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Extraction of natural dye from *Rosa damascena* Miller. - a cost effective approach for leather industry

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Abstract

Floral waste is considered an inexpensive source of natural dyes. Keeping in view the detrimental effects of synthetic dyes on human health and climate, the study was devised to utilize the waste petals of *Rosa damascena* as natural dye and assessed its potency on chrome tanned goat crust leather. Eco-friendly, aqueous method was adopted for the extraction of the dye. Various shades were obtained with pre-mordanting and post-mordanting methods using ten different mordants. Spectrophotometer (Spectraflash SF-650X) was used for evaluation of colour coordinates of dyed substrates. Very good colour fastness properties with respect to rubbing and light were found with copper sulphate, ferrous sulphate, ferric chloride and acetic acid mordants. Good colour fastness properties were also obtained without mordant. The findings of the study reveal that *R. damascena* petals are a good source of natural dye for leather dyeing, which will help local tanning industry to minimize environmental problems, lessen dermal issues by providing eco-friendly, non-carcinogenic and non-allergic dyes at low cost.

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Introduction

The leather industry is one of the biggest consumer of azo dyes (Sudha *et al.,* 2014) which belong to the chemical class of synthetic dyes (Saratale *et al.,* 2011). Basically, synthetic dyes are produced from cheap petroleum and coal-tar sources (Siva, 2007; Devi *et al.,* 2013) which not only pollute the environment during their synthesis, but also release effluents into water bodies after dyeing process (Zaharia *et al.,* 2012; Gupta *et al.,* 2013). They are injurious to health, destroying eco-system (Thiyagarajan *et al.,* 2015) and toxic to aquatic biodiversity (Danazumi and Bichi, 2010). Owing to mutagenic and teratogenic impacts of azo dyes (Abramian and El-Rassy, 2009) Germany was the first country which imposed ban on carcinogenic dyes (Sankat and Siddique, 2008). European Union, India, France, Turkey and Holland also banned their use (Nadigera, 2001; Kadolph, 2008; Sankat and Siddique, 2008). So, the consequences of synthetic dyes and the strict environmental standards (Purohit *et al.,* 2007) increase the demand of natural colourants (Bose, 2012).

Characteristically, natural dyes have wide variety, renewable, non-toxic, non-carcinogenic, non-poisonous, biodegradable, and non-hazardous to life (Saravanan *et al.,* 2013; Thiyagarajan *et al.,* 2015). Natural dyes enrich with aesthetic properties (Vankar, 2007), helpful to generate employment, safe for ecology (Mahanta *et al.,* 2005), provide economic benefits through sustainable yield and harvest of dye bearing plants (Saravanan *et al.,* 2013). On the other side, commercialization of natural dye can help to boost up the economy of the country (Upadhyay and Choudhary, 2014).

The leather industry is well known trade wise all over the globe including Pakistan. In Pakistan, the leather industry has a significant economic importance (Chattha and Shaukat, 1999), exporting 95% of their saddlery items to international markets and ranks high in employment opportunities (Saif, 2012). Thus, by keeping into account the economic importance of leather and carcinogenic impacts of synthetic dyes, the present study is designed to extract dye from *R. damascena* waste petals as plant waste is considered a cheap source for the extraction of dye (Ahmad *at el.,* 2011). Therefore, in this study the simple eco-friendly methodology was employed for the extraction of dye from the *R. damascena* waste petals. Moreover, the dye potency was assessed on chrome tanned goat crust leather with ten different mordants.

Rosa damascena is commonly known as the Rose (Hussain *et al.,* 2012) considered as a symbol of love, affection (Aaserud, 2013), purity (Boskabady *et al.,* 2011) and spirituality (Ahmad *et al.,* 2010). It is the oldest, most popular flower in cultivation worldwide (Katsumoto *et al.,* 2007), belongs to Rosaceae, native to Central Asia (Aaserud, 2013) and some of its species are native to Europe, northwest Africa and North America (Ekrami *et al.,* 2011). King of flowers (Peter Bealis, 1990) also grows in Pakistan as a commercial crop (Usman, 2015) and locally called 'Gulab' (Hussain *et al.,* 2012). Rose is used for decorative purpose (Masure and Patil, 2014), in weddings, funerals (Hussain *et al.,* 2015), social (Usman, 2015) and religious ceremonies (Boskabady *et al.,* 2011). *R. damascena* has numerous medicinal properties (Rusanov , 2005; Yashaswini *et al.,* 2011; Jitendra, 2012; Hussain *et al.,* 2012,). The colouring matter in the petals is called anthocyanin (Ekrami *et al.,* 2011) and its dye also reported for dyeing wool (Ekrami *et al.,* 2011; Karaboyaci, 2014).

