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Dyeing of Cotton Fabric using Pomegranate (*Punica granatum*) Aqueous Extract

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The decline in the use of artificial colourants due to their toxicity in food and textile industry, put forward by international market has increased the importance of natural raw materials. From those, pomegranate peel (*Punica grantum*) with solid applications is one of the most important sources of natural dyes. The major colouring component in pomegranate is tannins, extracted from the fresh and dried peels. The aim of present work is to evaluate pomegranate powder as natural textile dyestuff. The work consists of three steps, *i.e.* extraction, characterization and dyeing processes. The dye extraction procedure is conventional and traditional. Dyeing of different textiles, synthetic and natural fabrics with the extract of pomegranate powder has been carried out and dyeing has been optimized. Finally, dyed fabric have been subjected to different textile laboratory tests *e.g.*, colour fastness, light fastness, washing fastness and rubbing fastness (dry and wet).

Key Words: Tannin, Extraction, Cotton, Fastness properties, Textile dyestuff.

INTRODUCTION

Dyes are generally used in textile, paper, cosmetic, food, pharmaceutical and leather industries[1,2]. Water pollution due to discharge of non-biodegradable coloured effluents from textile dye manufacturing and textile-dyeing sill is one of the major environmental concerns in the world today. Strong colour imparted by synthetic dyes to the receiving aquatic ecosystems poses aesthetic and serious ecological problems such as inhibition of benthic photosynthesis and carcinogenicity[3,4]. Textile industry uses and rejects high amounts of water, its wastewater being the main way by which dyes are discharged into the environment. Textile effluents are characterized by strong colour and high concentrations of organic and inorganic compounds caused by residual dyes that were not fixed to the fibers during the dyeing process[5]. The serious environmental problems of public health concern related to coloured wastewaters containing synthetic dyes have diverted researchers promptly to look for eco-friendly products. Hence, there is a world's movement to return to the natural

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dyes[6]. Natural dyes of comprise those colourants that are obtained from animal or vegetable matter without chemical processing. Colouring agents of plants are derived from roots, leaves, barks and trunks or fruits. Henna, madder, pomegranate, turmeric, kamala, eucalyptus *etc.* are well-known examples of natural dyes. The introduction of natural dyes in textile dye houses is coupled to several requirements which have to be fulfilled *i.e.*, adaptation of traditional processes on modern equipments, appropriate supply of plant materials to dye houses and selection of material leading to product with acceptable fastness properties[7,8]. Besides that natural dyes possess some limitations such as lesser availability, poor colour yield, complexity of bond process and non-reproduce ability of shades. They offer much more advantages including renewable sources, minimal health hazard, mild reaction conditions, no disposal problems and harmonization with nature[9].

In this work, pomegranate (*Punica granantum*) was chosen as a representative source for a plant-based yellow dye. The dyes found in pomegranate are tannin; along with some parts of pelletierine called tanante of pelletierine. The rind of the fruit also contains a considerable amount of tannin, about 19 % along with pelletierine. Some of its isomers are also present along with agar, gel like materials and glycosides in rind of fruit[10-12]. The aim of the present work is to investigate the suitability of pomegranate in form of fine powder form for the extraction, dyeing and characterization in textile field.

EXPERIMENTAL

Aqueous extract of fresh and dried rind of pomegranate (commonly known as tanante of pelletierine) was chosen as colouring material (University of Agriculture, Faisalabad, Pakistan) for dyeing plain and weaved white cotton obtained from Sitra Fabrics Mills (Faisalabad, Pakistan). Harvested plant material was grounded using a food blender and sieved through a vibratory screen having mesh size 20 to obtain homogenous particles of uniform size. The powder was then stored in opaque, airtight plastic jars for further experiments. In order to ensure that homogeneous samples were collected, standard sampling techniques were applied.

Extraction procedure: For extraction of dye, one part of powder was mixed in 10 parts of DDW at 30 ± 1 °C and 60 ± 1 °C for 1 h[13]. The obtained extract was boiled at 100 ± 1 °C at atmospheric pressure for 1 h followed by soaking at 30 ± 1 °C for 1 h with constant stirring at 50 rpm. Finally, the extract was separated from the biomass by filtration through cotton cloth to remove insoluble residues[14,15]. This filtrate was subsequently used for cotton dyeing.

