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WOOL DYEING WITH EXTRACTS FROM AGRO INDUSTRIAL WASTES: A BOOST TO SUSTAINABILITY

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ABSTRACT

The purpose of this work envisages developing an eco-friendly technology by the application of renewable biomaterials as a source of natural colorants for dyeing textile substrates. In this study, natural dyeing extracts for textile fibres were obtained from grape pomace, a by-product generated in the winemaking process, and from olive trees pruning wastes, a native species from the Mediterranean basin.

The wool fabrics were dyed with different extracts (acidic, aqueous and alkali extracts). Olive leaves extracts gave greenish shades and grape pomace extracts gave brownish shades.

The wash and rubbing fastnesses of dyed wool samples were found to be within the range of 4 to 5 means fairly good to very good level.

The ultraviolet protective properties of the textiles fabrics dyed by grape pomace and olive leaves aqueous extract were also investigated.

KEYWORDS

Sustainability; Grape pomace; Olive leaves; Extraction; Ultraviolet protective properties.

1. INTRODUCTION

Recently a revival interest in the use of natural dyes in textile coloration has been growing. This is a result of the stringent environmental standards imposed by many countries in response to the toxic and allergic reactions associated with synthetic dyes (Gupta et al., 2001; Nagia and El-Mohamey, 2007).

The high processing cost and the low availability of natural coloring substances are the most important reasons for preventing these products from being more popular. The industries are continuously looking for cheaper and more environmentally friendly routes to existing dyes.

The attempts to reduce prices have mainly focused on the discovery of newer natural coloring substances especially the valorization of several colored plant wastes from industrial food and beverage production (Baaka, et al., 2015; Baaka et al., 2017).

Agriculture, food industry, and timber industry produces large volumes of wastes, both solids and liquids, resulting from their cycle of production. These wastes pose increasing disposal and potentially severe pollution problems (Laufenberg, et al., 2003) and represent a loss of valuable biomass and nutrients.

In the other hand, these wastes have attracted the attention of researchers as a source of natural textile dyes due to their abundance and availability at minimal costs (Meksi et al., 2012: Baaka et al., 2015). Additionally, their uses can promote the idea of “zero emissions”, based on the concept that wastes from one industry can be converted to raw material for another one, along with the added economic value to those wastes and minimizing their inherent environmental impacts.

Winemaking is one of the most important agricultural sector contributing substantially to national economies around the world. Total worldwide wine production is around 260 million hL, 60% of which is produced by European Union countries. Although winemaking is a seasonal activity of great social, economic and environmental relevance in the producing countries, it also leads to the generation of large

quantities of by-products. Grapes pomaces (Figure 1) are the main wastes generated in the winemaking process (Antonia, and Jaime, 2008). This industry wine by-product is a solid residue consisting of skins, pulp, stalks, and seeds (Shrikhande, 2000) which account for 25−35 Kg/hL of produced wine. The utilization of wine by-products is gaining increasing attention due to the environmental concerns and potential profitable applications.

Figure 1: Grape pomace after the pressing stage

The olive industry produces, in addition to olive oil as the main product, large quantities of liquid (margines) and solid wastes (pomace, leaves and twigs). During harvesting, the leaves can be picked with olives. These by-products are removed from olives in olive groves (Figure 2b) but also in oil mills (Figure 2c). The leaves are estimated at 10% of the total mass of harvested olives (Bouaziz et al., 2008). To this amount is added the biomass of tree pruning (Figure 5a). Indeed, after the olive harvest, olive trees generally undergo severe pruning every two years and a slight one the following year. The amounts of pruning process products were estimated at 25 kg of leaves and twigs (diameters smaller than 3 cm) produced per year per tree, according to Nefzaoui (1995). The valorization of these residues, generated in large quantities, has become a necessity to improve the profitability of the olive sector. Olive leaves are usually used as animal feed, and which could be used for antioxidant or olive-leaf extract production (De Leonardis et al., 2008; Fki et al., 2005; Lafka et al., 2011; Taamali et al., 2012).

