Pak. J. Sci. Ind. Res. 2008 **51**(3) 131-135

Relative Study of the Colour Fastness of Cotton, Woolen and Silk Fabrics Dyed With Walnut Bark Dye

Bushra Khalid*, **Azra Yaqub**, **Lubna Liaquat and Mohammad Sohaib**

Applied Chemistry Research Centre, PCSIR Laboratories Complex, Lahore-54600, Pakistan

(received February 19, 2008; May 27, 2008; accepted June 12, 2008)

Abstract. Natural walnut dye was extracted from walnut bark and applied to cotton, woolen and silk fabrics with the same depth of colour and colour fastness was assessed. Walnut dye had good saturation on all the three fabrics and its colour fastness properties ranged between good and excellent.

Keywords: walnut bark dye, *Juglan regia*, natural dye, colour fastness

Introduction

Natural and synthetic dyes are used for dyeing of fabrics and pottery. Some synthetic dyes such as disperse dyes and azo amine dyes have harmful effects on human beings causing allergy, cancer etc., and are anti-environment (IARC, 1975; Scott, 1952). Natural dyes are less allergenic, non-toxic and environment friendly and can be used in textile, pharmaceutical, food and cosmetic industry safely (Ali *et al.*, 2007). Shades produced with most of the natural dyes are not bright, so mordants are used to produce fast and bright colours (Gulrajani and Gupta, 1992), while some dyes are substantive and can be directly applied on the textile fabrics, wool and leathers, without any need of mordants.

The drawback associated with natural dyes is that there are no suitable standard shade cards and standard test procedures relating to their extraction and other dyeing properties. A lot of work is, therefore in progress to improve poor reproducibility and lack of desirable properties of natural dyes (Ali *et al.*, 2007).

The present work is concerned with the extraction of natural dye from the *Juglan regia* (walnut), dyeing of various fabrics (cotton, woolen and silk) with it and then studying the fastness properties.

Juglan regia (walnut) belongs to the family Junglandaceae. It is a slow growing tree in northern parts of Pakistan. It is planted mainly for timber and nuts. The husk is smooth and nuts are easy to split (Cannon and Cannon, 1994). Green hulls or rinds of walnut were used for dyeing. The roots, inner bark referred to as walnut bark, was also used even though it had less potency of colour than the rind (Rita, 1971). Fruit is excellent for eating and baking. It is often used in confectionery and ice cream.

*Author for correspondence; E mail: pcsir@brain.net.pk

All parts of the tree especially bark and nuts contain a substantive brown dye. This dye can be used to give various shades of brown and yellow. The colours are fast and permanent. Mordant may be used to produce a range of shades particularly with chrome, copper, alum and iron etc. The bark dye gives a pure colour to wool by applying bismuth and tin as mordant and brown violet on long simmering (Cannon and Cannon, 1994).

The most important dye pigment in walnut is Juglone, which is a derivative of naphthoquinones. Purified Juglone gives a red orange dye, which can be modified by the presence of tannins and flavonoids in the plant. Biologically active naphthoquinones are secondary metabolities of many plants. Juglone (5-hydroxy-1,4-naphthaquinone) is a naturally occurring naphoquinone that forms derivatives which are more extractable substances of the roots, leaves and green skin of walnut. Juglone and its derivatives have a wide spectrum of applications in folk medicine, cosmetics, pharmaceuticals and agro-eco system protection. Naphthoquinone derivatives have been used as antiviral and antifungal constituent of man preparation for skin colouring and hair colour dyes (Tomaszkiewicz and Vogt, 2004).

Materials and Methods

Instruments. D400 IR dyeing machine (SDL Atlas England); Launderometer (Roaches), Perspirometer kit (SDL Atlas England); oven, Ci 3000 + Xenon; weatherometer (Atlas England); water bath; grey scales for staining (ISO 105 A03); grey scale for change in shade (ISO 105 A02); crockmeter (SDL Atlas England); multifiber (DW).

Chemicals. Detergent ECE (without optical brightener), sodium per borate, l-histidine monochloride monohydrate, sodium dihydrogen orthophosphate, distilled water, sodium

carbonate, sodium hydroxide, acetic acid, sulphuric acid, and perchloroethylene solvent.

All the chemicals and solvents used were of AR grade.

Collection of bark and extraction of colour. The most often used parts of plant for dyeing are leaves and fruit husk (Onal *et al.,* 2004). In this study only bark was used for extraction of the dye.