Dyeing method: Dyeing was performed at 60 ± 1 °C for 1 h using fixed amount (M:L 1:15, for 1 g cotton fabric, 15 mL of extract was taken) of each extracts in dyeing glass. Dyed samples were extensively washed with cold and hot water to remove any unfixed dyed material and finally dried at ambient temperature[16].

Optimization of dyeing conditions: In all sets of experiments, cotton fabric was dyed using fixed volume of selected aqueous extract (M:L 1:15). To check the influence of experimental conditions such as different temperatures (40, 50, 60, 70, 80 and 90 ± 1 °C), salt concentrations (1, 2, 3, 4 and 5 g/L) and contact time (30, 60, 90 and 120 min) were applied[17].

Colour measurement and fastness properties: Colour changes induced during cotton dyeing were measured using Spectra Flash-Data Colour, SF-600 The colour values were obtained in terms of CIELAB with illuminant D6510°[18]. Colour fastness of dyed samples (M:L 1:15) were evaluated by washing with non-ionic soap (1 g/L) at 60 ± 1 °C for 30 min. Light fastness was analyzed by exposing dyed materials to direct sunlight for 48 h. For evaluation rubbing fastness of dyed cotton fabrics, samples were rubbed at 50 strokes/min at crock-meter using both dried and wet processes. Dyed samples were graded after each experiment[19,20].

RESULTS AND DISCUSSION

Optimization of extraction condition: An extraction for the period of 1 h at 30 ± 1 °C was used as a standard to optimize the extraction conditions during analysis of LAB and colour strength values. The optimized extraction time was boiling for 1 h (Fig. 1). From the results obtained, it can be concluded colour strength was highly dependent on the extraction time. Colour strength increased with increase in stirring time. This may be due to the colourant solubility in water, which increased with stirring time. For long time stirring and heating the some insoluble impurities cause unevenness in dyeing. Overnight soaking followed by stirring after 24 h resulted in more coloured material. The colour strength values might increase due to presence of tannins having acidic nature and some other liable constituents.

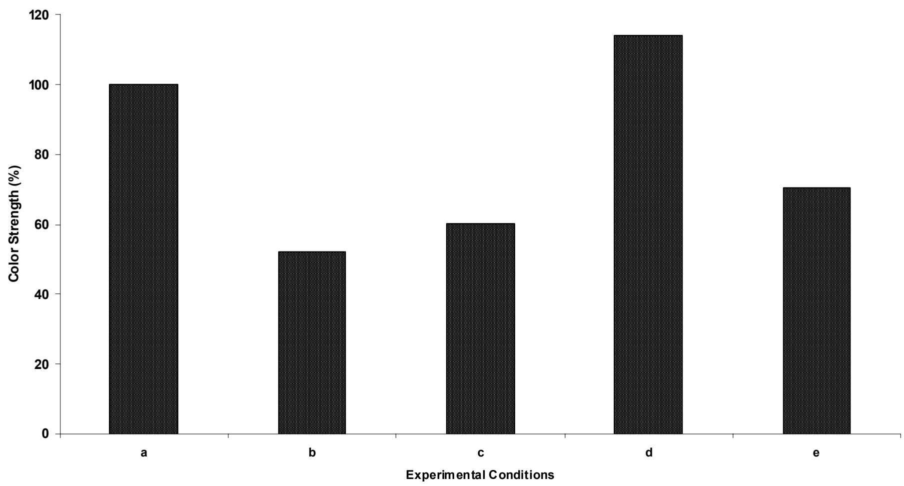

Fig. 1. Effect of different experimental conditions on the colour strength of the fabric. A = Stirring (1 h) at 30 ± 1 °C, b = Soaking at 30 ± 1 °C followed by stirring (1 h), c = Stirring (1 h) at 60 ± 1 °C, d = Boiling (1 h) at 100 ± 1 °C, e = Soaking (1 h) than boiling (1 h) at 100 ± 1 °C

Heating effect was also studied during extraction because low heating cause incomplete extraction during stirring and boiling for more time may cause decomposition of colourant into its constituents like gallic acid or elagic acid[21,22]. The colour strength values either decreases of the unevenness occurs upon dyeing because of involvement of impurities in extraction and upon filtering their appearance in filtrate cause unevenness during dyeing processes. After a long series of extraction process the optimized extraction condition was found 1 in boiling extract.