In this work, Winemaking waste (grape pomace) and olive tree pruning wastes were used to obtain colouring extracts to be used as dyeing baths for textile fabrics. The wool fabrics were dyed with different extracts (acidic, aqueous and alkali extracts). Greenish and brownish shades were obtained. The wash and rubbing fastnesses of dyed wool samples were found to be within the range of 4 to 5 means fairly good to very good level.

Figure 2. Olive leaves, a by-product of the olive industry, generated in large quantities after pruning olive trees (a), in olive groves (b) and in oil mill (c)

2. METHOD AND MATERIAL

2.1. Materials

Grape residues were provided by a winery, located in Kélibia (Tunisia). They were a mixture of several grape varieties namely: Carignan and Alicante Bouchet of *Vitis vinifera* L. The air-dried grape pomace was stored at room temperature during the course of this study.

Olive leaves used in this study were collected from Monastir (Tunisia). Leaves were washed to remove impurities such as dust and then dried at room temperature. Subsequently, the dried material was ground giving a fine powder.

Conventionally desized, scoured and bleached plain weave wool fabric (120 g/m^2) was used for the study. Folin-Ciocalteau's reagent, vanillin ($C_8H_8O_3$), catechin ($C_{15}H_{14}O_6$), aluminium chloride ($AlCl_3$), hydrochloric acid (HCl), sodium nitrite ($NaNO_2$) and sodium hydroxide (NaOH) were purchased from Sigma Aldrich (Belgium). Gallic acid ($C_7H_6O_5$) and sodium carbonate (Na_2CO_3) were purchased from Fluka (Switzerland). All the chemicals were of analytical grade.

2.2. Extraction process

Extraction of colour components from natural dye sources is an important step for dyeing any textile substrate to maximize the colour yield. In this work, three methods were used to extract natural dyes from grape pomace and olive tree pruning wastes. The extraction was carried out in laboratory machine (Ahiba Datacolor International, USA).

2.2.1. Aqueous extraction

In the present work, 50 grams of the crumbled biomass (grape pomace, olive leaves) was soaked in distilled water, heated at 100˚C during 60 min. Afterwards, the resulting solutions were filtered to remove the residue and used for dyeing. Then the boiled solution was cooled and filtered. Finally, the pH of the solution was recorded for further uses.

2.2.2. Alkaline extraction

One percent alkaline solution with addition of 1 g of sodium hydroxide in 100 mL of water was prepared. The dye materials were boiled in the medium at 100 °C. Then, the boiled solution was cooled and filtered.

2.2.3. Acidic extraction

One percent acidic solution by adding of 1 mL of HCL in 100 mL of soft water was prepared. The dye materials were added to it and boiled at 100 °C. The pH of the solution was also recorded.

2.3. Characterization of the extracts

The analysis of the extracted dyes was carried out by determination of total phenolic content (TPC), the total flavonoid content (TFC) and the total condensed tannins.

2.3.1. Determination of total phenolic content

The total phenolic content (TPC) of the extracts was determined by the Folin-Ciocalteu method (Dewanto et al., 2002). Briefly, 0.5 mL of each extract was mixed with 5 ml Folin-Ciocalteu reagent (1:10 v/v distilled water) and 4 ml (75 g/L) of Sodium carbonate. After a reaction time of 30 min at room temperature, the absorbance was read at 765 nm. Total phenolic content values are expressed as mg/l TPC in extract (Leiter et al., 2012; el Ksibi et al., 2015).

2.3.2. Determination of total flavonoids content

The total flavonoid content (TFC) of the extracts was measured using a modified colorimetric method described above (Sun et al., 2011). A volume of 0.25 mL of a known dilution of extract was added to a test tube containing 1.25 mL of distilled water. To the mixture was added 0.075 mL of 5% sodium nitrite solution. After 5 min, 0.15 mL of 10% aluminum chloride was added, and this was allowed to stand for 5 min. Then, 0.15 mL of 10% aluminum chloride was added, and this was allowed to stand for 6 min. Then, 0.5 mL of 1 M sodium hydroxide was added, and the mixture was diluted with another 0.275 mL of distilled water. The absorbance of the mixture at 510 nm was measured. Total flavonoids content values are

expressed as mg/l TFC in extract. The assays were carried out in triplicate and the results were expressed as mean values ± standard deviations.

