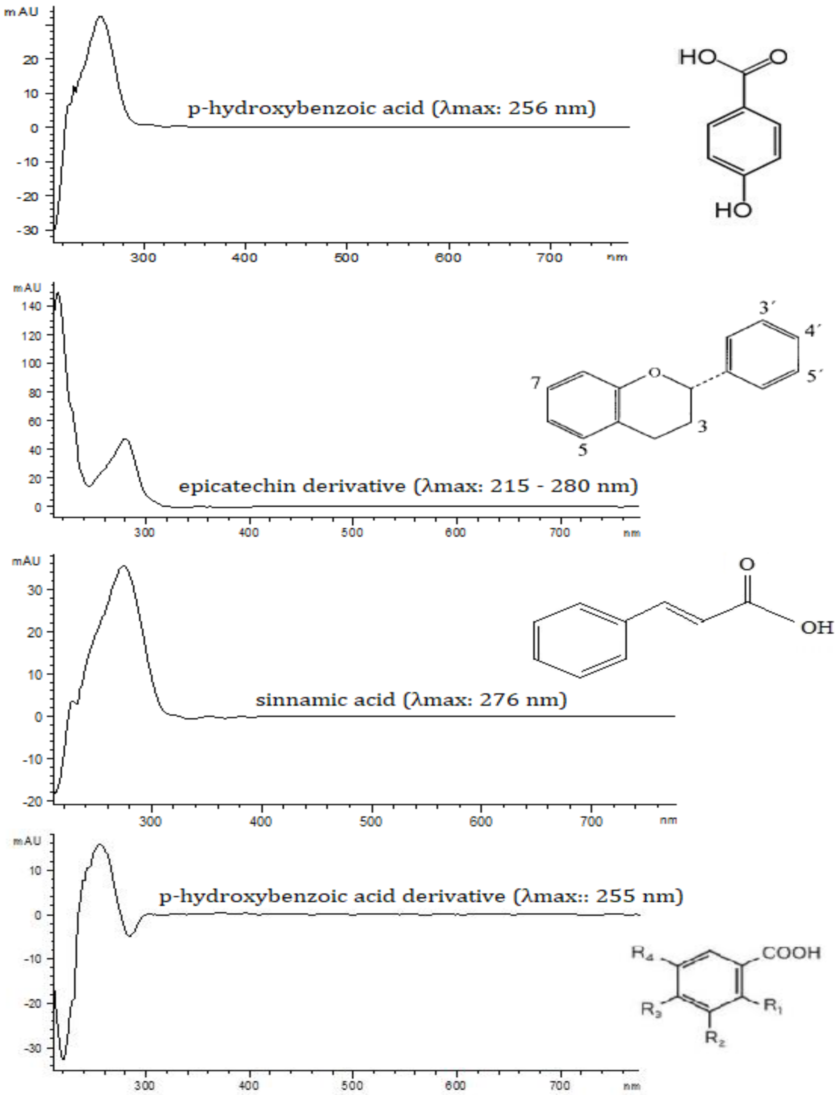

Figure 5. HPLC spectrum of p-hydroxybenzoic acid, epicatechin derivative, sinnamic acid and p-hydroxybenzoic acid derivative

Table 2. Color measurements for ferrous sulfate and alum mordant of wool fabric

Mordanting Method	Mordant Agent	Consantration (owf %)	Time (hour)	L*	a*	b*	K/S
Without Mordant	non	non	non	49.911	6.944	18.915	9.3975
Pre-mordanting	Ferrous Sulphate	2	2	43.188	9.147	18.586	12.881
		2	4	40.400	8.782	17.901	15.626
		2	16	41.342	8.611	17.519	13.766
		2	24	40.078	8.498	17.311	14.905
		4	2	41.551	8.648	17.523	13.426
		4	4	39.589	8.140	17.184	15.663
		4	16	39.663	7.888	17.318	16.118
		4	24	40.355	8.191	17.139	14.416
		6	2	41.461	8.229	18.135	14.872
		6	4	40.453	8.167	17.707	15.284
		6	16	40.348	7.303	16.879	14.788
		6	24	39.969	7.816	16.956	15.075
		8	2	41.436	7.796	17.820	14.528
		8	4	39.872	7.850	17.072	15.179
		8	16	39.911	7.783	17.192	15.498
		8	24	41.634	7.147	17.145	13.751
		10	2	41.936	7.874	18.209	14.544
		10	4	40.218	7.320	17.178	15.110
		10	16	39.834	6.645	16.711	15.337
		10	24	40.374	7.065	17.086	14.939
	Alum	2	2	43.591	10.487	19.570	14.092
		2	4	43.952	10.056	19.948	14.480
		2	16	42.918	10.508	19.542	14.641
		2	24	44.453	10.196	19.915	13.722
		4	2	43.444	10.409	19.520	14.229
		4	4	44.266	10.420	20.280	14.031
		4	16	43.189	10.386	19.758	14.448
		4	24	43.594	10.137	19.901	14.673
		6	2	44.197	10.199	20.184	13.809
		6	4	43.538	10.298	20.295	15.110
		6	16	42.177	10.478	19.730	15.626
		6	24	42.652	10.434	19.422	14.641
		8	2	44.525	10.932	20.040	12.881
		8	4	44.216	10.994	20.023	12.997
		8	16	44.707	10.885	20.311	12.830
		8	24	43.847	10.915	19.505	13.010
		10	2	45.712	10.585	21.528	14.001
		10	4	45.381	10.358	21.176	13.651
		10	16	44.787	10.610	21.183	14.183
		10	24	45.241	10.511	21.194	13.722

