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NAR KABUKLARI VE ZEYTİN YAPRAKLARI İLE KOMBİNE TABAKLAMA

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COMBINATION**

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TANNING EFFECT OF POMEGRANATE PEEL AND OLIVE LEAF TANNIN COMBINATION

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ABSTRACT: Agro-industrial wastes possess highly-valuable bioactive compounds. Recently, they have been taken into account as an alternative source that can be used as raw material for various industries. Such an approach can help to reduce both production cost and pollution load. In this study, the usability of pomegranate peels and olive leaves, defined to be agricultural and industrial residues, as a combined tanning agent in leather industry was investigated. For this purpose, first of all pomegranate peels and olive leaves were extracted for determining their tannin contents and then they were used in tanning process for evaluation their combined tanning ability. Shrinkage temperature (76.5 °C) resulted from this combination tanning and the mechanical properties of the tanned leather revealed that pomegranate peels and olive leaves can be evaluated as an alternative, satisfying combination of tanning material for leather industry.

Keywords: Agro-industrial waste, Pomegranate peel, Olive leaf, Tannin, Leather, Tanning

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ÖZ: Zirai-endüstriyel atıklar oldukça değerli biyoaktif bileşiklere sahiptir. Son zamanlarda, çeşitli endüstriler için hammadde olarak kullanılabilecek alternatif bir kaynak olarak dikkate alınmışlardır. Böyle bir yaklaşım hem üretim maliyetinin hem de kirlilik yükünün azaltılmasına yardımcı olabilir. Bu çalışmada, tarımsal ve endüstriyel artıklar olarak tanımlanan nar kabukları ve zeytin yapraklarının kombine bir tabaklama maddesi olarak deri endüstrisinde kullanılabilirliği araştırılmıştır. Bu amaçla, öncelikle nar kabukları ve zeytin yaprakları tanen içeriklerinin belirlenmesi için ekstrakte edilmiş ve daha sonra kombine tabaklama yeteneklerini değerlendirmek için tabaklama işleminde kullanılmıştır. Bu kombine tabaklama ile elde edilen derinin büzülme sıcaklığı (76,5 °C) ve mekanik özellikleri, nar kabuğu ve zeytin yaprağının deri endüstrisi için alternatif, tatmin edici bir tabaklama maddesi kombinasyonu olarak değerlendirilebileceğini ortaya koymuştur.

Anahtar Kelimeler: Zirai-endüstriyel atık, Nar kabuğu, Zeytin yaprağı, Tanen, Deri, Tabaklama

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1. INTRODUCTION

Skin is a connective tissue mostly made up of a fibrous collagen structure chained in the extracellular matrix. Tanning process is collagen matrix protection, by making it resistant to humidity, heat and microbial attacks [1]. In leather industry, current tanning technology is dominated by Cr (III) tanning due to its ability to produce high-quality leather such as excellent hydrothermal stability, better dyeing characteristics and softness. Nevertheless, the technology has been censured globally for its severe environmental detriments, unfavorable effects on human health and other organisms [2]. Therefore, environmental sustainability, renewable materials and greener tanning processing methods have been become important in leather industry more than ever. Recently, traditional vegetable tanning has regained its former popularity and vegetable tannins were highly recommended because of their less and manageable environmental effects [3-4].

Vegetable tannins, exist in roots, flowers, leaves and woody parts of many plants, have 500-3000 Da molecular weight [5] and contain sufficient hydroxyls and other suitable groups (carboxyl) to form effectively strong complexes with collagen [6-8] First criterion to be considered for evaluation of vegetable extract as tanning material in leather industry is contents of tannin and non-tannin. The effectiveness of an extract having higher tannin content is higher in tanning process. On the other hand, the availability of the respective plant is the decisive factor for commercial production of a vegetable tanning extract [9]. Mimoso, quebracho, sumac, tara, valonea and chestnut are the most common vegetable tannins used commercially in leather industry.

Recently, studies on alternative tannin sources from regional plants [10], residues of agricultural production [11] and industrial wastes [12] have increased. Although many of them could not find a place commercially in leather industry, leather products tanned with olive leaves and rhubarb extracts are available on the market [9,13,14]. The most important reason for researching new tannin sources is current supply of vegetable tannins is not sufficient to sustain global leather production, therefore there is a necessity for more sources of tannins to supplement current production of tannins [15].

On the other hand, both in developing and developed countries, plant wastes are currently an important issue. Agricultural-based industries produce enormous amount of residues every year. These residues may cause to environmental pollution and harmful effect on human and animal health in case released to the environment without proper disposal procedure. Most of the agro-industrial wastes are untreated and underutilized; they are disposed of either by burning, dumping or unplanned landfilling [16]. It is clear that using of these wastes as a source of raw materials in different industries will contribute to both economy and waste reduction. Olive leaves and pomegranate peels containing polyphenolic compounds are taken into consideration valuable agro-industrial wastes in the world and in our country.