2.3.3. Determination of condensed tannins

Condensed tannins were determined according to the Julkunen-Titto method (Julkunen-Tiitto, 1985). 50 µL of each extract were mixed with 1.5 mL of 4% vanillin and then 750 µL of concentrated HCl were added. The well-mixed solution was incubated at room temperature in the dark for 20 minutes. The absorbance against blank was determinate at 500 nm. Results were expressed as mg/l CTC in extract. The assays were carried out in triplicate and the results were expressed as mean values ± standard deviations.

2.4. Dyeing method

The dyeing experiments were performed in a laboratory dyeing machine (Ahiba Datacolor International, USA) with L: R of 40:1, following the temperature/time dyeing conditions shown in Figure 3.

The dyed fabrics were then rinsed with cold water and washed in followed by soaping with 2 g/L of a non-ionic soap, Cotoblanc RS (Bezema AG, Switzerland) at 60°C. Finally, the fabric samples were washed thoroughly with cold water, squeezed and dried.

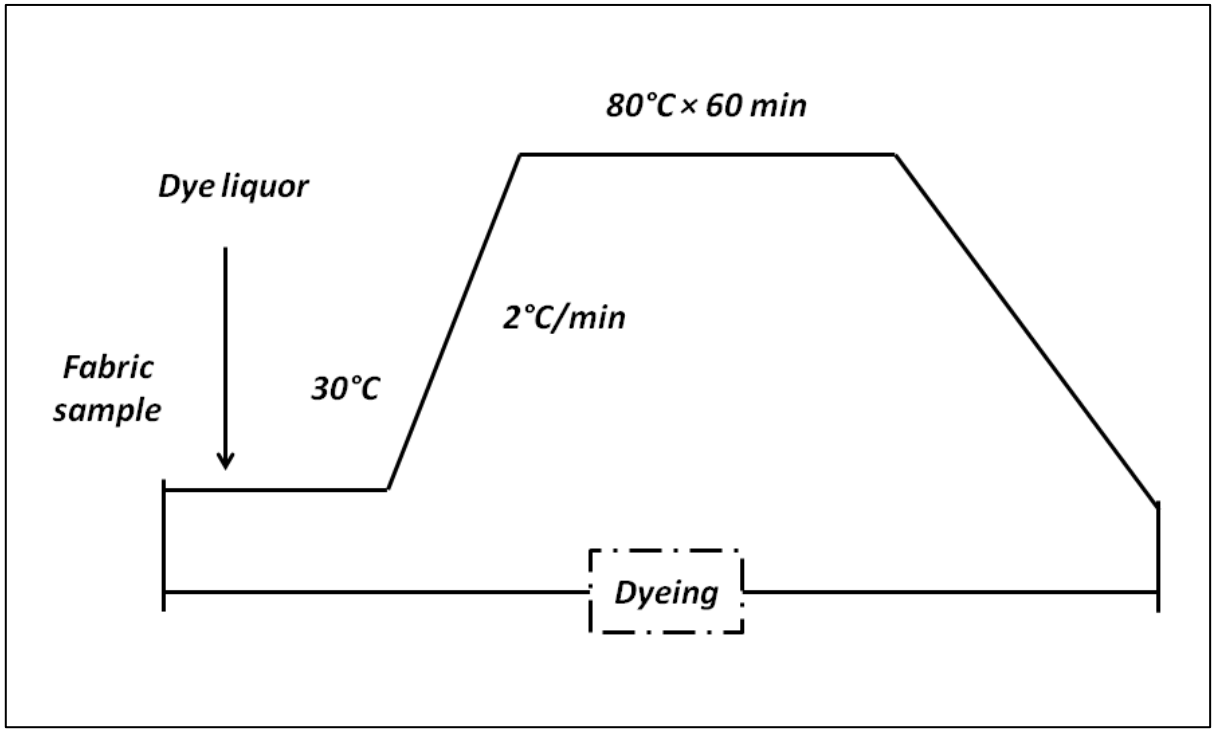

Figure 3: Dyeing process

2.5. Color measurements

Color measurements of dyed woolen fabrics were carried out by following standard procedures. Estimation of color parameter values in terms of K/S and CIE-L*a*b* values were recorded on SpectroFlash SF300 spectrophotometer with DataMaster 2.3 (Datacolor International, USA) using D and 10° standard observer.