Effect of temperature on dyeing: Temperature plays an important role in dyeing because at high temperatures dyes may undergo chemical degradation and at low temperatures, incomplete dyeing may occur. In general, best results can be obtained at moderate temperatures, *i.e.* between 40-60 °C. The effect of temperature on the dye ability of the cotton fabrics was conducted at different temperatures (40-90 °C) (Fig. 2). The colour strength values show that upon gradual raising the temperature more dye molecules are attached but at high temperatures dye components probably start to decompose resulting in uneven dyeing[23]. At 60 °C the affinity of dye molecules to the cellulosic fibers was maximum and even dyeing was observed. So that optimized dyeing temperature 60 °C was selected.

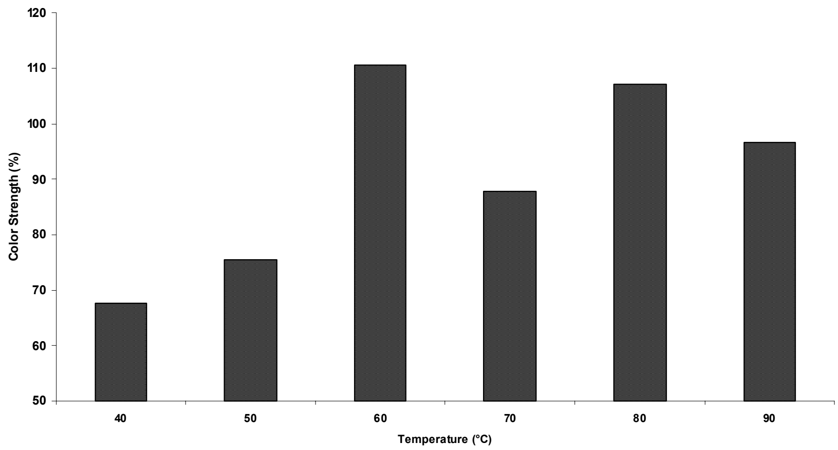

Fig. 2. Effect of dying temperatures on colour strength value of fabric using pomegranate extract

Effect of time on dyeing: In dyeing process time is very important parameter because long- and short-time dyeing gives the same effect as the variation of temperature. During constant heat for a long-time, decomposition of the dye material might occur while short-time causes incomplete dyeing. The colour strength values displayed in Fig. 3 demonstrate these effects. The samples obtained were either uneven or incompletely dyed. The optimized dyeing time is 1 h when the sample is evenly and completely dyed as the result of the maximal dye-uptake of the cotton fibers. The decline in the dyeability after 1 h may be attributed to the desorption of the dye molecules as a consequence of long dyeing time[24].

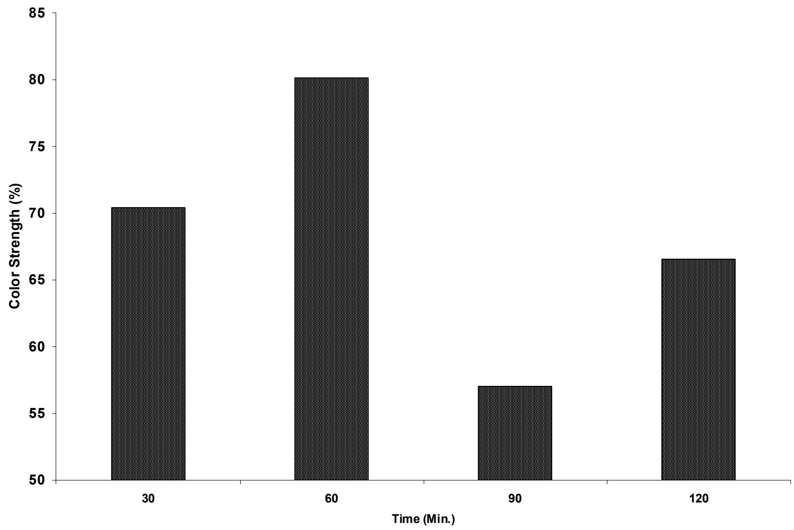

Fig. 3. Effect of dying time on the colour strength of fabric using pomegranate extract