Table 3. Color measurements for tannic acid and tartaric acid mordant of wool fabric

Mordanting Method	Mordant Agent	Consantration (owf %)	Time (hour)	L*	a*	b*	K/S
Without Mordant	non	non	non	49.911	6.944	18.915	9.3975
Pre-mordanting	Tannic Acid	2	2	43.773	10.062	18.922	13.357
		2	4	43.989	10.399	19.655	13.708
		2	16	44.139	9.730	19.015	13.195
		2	24	42.863	9.841	18.358	13.665
		4	2	43.957	9.918	19.071	13.468
		4	4	42.975	10.002	18.455	13.622
		4	16	43.09	9.855	18.533	12.971
		4	24	43.483	9.678	18.729	12.971
		6	2	46.512	9.360	19.789	12.556
		6	4	47.269	9.459	20.716	12.483
		6	16	45.867	9.312	19.857	12.932
		6	24	46.243	9.225	19.703	12.945
		8	2	45.392	9.955	20.451	13.371
		8	4	45.417	9.993	20.447	13.454
		8	16	44.466	9.738	19.900	13.665
		8	24	44.263	9.662	19.318	13.412
		10	2	47.178	9.733	20.554	11.872
		10	4	45.483	9.969	21.181	13.927
		10	16	45.947	9.653	20.341	12.716
		10	24	45.112	9.440	19.691	13.049
	Tartaric Acid	2	2	43.784	10.414	19.415	12.881
		2	4	44.689	10.537	20.052	12.459
		2	16	43.414	10.545	19.515	13.010
		2	24	44.770	10.646	20.476	12.779
		4	2	43.730	10.497	20.222	14.385
		4	4	43.479	10.690	21.071	15.284
		4	16	46.178	10.469	21.827	13.986
		4	24	44.065	10.470	20.603	14.464
		6	2	47.291	9.944	21.243	12.754
		6	4	46.698	10.366	21.329	13.249
		6	16	45.816	10.356	20.830	13.426
		6	24	45.807	10.359	20.920	13.780
		8	2	46.309	10.329	21.368	13.330
		8	4	47.574	10.108	21.325	12.495
		8	16	45.766	10.579	20.943	13.780
		8	24	45.430	10.457	20.696	13.751
		10	2	44.628	10.660	21.360	14.657
		10	4	46.054	10.688	21.887	14.092
		10	16	44.908	10.511	21.671	15.868
		10	24	45.523	10.665	21.482	13.927

Table 4. Fastness properties of pre-mordanting method (ferric sulphate)

				Washing Fastness							Perspiration Fastness															Rubbing Fastness		Water Spotting	
											Acidic							Alkaline											
					Staining							Staining							Staining										
Method	Mordant Agent	Concentration (%owf)	Time (hour)	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Light Fastness	Wet	Dry	After 2 min.	Dry
without mordant	non	non	non	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	3	4	3	3	5
pre-mordant	ferric sulphate	2	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		2	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		2	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		2	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		4	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		4	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		4	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		4	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		6	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		6	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		6	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		6	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		8	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		8	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		8	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		8	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		10	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		10	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		10	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5
		10	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	5	3	5

Table 5. Fastness properties of pre-mordanting method (alum)