Color strength (K/S) value was calculated by using Kubelka-Munk equation:

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \qquad (1)$$

Where K is the absorption coefficient, S is the scattering coefficient, and R is the reflectance of dyed samples.

2.6. Fastness determination

The fastness properties of dyed samples were tested according to ISO standard methods. The specific tests were as follows: ISO 105-C06 for color fastness to washing and ISO 105-X12 for color fastness to rubbing.

2.7. Evaluation of UV-protective properties of fabrics

The UV-protection properties of the fabrics were measured according to the Standard AS/NZS 4399:1996. Measurements were performed in a UV-Visible spectrophotometer system SDL; model M284, from 295 nm at an interval of one nm. The percentage brockings of UV-A (315 nm- 400 nm) and UV-B (295 nm- 315 nm) were calculated from transmittance data. The UV protection factor (UPF) was calculated using the following equation described in previous study (Kathirvelu et al., 2009):

$$UPF = \frac{\sum_{\lambda=295}^{\lambda=400} E\lambda \times S\lambda \times \Delta\lambda}{\sum_{\lambda=295}^{\lambda=400} E\lambda \times S\lambda \times T\lambda \times \Delta\lambda} \qquad (2)$$

Where Eλ is the relative erythemal spectral effectiveness,
Sλ is the solar spectral irradiate (in $W.m^{-2}.nm^{-1}$),
Tλ is the spectral transmission of the specimen,
Δλ is the measured wavelength interval (nm).

3. RESULTS

3.1. Characterization of the obtained extracts

Total phenolic, total flavonoid and condensed tannin contents were determined from the calibration curves of gallic acid, quercetin and catechin, respectively. The total phenolic, total flavonoid and condensed tannin contents among the different extracts are presented in figures 3 and 4.

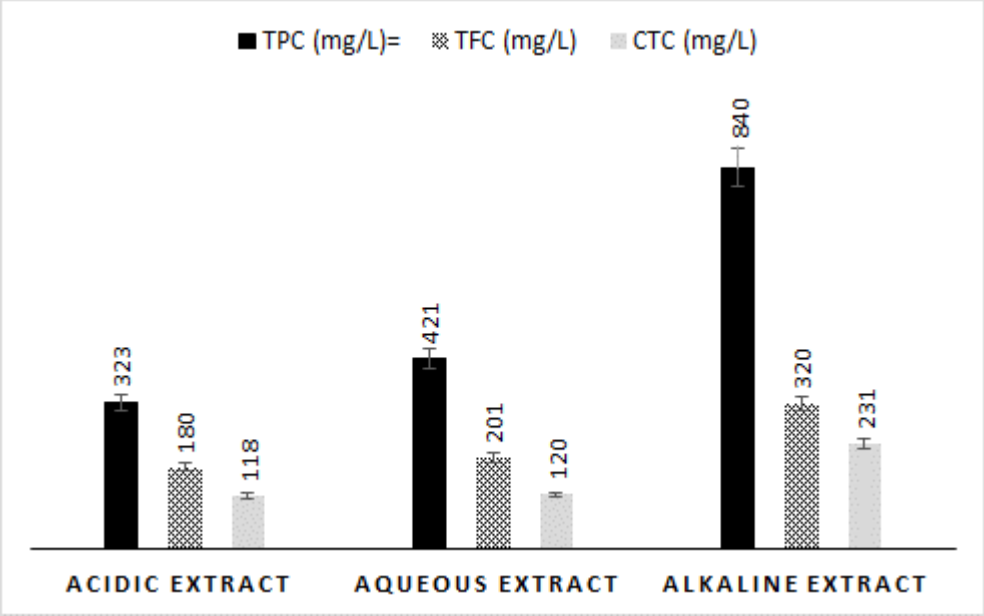

Figure 3: Total phenolic, flavonoids and condensed tannins contents of obtained extracts from olive leaves. Values represent mean, and standard error bars are on the top of each column.