Walnut bark was purchased from Murree market and thoroughly washed with water and dried. It was ground into powder and sieved through 22 mesh size strainer. 500 g bark powder was soaked in 5 litre water overnight, boiled for 2 to 3 h and then subjected to stirring for 3 to 4 h at simmering temperature. A dark brown coloured dye solution obtained was filtered and kept for dyeing and other tests (Kongkachuichay *et al.,* 2002).

Dyeing with walnut dye. 20 g Fabrics of cotton, wool and silk each were dyed with the same depth of walnut bark dye extract in the D400 IR dyeing machine (SDL Atlas England) with programmes to control temperature (100 °C), time 1 h and speed of circulation 1.5 rpm. The three dyed fabrics were used to study the colour fastness to washing, perspiration, rubbing fastness, light fastness, dry cleaning, fastness to water and sea water, heat fastness and spotting to alkaline and acid colour fastness (Paul *et al.,* 2003).

Fastness determination. Wash fastness test of all the three dyed fabrics was determined according to ISO 105 C06 method. Light fastness was examined according to ISO 105 standard method procedure B02. Rubbing fastness (dry and wet) test was carried out according to ISO 105x12 standard test procedure. Colour fastness tests to dry cleaning, water, sea water, and perspiration (acidic and basic) were carried out according to ISO 105: D01, E01, E02 and E04 methods, respectively. Colour fastness to spotting of acids and alkalies tests were performed according to ISO E05 and E06 methods, respectively (BS 1006: 1990).

Washing fastness: Washing fastness was determined by preparing the soap solution containing 4 g detergent and 1 g sodium perborate per litre of distilled water. Then pH was adjusted to 10.5±0.1 by addition of approx. 1 g of sodium carbonate. Cotton, woolen and silk fabric pieces of size. 10 x 4 cm were attached to multifiber DW of the same measurements by sewing along with one of the shorter sides. The three composite specimens were put into glasses of launderometer (Roaches) for 30 min at 60 °C having liquor ratio 50:1. Launderometer or Washtec consists of a water bath containing a rotatable shaft which supports radially, stainless steel container (75±5 mm diameter x 125±10 mm height) of capacity 550±50 ml, the bottom of the container being 45±10 from the centre of the shaft. The shaft/container assembly is rotated at a frequency of 40±2/min. After 30 min, samples were removed from the Washtec. Stitches were removed and the specimens were dried at temperature not more than 60 °C. The change in stain and in shade was assessed with the help of grey scale.

Colour fastness to perspiration. Tests were carried out by dipping the fabrics into l-histidine monohydrochloride monohydrate solution according to ISO 105 E04 method. Specimens of cotton, wool and silk of 4 cm x 10 cm measurement were attached to pieces of multifibre of the same measurement by sewing alongwith one of shorter sides and dipped separately into alkaline and acidic solutions for 30 min having liquor ratio 50:1. Then the cotton, wool and silk specimens were placed in the perspirometer kits and the desired pressure was applied. Perspirometer kits are test devices each consisting of a frame of stainless steel into which a weight piece of mass 5 kg and base of 60 mm x 115 mm is closely fitted so that a pressure of 12.5 kpa can be applied on test specimens measuring 40 mm x 100 mm, placed between glass or acrylic resin plates measuring 60 mm x 115 mm x 1.5 mm. The test device is constructed in such a way that a pressure of 12.5 kpa remains unchanged. The perspirometer kits (acidic and basic) for tests of the three fabrics were placed in the vacuum oven for 4 h and then the kits were removed from the oven and the stitches were opened except on one shorter side. Specimens were dried at 60 °C by hanging in air. Change in the colour of each specimen and staining of the adjacent fabric (DW) were assessed with grey scale.

Rubbing fastness. Dry rubbing on cotton was carried out with the help of crockmeter under a pressure of 9N in to and fro movements on standard rubbing cloth. Test sample cotton of 5 cm x 14 cm measurement was taken. Both warp and weft readings were noted. Same procedure was adopted for wool and silk and values were taken with the help of grey scale.

Wet rubbing. Wet rubbing on cotton fabric was done under the same conditions of crockmeter as in the dry rubbing except the standard rubbing cloth was soaked into 100% deionized water. Same procedure was repeated with woolen and silk fabrics and the change in colour and in stain was assessed with the help of grey scale.

