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Textile dyeing of cotton and wool textile material with natural dyes extracted from bluish purple grapes

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Fruits and vegetables contain considerable amounts of natural dyes which could serve in textile dyeing operations. A natural dye was extracted from fresh bluish purple grapes by aqueous extraction method. To get the maximum yield of natural dye from bluish grape, variation in temperatures and time was explored. The dyeing was carried out on wool and cotton textile material without, with a single and a mixed mordant system. Color strength; build up properties, CIE Lab coordinates and fastness properties were measured. The results indicated the potential of extractable natural dye on both textile materials, especially on wool. It was observed that mordant dosage and dyeing temperature were the key factors for good dyeing conditions with the natural dye. Regarding fastness, washing was good on all dyeing fabrics but light fastness was not up to the mark with respect to commercial dyeing requirements.

Keywords: Cotton and wool textile material, Dyeing, Bluish purple grapes, Natural dyes, Extraction, Mordant system.

INTRODUCTION

The plants, minerals, and animals are the major source of natural dyes, almost non-substantive and can be used with the help of a mordant (metallic salt). There is an edge to natural dyes as compared to synthetic dyes, as they are soft, shiny and comforting for human eyes [1]. The raw material of synthetic dyes is non-renewable; while the materials for the natural dyes are environmentally friendly, renewable, and biodegradable. The raw material for natural dyes comprises anti-allergy, safety with skin. Its importance is due to medicinal properties on skin especially for kids, and no hazardous elements found for human health [1]. Nature produces different sources of dyes such as insects and plants used since centuries. Plants are the key source of around all natural dyestuffs and sources of different colors - brown, black, yellow, red, and blue, as well as combinations of these [4]. Flowers, wood, seed, root, fruit, leaf, bark, etc. are parts of plants which are the main source of dyes production. For thousands of years, indigo was a very important natural dye in Arab countries [8, 14], the turmeric was the main source plant for natural dyes used in the Egyptian period about four thousand years ago. Indigo dyes can be used only once, due to vat dyes [8]. Insolubility of dyes in water is always a problem, while indigo reduced and converted in the form of white color and it is soluble in water. There are two other types of vat dyes such as biblical blue and Tyrian purple. For reduction of dyes the vat containing fabric/yarn can be applied [9]. Except for few restrictions, there are many advantages of natural dyes. In natural dyes the extraction time is a very important and tedious factor. The dyeing cost by natural dyes is higher as compared to synthetic dyes, due to the color components in the raw material. Among natural dyes some are fugitive and require mordant for improvement of fastness properties. Among these few mordants (metallic) are hazardous [6]. The collection of the plants, lack of the technical expertise for extracting, non-availability of standardization, lack of dyeing techniques by natural dyes is a problem the entire world is facing today. As compared to synthetic dyes the natural dye is less toxic, less polluting, less poisonous, and carcinogenic free. These dyes are soft in use, environmentally friendly and recyclable [6]. Due to mass/public awareness, the use and demand of natural dyes is increasing gradually, including use in food products, due to its lower toxicity and environmentally friendly nature. Many studies have been done for the assessment of benefits, availability of potential for use of natural dyes on pilot plant scale. Some new techniques including biotechnology are needed for the improvement in production of dyes quantity and quality [6]. Most of the natural dyes do not directly combine with the material to be dyed. They require chemical substances known as mordants, which are metallic salts that have an affinity for both fiber and dyestuffs. A mordant is considered equally a

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chemical, which can itself, be stable on the fiber and which also combines with dyestuffs. Mordanting can be carried out in three ways such as, pre-mordanting, simultaneous mordanting, post-mordanting. Mordants (metallic), oil mordant, and tannis are three important types of mordants [13]. The traditional way of extraction of the natural dyes is by soaking the plant or other material containing dye in water and boiling the solution in earthen or metal vessels (preferably copper, stainless steel). Generally, four methods are adopted for extraction such as, aqueous, alkaline, acidic, and fermentation [12].

MATERIAL AND METHODS

Experimental material

The sample 100% free optical brightener, bleached scoured woven fabric (cotton) (150 g/m^2, 1/1 plain weave) and 100% wool yarn was used for dyeing purpose. The chemicals and auxiliaries used such as gallotannic acid, potash alum, acetic acid, sodium chloride, sodium hydroxide, and hydrochloric acid were purchased from Merck. Soaping agent was used of commercial grades purchased from local market.

