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# Use of fermented dough extract in the dyeing of wool fabrics

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In this study, shells of onion (*Allium cepa* L.) have been used for the dyeing of the wool fabrics. Fermented dough extract (FDE) has been chosen as a natural mordant compound and the effect of pre-treatment with the FDE is examined in terms of color strength and fastness values. Dyeing experiments are carried out at *p*H 4 and *p*H 7 using three mordanting techniques. The chromaticity co-ordinates are measured in CIELab system. The results of untreated samples are compared with the dyed samples pre-treated with FDE in terms of color strength and fastness properties. It is observed that the pre-treatment with FDE enhances not only the color strength of the dyed fabrics but also their brightness.

**Keywords**: *Allium cepa*, Dyeing, Fermented dough extract, Wool

In recent years, not only researchers but also practitioners have been looking for various types of treatments in order to obtain improvement in color, fastness and functional characteristics of the dyed fabrics in the frame of environmental consciousness[1]. Therefore, natural coloring materials and the methods of their applicability on different types of fibers are of considerable interest of chemists. A number of studies have been focused on the amount of dye found in natural sources, effect of mordants, improvement of fastness values and color strength of the dyed fibres[2-8].

Improving the dyeing and fastness properties of the textile fabrics have been the subject of various studies[9,10]. Recent developments in natural textile dyeing are focused on the modification of natural and synthetic fabrics using pre- and post-treatment agents in order to improve the color, fastness, and functional characteristics of the dyed fabrics[1]. A number of studies revealed that cationic[11,12] and anionic[13,14] agents are effective in the dyeing properties of fabrics. Alam and Bendak[15] reported that improvement in the dyeability of wool, silk and polyamide fabrics with cationic dye can be achieved by pre-treatment of fibres with saccharin sodium salts. Yuhan *et al.*[16] developed a new method for exhausting the dyestuff to the wool fabric. Onal *et al.*[17] studied on the improvement of the dyeability of wool, cotton and feathered leather. For this purpose, calcium oxalate in alkaline medium and willow extracts have been applied on to wool fibre, feathered-leather and cotton as a pre-treatment process. Results of the previous studies indicated that pre-treatment of fibres with different types of agents enhances not only the color strength of the fabrics but also their fastness properties[18,19].

The main colorant of the onion shells is Quercetin, which is chemically known as 3,5,7,3′,4′-pentahydroxy flavones[20,21]. It is reported that quercetin exhibits anti-carcinogenic, antioxidant and good dyeing properties due to its auxochrome (-OH) group and other chromogen groups present in its structure[22-24].

This study evaluates the effects of fermented dough extract pre-treatment on wool samples in weak acidic and neutral media. This novel organic mordant has been used for the dyeing of wool fabric to increase its fastness and color strength.

## Experimental

The wool fabrics were supplied from Toga Textile Company (Tokat, Turkey). The wool fabric of 117 $g/m^2$ (25 warps /cm, 11 wefts/cm) was washed with solution containing 0.5 g/L sodium carbonate and 2 g/L non-ionic detergent (Labonene) followed by heating at 45-50°C for 30 min. After the pre-treatment, the material was washed with distilled water and dried at room temperature.

Dried onion shells were purchased from local market of Tokat, Turkey. Alum [$AlK(SO_4)_2 \cdot 12H_2O$], copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$), potassium dichromate ($K_2Cr_2O_7$), acetic acid and sodium hydroxide were supplied from Merck. These mordant salts were used in order to obtain different color and color shades. Wheat flour was obtained as natural wheat seeds from Tokat. Soxhlet apparatus was used to extract the dyestuff from the onion skin. Deionised water was used in the experiments.

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#### Color Measurements and Fastness Tests

The color yield of the dyed fabrics was evaluated by Premier Colorscan SS 6200A Spectrophotometer in terms of CIELab values ($L^*$, $a^*$, $b^*$, $C^*$) and color strength ($K/S$) values. The color strength values of the dyed fabrics were calculated using the Kubelka-Munk equation.

The crock, light and wash fastness values of all dyed samples were determined by a 225 model crock meter, a fadometer (Xenotest), and a LHTP model Atlas Launderometer respectively. Moreover, crock, light, and wash fastness were established according to DIN 54021, DIN 54004 and ISO 105-C06, CIS respectively.

