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Environmental friendly leather dyeing using *Tagetes erecta* L. (Marigold) waste flowers

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Abstract

In the present study, waste flowers of *Tagetes erecta* L. (Marigold) were used for the extraction of dye and the efficiency of yellow dye was evaluated on goat leather. Cost efficient, environment friendly aqueous method was employed to extract dye. Twenty various shades were obtained with pre-mordanting and post-mordanting methods. Very good to excellent colour fastness (mild washing, rubbing) have been shown with all mordants including without mordant whereas colour fastness (daylight) results have been recorded fair to good. Results of study revealed that *T. erecta* floral dye has been found a new addition in natural leather dyeing and can be served as a good source of fashion colours at economical rates.

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Introduction

Natural dyes have been used for coloring various substrates such as leather and textiles (wool, cotton, and silk) since ancient times (Cristea and Vilarem, 2006). Primitive people used different parts of plants for dye extraction such as barks, flowers, fruits, leaves, roots, seeds and stems (Angelini *et al.*, 2003; Adeel *et al.*, 2009). Among all plant parts, the floral waste is preferred to extract natural colorant (Singh *et al.*, 2017) as it is considered economical (Jadhao and Rathod, 2013; Pervaiz *et al.*, 2016a). Moreover, dye extraction from floral parts is relatively easy and fast as compared to other plant parts (Annapoorani and Sundarraj, 2014).

Among flowers, *Tagetes erecta* (Marigold) is well known and popular flower and commonly found in Asia and other continents of the world. It belongs to a plant of genus *Tagetes* and the family Asteraceae (Vankar *et al.*, 2009). Significantly, the ancient flower is considered sacred (Belemkar and Ramachandran, 2015) in Hinduism, Buddhism, Christianity, and known for its purity and victory. *T. erecta* has different shade variations such as lemon yellow, maroon orange and golden (Shetty *et al.*, 2015). It is cultivated easily and it has potential to grow in variety of soils and compatible with different climatic conditions. Most importantly, the plant bears flowers for a longer period. *T. erecta* is used for decorative purposes and most popular in making garlands (Jothi, 2008), worship idols (Vankar *et al.*, 2009; Singh and Swami, 2016), coloring food (Vasudevan *et al.*, 1997), dairy products and egg yolks (Bosma *et al.*, 2000). In addition, *T. erecta* waste flowers were used as a dyeing agent and reported to dye silk (Chavan and Ghosh, 2015), wool (Montazer and Parvinzadeh, 2007) and cotton (Sundrajana *et al.*, 2011; Farooq *et al.*, 2013; Khattak *et al.*, 2014; Sadi *et al.*, 2016).

By considering the present scenario of environmental pollution and human health risks (Khattak *et al.*, 2014) generated by synthetic dyes (Arunkumar and Yogamoorthi, 2014) the demand of eco-friendly natural dyes is increasing day by day (Haji, 2010). Furthermore, in order to achieve sustainable development goal, the eco-solution to extract dye

from plant waste (Ghorpade *et al.*, 2000; Vankar *et al.*, 2009) which helps to elevate the income of florists, flower exporters and also replenish the demand of eco-friendly, non-carcinogenic, non-toxic, non-hazardous and non-poisonous green dyes (Pervaiz *et al.*, 2016b; Pervaiz *et al.*, 2016c). So, keeping the above points in view, the present research was carried out in Punjab to utilize *T. erecta* floral waste for dye extraction as they are abundantly available at low price in the province.

Moreover, the study was designed with the objectives to extract natural dye from *T. erecta* floral waste and assess its dye-ability on goat leather.

Materials and methods

Collection of dyeing material

T. erecta waste garlands of yellow colour were collected from the local flower market at the rate of 0.10 United States Dollar (USD).

Goat leather

The goat crust leather treated with chrome was procured from the Sialkot at the rate of 7.63 USD.

Mordants & chemicals

The commercial grade mordants *i.e.* acetic acid, aluminium sulphate, copper acetate, copper chloride, copper sulphate, ferric chloride, ferrous sulphate, potash alum, potassium dichromate, tartaric acid, and chemicals *i.e.* formic acid, sodium bicarbonate and sodium formate were used in the study.

Dye extraction

Flowers were separated from garlands and petals were plucked from flowers. Petals (100 g) were weighed using digital balance and then rinsed with water to remove the adhering soil and impurities (Sundari, 2015; Pervaiz *et al.*, 2016a; Pervaiz *et al.*, 2016b).

The dye was extracted in aqueous medium by steeping petals for 72 hours at room temperature. The maximum colorant from immersed petals was extracted at 40°C for 40 minutes. Thereafter, the solution was left to cool and filtered through filter paper (Pervaiz *et al.*, 2016a; Pervaiz *et al.*, 2016b).
Leather dyeing and mordanting.

Leather specimen of 10g was weighed and soaked in the distilled water for 12 hours. Soaked leather was treated with 1% solution of sodium bicarbonate and sodium formate in a micro-steel drum for 30 minutes. Thereafter, the treated leather specimens were washed with tap water.