Pomegranate peel is considered as major waste fraction that accounts for around 50% of whole fruit weight during industrial processing of pomegranate fruit to juice, and disposal of which has become a serious environmental problem [17,18]. However, pomegranate peel comprises notable polyphenolic compounds such as phenolic acids, anthocyanins and tannins (ellagitannins, punicalagin, punicalin and gallotannins) [19]. Globally, the pomegranate cultivation area is estimated to be around 300,000 hectares with a yield of 3 million metric tons [20,21]. The global total production of pomegranate is approximately 1.5×10^6 tons in 2014 according to the Food and Agricultural Organization [22]. It also is specified that this amount has fairly increased in the past decade [23,24]. Leading pomegranate producing countries in 2019 were India, Iran, Turkey, Egypt, USA, respectively [25]. In Turkey, 647.676 tons of pomegranates were produced in 2021 [26], while 465.200 tons of pomegranate were produced in 2016 and it has been estimated that 35-55% of these pomegranates turned into waste in the same year [27].

Olive tree provides three main products, they are olive fruit, olive oil, and olive leaves (represent about 5% of the weight of olives) [28]. Olive fruit is mainly grown to produce olive oil (%90) and table olives (%10) in the world [28,29]. In Turkey, total olive production remarked to be 1.739.000 tons in 2021 [30]. In the last 60 years, the production volume of olive oil has tripled globally and it reached to 3.12 million metric tons in 2019/20. The top five producers of olive oil are Spain, Greece, Italy, Turkey and Morocco [31,32] During the olive oil production process, olive leaves are mainly found in two different points. Firstly, they are found during olive tree pruning (they constitute 25% of the pruning), and secondly in the olive oil production facility (where leaves are separated from the olives by a blower machine) [33]. It is estimated that an annual 1.25 million tons of olive leaf waste are generated in Spain alone; it is around 50% of the total world production [32]. Olive leaves contain secoiridoids (oleuropein, ligstroside, dimethyloleuropein and oleoside), flavonoids (apigenin, kaempferol, luteolin), and phenolic compounds [34].

In this study, it was aimed to investigate possible use of pomegranate peels' and olive leaves' tannins as an alternative combination of tanning agent from agro-industrial wastes and determine their synergistic tanning effect.

2. MATERIALS AND METHODS

2.1 Materials

The pomegranate peels and olive leaves were supplied from a local market in Izmir and a manufacturer in Akhisar/Manisa, respectively. Before experiments, pomegranate peels were rinsed, dried and broken into pieces about 0.5-1 cm, while olive leaves were separated from branches. Pickled sheep skin (breed of Métis) was used in tanning trial.

2.2. Methods

2.2.1. Determination of moisture contents of pomegranate peels and olive leaves

The moisture contents of pomegranate peels and olive leaves were determined according to SLC 113 [35] test method with three replications and mean data were given as results.

2.2.2. Extraction processes

20 g dehumidified (40 °C for 48 h in an oven) pomegranate peels were subjected to Koch extraction for a total 8 h, with the first 6 h at 70 °C and the last 2 h at 90 °C in order to extract more solid matter. Distilled water was used as solvent. Olive leaves were extracted in like manner. The extractions were repeated three times for both of them. Extraction yields of the extract solutions were calculated according to the Formula 1.

$$(\%) \text{Extraction Yield} = \frac{\text{extract obtained (g)}}{\text{amount used in extraction (g)}} \times 100 \quad (1)$$

2.2.3. Determination of tannin contents of extract solutions

The amounts of total solids, total solubles, non-tannin constituents and tannin matter absorbable by hide powder of each extract solutions obtained from Koch extractions were determined according to the SLC 114 [36], SLC 115 [37], SLC 116 [38] and SLC 117 [39] test methods, respectively.

2.2.4. Combined tanning with pomegranate peels and olive leaves

For pilot-scale tanning process, high volumes of extract solutions are required and they should be concentrated in an oven for a long

time before using, meanwhile, fungal growth which can cause changes in composition of the extract solutions may occur. For this reason, it was decided to use ground forms of pomegranate peels and olive leaves in the tanning process in terms of practicality. The amounts to be used in tanning process were calculated by taking into account their tannin contents.

In accordance with this purpose, pomegranate peels and olive leaves were ground by a Super Mixer SM 108 Model micro mill and turned into powder form. In order to determine the combined tanning effect of pomegranate peels and olive leaves, equal active tannin matter was calculated for both of them. In other words, 6% active tannin matter was calculated separately for pomegranate peels and olive leaves, therefore the amount of active tannin used in the tanning process corresponded to 12%. Weighed pomegranate peels and olive leaves were combined and given in three portions during tanning process. A basic tanning recipe was applied and given in Table 1.