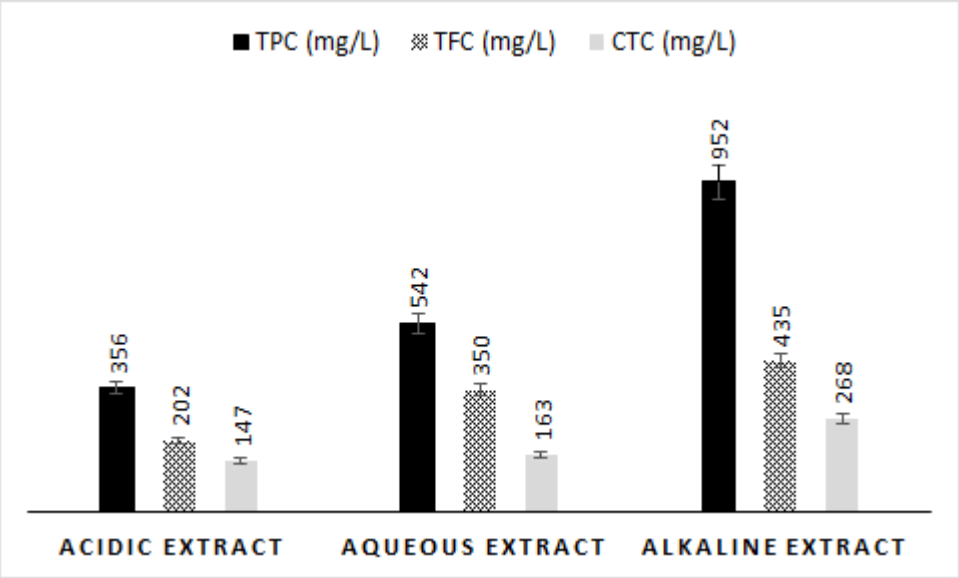

Figure 4: Total phenolic, flavonoids and condensed tannins contents of obtained extracts from grape pomace. Values represent mean, and standard error bars are on the top of each column.

The results showed that grape pomace extracts possessed higher phenolic, flavonoid and condensed tannin components than olive leaves extracts. In addition, the highest phenolic compounds were obtained in the case of alkaline extraction. In fact, in this condition of pH, the phenolic groups reacted with base and formed the more soluble salts in the water.

This is could be due to the presence of phenolic groups (acid group) in the grape pomace and olive leaves extracts. In fact, the fraction of phenolates (phenoxide anions) increases in highly alkali aqueous solution (Naczk & Shahidi, 2006) and in this condition of pH, the phenolic groups reacted with base and formed the more soluble salts in the water. This is due to the presence of phenolic groups (acid group) in the grape pomace and olive leaves extracts. Thus, these phenolic groups react with caustic soda (NaOH) and form more soluble salts in water. Indeed, in an alkaline aqueous medium, it will have an acid-base reaction between the hydroxyl groups (El-Nagar et al., 2005) of the polyphenol (Example: Quercetin) and the caustic soda for obtaining salts which are more soluble in water than the departure polyphenols (Khaliq et al., 2015; Gomez-Alonso et al., 2007). This reaction is represented by figure 5.