Light fastness. Light fastness was carried out according to ISO 105 standard procedure B02; in weatherometer by Atlas. Xenon arc lamp was used which is an artificial light source representative of natural day light D65. Fabrics of measurement 7 cm x12 cm of cotton, wool and silk were exposed to

Xenon arc lamp for 24 h, at standard testing conditions using blue wool as standard reference fabric. The above three treated fabrics were compared with grey scale for evaluation.

Colour fastness to dry cleaning. Undyed cotton twill bags of 10 cm x 10 cm measurement were stitched around three sides and cotton, woolen and silk pieces of 4 cm x 10 cm measurement were placed into separate bags alongwith 12 non-corrodable steel disks and the fourth side of the bag was sewed. Then the bags were placed in separate containers of Washtec containing 200 ml of perchloroethylene solvent and agitated for 30 min at 30±2 °C. Afterwards the bags were removed from the container. The samples were squeezed to remove surplus solvent and dried in the air by hanging them at a temperature of 60±5 °C. Assessment of change in colour of samples and change in colour of solvent was carried out with the help of grey scale.

Colour fastness to water and sea water. Colour fastness to water and sea water was evaluated in the same manner as for the colour fastness to perspiration. ISO-105 E01 and E02 methods were used for water and sea water, respectively. In case of water, fabrics were dipped in deionized water, while for colour fastness to sea water, fabrics alongwith multifibers were dipped in NaCl solution (30 g/l) for 30 min. For both water and sea water the above three treated composite fabrics were put in perspirometer kit. These kits were placed in the oven for 4 h at 37±2 °C. Then the specimens were dried at temperature not more than 60 °C. Change in shade and in stain were noted with the help of grey scale.

Colour fastness to dry heat. Dry hot pressing was done according to ISO 105 XII. Specimens of cotton, wool and silk were pressed at temp. 110±2 °C with hand iron and change in colour was assessed with grey scale.

Colour fastness to spotting acids and alkali. Spots of acetic acid 300 g/l, sulphuric acid 50 g/l, tartaric acid 100 g/l and Na_2CO_3 100 g/l of water were put on the specimens and change in shade was assessed with ISO-105 A02 grey scale.

Results and Discussion

Change in staining. When the washing fastness properties of cotton, woolen and silk fabrics were compared, it was observed that on diacetate band of multifiber DW for cotton fabric, grey scale gave good (4-5) rating, while for woolen and silk fabrics, staining was excellent (5). For cotton band of multifiber, staining was good for both woolen and silk fabrics (4-5) rating and (4) for cotton fabric. For nylon band of multifiber, cotton fabric gave satisfactory (3-4) results, woolen gave (4) rating and silk gave good (4-5) rating. For polyester band all the fabrics gave the same rating of 4. For polyacrylic and wool bands, all the three fabric gave excellent (5) rating for staining (Table 1).

Change in shade. Results for change in shade gave satisfactory (3-4) rating for cotton, (4) for woolen and (2-3) rating for silk which is poor as compared to the other two fabrics. Results for change in shade for cotton, woolen and silk dyed with walnut bark extract are given in Table 1.

Colour fastness to acidic and basic perspiration. Results of acidic and basic perspiration can also be seen in Table 1.

Table 1. Washing fastness, alkaline and acidic perspiration fastness of fabrics

Fabric	Washing fastness rating						
	Diacetate	Cotton	Nylon	Polyester	Polyacrylic	Wool	Change in shade
Cotton	4-5	4	4	5	5	5	3-4
Wool	5	4-5	4	5	5	5	4
Silk	5	4-5	4	5	5	5	2-3
Fabric	Basic perspiration						
Cotton	4-5	4	4	5	4-5	4-5	4-5
Wool	5	4-5	4	5	5	5	4-5
Silk	5	4-5	4-5	5	5	4-5	5
Fabric	Acidic perspiration						
Cotton	4-5	4	4	5	5	4-5	4
Wool	5	4-5	4	5	5	4-5	4-5
Silk	4-5	5	4-5	5	4-5	4-5	4-5

***Cotton fabric*.** On diacetate band of cotton fabric the results of acidic and basic perspirations were good (4-5). For cotton and nylon band acidic and basic perspiration results for staining were 4. For polyester band both basic and acidic perspiration gave excellent (5) results. For polyacrylic band it was found excellent (5) for acidic perspiration and good (4-5) for basic perspiration. For wool band, results for both perspirations were good (4-5). Change in shade for acidic perspiration was 4, while it was good (4-5) for basic perspiration for cotton fabric.