#### Preparation of Dough Extract and Pre-treatment Processes

First day, wheat flour (1200 g), deionised water (700 g) and salt (NaCl) 20 g were mixed and kept as such for 24 h at room temperature. Second day, 1000 g of flour, 600 g of deionised water and 10 g of salt were mixed and added to above mixture and allowed to remain as such for 24 h at room temperature. Third day, 1600 g of flour and 800 g of deionised water were mixed and added to this mixture. The content was allowed to remain as such for 24 h at room temperature. Fourth day, 1600 g of flour and 800 g of deionised water were mixed and added to this mixture, and whole content was allowed to remain as such for 24 h at room temperature.

Three (3) kg of dough was mixed in 5 L of deionised water. Extract was filtered in order to obtain clear solution. Half of the fabrics was placed into this extract for 24 h before dyeing procedures. After that, the samples were rinsed with deionised water and air-dried. The dyeing procedures were performed on both pre-treated samples with fermented dough extract (FDE) and control samples that were not subjected to FDE.

#### Extraction and Dyeing Methods

Dry shells of onion (100 g) were crushed and extracted with deionised water (1000 mL) by soxhlet apparatus until colorless, at its boiling point. The fractions were combined and then used for the dyeings.

For pre-mordanting, the control samples and the fabrics pre-treated with FDE were heated in 0.1 M mordant solution (100 mL) for 1 h at 90ºC. After cooling of the sample, it was rinsed with deionised water and put into the dye-bath solution (100 mL). It was then heated at 90 ºC for 1 h. Finally, the dyed material was removed, rinsed with deionised water and air-dried. In meta-mordanting method, both mordant (in solid state equivalent to 0.1 M mordant solution) and dyestuff solution are poured into a flask and the sample was placed into this mixture. The content was heated at 90 ºC for 1 h. After cooling, it was rinsed and dried. On the contrary to pre-mordanting method, the materials were firstly treated with the dyestuff solution for 1 h at 90 ºC in post-mordanting method. After cooling, it was rinsed with distilled water and put into 0.1 M mordant solution (100 mL) and heated for 1 h, at 90 ºC. Finally, the dyed material was rinsed with deionised water and dried.

### Results and Discussion

The dyeing processes of the wool fabrics, pre-treated and un-pretreated with FDE using the extract of onion shells, have been carried out using some transition metal salts at various *p*H values by three mordanting methods. Eighteen samples were pre-treated with FDE and for comparison eighteen content without FDE pretreatment are used. Dyeing of the fabrics was carried out at *p*H 4 and *p*H 7 respectively.

#### Proposed Dyeing Mechanism

Wool molecules consist of amino acid units and hence can be considered as an amphoteric compound because of the free amino and carboxyl groups in its structure[25-28].

Quercetin molecule has three possible chelating sites, including 3-hydroxychromone, 5-hydroxychromone and 3′-4′-dihydroxyl group[26]. Hydroxyl (-OH) and carbonyl (C=O) groups in the dyestuff molecule form coordinate covalent bonds with mordant cation[27], such as $Cu^{2+}$. The possible structures of the complexes with copper that may occur between wool fibres and onion shells can be considered, as shown in Fig. 1.

After the interaction of the mordant and the dyestuff with the fibre, some coordination sites may remain unoccupied in the presence of Al (III) and Cr (III) metals. These sites may be occupied with the functional groups of the fibre[28], such as amino and carboxylic groups or chelating cites of the dyestuff. Additionally, these metals form a bridge between the fibre and the dye.

#### Fastness Values

The wash, rub and light fastness values of the dyed wool fabrics with onion shells were given in Tables 1-3. The rub and wash fastness levels are determined by grey scale, while light fastness values are established using blue scale. The good to excellent wash fastness of the samples dyed with onion shells is due to the strong covalent bonds between the dye molecule and the fabric. Rub fastness of the dyed samples is determined in both dry and wet forms. The light fastness values of the dyed wool fabrics pretreated with FDE range between 3 and 7, i.e. moderate to excellent. It is indicated that wool samples pre-treated with FDE exhibit better light fastness values as compared to control samples.