Effect of salt concentration: Addition of electrolytes results in enhanced rate of exhaustion and decrease in diffusion. The efficiency of increase in order of exhaustion of dye bath is of order KCl < Na_2SO_4 < NaCl[25]. During the dyeing process sodium sulphate was used as exhausting agent and hence added at later stage of dyeing from point of view of even dyeing and maximum exhaustion of the dye bath. Inorganic salts have two main functions one is improvement of dyestuff affinity and second is the acceleration of the dyestuff associates and lowering solubility. The sodium ion being present in Glauber's salt interacts with negative charge side of cotton fabric and neutralizes it. The fabric loses its proton of hydroxyl group and become negatively charged. As a result water becomes slightly more acidic. This negative charged layer is produced on fabric and then is entered to attach the colourant on cotton fiber form the dye bath. When sodium ion neutralizes the negative charge of fiber, it facilitates the approach of dye molecules to approach the fiber with in range of hydrogen bond already formed between the fiber and dye molecule. As a result maximum colour is attached to fiber. But as the salt concentration increased, the dye uptake by fiber was reduced. This was because of interaction of complex formation with fiber. This complex formed aggregate on the fiber and it not only causes unevenness but also dye uptake as shown in Fig. 4. Thus the optimized salt concentration is 1 g/L because too high concentration of salt has given uneven samples and it has given maximum affinity for colourant to attach the fiber that resulted in uneven dyeing.

Fastness properties: Table-1 shows that the fabric dyed with the extract of pomegranate rind exhibits good fastness properties[26]. The superior washing property of the colourant is due to the kinetics and thermodynamic effects of the metal complex formation and alkaline media used in dying[27]. In alkaline conditions, tannins are neutralized and good hue can be observed. The good rubbing properties show that the dyeing was properly done and tannins have strong affinity to fabric because on rubbing less colour was detached.

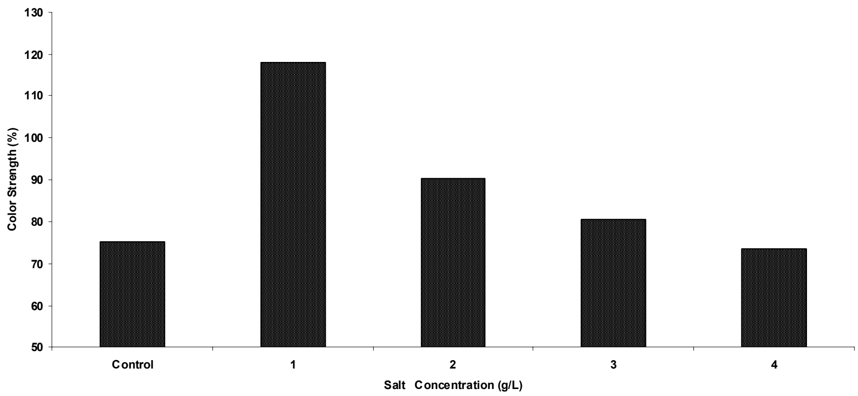

Fig. 4. Effect of various salt concentrations on colour strength of fabric using pomegranate extract. (Control = 0 g/L, 1 = 1 g/L, 2 = 2 g/L, 3 = 3 g/L and 4 = 4 g/L)

TABLE-1
COLOUR FASTNESS PROPERTIES OF DYED FABRIC USING AQUEOUS EXTRACT OF POMEGRANATE RIND POWDER

Optimized sample / properties	WF	LF	Dry Rubbing	Wet Rubbing
Boiling (1 h, ext)	4-5	5	4-5	3-4
60 ºC dyeing temp	4-5	5	4-5	4
60 min dyeing time	4-5	5	4	4
Salt concentration (1 g/L)	4-5	5	4-5	3-4

Conclusion

The natural dye extracted from pomegranate rind might be used as a possible substitute of synthetic dyes having banned aryl-amine moieties. A systematic study of extraction, characterization and improving the properties of the dye is must to minimize the cost investment, yield maximization and dye purity. The dyes are eco-friendly and safe only when they are easily biodegradable, having no health hazard effects and eco-label. Pomegranate rind dye (tannin) is one of dyes that provide such beneficial properties. The present results have demonstrated the potency of pomegranate rind as a source of natural colouring agents for dyeing of cotton textiles by using both traditional and modern techniques. There are still important aspects, however, which warrant further studies including the selection of plant materials with better fastness properties, further optimization of extraction and influence of the substrates (cotton, wool, silk) on shades and dyeing properties.