Materials and methods

Collection of raw material

R. damascena waste petals were purchased from the local florist's shop, Lahore, Pakistan at the rate of 0.19 USD.

Leather

The chrome tanned goat crust leather was purchased from the Sialkot, Pakistan at the rate of 7.63 USD.

Chemicals

Commercial grade sodium formate, sodium bicarbonate, sodium hydroxide, formic acid, alum, potassium dichromate, copper sulphate, ferrous sulphate, aluminium sulphate, copper acetate, ferric chloride, acetic acid, copper chloride and tartaric acid were purchased from the local chemical market.

Dye Extraction

100 g *R. damascena* waste petals were weighed and washed thoroughly with water to remove soil and other impurities.

The dye was extracted with distilled water (Antima *et al.,* 2012). Pre-weighed 100 g *R. damascena* waste petals were immersed in 1000 mL distilled water and kept for 36 hours at room temperature. In order to obtain maximize dye yield, the soaked petals were heated gently at 40 ˚C for 40 minutes.

Leather dyeing preparation

10 g goat crust leather was weighed and soaked in the tap water for over-night. Then the wetback crust leather was processed with the solution of sodium bicarbonate and sodium formate in a micro steel drum (Cheng Zhang) for 30 minutes. After this, the processed leather specimens were rinsed with tap water.

Mordanting and dyeing

Pre-mordanting and post-mordanting methods were selected for treating crust leather with ten different mordants. 1 M solution of each mordant was used for mordanting for 60 minutes.

The leather samples were dyed with extracted dye in two steps. In the first step, the crust leather was dyed with 250 ml dye extract for 30 minutes. After 30 minutes, pH of the dye extract was adjusted at 3.0 with formic acid for dye fixation. In the second step, the same process was repeated again.

Drying

The dyed samples were spread under shade for drying.

Colour coordinates of dyed leather specimens

Colour coordinates of goat crust leather of undyed and dyed specimens were determined through spectrophotometer (Spectraflash SF-650X). Colour coordinates L*, a*, b*, C* and h were obtained from the grain side of the specimens. According to colour coordinates, L* refers to 100-0 values by presenting light to dark colours. The value of negative a* expresses the green colour and positive a* value shows red colour. Whereas, negative b* value describes the blue colour and positive value of b* denotes yellow colour. The C* shows the chroma by describing the vividness and dullness of colour while h represents hue which expresses the tones of shades (Torskangerpoll and Anderson, 2005).

Colour fastness properties

The strength of dye affinity with the crust leather was studied by performing colour fastness to rubbing and light using standard methods. The staining and colour change grades were assessed with the ISO grey scales having interpretation range of 1-5, where 1 stands for very poor, 2 for poor, 3 for good, 4 for very good and 5 for excellent (Paschal *et al.,* 2015).

Colour fastness to rubbing

Colour fastness to rubbing of dyed leather specimens was carried out according to SLF-5 (BS-1006) using SATRA rub fastness tester. Colour fastness in dry and wet state was determined from the grain side of the dyed leather specimens. For dry rubbing, grain side of the specimen was rubbed with a dry circular wool felt pad by setting 512 cycles. For wet rubbing, the circular wool felt pads were soaked in boiling water for two minutes and then cooled down. The weight of the soaked wool felt pad was adjusted according to the standard method by squeezing the excess water. The specimens were rubbed with the wet wool felt pads by circulating 256 times on the grain side. The wool felt pads were dried and the extent of staining was evaluated using ISO grey scale for staining (Table 4).

Colour fastness to light

Colour fastness to light of dyed leather specimens was determined by exposing the specimens in sunlight for 24 hours and 48 hours. The pre- and post-mordanted dyed leather specimens of size 3 × 3 cm^2 were exposed to direct sunlight for 24 hours between 11 am to 2 pm. After 24 hours of exposure to sunlight, each specimen was cut into two portions. One portion of each specimen was further exposed to sunlight for 24 hours, hence for a total period of 48 hours. The change in colour of each exposed specimen in comparison to unexposed specimen was assessed using ISO grey scale for change in colour (Table 5).