Table 1 — Wash fastness values of the dyed fabrics
[Scale — 5 (good) to 1 (poor)]

| Dyeing method | Mordant | FDE | | Control | |
|---|---|---|---|---|---|
| | | *p*H 4 | *p*H 7 | *p*H 4 | *p*H 7 |
| Pre mordanting | Cu | 4 | 4 | 4 | 4 |
| Pre mordanting | Al | 5 | 4 | 4 | 4-5 |
| Pre mordanting | Cr | 5 | 4 | 4 | 4 |
| Meta-mordanting | Cu | 4-5 | 4 | 4 | 4-5 |
| Meta-mordanting | Al | 4 | 4 | 4 | 4 |
| Meta-mordanting | Cr | 3 | 3 | 4 | 4 |
| Post-mordanting | Cu | 4 | 4 | 4 | 4 |
| Post-mordanting | Al | 4-5 | 4-5 | 4 | 4 |
| Post-mordanting | Cr | 3 | 3 | 4 | 4 |

Cu - $CuSO_4 \cdot 5H_2O$; Al - $KAl(SO_4)_2 \cdot 12H_2O$; Cr - $K_2Cr_2O_7 \cdot 7H_2O$.

**Color Coordinates and Color Strength**

It is observed that the dye uptake ability depends on *p*H, mordant and treatment type. *K*/*S* values of the dyed wool fabrics are given in Fig. 2. The optimum dyeing conditions for the wool fabrics pre-treated with FDE are obtained at *p*H 4, using meta-mordanting method, in the presence of $CuSO_4$ mordant. Generally, wool fibres exhibit high *K*/*S* values. It is clear that pre-treatment of wool fibres with FDE helps to improve the *K*/*S* values of the dyed samples.

The CIE *L** *a** *b** color coordinates of the dyed wool fabrics are presented in Table 4. Green and khaki tones are obtained in the dyeing of wool fabrics. The results indicate that different color and shades are achieved in all dyeing experiments.

In this work, dyeing properties and fastness values of the dyed wool samples pre-treated with FDE and un-pretreated with FDE (control) are compared. The fastness values, brightness and color strength values of the dyed samples pre-treated with FDE are found higher than those of the control samples. These results are very important for textile industry. The effect of FDE on *K*/*S* and fastness values can be explained with the composition of FDE. Dough extract consists of 0.5% oil,

Table 2 — Rub fastness values of the dyed fabrics
[Scale — 5 (good) to 1 (poor)]

| Dyeing method | Mordant | FDE | | | | Control | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Dry | | Wet | | Dry | | Wet | |
| | | *p*H 4 | *p*H 7 | *p*H 4 | *p*H 7 | *p*H 4 | *p*H 7 | *p*H 4 | *p*H 7 |
| Pre mordanting | Cu | 4-5 | 4-5 | 3 | 4 | 4 | 4 | 4 | 4 |
| Pre mordanting | Al | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 |
| Pre mordanting | Cr | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 | 4 | 4 |
| Meta-mordanting | Cu | 4-5 | 4-5 | 3 | 5 | 4 | 3-4 | 4 | 4 |
| Meta-mordanting | Al | 4-5 | 4 | 4-5 | 4 | 4 | 4-5 | 4 | 4 |
| Meta-mordanting | Cr | 4-5 | 4-5 | 3 | 4 | 4 | 4 | 4 | 4 |
| Post-mordanting | Cu | 4 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4 | 4 |
| Post-mordanting | Al | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 |
| Post-mordanting | Cr | 4 | 4-5 | 3 | 4 | 4 | 4 | 4-5 | 4 |

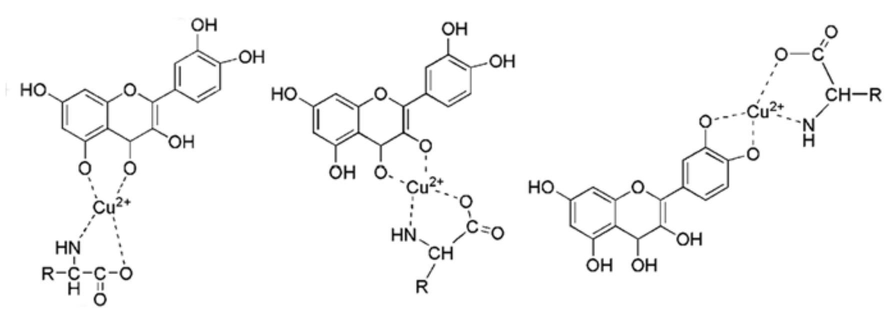

Fig. 1 — Possible structures of $Cu^{2+}$ complexes that may occur between the wool fibre and the onion shell