Equipment

Dyeing machine (AHIBA IR) and Datacolor (SF 650X) was used for the dyeing experiments and data color was used for color match and for colorimetric data evaluation. Light fastness was determined on a Weather-Ometer. Samples were preserved on a curing machine of Rapid. Fastness rating was assessed on a color matching cabinet by comparing with grey scale and blue wool scale for light fastness rating.

Extraction method

A weighed amount of grapes fruit was extracted with distilled water in a beaker. In the standard procedure the mass of grapes / volume of liquid ratio were set at 5:20, i.e. 5 g of grapes was extracted with 20 ml of water. The extraction process was done at different temperatures (60, 95°C) with variation of time (40, 60, 80, 100 min) to get the maximum yield of dyeing extract.

Dyeing experiment

Exhaustion was used for dyeing operations (liquid ratio) 1:30, i.e. for 1 g of textile material a dye-bath of 30 ml was used. As per standard method, the dyeing trials were executed at 95°C. Different dyeing procedures were tested with regard to mordanting and dyeing temperature. Either 10 g of bleached wool yarn or 10 g of bleached cotton fabric was used as textile material. Both cotton and wool textile materials were tried in a sequence.

- Experiment No. 1,8,15: Standard dyeing, without mordant at 60°C, 80°C, 95°C for 60 min, L: R 1:30.
- Experiment No. 2,3,9,10,16,17: Pre-dyeing with mordant (5-15%) gallotannic acid at 60°C, 80°C, 95°C for 60 min, L: R 1:30.
- Experiment No. 4,5,11,12,18,19: Dyeing with mordant used alum (2-4g/l) at 60°C, 80°C, 95°C for 60 min, L: R 1:30.
- Experiment No. 6,7,13,14,20,21: Pre-dyeing with mordant using gallotannic acid and dyeing with alum at 60°C, 80°C, 95°C for 60 min, L: R 1:30.

The alum ($KAl(SO_4)_2.12H_2O$) used as a mordant, was added to the dye bath to a final dye bath concentration 3-4 g/l.

Optimum procedure of pre-mordanting with gallotannic acid ($C_{76}H_{52}O_{46}$) was used. A mass of gallotannic acid corresponding to 5-15 % of the mass of textile material was dissolved in water at 50°C. The textile samples were then impregnated at 80°C for 1h at a liquor ratio of 1:20. The mordanted samples were washed with water, and dried before dyeing. After that, unfixed dyestuff was detached by washing 3 times with cold water.

Measurement of dye fixation

The fixation of the dye in % was calculated first by determining the reflectance (R) of the dyed samples at the wave length of minimum reflectance (maximum absorbance) on an SE-650 spectrophotometer. The color yield (K/S) value was then calculated by using the Kubelka-Munk equation (eq. 1) and the dye fixation (%) was evaluated using eq. 2 [5].

$$K/S = (1-R)^2/2R \quad (1)$$

$$\%\ \text{Dye fixation} = \frac{\text{K/S value of sample after soaping}}{\text{K/S value of sample before soaping}} \times 100 \quad (2)$$

Fastness testing

The fastness properties were determined according to the international standards. The specific tests used were ISO-105-CO3 [2] (color fastness to washing), ATCC – 16 E [3] option-3 (color fastness to light).

RESULTS AND DISCUSSION

Effect of time and temperature on color extraction

The effect of temperature was observed with different time intervals on the color extraction of grapes as shown in Fig. 1.

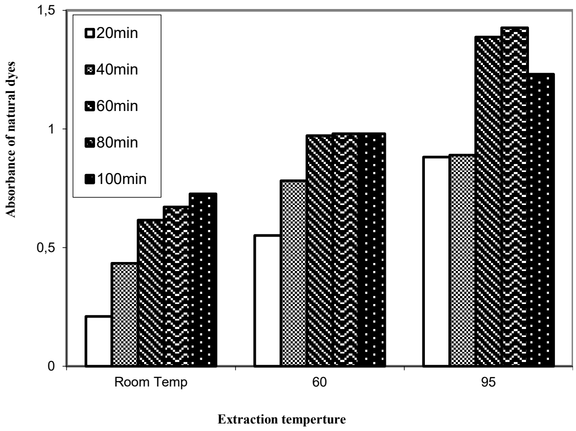

Fig 1. Absorbance of natural dyes at various extraction temperatures as a function of time.

The increase in the absorbance may be regarded as a function of extraction time and temperature [10], absorbance at maximum wavelength (λ_{max}) of the extracted dyes from grapes was found to be at 540 nm at 60°C, the absorbance slightly increased as compared to room temperature. At 95°C the absorbance was found to be maximum (1.388), the majority of the color substances were extracted within the first 60 min of the extraction time. After 60 min there is no significant effect on absorbance.