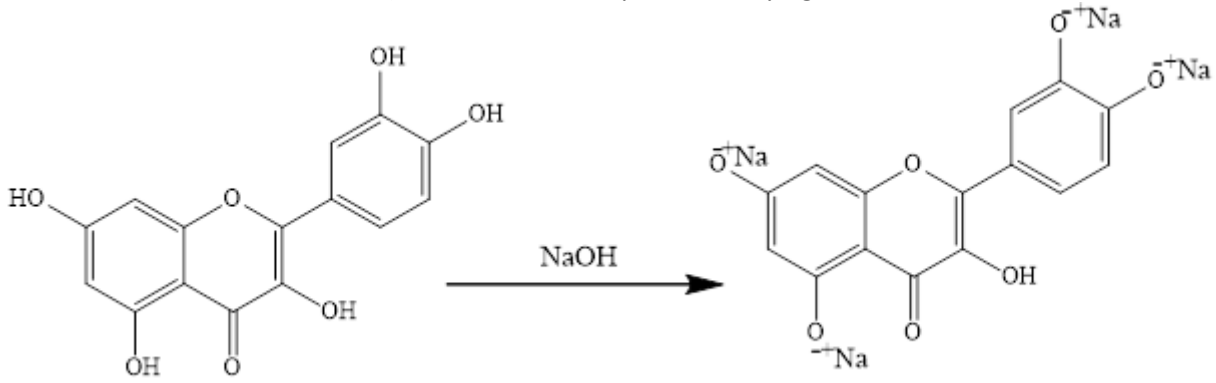

Figure 5: General reaction of quercetin components with caustic soda

3.2. Effect of the dye concentration

Wool fibers dyed using different extracts (acidic, aqueous and alkaline) of grape pomace and olive leaves powder were studied in order to evaluate their color measurement parameters viz., L, a* and b* values. Different dye concentrations (1, 3, 5, 8 and 10%) were used and the results are collected in Table 2.

Table 2: CIE L*a*b* and K/S values of wool fabrics dyed with different olive leaves and grape pomace dye concentrations

Condition	Olive leaves				Grape pomace			
	K/S	L*	a*	b*	K/S	L*	a*	b*
1 % Acidic extraction	1.13	65.05	5.47	14.22	1.09	64.46	3.04	11.27
3% Acidic extraction	1.50	65.47	6.17	12.24	2.01	63.53	3.94	9.68
5 % Acidic extraction	3.48	50.26	6.81	10.96	4.36	58.24	5.77	9.21
8% Acidic extraction	3.37	45.64	5.68	10.54	4.33	52.18	7.09	10.59
10% Acidic extraction	3.25	41.09	7.14	9.41	4.47	45.43	5.49	8.35
1% Aqueous extraction	1.27	63.38	3.49	16.7	2.39	62.54	3.21	11.09
3% Aqueous extraction	1.29	58.53	8.91	15.16	3.01	54.95	3.77	9.2
5 % Aqueous extraction	4.88	48.36	5.02	10.96	6.62	50.71	5.79	8.86
8% Aqueous extraction	4.46	41.8	4.7	13.04	6.36	40.54	5.71	8.98
10% Aqueous extraction	4.90	38.02	5.13	12.67	6.26	41.25	4.04	7.46
1% Alkaline extraction	1.57	59.21	7.82	13.01	3.41	56.57	7.52	14.04
3% Alkaline extraction	2.43	50.26	8.91	15.16	7.32	50.41	8.25	13.81
5% Alkaline extraction	8.95	48.36	6.81	8.62	10.28	46.35	9.31	14.09
8% Alkaline extraction	8.46	40.07	9.41	8.33	10.09	38.75	9.03	12.62
10% Alkaline extraction	8.52	38.86	9.25	8.06	11.49	38.27	9.67	13.29

Comparison of the dyeing results (according to Table 2) shows that, for both grape pomace and olive leaves, the increase of the dye concentration increases the the colour yield (K/S) of dyed fabrics. The best value of (K/S) was obtained for 5% alkaline extraction condition. In Table 2, it can be also seen that decreasing alkaline conditions of extraction enhanced the L* as a result of the decline of dyeability.
The colorimetric parameters a* and b* exhibited different behaviors to that of the colour yield (K/S). However, it could be confirmed that olive leaves extracts gave greenish shades and grape pomace extracts gave brownish shades.

3.3. Color fastness properties

In this part, wool fabrics were dyed with different extracts in the optimized dye concentration (5%). The obtained shades for different dyed fabrics were presented in Figure 6.