***Woolen fabric*.** Acidic and basic perspiration gave excellent (5) rating for diacetate bands of multifiber. For cotton bands change in stain was good (4-5) for acidic and basic perspirations. For nylon bands staining was same i.e. (4). For polyester and polyacrylic bands staining for both perspiration were excellent (5). Wool band also gave excellent staining (5) for basic perspiration and good (4-5) for acidic perspiration; change in shade were good (4-5) for both acidic and basic perspiration.

***Silk fabric*.** For silk fabric dyed with walnut dye, diacetate band showed excellent (5) rating for basic perspiration and good (4-5) rating for acidic perspiration. For cotton bands of multifiber, change in stain was good (4-5) for basic perspiration and excellent (5) for acidic perspiration. For polyester band rating for staining was excellent (5) for both acidic and basic perspiration. For polyacrylic band it was excellent (5) for basic perspiration and good (4-5) for acidic perspiration for silk fabric. For nylon band basic and acidic perspiration gave good (4-5) rating results on silk. Wool band also gave good (4-5) rating for silk fabric for acidic and basic perspiration. Change in shade gave excellent rating (5) for basic perspiration and good rating (4-5) for acidic perspiration.

Light fastness. Results of light fastness are shown in Table 2 for cotton, wool and silk dyed with walnut bark dye extract. Cotton and silk gave rating (4) while wool gave good rating (4-5).

Dry cleaning. Results of change in colour were excellent (5) for cotton, woolen and silk fabrics. Change in colour of solvents was also excellent (5) for all the three fabrics.

Rubbing fastness: *Dry rubbing fastness*. Cotton and wool both gave good rating (4-5) for dry rubbing fastness along warp and weft whereas the silk fabric showed excellent rating (5) for dry rubbing fastness along warp and weft.

***Wet rubbing fastness*.** For cotton fabric, wet rubbing along warp was 3-4 which was satisfactory and acceptable. Along weft wet rubbing was 4. For woolen fabric wet rubbing fastness along warp and weft was 3-4, which is also acceptable. Silk fabric gave wet rubbing fastness rating good (4-5) along warp and weft. Results are shown in Table 2.

Table 2. Rubbing fastness, light fastness and colour fastness to dry cleaning

Fabric	Rubbing fastness				Light fastness	Colour fastness to dry cleaning	
	dry rubbing		wet rubbing		change in shade of fabric	change in shade of fabric	change in shade of solvent
	warp	weft	warp	weft			
Cotton	4-5	4-5	3-4	4	4	5	5
Wool	4-5	4-5	3-4	3-4	4-5	5	5
Silk	5	5	4-5	4-5	4	5	5

Colour fastness to water: *Change in stain*. For cotton fabric diacetate band gave good (4-5) rating. For woolen and silk fabric the rating of change in stain were excellent (5). For cotton band of multifiber DW all three fabrics showed same rating i.e. 4-5 which was good. For nylon band, cotton and silk gave rating 4 for staining while woolen gave good (4-5) rating for staining. For polyester band results were excellent (5) for all the three fabrics. Polyacrylic band showed good rating (4-5) for cotton and silk and excellent rating (5) for wool. Wool band of multifiber gave rating of 4 for staining of cotton, 4-5 rating for woolen and rating 5 for silk fabric (Table 3).

***Change in shade*.** Rating for change of colour fastness to water for all the three fabrics was good (4-5). Results are shown in Table 3.

Colour fastness to sea water. Results of colour fastness to sea water are also given in Table 3.

Staining. For diacetate band cotton fabric showed rating of 4, wool showed excellent (5) rating and silk showed good (4-5) rating for staining. Cotton band of multifiber showed excellent (5) rating for staining of cotton, wool and silk with walnut bark dye. Nylon band gave good (4-5) results for wool and silk and excellent (5) rating for cotton fabric. Polyester band gave good rating for staining (4-5) for cotton and excellent rating (5) for both wool and silk. Polyacrylic band showed excellent staining (5) for both wool and silk and good (4-5) rating for cotton fabric. Wool band of multifiber (DW) gave 5, 4-5 and 4 stain rating for cotton, wool and silk, respectively (Table 3).

