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EXTRACTION OF DYESTUFF FROM ONION (*Allium cepa* L.) AND INVESTIGATION OF DYEING PROPERTIES OF COTTON AND WOOL FABRICS USING (urea+ammonia+calcium oxalate) MIXTURE

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Abstract - The dyestuff from onion (*Allium cepa* L.) was extracted using Soxhlet apparatus with distilled water. Wool and cotton fabrics were pretreated with (urea+ammonia+calcium oxalate) mixtures, artificial animal urine system (AAUS) before dyeing. The solutions 0.1 M of $CuSO_4$, $FeSO_4$ and $AlK(SO_4)_2.12H_2O$ were used as mordant agents. Pre-mordanting, together mordanting and last mordanting methods were applied at pH =4 and pH= 7 for dyeing of fabrics. According to the fastness results, the best dyeing method was determined as together mordanting method at pH=4 for wool and last mordanting method at pH= 7 for cotton fabric. The results also reveal that the onion containing Quercetin dyestuff shall probably be an important raw material for dyeing process of natural textile fibers.

Key words - *Wool, Cotton, Oxalate, Dyeing, Fastness*

1 Introduction

Natural dyes have high importance in producing hand made carpets, kilim and similar industrial dyeing applications before of their advantage of high colour fastness, cheapness, long term colour stability and authentic properties. Nowadays, the natural dyes are being produced in Asian countries such as Turkey, Iran, India, Azerbaijani, and natural dye products are being used most countries of the world [1].

There are many industrial plants which contain natural dyes such as onion(*Allium cepa* L.) which has odoriferous, and is used as spices plant, commonly. Onion has major flavone molecule which can be used as dyestuff of 3,5,7-tri hydroxy-2-(3’,4’ dihydroxy chromen-2-on called as Quercetin. [2] (Figure 1). The molecule structure of Quercetin play important role on dyeing process of natural fabric.

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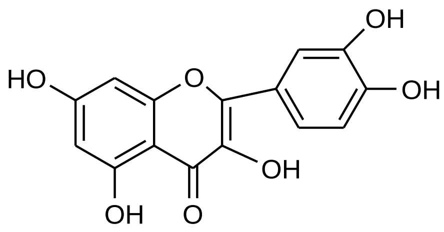

Figure 1. Chemical structure of quercetin

The acquired dyeing and fastness properties of woolen and cotton fabrics are very important characteristic in terms of user. The interaction of mordant compounds with wool and cotton fibers effects the affinity to fibers of dyestuffs. Improving the dyeing and fastness properties of textile fibers constitute the main subject of various studies [4,5,6]. In another different and last study, Onal-1 mordant mixtures in alkalin medium had been applied to wool fiber, feathered leather and cotton as a pretreatment process using *Rubai tinctorum* L. and *Hyperium scabrium* L. [7,8].

This study evaluates the average of dyeing properties of wool and cotton fabrics using Onion (*Allium cepa* L.*)* and the effect of (urea+ammonia +calcium oxalate) mixtures for each fabrics.

2. Experimental

Preparation of mordant solutions and dye-bath

Wool and cotton samples were treated with artificial animal urine system (AAUS). The stem and leaves of Onion (*A.cepa*) were supplied Plant Research Laboratory, Gaziosmanpasa University, in June, 2010. It was dried in shade, cleaned and powdered by grinder before the experiments. Extraction *of A. cepa* was performed by soxhlet apparatus with distilled water. 1 L of distilled water was used (for 100 g plant material) then the dyestuff was transferred to the aqueous media.

Reagents and equipments

All chemicals used in this work, were purchased from Merck. Distilled water was used for all steps. $FeSO_4.7H_2O$, $AlK(SO_4)_2.12H_2O$ and $CuSO_4.5H_2O$ were purchased from Merck. Extraction was performed by using soxhlet apparatus. Colour codes were determined by Pantone Colour Guide. The wash-, crock- (wet, dry) and light fastness of all dyed samples were established according to ISO 105-C06 and to CIS, respectively, and fastnesses were determined by Atlas Weather-ometer, a Launder-ometer and a 255 model crock-meter, respectively [9].

