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EXTRACTION OF NATURAL COLORANTS FROM TWO VARIETIES OF SAFFRON (*CROCUS SATIVUS*) AND THEIR DYEING EVALUATION ON GOAT LEATHER

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ABSTRACT

The colorant was extracted from Iranian and Italian *crocus sativus* stamen by maceration, mixing both the samples separately in 300 mL methanol (solvent). Mixture was kept in dark for 36 hours, filtered and then each filtrate was washed with separate 300 mL *n*- hexan to remove fatty acids. Pure lower layer of *crocus sativus* was separated and applied for further studies. The colorants were applied to the goat leather surface and its color fastness to mild washing evaluation was done according to standard procedure. Color fastness to mild washing evaluation showed almost no color change in red *crocus sativus* and same with sodium dichromate (4) and slight color change (3-4) in all other samples which showed the retaining capacity of colorant at leather surface.

KEYWORDS: *Crocus sativus*; Extraction; Mordants; Leather; Color fastning.

INTRODUCTION

A kind of spice, saffron is obtained from *crocus sativus* flower that is mostly known as saffron crocus. It is an utmost advantageous plant amongst the Iridaceae family in the world. About for the last thirty five hundred years saffron crop is being grown as a spice. Saffron's dried stigmas are the much expensive which have been fascinating since beginning, owing to its medicinal benefits (Plessner *et al*., 1989). Both spice and the plant are commonly known

with the name of saffron. The domestic use of saffron dates back two thousand to fifteen hundred years BC as per study of Archeology and history (Grilli, 2004). Its colour is due to crocin, smell is due to safranal and the special bitter taste is due to the presence of glycoside picrocrocine (Basker *et al.*, 1999). Its taste and idoform or hay like scent is due to the chemicals picrocrocine ($C_{16}H_{26}O_7$) that is considered chief sour constituent of *crocus sativus*. Picrocrocine function as a monoterpene glycosides predecessor for safranal ($C_{10}H_{14}O$) that is volatile oil causing aroma (Lozano *et al.*, 2000). Its odour is sometimes called as sea air. Due to its powerful yellow color it is applied in saffron rice and other dishes. A carotenoid dyestuff, crocin is also found in saffron that imparts rich golden shade to textiles and dishes. Almost ninety percent of the saffron is produced in Iran. There are many synthetic dyes which discharge carcinogenic amines and harmful for human skin. In 2003 AZO dyes were banned by European Union because of their carcinogenic and injurious effect on human reproductive system.

Thus by considering these facts natural dyes have attracted human attention and as an alternate to synthetic dyes, natural dyes are being produced. These dyes don't have toxic residue as synthetic dyes and their waste water can be used on agricultural land to deliver nutrients to harvests. They are renewable, non-toxic, non-carcinogenic, non-poisonous and non-hazardous to life. For the natural dyes several plants have been studied for dyes extraction and experimented on silk, wool cotton and leather for instance pomegranate, capsicum, tamarind, walnut, henna, euclyptus bark, and onion peel (Inayat et al., 2010). Similarly natural colors are used for dyeing leather (Musa *et al.*, 2009). Beet root (Sivakumar et al., 2009) Mucuna pruriens (Sundari et al., 2015), Justicia carnea hooker (Paschal *et al.*, 2010), Rubiatinctorum (Onem *et al.*, 2011), Bixaorellana seeds (Selvi *et al.*, 2013).

Thus the current study was designed and colorants were extracted from the two varieties of crocus stivus and their dyeing properties on the leather were studied.

MATERIALS AND METHODS

The experiment was designed for the extraction of environment friendly colorants from the two varieties of *crocus sativus* for application on goat leather.

COLLECTION OF SAMPLE

The samples of Iranian brand and Italian brand crocus sativus were collected from local market at Lahore (Pakistan).

EXTRACTION OF COLORANTS FROM SAMPLE

Pre weighed 30g red (Iranian) and 30g brown (Italian) *crocus sativus* were separately mixed with 300 mL methanol and kept in dark for 36 hours with occasional shaking. After 36 hours both samples were filtered and filtrates were separately washed with 300 mL *n*-hexane to remove fatty acids. Pure colorant extracts of *crocus sativus* in lower layer was separated and stored for further studies.

Table 1: Extraction of colorants from sample.

Sample Type	Weight of sample (g)	Maceration solvent	Washing solvent	Extract (Colorant)
Red saffron (Iranian)	30 g	300 mL Methanol	300 mL *n*-hexan	150 mL
Brown saffron (Italian)	30 g	300 mL Methanol	300 ml *n*-hexan	150 mL

PROCESSING OF LEATHER

Wet blue goat leather was taken and further converted to crust blue.Goat skin with pH range 3.9-4.0 was chosen for experimentation because of better penetration of dye under normal condition. Two strips of wet blue leather with 30 cm length and 15 cm width were taken and the following steps were preceded to dye the leather.