				Washing Fastness							Perspiration Fastness															Rubbing Fastness		Water Spotting	
					Staining						Acidic							Alkaline											
												Staining							Staining										
Method	Mordant Agent	Concentration (%owf)	Time (hour)	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Light Fastness	Wet	Dry	After 2 min.	Dry
without mordant	non	non	non	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	3	4	3	3	5
Pre-mordant	alum	2	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		2	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		2	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		2	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		4	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		4	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		4	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		4	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		6	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		6	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		6	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		6	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		8	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		8	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		8	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		8	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4	5	2	4/5	5	3	5
		10	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		10	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		10	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		10	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5

Table 6. Fastness properties of pre-mordanting method (tannic acid)

				Washing Fastness							Perspiration Fastness															Rubbing Fastness		Water Spotting	
					Staining						Acidic							Alkaline											
												Staining							Staining										
Method	Mordant Agent	Concentration (%owf)	Time (hour)	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Light Fastness	Wet	Dry	After 2 min.	Dry
without mordant	non	non	non	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	3	4	3	3	5
Pre-mordant	tannic acid	2	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		2	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		2	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		2	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		4	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		4	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		4	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		4	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		6	2	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		6	4	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		6	16	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		6	24	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		8	2	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		8	4	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		8	16	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		8	24	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		10	2	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		10	4	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		10	16	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5
		10	24	5	5	5	5	5	5	5	4	5	4/5	4/5	4	4/5	5	4	4/5	4	4	4	4	4/5	3	4/5	5	3	5

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Table 7. Fastness properties of pre-mordanting method (tartaric acid)

				Washing Fastness							Perspiration Fastness															Rubbing Fastness		Water Spotting	
					Staining						Acidic							Alkaline											
												Staining							Staining										
Method	Mordant Agent	Concentration (%owf)	Time (hour)	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Color Change	Wo	PAN	PET	PA6.6	Co	CA	Light Fastness	Wet	Dry	After 2 min.	Dry
without mordant	non	non	non	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5	4/5	4/5	5	3	4	3	3	5
Pre-mordant	tartaric acid	2	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		2	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		2	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		2	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		4	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	4/5	3	5
		4	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	4/5	3	5
		4	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	4/5	3	5
		4	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		6	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		6	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		6	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		6	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		8	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		8	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		8	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		8	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		10	2	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		10	4	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		10	16	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5
		10	24	5	5	5	5	5	5	5	4/5	5	4/5	4/5	4/5	4/5	5	4	4	4/5	4/5	4/5	4/5	5	2	4/5	5	3	5

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4. Discussion and Conclusion

The products that emerge during the olive oil production processes produce bad odour, high organic content (COD: 220 g/l, COD/BOD5: 2-5, hard to decompose), creating an acidic environment and high phenolic and solid content. These problems create a huge environmental problem. Many studies have been carried out to eliminate the environmental problem of olive oil production wastes. Many studies have been carried out to eliminate the environmental problem of olive oil production wastes. Olive oil production wastes contain phenolic compounds that make up natural dyestuffs. The high phenolic content of these wastes (more than 80 g/l phenolic substance content) has led to the investigation of the possibilities of using olive oil production wastes as natural dyestuffs.

When the wool fabric in dyeing with the mordanted in various mordant concentration and duration K/S values were analyzed, K/S values of ferrous sulphate > tartaric acid > alum > tannic acid has been shown to decrease with the mordant usage. Mordant dyeing of increasing concentrations of K/S values has resulted in very little increase, while the mordant time did not cause too much impact. This is going to be saturated with the fibers or dyes may be a result of being connected to all the mordant agents. The colors occurring in the material varies by the mordant has been generally in dark brown and light brown tones. As a result of fastness tests, ferrous sulphate was found to give the best results in all fastness tests. Alum and tartaric acid has poor light fastness. Fastness test results showed that tannic acid has middle fastness results.

Performed HPLC-DAD analysis the dyestuff as a result of the ten compounds were identified, seven of them has defined. P-hydroxybenzoic acid (retention time: 4.26), epicatechin derivatives (retention time: 7.32), p-hydroxybenzoic acid derivative (retention time: 9.56), cinnamic acid (retention time: 22.787) were identified dye stuffs. Dyestuffs which at wool fabric have been already taken by fiber in the beginning of dyeing temperature. Until the end of the dyeing, fibers have formed bonds with the dye molecules.

The good results of color and fastness analyses and the uptake of dyestuff molecules by the fiber show that olive oil production wastes can be used as an alternative natural dyestuff source. As a result of the studies, it has been shown that the high phenolic contents contained in olive oil production wastes can be used as natural dyestuffs.

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