Dyeing procedures

Dyeing procedures of wool and cotton samples were firstly treated with artificial animal urine system (AAUS). The undyed materials were kept into AAUS included NH_3 (3%, v/v), CaC_2O_4 (3%, m/v) and urea (3%, m/v) for 24 h, at room temperature before dyeing procedures. At the end of the time, the samples rinsed with distilled water and dyed according to the dyeing methods that mentioned below.

Pre-mordanting method

The undyed material (1 g) which was treated with willow solution and AAUS for 24 h at room temperature, separately, was heated in 0.1 M mordant solution (100 mL) for 1 h at 90ºC. After cooling of sample, it was rinsed with distilled water and put into dye-bath solution (100 mL). It was heated at 90ºC for 1 h, at the end of the period, the dyed material removed, rinsed with distilled water and dried.

Together-mordanting method

Both mordant (in solid state which equivalent to 0.1 M mordant solution) and dyestuff solution poured into a flask and the sample placed in this mixture. The complication was heated at 90ºC for 1 h. After cooling, it was rinsed and dried.

Last-mordanting method

On the contrary to pre-mordanting method, the undyed material (1 g) was first treated with dyestuff solution for 1 h at 90ºC. After cooling the sample, it was rinsed with distilled water and put into 0.1 M mordant solution (100 mL) and heated for 1 h at 90ºC. Finally, the dyed material was rinsed with distilled water and dried.

3. Results and Discussion

Proposed dyeing mechanism

As the hydroxy (-OH) and carbonyl (C=O) groups forms coordine covalent bonds with mordant cation, such as Cu^{2+} (Figure 2, Figure 3 and Figure 4).

The dyeing mechanisms of wool with Salvigenin by pre-mordanting (1), together-mordanting (2) and last-mordanting (3) methods can be considered as follows [10] :

(1) Wool...........................Mordant (Me^{n+})........................Dyestuff

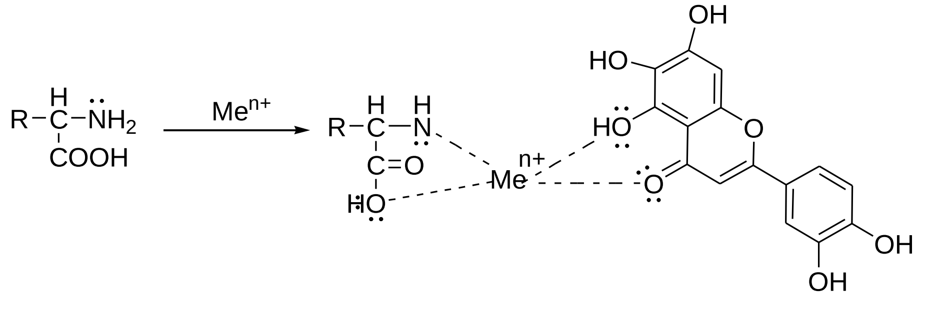

Figure 2. Proposed mordant-dye complex according to pre-mordanting method in dyeing of wool fibers

(2) Dyestuff......................Mordant (Me^{n+}).....................Dyestuff

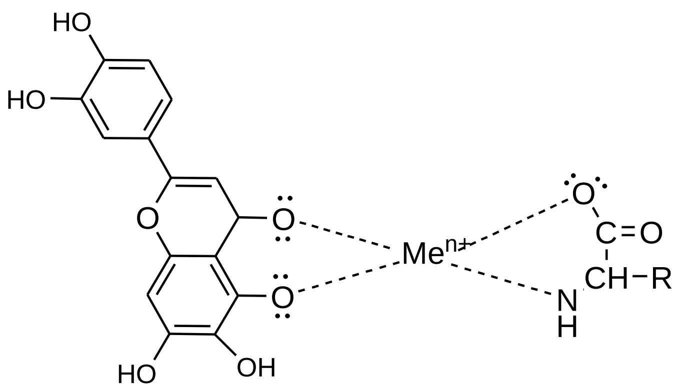

Figure 3. Proposed mordant-dye complex according to together-mordanting method in dyeing of wool fibers