NEUTRLIZATION

As the animal leather has low pH so to attain the pH equal to 7, neutralization was done as at high pH leather structure open and becomes more flexible. For neutralization two leather strips (named A and B) were separately treated with water, 2% neutralizing syntan (Protan 114P and Protan 150P) and 1.0% sodium bicarbonate and these strips were left to rotate in a drum for 45 minutes. The pH of leather was tested by using two liquefied indicators BCG (Bromocresol Green) and BCP (Bromocresol purple). Then exhausted and splashed healthy.

DYEING

Strip ‘A’ was dyed with Red *crocus sativus* colorant and strip ‘B’ was treated with Brown *crocus sativus* colorant for 60 minutes in two drums separately.

FAT LIQUORING

Three different kinds of oils (Pelantola BA, Olindol C×70 and Saltostate KSB), each 10 gram, were added to lubricate and soften the leather fibre and the drums were rotated for 45

minutes. After that 3% formic acid solution was added in three turns with 10+10+25 minute's difference in both the drums equally for the fixation of pH i.e. pH 3.5.Then dried and rinsed.

APPLICATION OF MORDANTS

Each dyed strip was cut into three equal parts and dipped in three mordant like potassium dichromate (5 g $KMnO_4$ in distilled water up to 200 mL), aqueous solution of sodium dichromate (5 g $Na_2Cr_2O_3$ in distilled water up to 200 mL) and formic acid separately and left for 24 h. Then rinsed and dried.

COLOUR FASTNESS TO MILD WASHING (ISO 15703:1998) OF LEATHER SAMPLES

According to the ISO 15703:1998 the color fastness to mild washing of the leather samples was proceeded. To prepare washing liquor 5.0 gram of detergent per litter of water was mixed and stirred vigorously up to there was no any solid part visible. Then, a drum having 100ml of washing liquor and 20PTFE rods was heated to $30^oC \pm 2^oC$. Each sample was employed in the container and the container was revolved at 40 rpm ± 5 almost for thirty minutes. During this process the temperature was maintained at $30^oC \pm 2^oC$. Then the washing liquor was replaced with the 100ml of fresh water at the maintained temperature ($30^oC \pm 2^oC$) for rinsing purpose. The process of rinsing was repeated once more using fresh water. After removing the processed leather sample from the drum, it was kept in between the sheets of blotting paper which were then kept in between the glass plates. These glass plates were kept under pressure for one minute by putting the weight of 4.5 kg on it. Then the samples were taken out, unfasten and were opened out on the horizontal grill in such a way that there was no touching in between sample leather and adjacent fabric. Then the sample was let dry in air under controlled standard conditions as described in ISO 2419 ($20^o C \pm 2^oC$ and 65 ± 4% RH). The leather sample was ventured lightly. The assessment of the grain side of the leather for visual change was done according to the ISO 105 A02 and also the visual assessment of the staining of the adjacent fabric was done according to ISO 105 A02. The same procedure was followed to check the color fastness to mild washing of all the remaining seven samples and their reading sand change in color was noted down.

RESULTS AND DISCUSSION

Two varieties of *crocus sativus* (red and brown) were collected and the dyes were extracted through maceration using methanol as solvent and washing with *n*- hexane. These dyes were stored in dark at room temperature.

Table 1: Leather colors of *crocus sativus* colorant.

Sr.No	Colorant	Solvent	Mordant	Leather color
1	Red *crocus sativus* extract	Water	-	
2	Brown *crocus sativus* extract	Water	-	
3	Red *crocus sativus* extract	Water	Potassium dichromate	
4	Brown *crocus sativus* extract	Water	Potassium dichromate	
5	Red *crocus sativus* extract	Water	Sodium dichromate	
6	Brown *crocus sativus* extract	Water	Sodium dichromate	
7	Red *crocus sativus* extract	Water	Formic acid	
8	Brown *crocus sativus* extract	Water	Formic acid	

Table 2: Color fastness to mild washing of *crocus sativus* colorant dyed leather.