2.2.5. Determination of tanning ability

Shrinkage temperature of leather is the most important parameter to identify tanning effect of tanning agent. The shrinkage temperatures of pickled pelt and the leather tanned with pomegranate peel & olive leaf tannin combination were measured according to ISO 3380 standard [40] with three repetitions and the obtained data were given as mean values.

Table 1. Tanning Recipe

Process	Amount (%)	Product	Temperature (°C)	Duration (min.)	pH
Depickling	150	Water (9 °Be' NaCl)		15	
	1	HCOONa		45	
	0.5	NaHCO ₃		60	4.5
Draining					
Tanning	200	Water	30		
	X	Pomegranate Peel & Olive Leaf Powder		45	
	X	Pomegranate Peel & Olive Leaf Powder		45	
	X	Pomegranate Peel & Olive Leaf Powder	37	90 (left in bath overnight)	
	0.1	Fungicide		20	
	1	HCOOH		60	3.7
	0.5	MgSO ₄		15	
Washing	200	Water		10	
Draining		Aging for 1 week - Shrinkage Temperature Measurement			
Retanning-Fatliquoring	200	Water	35		
	1	Neutralizing Syntan		30	4.5-5.0
	3	Amphoteric Acrylic Polymer		30	
	5	Synthetic & Natural Fatliquor	45	75	
	3	Synthetic Fatliquor			
	2	Lecitin Fatliquor			
	1.2	HCOOH		90	3.5
Washing	200	Water		10	

2.2.6. Measurement of color

The color and color components of the leather tanned with pomegranate peels and olive leaves tannin combination were measured by Minolta CM-2600d Spherical Spectrophotometer according to the CIE*Lab color system. Color measurements were carried out from ten different parts of leather and the obtained data were given as mean values.

2.2.7. Physical Properties of Tanned Leather

In order to evaluate mechanical properties of the obtained leather; tensile strength, percentage of elongation [41] and tear load [42] were examined according to related standards and the obtained data were given as mean values.

3. RESULTS AND DISCUSSION

3.1. Moisture Contents, Extraction Yields and Tannin Contents of Pomegranate Peels and Olive Leaves

The moisture contents of pomegranate peels and olive leaves were determined to be 11.1% and 5.9%, respectively. Extraction yields and tannin contents of pomegranate peels and olive leaves extract solutions were given in Figure 1. It was obviously seen that extraction yields was similar, however the tannin content of pomegranate peels (52%) was determined to be higher than olive leaves (28.2%). It is necessitated that the amount of olive leaves to be use in tanning process must be more than pomegranate peels due to its lower tannin content. As a matter of fact, as mentioned before, the amount of ground material taking in consideration the amounts of tannin contents was calculated and 6% active tannin matter bearing plant material powder was weighed from each vegetable source and used in tanning process.

From the Koch extraction of olive leaves, the extracted solid matter content was determined to be 36.1%, and the tannin content in solid matter was determined to be 28.2%. Considering these data, it was calculated that the amount of tannin in leaf corresponds to 10.2%. Similarly, from the Koch extraction of pomegranate peels, the extracted solid matter content and the tannin content in solid matter were found to be 36.5% and 52%, respectively and the amount of tannin in peel was calculated to be 19%. From the results, it was determined that the wastes of pomegranate peels and olive leaves contain considerable and commercially reasonable amounts of tannins when compared with the commonly used tannin sources in leather industry (plant materials i.e. barks of acacia (36%), wood of quebracho (17-25%), wood of chestnut (6-12%)).

Lorini et al. reported that hydrolyzed tannin contents of olive leaves in different cultivars were determined to be 26.09 and 30.59 mg GAE/g DS (mg equivalent of gallic acid per g of dry sample), however, the condensed tannin content was not detected in these olive leaves [43]. On the other hand, condensed and hydrolysable tannin contents of pomegranate peels from various cultivars were investigated by Saad et al. [44]. Authors reported that hydrolysable tannin contents of pomegranate peels were between 421.6 and 504.8 mg TAE/g DW (mg equivalent of tannic acid per g of dry weight), while condensed tannin contents were between 1.5 and 7.7 mg CE/g DW (mg equivalent of catechin per g of dry weight). Similarly, it was seen that the tannin content of pomegranate peels was determined to be higher than olive leaves in these studies. In addition, the results indicated that both pomegranate peels and olive leaves belong to the sort of hydrolysable tannins.