Results and discussion

Effects of aqueous dye extraction and the significance of floral dyeing

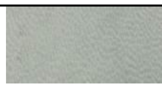
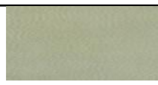
The results of study revealed that aqueous method is effective for the extraction of floral dye (Deshpande and Chaturvedi, 2014) and economical (Ibrahim *et al.,* 1997). Moreover, it has been analysed from results of Fig.1 that water soluble dye extracted from steeped petals within 36 hours produced good results. Furthermore, the obtained results demostratred that dyed specimens found fragrant after dyeing which is reported dynamic quality of floral dyeing (Singh and Srivastava, 2015).

Shades obtained with and without mordants

Results below in Table 1 showed the application of mordants produced 20 varieties of colours. Moiz *et al.,* (2010) carried out a study regarding dyeing of leather with henna also reported the different shades were obtained with different mordants. Furthermore, the analysis of results given in Table 1 also exhibited shade without mordant due to the chrome tanning attribute (Eitel *et al.,* 1984). Analysis of results inferred that 11 out of 20 mordants produced soft fashion shades (Kamel *et al.,* 2011). Table 1 also demonstrated that ferrous sulphate which is a well known mordant and considered for grey to black shades gave one of the significant colour strength (Bhanawat and Mohta, 2014).

The general appearance of dyed leather specimens can be assessed easily. It was found from the visual inspection that dyed leather specimens exhibited a good variety of shades with pre- and post-mordanting techniques. All the specimens were found in good and fair levelness of shade.

Table 1. Shades obtained on dyed leather specimens with and without mordants.

Mordant	Pre-mordanting	Post-mordanting
Without mordant		
Alum		
Potassium Dichromate		
Copper Sulphate		
Ferrous Sulphate		
Aluminium Sulphate		
Copper Acetate		
Ferric Chloride		
Acetic Acid		
Copper Chloride		
Tartaric Acid		

In addition, the post-mordanted dyed substrates produced dark colours. Moreover, good levelness of shade was also found without mordant dyed specimen (Table 1). Overall, *R.damacena* dye was found good in terms of colour evenness from both grain and flesh side of leather.

Colour coordintes of dyed leather specimens

The low values of L* were obtained with post-mordanted method except with acetic acid mordant. Ferrous sulphate was dominant of all mordants in post mordanting method having lower value of L* 37.38 and the ferric chloride was the second highest mordant to produce dark shade having 42.34 L* results with the post mordanting method. Moreover, potassium dichromate evaluated in deep shade as compared to alum, acetic acid, copper chloride and tartaric acid. It was also noticed that lower L* value 49.05 obtained without mordant (Table 2).

Table 2. Colour coordinates of dyed leather specimens with pre-mordanting method.

Mordants	L*	a*	b*	C*	H
Without Mordant	49.05	-0.85	2.45	2.59	109.18
Alum	65.94	-4.15	5.58	6.95	126.61
Potassium Dichromate	65.51	1.30	24.77	24.81	87.01
Copper Sulphate	62.66	-2.85	13.39	13.69	102.03
Ferrous Sulphate	47.55	-1.36	7.50	7.62	100.31
Aluminium Sulphate	65.08	-3.58	13.94	14.39	104.39
Copper Acetate	67.55	-2.53	8.91	9.26	105.83
Ferric Chloride	41.73	-1.14	-2.21	2.54	243.26
Acetic Acid	65.73	-2.39	8.87	9.91	105.11
Copper Chloride	58.56	-1.43	5.72	5.90	104.01
Tartaric Acid	55.41	2.31	5.72	6.17	68.05

Effects of mordanting methods

From the obtained results, it can be analyzed that colour coordinates were better obtained with the post mordanting technique. The results of earlier study of Musa *et al.*, (2009) also concluded that the post-mordanting method characteristically bestowed better results on leather specimens as compared to pre-mordanting method.

Table 3. Colour coordinates of dyed leather specimens with post-mordanting method.

Mordants	L*	a*	b*	C*	h
Without Mordant	49.05	-0.85	2.45	2.59	109.18
Alum	66.33	-6.70	12.99	14.62	117.28
Potassium Dichromate	60.82	3.53	24.55	24.90	81.86
Copper Sulphate	58.87	-2.96	13.41	13.74	102.44
Ferrous Sulphate	37.38	-1.52	-2.25	2.72	235.95
Aluminium Sulphate	66.39	-5.72	13.43	14.60	113.06
Copper Acetate	64.00	-2.10	19.96	20.07	96.01
Ferric Chloride	42.34	-0.62	0.57	0.84	137.37
Acetic Acid	71.30	-3.39	12.03	12.50	105.76
Copper Chloride	63.07	-1.65	16.93	17.01	95.57
Tartaric Acid	65.73	-2.68	9.82	10.18	105.29

Table 4. Colour fastness to rubbing of dyed leather specimens.