Table 3. Results of colour fastness to water and sea water, dry heat fastness and spotting to acids and alkali

	Colour fastness to water						
Fabric	Diacetate	Cotton	Nylon	Polyester	Polyacrylic	Wool	Change in shade
Cotton	4-5	4-5	4	5	4-5	4	4-5
Woolen	5	4-5	4-5	5	5	4-5	4-5
Silk	5	4-5	4	5	4-5	5	4-5
	Colour fastness to sea water						
Fabric	Diacetate	Cotton	Nylon	Polyester	Polyacrylic	Wool	Change in shade
Cotton	4	5	5	4-5	4-5	5	4-5
Woolen	5	5	4-5	5	5	4-5	4-5
Silk	4-5	5	4-5	5	5	5	4-5

	Colour fastness to spotting			
Fabric	Dry heat fastness (at 110 °C)	Acid spotting		Alkali spotting Na_2CO_3 (100 g/l)
		Acetic acid (300 g/l)	Sulphuric acid (150 g/l)	
Cotton	4-5	4-5	4	3-4
Wool	4-5	4-5	3-4	3
Silk	4-5	4-5	4	4

Change in shade for sea water. Change in shade was good (4-5) for all the three fabrics dyed with walnut bark dye extract for sea water (Table 3).

Dry heat fastness: At 110 °C cotton, woolen and silk fabrics gave good (4-5) rating during dry hot pressing. Results are given in Table 3.

Colour fastness to acidic and basic spotting. Change in shade for cotton, woolen and silk fabrics, on spotting with acetic acid (300 g/l) gave good rating (4-5). Sulphuric acid (150 g/l) spotting gave rating 4 for cotton and silk and satisfactory (3-4) rating for wool. Alkali spotting (100 g/l Na_2CO_3) gave satisfactory rating (3-4) for cotton and 3 for wool, while rating of 4 for silk fabric. Results are tabulated in Table 3.

Conclusion

Results showed that natural walnut bark dye has excellent to good colour fastness properties. Colours are fast and permanent. Dye can be applied without mordant and has good saturation on cotton, woolen and silk fabrics. It is environment friendly and has no health hazard effects.

References

Ali, S., Nisar, N., Hussain, T. 2007. Dyeing properties of natural dyes extracted from eucalyptus. *J. Textile Inst.* **98:** 559-562.

BS 1006:1990. *Methods of Test for Colour Fastness of Textile and Leather*, ISO 105-C06 (1-3), D01 (1-2), X12 (1-2), E01 (1-2), E02 (1-2), E04 (1-3), B02 (9-10), E05 (1-2) and E06 (1-2), 5th edition, Society of Dyers and Colourists, Bradford, USA.

Cannon, J., Cannon, M. 1994. *Dye Plants and Dyeing*, pp. 29-30, Timber Press, Portland in Assoc. with The Royal Botanic Gardens, Kew, UK.

Gulrajani, M.L., Gupta, D. (eds.) 1992. *Natural Dyes and Their Applications to Textiles*, Indian Institute of Technology, New Delhi, India.

IARC, 1975. *IARC Monographs on the Evaluation of Carcinogenic Risk of Chemicals to Man*, vol. **9**, 286 p., International Agency for Research on Cancer, Lyon, France.

Kongkachuichay, P., Shitangkoon, A., Chinwongamom. N. 2002. Sudies on dyeing of silk yarn with lac dye: effects of mordants and dyeing conditions. *ScienceAsia* **28:** 161-166.

Onal, A., Camci, N., Sari, A. 2004. Extraction of total dye stuff from walnut leaves (*Juglan regia* L.) and its dyeing conditions for natural fibers. *Asian J. Chem.* **16:** 1533-1539.

Paul, S., Sharma, A., Grover E. 2003. Standardization of dyeing variables for dyeing of cotton with walnut bark. *Man-Made Textiles in India* **46:** 152-155.

Rita J.A. 1971. *Natural Dyes and Home Dyeing: A Practical Guide with over 150 Recipes*, Dover Publications Inc., New York, USA.

Scott, T.S. 1952. The incidence of bladder tumours in a dye stuffs factory. *Br. J. Ind. Med.* **9:** 127-132.

Tomaszkiewicz-Potepa, A., Vogt, O. 2004. Juglone - a biological active metabolite from plants of family Juglandaceae. *Wiad. Chem.* **58:** 881-894.