The processed leather specimens were mordanted with ten selected mordants using pre-mordanting (before dyeing) and post-mordanting (after dyeing). 1M solution of each mordant was used for mordanting leather specimens for 60 minutes. The leather specimen was dyed step wise with 250mL of dye extract. In the first step, the leather specimen was dyed for 30 minutes and for the fixation of dye, formic acid was used. In the second step, the same process was repeated again and then dyed leather samples were spread in open air for drying (Pervaiz *et al.*, 2016a; Pervaiz *et al.*, 2016b; Pervaiz *et al.*, 2016c).

Colour values of dyed leather specimens

Colour values (L^* , a^* , b^*) of dyed and undyed leather specimens were ascertained from the grain side of leather through a spectrophotometer (Spectra flash SF-650X) (Pervaiz *et al.*, 2016a; Pervaiz *et al.*, 2016b).

Colour fastness of dyed leather specimens

The strength of dye affinity on leather was evaluated by performing colour fastness to mild washing (ISO15703:1998), rubbing (BS1006:1990) and daylight (ISO 105-B01:1999).

Results and discussion

General appearance of dyed leather specimens

The general appearance of dyed leather specimens can be visualized from Table 1. It was noticed from the visual inspection that dyed leather specimens exhibited a good leveled smooth dyeing having attractive appearance with and without mordants (Pervaiz *et al.*, 2016b). A fair levelness of dye was obtained with ferrous sulphate and ferric chloride mordants using pre-mordanting method whereas; with the post-mordanting method, fair to good dye uniformity was observed with all selected mordants. The dye penetration level was visually observed and found to be good to very good with and without mordants. Results of previous study of (Musa *et al.*, 2009) showed the similar output with the natural dyes.

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

Without mordant		
Mordants	Pre-Mordanting	Post-Mordanting
Acetic acid		
Aluminium sulphate		
Copper acetate		
Copper chloride		
Copper sulphate		
Ferric chloride		
Ferrous sulphate		
Potash alum		
Potassium dichromate		
Tartaric acid		

Shade obtained without mordant

It was clearly observed from the results presented in Table 1 that un-mordanted dyed leather specimen rendered beautiful pale yellowish shade with the extracted yellow dye which was the reported shade of *T. erecta* (Verghese, 1998; Jothi, 2008; Pervaiz *et al.*, 2016b; Singh and Swami, 2016).

Shades obtained with pre-mordanting and post-mordanting

From Table 1, it was found that soft pleasing shades were obtained with the *T. erecta* dye. Most of the shades obtained were in variation of light yellow colour with pre-mordanting method.

Besides pale yellow shades, beautiful variety of cardamom green, yellowish green, and light brown were also exhibited with different mordants. Shade of yellow was developed with aluminum sulphate which is the reported shade on cotton and silk (Jothi, 2008).

Moreover, similar result of light yellow shade is reported on cotton with copper sulphate mordant (Kanchana *et al.*, 2013). Whilst ferrous sulphate produced light brownish shade which is similarly reported shade on cotton (Jothi, 2008; Chavan and Ghosh, 2015). Moreover, acetic acid exhibited bright yellow shade, copper acetate and potash alum produced light yellow whereas ferric chloride developed light brown.

In post-mordanting method, an assortment of pale yellow shades was developed with the *T. erecta* yellow dye (Ratnapandian *et al.*, 2011; Sadi *et al.*, 2016).

From the comparative analysis of all shades, it has been revealed that most of obtained shades were light and soft with aluminium sulphate, copper sulphate and potash alum (Kamel *et al.*, 2011). Furthermore, brownish shade was obtained with ferric chloride mordant which is reported by (Khattak *et al.*, 2011).

Effects of mordanting

Mordants played significant role to fix the dye on substrate (Bose and Nag, 2012). In this study, ten different mordants were chosen for the evaluation of *T. erecta* dye.

From the obtained results, it was analyzed that dark shades were obtained with metal mordants. Analyzing all mordants results, it was found that ferric chloride mordant produced dark reported shade (Khattak *et al.*, 2014; Pervaiz *et al.*, 2016b).

In addition, dark shade was also observed with ferrous sulphate (Jothi, 2008) which is reported quality of metal mordant to make coordination with the dye (Ratnapandian, 2013) and in the present study, it developed good bonding with the *T. erecta* dye. Besides ferrous sulphate mordant, potash alum was also found in dark shade (Khattak *et al.*, 2014).

Aluminium sulphate mordant was found significant to develop yellow shade. Moreover, beautiful range of yellow shade was developed with selected mordants.

Colour coordinates of dyed leather specimens with pre-mordanting and post-mordanting

Combination of low and high values of L* were obtained with selected mordants. Assessing the results of *T. erecta* in Table 2, it has been revealed lowest values (L*) recorded with ferrous sulphate and copper chloride mordants with pre-mordanting method.

Observing results of colour coordinates, it was noted that most of lowest values of lightness were obtained with pre-mordanting method. Potassium dichromate observed dominant to produce yellowish shades having positive b* values with both mordanting techniques.