Mordants	Pre-mordanting		Post-mordanting	
	Staining			
	(Dry)	(Wet)	(Dry)	(Wet)
Without mordant	4	4	-	-
Alum	4-5	4-5	4	3-4
Potassium Dichromate	4-5	4	4	4
Copper Sulphate	4-5	4-5	4-5	4
Ferrous Sulphate	4	3-4	3-4	3-4
Aluminium Sulphate	4	4-5	4-5	4
Copper Acetate	4-5	4	4	4-5
Ferric Chloride	4	3-4	4	4
Acetic Acid	4-5	4	4	3-4
Copper Chloride	4-5	4	4-5	4
Tartaric Acid	4-5	4-5	4-5	4-5

Colour fastness to rubbing

Staining grades of colour fastness to rubbing revealed that the good values were obtained with pre-mordanting method. Comparatively, it was also noticed from the results obtained from both mordanting techniques that colour fastness to rubbing in dry state produced better results than the colour fastnss to rubbing in wet state. Besides the rubbing in dry state, the good staining grades (4-5) were also obtained in rubbing (wet) with alum, copper sulphate, aluminium sulphate and tartaric acid in pre-mordanting and with copper acetate and tartaric acid in post-mordanting. The results of wet rubbing with pre- and post-mordanting methods showed that majority of the mordants had good staining grades in the range of 4-5 and 4 and the overall results of colour fastness to rubbing were found good to very good with both mordanting techniques.

Table 5. Colour fastness to light of dyed leather specimens.

Mordants	Pre-mordanting		Post-mordanting	
	Change in Colour			
	24 hrs.	48 hrs.	24 hrs.	48 hrs.
Without Mordant	4	3	-	-
Alum	2-3	2-3	3	3
Potassium Dichromate	2-3	2	2-3	2
Copper Sulphate	4	3	3-4	3
Ferrous Sulphate	4-5	4	4-5	3
Aluminium Sulphate	4	3	4	4
Copper Acetate	4	4	3-4	3-4
Ferric Chloride	4-5	4-5	3-4	3-4
Acetic Acid	4	4	4	3-4
Copper Chloride	5	4	4-5	4-5
Tartaric Acid	3-4	3-4	3	3

Colour fastness to light

The results of colour fastness to light with pre- and post-mordanting methods were observed good to very good after 24 hrs. of sunlight exposure (Table 5). Excellent change in colour (5) was obtained with copper chloride mordant in pre-mordanting method. Application of ferrous sulphate and ferric chloride mordants also produced very good change in colour (4-5) after 24 hours of exposure in pre-mordanting. Change in colour (3-4) was obtained with tartaric acid after 24 hours of exposure in pre-mordanting which was also a good value. Potassium dichromate gave colour change (2-3) after 24 hours with both mordanting method whereas alum produced better results of colour change (3) after 24 and 48 hours in post mordanting technique.

Table 6. Feasibility study of *R. damascena* liquid dye.

Sr.No.	Description	Dollars
1	Raw Material (1 Kilogram waste petals)	0.19
2	Utilities	0.58
3	Labour	0.48
4	Transportation	0.38
5	Packing	0.14
6	Overheads (Miscellaneous)	0.10
Total Cost		1.86 USD

Feasibility study of R. damascena liquid dye

The production cost of liquid dye was estimated by taking into account the raw material (waste petals), utilities, labour, transportation, packing and overhead miscellaneous (Table 6). The extraction cost of liquor dye of 1kg waste petals was 1.86 USD which can be used for dyeing three goat skins and the per skin liquor cost was 0.62 USD. So, *R. damascena* extract from waste petals was not only significant in terms of environment safety but also economical viable as compared to toxic synthetic dyes.

Fig. 1. Extracted dye of *R.damascena*.

Conclusion

The findings of the study show that *R. damascena* waste petals possesses good potential of dye and its good colouring attributes was observed good on leather. Results of the study revealed that liquid dye produced different soft and dark shades with and without mordants. Beside this, a use of natural dyes from waste petals was found economical according to industrial point of view. Conclusively, the *R. damascena* liquid dye was also found good in terms of colour fastness to rubbing and light. Moreover, in the prevailing scenario of global environmental concerns, Pakistan has a good opportunity to promote green economy using natural dyes.

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