Sr.No	Sample type	Mordant	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	Change in Color
1	Brown *crocus sativus*	-	5	5	5	5	5	5	3-4
2	Red *crocus Sativus*	-	5	5	5	5	5	5	4
3	Brown *crocus sativus*	Sodium dichromate	5	5	5	5	5	5	3-4
4	Red *crocus*	Sodium dichromate	5	5	5	5	5	5	4

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5	Brown *crocus sativus*	Formic acid	5	4	5	5	4	5	3
6	Red *crocus sativus*	Formic acid	5	5	5	5	4	5	3-4
7	Brown *crocus sativus*	Potassium dichromate	3	3	4	3	3	3	3
8	Red *crocus sativus*	Potassium dichromate	3	3	3	3	4	3	3

The pH of goat leather was increased by neutralization process as low pH (3.9-4) of animal skin has less penetrating ability and at high pH the pores of leather structure opens and becomes more flexible which increases the dye penetrating ability. Leather is a protein textile composed of long chains of amino acids which coil up on themselves because of hydrogen bonding and other intermolecular forces. Leather protein consists of two major functional groups (-NH_2 and –COOH) which affect the ability of dye interaction. The functional group -NH_2 protonate to give positively charged group and the other functional group –COOH deprotonate to give negatively charged functional group. Although the colorant applied on leather gave good penetration and represented excellent results of color fastness to mild washing with color change range 4 of red *crocus sativus* and color change range 3-4 of brown *crocus sativus* which were evaluated against acetate, cotton, nylon, polyester, acrylic and wool.

For color fixation of basic dyes on leather with less pH three mordant were used:-The mordant Sodium dichromate on leather was evaluated against grey scale with the upper given same properties. The color change range of red *crocus sativus* with Sodium dichromate was 4 and with brown sample it was 3-4 showing that red saffron sample is excellent as compare to brown one. The color change range of formic acid mordant with red *crocus sativus* colorant was 3-4 and with brown one it was 3, representing good results of red one with formic acid as compare to brown one. The color change range of potassium dichromate mordant with red *crocus sativus* was 3 and the same was observed with brown one, showing equal tendency.

CONCLUSION

Two natural colorants of red and brown saffron were selected and dyes were extracted through maceration using methanol as solvent. The dyes of red and brown saffron were

applied on wet blue goat leather by using mordant potassium dichromate, sodium dichromate and formic acid. The colorant were applied to the leather surface and its color fastness to mild washing evaluation was done according to standard procedure. Color fastness to mild washing evaluation showed almost no color change in red *crocus sativus* and same with sodium dichromate (4) and slight color change (3-4) in all other samples which showed the retaining capacity of colorant at leather surface. The best results were represented that of red saffron with and without mordant as compare to brown saffron.

REFERENCES

1. Plessner, O., M. Negbi, M. Ziv,D. Basker., Effects of temperature on the flowering of Crocus (*Crocus sativus L.): Induction of hysteranthy. Israel J. Bot*, 1989; 38: 1-7.
2. Grilli Caiola, M., Saffron reproductive biology.*Acta Hort*., 2004; 650: 25-37.
3. Basker, D, M. Negbi., The uses of saffron.*Econ. Bot*, 1999; 3: 228-236.
4. Lozano P, Castellar M, Simancas M, Iborra L Quantitative high-performance liquid chromatographic method to analyse commercial saffron (Crocus sativus L.) products. Journal of Chromatography. 1999; 830: 477-483.
5. Inayat, A., Khan, S. R., Waheed, A., Deeba, F., "Applications of Eco-friendly Natural Dyes on Leather using Different Modrants.Proceeding Pakistan Academy Science," 2010; 47(3): 131-135.
6. Musa, A. E., Madhan, B., Madhulatha, W., Raghava Rao, J., Gasmelseed, G. A., Sadulla, S. Coloring of leather using henna-Natural alternative material for dyeing. The *Journal of the American Leather Chemists Association*, 2009; 104(5): 183-190.
7. Sivakumar, V., Anna, J. L., Vijayeeswarri, J. and Swaminathan, G., "Ultrasound Assisted Enhancement in Natural Dye Extraction From Beetroot for Industrial Applications and Natural Dyeing of Leather,"Ultrasonics Sonochemistry, 2009; 16(6): 782-789.
8. Sundari, N., "Extraction and Optimization of Mucuna pruriens for Dyeing of Leather," Polish Journal of Chemical Technology, 2015; 17(2): 57-63.
9. Paschal, F. D., Danladi, A. A., Myek, B., Sunday, D. J., Joshua, I., Ferdinand, N., "Extraction of Dyestuff from Justicia carnea hooker and its Application in the Dyeing of Wool, Leather and Cotton," International Journal of Mordern Chemistry, 2015; 7(2): 81-90.
10. Onem, E., Gulumser, G.s, Ocak, B. Evaluation of natural dyeing of leather with Rubia tinctorum Extract. *Ekoloji Dergisi*, 2011; 20(80).

11. Selvi, A. T., Aravindhan, R., Madhan, B., Rao, J. R., "Studies on the Application of Natural Dye Extract from Bixa orellana Seeds for Dyeing and Finishing of Leather," Industrial Crops and Products, 2013; 43: 84-86.