Color strength and dyeing quality

The K/S values calculated from the colorimeter data for the dye applied on cotton and wool using both mordants, i.e. gallotannic acid and alum, are shown in Table 1.

It was observed that with increase in different conditions, i.e. temperature and mordant, the value of K/S increases which shows good build up properties of the dye. At a low temperature 60°C and without mordant the K/S values of cotton and wool were 0.75 and 0.97, respectively, but these values reached a maximum when the best conditions were applied. The difference in color strength of cotton and wool was significant, i.e. at 95°C; it was 1.65 and 3.47, respectively.

Table 1. Results of dyeing experiments on cotton and wool textile material**.** Temperature of dyeing (60, 80, 95°C), type of mordant (gallotannic acid, alum), CIE Lab Coordinates and selected fastness properties (Wet fastness: Color/ bleeding: 1 = poor, 5 = excellent, fastness to light: 1 = poor, 8 = excellent).

Exp No.	Temp. (°C)	Gallo-tannic acid (%)	Alum (g/l)	Textile material	K/S	L	a	b	Fix (%)	Washing CO3 Change in Staining Shade		Light Fast-ness
1.	60	0	0	Cotton	0.75	76.73	4.62	-1.96	66	3/4	3/4	1/2
				Wool	0.97	65.31	5.84	-2.56	72	4	3/4	2
2.	60	0	2	Cotton	0.92	72.21	4.73	-1.98	67	4	3/4	2
				Wool	1.20	64.24	8.12	-2.12	73	4	3/4	2
3.	60	0	4	Cotton	1.16	70.37	5.06	1.92	68	4	3/4	2/3
				Wool	1.34	64.16	8.38	-1.90	74	4	3	2/3
4.	60	5	0	Cotton	1.15	70.24	5.24	-2.16	68	4	3/4	2/3
				Wool	1.34	63.21	8.92	-2.22	75	4/5	3/4	3
5.	60	15	0	Cotton	1.22	70.13	5.47	-2.31	68	4	3/4	½
				Wool	1.76	62.33	9.16	-2.67	75	4/5	3/4	2/3
6.	60	5	2	Cotton	1.22	71.06	5.72	-2.55	70	4/5	3/4	2/3
				Wool	1.75	61.78	9.24	-2.78	78	4	3	3
7.	60	15	4	Cotton	1.42	70.26	5.88	-2.61	72	4/5	3/4	2/3
				Wool	1.96	61.24	9.76	-2.83	80	4	3/4	¾
8.	80	0	0	Cotton	0.92	74.21	5.42	2.77	68	4	4	2
				Wool	1.13	63.42	8.88	-2.68	74	4/5	4	2/3
9.	80	0	2	Cotton	1.34	71.86	6.36	-2.80	68	4	4	2/3
				Wool	1.56	62.54	8.92	-2.81	76	4/5	4	3
10.	80	0	4	Cotton	1.51	69.38	6.72	2.43	70	4/5	3/4	2/3
				Wool	1.80	61.30	9.74	-3.56	78	4/5	4	3
11.	80	5	0	Cotton	1.22	69.83	6.55	-2.46	70	4	3/4	½
				Wool	1.58	61.07	9.12	-2.98	78	4/5	4	2/3
12.	80	15	0	Cotton	1.41	69.76	7.66	-2.73	72	3/4	3/4	2/3

				Wool	1.89	60.35	11.78	-4.23	80	4	3/4	2/3
13.	80	5	2	Cotton	1.55	69.81	7.68	-3.24	76	4	3/4	2/3
				Wool	2.04	60.11	12.37	-7.64	80	4/5	4	3
14.	80	15	4	Cotton	1.73	69.24	7.88	-4.56	78	4	3/4	3
				Wool	2.56	59.96	14.15	-9.77	82	4/5	4	¾
15.	95	0	0	Cotton	1.21	72.63	8.23	-2.81	68	4/5	4	2
				Wool	1.46	62.10	9.21	-3.49	75	4/5	4	2/3
16.	95	0	2	Cotton	1.50	70.38	5.43	-3.12	70	4/5	4	3
				Wool	1.82	61.34	10.22	-4.25	78	4/5	5	¾
17.	95	0	4	Cotton	1.66	68.72	6.21	-4.37	76	4/5	4	3
				Wool	2.21	58.67	13.43	-7.09	80	4/5	4	¾
18.	95	5	0	Cotton	1.64	68.13	6.82	-3.12	76	4/5	3/4	3
				Wool	2.82	57.16	13.76	-4.15	82	4/5	3/4	¾
19.	95	15	0	Cotton	1.67	68.14	6.88	-3.55	76	4/5	4	¾
				Wool	3.18	57.12	14.26	1.28	82	4/5	4	¾
20.	95	5	2	Cotton	1.65	68.27	6.82	-2.21	76	4/5	4	¾
				Wool	3.42	57.15	14.57	-4.56	84	4/5	4	3
21.	95	15	4	Cotton	1.65	68.38	7.21	-2.86	76	4/5	4	3
				Wool	3.47	57.06	15.86	-5.54	86	4/5	4	¾