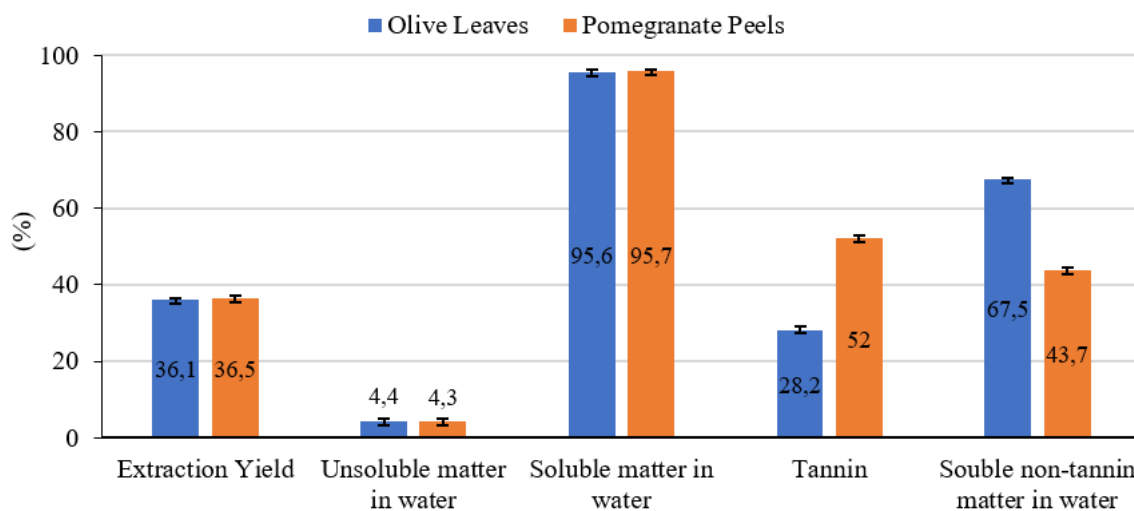


Figure 1. Tannin/non-tannin, soluble/insoluble matter contents and extraction yields of extract solutions obtained from pomegranate peels and olive leaves.

3.2. Shrinkage temperature and physical properties of tanned leather

The shrinkage temperature of the leather tanned with pomegranate peels and olive leaves tannin combination was determined to be 76.5 °C. Comparing with the shrinkage temperature of pelt (42 °C), 34.5 °C of increments achieved in the shrinkage temperature of the leather by tanning process. The shrinkage temperatures of leathers tanned with commercially used hydrolyzed tannins generally vary between 70 and 75 °C.

The physical test results of the leather tanned with pomegranate peels and olive leaves tannin combination were given in Table 2. Acceptable quality standards recommended by United Nations Industrial Development Organization (UNIDO) [45] for vegetable tanned upholstery leathers (<2 mm of thickness) are 10 N/mm² for tensile strength and 15 N/mm for tear strength. Compared with the recommended standards; it was seen that recommended values were met by the produced leather. In addition, the obtained values also met the quality requirements for garments leathers recommended by UNIDO regardless of the tanning method (min. tensile strength of 12 N/mm² and min. tear strength of 20 N/mm).

Table 2. Physical test results of the tanned leather

Tensile Strength (N/mm ²)	Elongation (%)	Tear Strength	
		Maximum Force (N)	Thickness (mm)
15.4(±2.0)	60.8(±0.5)	27.8(±0.28)	0.7(±0.01)

3.3. Color of tanned leather

Since vegetable tannins are also natural dyes and possess their natural colors to the leather, vegetable tanned leathers are generally not dyed; they are left in their natural colors. Therefore, it is important to determine the color gained to the leather by pomegranate peels and olive leaves tannins. Color measurements (L, a, b values) of the tanned leather were given in Table 3. From the examination of the data, it was determined that the color of the leather is predominantly in yellow tone and tenuously in red tone. In addition, obtained pseudo color of the tanned leather from spectrophotometer was given on the left side of Table 3.

Table 3. Color measurements of the tanned leather

Pseudo Color(D65)	L*(D65)	a*(D65)	b*(D65)
		65.59	8.18

Lightness/brightness (L=0 black, L=100 white)
 Red/green color (+a red, -a green)
 Yellow/blue color (+b yellow, -b blue)

4. CONCLUSION

In this study, olive leaves and pomegranate peels which are defined as agro-industrial wastes were investigated in terms of usability as an alternative tanning agent combination in leather industry. From the Koch extraction of olive leaves and pomegranate peels it was determined that these wastes contain considerable and commercially reasonable amounts of tannins when compared with commonly used tannin sources (plant materials i.e. barks of acacia, wood of quebracho and chestnut). 76.5 °C of shrinkage temperature gained to the leather by pomegranate peels and olive leaves tannin combination tannage which is quite satisfying for a hydrolysable type of tannin furthermore it was also revealed that the physical properties of the resulted leather met the quality requirements expected from a vegetable tanned upholstery leathers and garments leathers regardless of the tanning method.

As a result, it has been revealed that pomegranate peels and olive leaves, which do not have a significant commercial value and are even considered as wastes, can be utilized as new sources of vegetable tanning material for the leather industry.

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