(3) Wool............................Dyestuff (Me^{n+})..............................Mordant

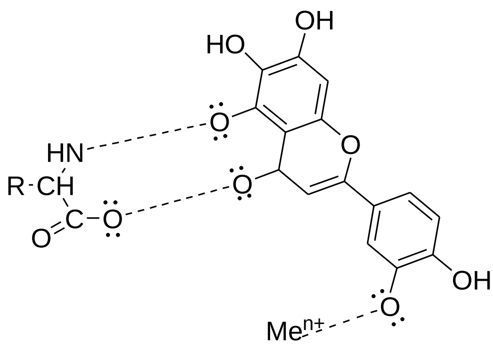

Figure 4. Proposed mordant-dye complex according to last-mordanting method in dyeing of wool fibers

Because of cotton has cellulosic structure, coordine covalent bonding occurs between CH_2O- groups of cellulose and metal cation. The suggested mechanism is given below (Figure 5)

The variation of average fastness for wool with respect to the mordant agent at Fig.6 and the variation of average fastness for cotton with respect to the mordant agent at fig.7.

As seen from the curves in Fig. 6 the average fastness for wool samples decreases in the order of Fe(II)>Cu(II)>Al(III). Best values for wool samples obtained by using Pre-mordanting method with Fe(II) and Al(III) mordants.

It can be clearly observed from the Fig.7, there is no considerable difference between together- and last-mordanting method with the use of Fe(II) and Al(III) mordants in dyeing of cotton fibers.

In general, from the Figures 6 and 7, the most effective mordant agent is Fe(II) and the most effective dyeing procedures are together- and last-mordanting method. This situation can be explained by the high stability of Fe(II) complex. Based on the results, it can be noted that treatment of natural fibers with AAUS assists to strenght the coordinate covalent bonding of Fe(II) salt to natural fiber.

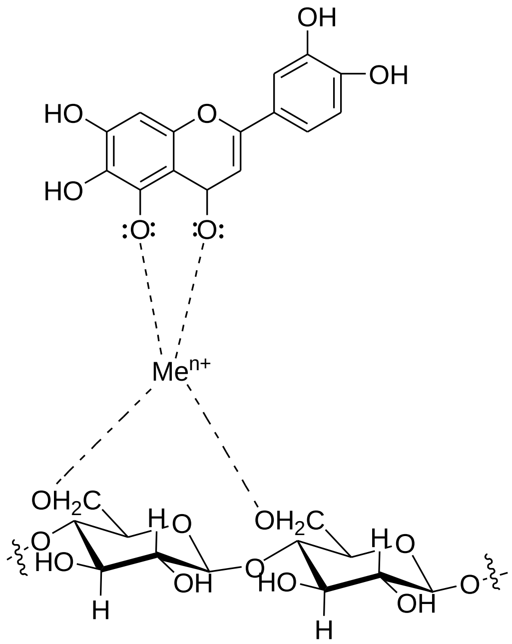

Figure 5: Proposed mordant-dye complex according to together-mordanting method in dyeing of cotton

When evaluated the dyed wool samples, green , brown and its tones were obtained in the presence of pre- and together-mordanting methods by $CuSO_4$ and $FeSO_4$ salts, and yellow tones were obtained by $AlK(SO_4)_2.12H_2O$ for three mordanting methods.

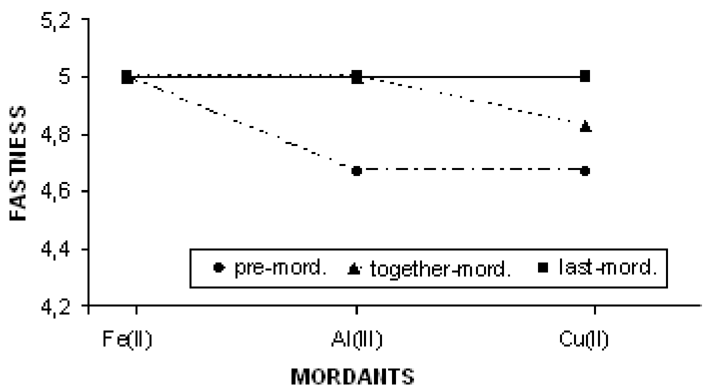

Figure 6. The variation of average fastness for wool with respect to the mordant agent

In dyeing of cotton samples, gray, light gray and cream tones were occured. According to the experimental results, however, the colours fastness of dyed cotton and wool samples have good degrees.