Effect of temperature

The effect of temperature has been investigated in detail in Table 1. With an increase in temperature; the strength of the color (K/S value) will also increase. This may be a result of the increase in the disaggregation of the dye molecules and/ or increase in the rate of dye penetration into the fiber [7]. With reference to Fig. 2, without mordant the behavior of both textile materials are the same with leading the strength of wool than cotton. While at maximum mordant the maximum K/S of cotton was obtained at 80°C it rapidly increased in wool between 80°C to 95°C, which can be attributed to the optimum temperature for wool dyeing.

Effect of mordant

Table 1 shows that despite the significant role of temperature in color strength, the concentration of mordants plays a vital and major role. The results indicate that the increase in concentration of mordant shifted the reaction towards more dyeing opportunities producing better yield and fixation. The gallotannic acid is very crucial for dyeing purpose and is used for cotton preparation and permanent retaining of color material [11]. Alum has no effect on color, which helps evenness and brightens slightly the textile material.

In this regard, by using both mordants maximum yield and fastness results were observed at all dyeing conditions. At 95°C without mordant the K/S for cotton was 1.21, which increased to 1.65 at maximum mordant while for wool it increased from 1.46 to 3.47.

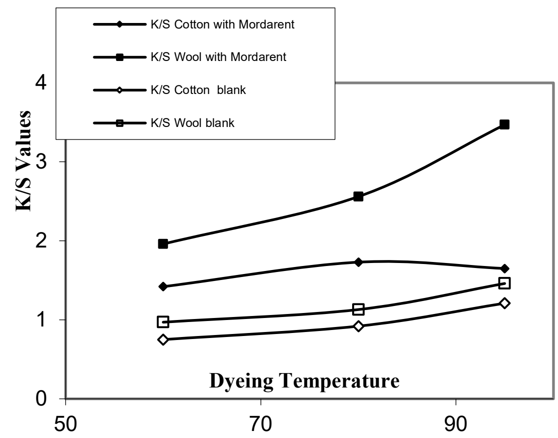

Fig. 2. Color strength at different dyeing temperatures on cotton and wool textile material at optimum mixed mordant system

Fastness properties

Fastness properties of textile material dyed under optimized conditions are shown in Table 1. As can be seen, the dye has fairly good wet fastness properties. As presented, a minor difference in fastness properties was found among the different dyeings of grapes extract. The dyeing experiments carried out without mordant showed no significant difference in light fastness at a higher temperature of 95°C in the case of cotton. But in the case of wool the light fastness slightly increased from 2 to 2/3 with increasing the dyeing temperature. On dyeing with mordant, the light fastness slightly increased in the case of cotton but for wool textile material it was better and became stable at 3/4 rating. The light fastness of wool is not up to the mark of textile standards but it is better as compared to the light fastness of cotton dyed with grapes extract. The washing fastness properties in both cotton and wool textile material are better and can be comparable with most of the available commercial direct dyes. However, the washing fastness of wool dyed fabric is better as compared to the cotton dyed fabric. In natural dyeing the mordanting agent directly influences its dyeing properties, as well as fastness properties.

CONCLUSION

The colorimetric data revealed that the bluish purple grape extract is a good and potential natural colorant source for dyeing wool, as well as cotton textile material. The maximum concentration of extractable dye materials was obtained at 95°C with maximum time, which were sufficient for their use as textile dyes. The shades of the dyeing obtained on cotton and wool were beige and pink, respectively, which are interesting in textile dyeing. The dyeing process was performed in the presence of mordant with different concentration and system i.e. gallotannic acid and alum, however the optimal dyeing was carried out using a mixed mordant system consisting of 15% of gallotannic acid and 4 g of alum. Regarding wet fastness both textile materials showed good results but in various cases the fastness to light was insufficient; therefore, research work optimization needs to be explored further. These experiments indicate that the quality of dyeing and fastness of wool is slightly better than of cotton.

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