Table 3 — Light fastness values of the dyed fabrics
[Scale: 8 (maximum) to 1 (very poor)]

| Dyeing method | Mordant | FDE | | Control | |
|---|---|---|---|---|---|
| | | *p*H 4 | *p*H 7 | *p*H 4 | *p*H 7 |
| Pre mordanting | Cu | 6 | 7 | 3-4 | 3-4 |
| Pre mordanting | Al | 3 | 6 | 3 | 3-4 |
| Pre mordanting | Cr | 4 | 6 | 3 | 3 |
| Meta-mordanting | Cu | 5 | 7 | 3 | 3-4 |
| Meta-mordanting | Al | 3 | 6 | 3 | 3-4 |
| Meta-mordanting | Cr | 4-5 | 4 | 3 | 3 |
| Post-mordanting | Cu | 7 | 7 | 3 | 3-4 |
| Post-mordanting | Al | 5 | 5-6 | 3 | 3-4 |
| Post-mordanting | Cr | 5-6 | 5 | 3 | 3 |

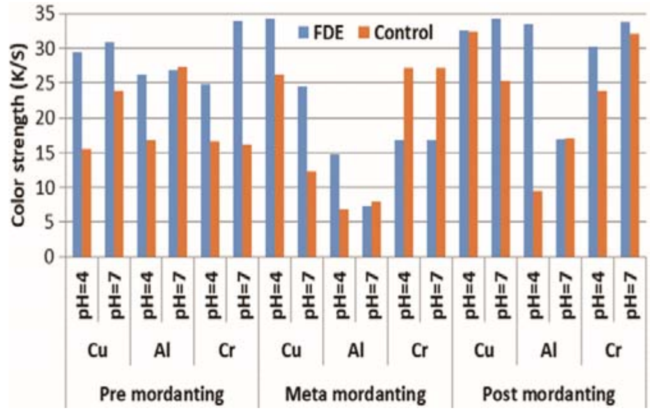


Fig. 2— Color strength values of the dyed wool fabrics

Table 4 — Color coordinates and color strength values of the dyed wool fabrics

| Method | Mordant | FDE | | | | | | Control | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | *p*H 4 | | | *p*H 7 | | | *p*H 4 | | | *p*H 7 | | |
| | | ***L**** | ***a**** | ***b**** | ***L**** | ***a**** | ***b**** | ***L**** | ***a**** | ***b**** | ***L**** | ***a**** | ***b**** |
| PM | Cu | 34.84 | 11.24 | 25.68 | 36.33 | 8.66 | 24.58 | 49.85 | 11.11 | 27.32 | 41.65 | 4.24 | 22.95 |
| PM | Al | 45.51 | 12.54 | 37.41 | 45.73 | 12.00 | 32.82 | 49.36 | 11.46 | 27.35 | 46.89 | 12.12 | 45.36 |
| PM | Cr | 44.23 | 2.94 | 21.65 | 36.78 | 5.20 | 22.91 | 49.73 | 11.39 | 27.21 | 44.96 | 3.68 | 22.69 |
| MM | Cu | 33.31 | 6.29 | 20.32 | 42.41 | -4.16 | 18.32 | 41.03 | -0.64 | 21.20 | 48.87 | -1.60 | 20.89 |
| MM | Al | 51.03 | 9.46 | 31.21 | 56.78 | 6.87 | 26.76 | 58.34 | 7.33 | 30.72 | 56.26 | 6.28 | 26.93 |
| MM | Cr | 46.61 | 3.66 | 20.84 | 47.66 | 4.05 | 22.45 | 40.21 | 3.35 | 21.57 | 45.06 | 5.86 | 32.12 |
| PSM | Cu | 26.36 | 11.22 | 15.61 | 29.89 | 5.60 | 17.84 | 35.39 | 6.77 | 20.10 | 35.97 | 6.53 | 22.38 |
| PSM | Al | 29.06 | 10.79 | 19.29 | 48.99 | 10.73 | 33.32 | 55.25 | 7.89 | 32.84 | 48.83 | 10.38 | 37.45 |
| PSM | Cr | 43.51 | 15.68 | 31.85 | 33.64 | 6.42 | 21.29 | 41.62 | 4.63 | 20.81 | 31.73 | 8.47 | 20.43 |

2.3% inorganic compounds, 68-73% water, 14% protein, 10% carbohydrate as well as enzymes and vitamins. During fermentation process, the starch molecules of dough extract start degrading to the glucose, and then glucose units are transformed in to the carbon dioxide and ethyl alcohol. However, lactic acid occurs as a final product of the fermentation process[29].

Consequently, it can be said that one or more components in the dough extract affect the absorption of dye molecules on to the fabrics and form the strong chemical bonds between dyestuff and fabric molecules.

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