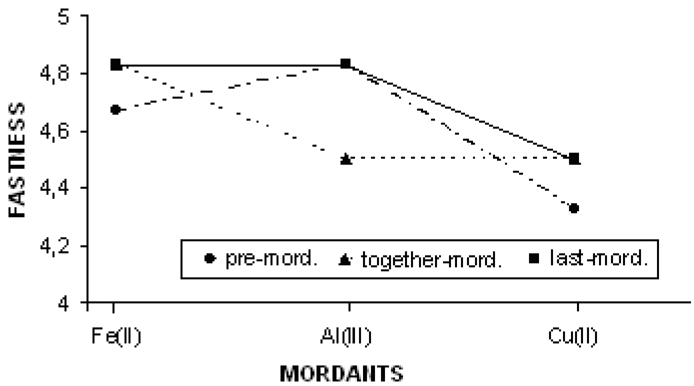

Figure 7. The variation of average fastness for cotton with respect to the mordant agent

The effect of AAUS was explained by Onal in 1996. Shortly, the components of AAUS (ammonia+ urea+ oxalate) have a great importance on the fastness of dyed fibers [10]. Here, ammonia helps the expanding of fiber misels so it facilitates the penetration of dye to the fiber. Urea serves as a pH regulator, and as last, oxalate plays an important role during the formation of complex structure which occurs between dye and natural fiber. It makes this complex very stable, and so the fastness values of the dyed samples increase in the presence of AAUS.

All the fastness values and colour codes are presented in Table I, Table II, for wool and cotton samples, respectively.

Table 1. Fastness values and color codes of dyed wool fabric (average values)

Mordant	**Dyeing Method**	**Wash-Fastness**	**Crock Fastness**		
			Wet	**Dry**	**Light fastness**
$FeSO_4.7H_2O$	Pre-mordanting	4	5	5	6
	Together-mordanting	4-5	5	5	6-7
	Last-mordanting	4-5	5	5	6
$CuSO_4.5H_2O$	Pre-mordanting	4	5	5	6
	Together-mordanting	3	5	5	7
	Last-mordanting	4-5	4-5	4-5	6
$AlK(SO_4)_2.12H_2O$	Pre-mordanting	3	5	5	6-7
	Together-mordanting	3-4	5	5	7
	Last-mordanting	4-5	4-5	4-5	6-7

It can be clearly seen that wet and dry fastness values are very good for dyed wool and cotton fibers .

Table 2. Fastness values and colour codes of dyed cotton fabric (average values)

Mordant	Dyeing Method	Wash-Fastness	Crock Fastness		
			Wet	Dry	Light fastness
$FeSO_4.7H_2O$	Pre-mordanting	5	5	5	5-6
	Together-mordanting	3-4	5	3-4	5-6
	Last-mordanting	5	5	5	6-7
$CuSO_4.5H_2O$	Pre-mordanting	4-5	5	4-5	6
	Together-mordanting	4-5	5	4	5-6
	Last-mordanting	5	5	5	6-7
$AlK(SO_4)_2.12H_2O$	Pre-mordanting	4	5	5	5
	Together-mordanting	3-4	4	4	5-6
	Last-mordanting	5	5	5	6-7

Consequently, the best dyeing conditions of wool materials are obtained with Fe(II) and Cu(II) mordants using pre- and together mordanting method. Generaly green and brown colour tones were obtained for wool samples. On the contrary to wool, the highest fastness values obtained for cotton fibers with Fe(II) according to all mordanting methods. The colours of cotton fibers are gray, yellow and cream tones. In addition, AAUS contributes the brightness of natural fibers dyed samples.

A. cepa may be evaluated as an important natural dyestuff source. However, AAUS which called as Onal-1 mordant system, may be used as pre- mordanting mixtures for cellulosic and protein fibers to increase the fastness and brightness of the textile products.

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