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REFERENCES

1. M.S. Chiou and H.Y. Li, *J. Hazard. Mater.*, **93**, 233 (2002).
2. I. Banat, P. Nigam, D. Singh and R. Marchant, *J. Bioresour. Technol.*, **58**, 217 (1996).
3. E. Forgacs, T. Cserhati and G. Oros, *Environ. Internat.*, **30**, 953 (2004).
4. T. Robinson, G. McMullan, R. Marchant and P Nigam, *J. Bioresour. Technol.*, **77**, 247 (2001).
5. P. Cooper, Color in Dye House Effluent, Society of Dyers and Colorists, Bradford (1995).
6. S. Tawfik, *J. Asian Text. Assoc.*, **63**, 165 (2002).
7. T. Bechtold, A. Turcanu, S. Geissler and E. Gangleberger, *Bioresour. Technol.*, **81**, 171 (2002).
8. T. Bechtold and A. Turcanu, *J. Clean Prod.*, **11**, 499 (2003).
9. M.D. Teli, R. Paul and P.D. Pardehsi, *J. Colourage*, **47**, 43 (2000).
10. P.S. Vankar, A. Mishra, V. Tiwari and B. Ghorpade, *Asian Textile J.*, **8**, 30 (2001).
11. D. Gupta, S.K. Khare and S. Laha, *J. Color Technol.*, **120**, 167 (2004).
12. M. Montazer, M. Pravinzadeh and A. Kiumarsi, *J. Color Technol.*, **120**, 161 (2004).
13. P.S. Vankar, V. Tiwari and B. Ghorpade, *Colourage*, **3**, 21 (2000).
14. X.X. Feng, L.L. Zhang, Y.J. Chen and Y.C. Zhang, *J. Clean Prod.*, (1-7) (2005).
15. D. Gupta, M. Gulrajani and S. Kumari, *J. Color Technol.*, **120**, 205 (2004).
16. H.M. Zody, *J. Rad. Phys. Chem.*, **7**, 1 (2004).
17. K. Sachan and V.A. Kapoor, *J. Colourage*, **9**, 1 (2004).
18. M.L. Gulrajani, R. Srivasatava and M. Goel, *J. Color Technol.*, **117**, 1 (2001).
19. H.T. Lokhand and V.A. Dorugade, *J. Am. Dyestuff Reporter*, **88**, 29 (1999).
20. Anonymous, The Society of Dyers and Colorists, Standard Methods for the Assessment of Color, Third Report of Fastness Test of Textile Coordination Committee Bradford, England, pp. 24, 37, 63, 71 (1990).
21. A.A. Ansari and B.D. Thakur, *J. Colourage*, **47**, 15, 18 (2000).
22. B.S. Bhal and A. Bhal, Advanced Organic Chemistry, Aromatic Carboxylic Acids and Hteri Derivatives, edn. 2, Vol. 44, p. 1123 (1983).
23. D. Gupta, S. Kumar and M.L. Gulrajani, *J. Color Technol.*, **17**, 328 (2001).
24. H.G. Prabu and M. Sundrarajan, *J. Color. Technol.*, **118**, 131 (2002).
25. V.A. Shenai, *Direct Cotton Dyeing IIT*, *India*, **5**, 161 (1992).
26. R. Shamey and T. Hussein, Textile Progress (Critical Solutions in the Dyeing of Cotton Textile Material) Wood Head Publisher, 2002; Vol. 1 and 2, p. 37 (2005).
27. V.A. Popoola, *J. Appl. Polym. Sci.*, **77**, 752 (2000).

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