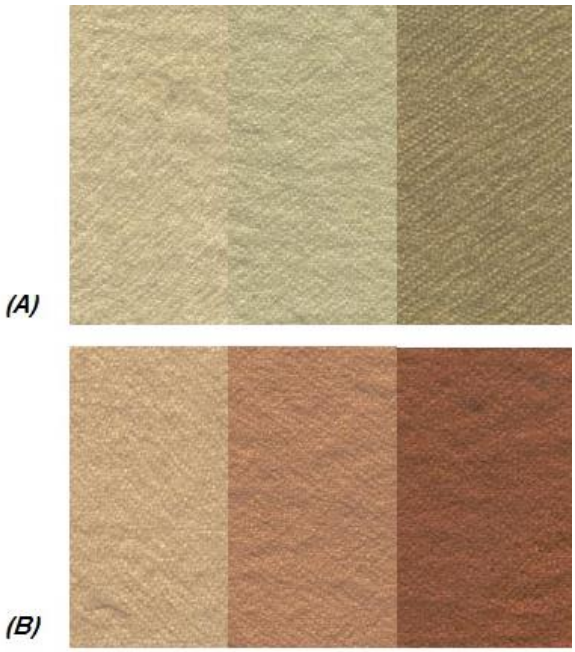

Figure 6: Dyed wool fabric. (A) Olive pruning wastes (acidic, aqueous and alkaline extracts). (B) Grape pomace (acidic, aqueous and alkaline (dye concentration (dye concentration =5%)

Olive leaves extracts gave greenish shades and grape pomace extracts gave brownish shades. The washing and rubbing fastness properties of all dyed wool samples are presented in Table 3.

Table 3: Fastness properties of dyed wool fabrics with 5% dye concentration

	Condition	Wash	Rubbing	
			Dry	Wet
Olive leaves	Acidic extraction	4	4-5	3-4
	Aqueous extraction	4	4	3-4
	Alkaline extraction	4-5	4-5	4
Grape pomace	Acidic extraction	4	4	3-4
	Aqueous extraction	4-5	4-5	4
	Alkaline extraction	5	5	4

From table 3, the wash fastnesses of dyed wool samples were found to be within the range of 4 to 5 means fairly good to very good level.
Color fastness to rubbing was found to be also within the range of 3-4 to 5 means fairly good to good level in all dyed wool samples.

3.4. UV protection properties

To investigate the UV-protection properties of grape pomace and olive leaves extracts dye, the UPF values of the wool fabrics with different extracts were evaluated.
Table 4 shows the UPF values and protection class of the wool fabrics dyed with olive leaves and grape pomace extracts.

Table 4: UPF values and protection class of dyed wool fabrics with olive leaves and grape pomace extracts (5% dye concentration)

	Condition	UPF	Class
	Undyed	5	Insufficient
Olive leaves	Acidic extraction	15	good
	Aqueous extraction	28	Very good
	Alkaline extraction	50+	Excellent
Grape pomace	Acidic extraction	25	Very good
	Aqueous extraction	40	Excellent
	Alkaline extraction	50+	Excellent

According to AS/NZS 4399:1996, UPF values of less than 15, between 15 and 24, between 25 and 39, and above 40 are classified as bad, good, very good, and excellent protection against solar ultraviolet radiation, respectively.
The wool fabrics have low UPF value (equal to 5). This value is less than 15 which cannot offer any degree of protection, while the dyed samples of wool fabrics with olive leaves and grape pomace acidic extracts have a UFP superior than 15. This means that these samples are classified to have a good to very good protection properties. Excellent UV protection was observed in the wool fabrics dyed with alkaline extracts.

3. CONCLUSION

With an increasing interest by numerous industrialists, the use of natural dyes is on the rise. Hence, researchers are attempting to find new natural dyes particularly by the valorization of food and beverage by-products.
This study intended to explore natural dyes application as an alternative to synthetic dyes in textile application. Olive leaves and grape pomace were used to obtain a colouring extracts as dyeing baths for wool fabrics. Based on the achieved results, the best extraction conditions are obtained in alkaline medium. From this study, one can conclude that olive tree pruning wastes and wine industry by-product extracts can be used as dyeing bath with a low environmental impact. The dye extracts are obtained without any chemicals additives and different shades were achieved only by pH changing. The UV protection properties

of the dyed samples were good to excellent. Therefore, this colorant could be used in industrial dyeing process for its promising dyeing and UV protective properties.
Textile factories will face the requirement of dwindling water, energy and chemicals uses and reducing the environmental impact of the textile industry.

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