While acetic acid recorded with b* values (24.30) and (18.69) using pre- and post-mordanting methods. Furthermore, it was noted that most of a* values in negative form which showed the greenish shade with pre-mordanting and post-mordanting methods. By the comparison all results of pre-mordanting and post-mordanting, it was revealed that *T. erecta* produced dynamic positive b* values.

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

Mordants and Without Mordant	Pre-mordanting			Post-mordanting		
	L*	a*	b*	L*	a*	b*
Without mordant	74.06	-3.34	18.10	74.06	-3.34	18.10
Acetic acid	75.11	-4.00	23.96	75.87	-2.46	18.53
Aluminium sulphate	78.59	-5.60	23.97	81.03	-3.20	12.65
Copper acetate	76.89	-3.69	15.09	75.75	-2.05	16.45
Copper chloride	58.66	0.80	10.46	74.24	-3.77	21.96
Copper sulphate	75.42	-3.83	16.06	80.71	-3.62	11.17
Ferric chloride	79.00	-2.68	9.78	67.63	3.20	9.69
Ferrous sulphate	58.67	-1.34	10.52	66.59	0.77	8.31
Potash alum	78.06	-5.22	23.88	81.66	-2.89	11.29
Potassium dichromate	75.50	-1.23	16.38	75.31	-2.10	24.47
Tartaric acid	75.65	-2.82	13.63	78.61	-2.22	6.88

Colour fastness to mild washing with pre-mordanting and post-mordanting

Leather specimens dyed with selected mordants showed good to excellent tendency in colour fastness to mild washing with the *T. erecta* dye (Table 3&4). The results demonstrated that after mild washing, no staining was observed on adjacent multifiber with and without mordants using pre-mordanting and post-mordanting methods. Furthermore, the results elucidated that the *T. erecta* dye showed good bonding therefore,

the wash fastness properties obtained (4-5) to (5). In addition, it was noted that the negligible change in colour observed after washing which rendered that dye was colour fast on leather. Analysis of pre- and post-mordanting techniques showed that *T. erecta* dye has good potential for dyeing leather and mild washing results were found encouraging regarding industrial point of view except leather specimen which post-mordanted with tartaric acid recorded with noticeable change in colour result.

Table 3. Colour fastness to mild washing with pre-mordanting.

Mordants and Without Mordant	Staining						Change in Colour
	CA	CO	PA	PES	PAN	WO	
Without mordant	5	5	5	5	5	5	4-5
Acetic acid	5	4	4	4-5	4-5	4-5	4
Aluminium sulphate	4-5	5	5	5	5	5	4-5
Copper acetate	5	5	5	5	5	4	4-5
Copper chloride	5	4-5	5	5	5	4	4
Copper sulphate	4-5	4	5	5	5	5	4
Ferric chloride	4-5	4-5	5	5	5	5	3
Ferrous sulphate	4-5	5	5	5	5	5	4-5
Potash alum	5	5	5	5	5	4-5	3-4
Potassium dichromate	4-5	4-5	5	5	5	5	5
Tartaric acid	4-5	5	5	5	5	5	2

CA = Diacetate, CO = Cotton, PA = Polyamide, PES = Polyester, PAN = Acrylic, WO = Wool

5= Excellent, 4= Good, 3= Average, 2= Poor, 1= Very Poor

Table 4. Colour fastness to mild washing with post-mordanting.

Mordants and Without Mordant	Staining						Change in Colour
	CA	CO	PA	PES	PAN	WO	
Without Mordant	5	5	5	5	5	5	4-5
Acetic acid	5	4-5	4-5	5	5	5	4-5
Aluminium sulphate	3-4	5	4-5	4-5	5	3-4	4
Copper acetate	4-5	4-5	4	5	5	5	5
Copper chloride	4	5	4-5	4-5	4-5	4-5	4-5
Copper sulphate	4-5	4-5	4-5	4	5	4-5	5

Mordants and Without Mordant	Staining						Change in Colour
	CA	CO	PA	PES	PAN	WO	
Ferric chloride	5	4-5	4	5	5	5	3-4
Ferrous sulphate	4-5	4-5	4-5	4	5	4-5	3-4
Potash alum	4-5	4-5	4-5	4	5	4-5	5
Potassium dichromate	4-5	4-5	4-5	4	5	5	5
Tartaric acid	5	5	4-5	5	5	4-5	4-5

CA = Diacetate, CO = Cotton, PA = Polyamide, PES = Polyester, PAN = Acrylic, WO = Wool

5= Excellent, 4= Good, 3= Average, 2= Poor, 1= Very Poor

Colour fastness to rubbing with pre-mordanting and post-mordanting

Based upon results Table 5, leather mordanted with selected mordants showed very good to excellent colour fastness to rubbing (dry & wet). Moreover, change in colour results with pre-mordanting method bestowed better results than post-mordanting method. Staining grades of colour fastness to rubbing revealed that excellent (5) to very good (4-5) fastness grades obtained with pre-mordanting and post-mordanting methods. Comparatively, it was also observed from the results obtained from both mordanting techniques that colour fastness to rubbing in dry state produced

excellent results. Besides the rubbing in dry and wet state, maximum excellent (5) grades obtained with change in colour results using pre-mordanting method.

The reason of very good to excellent rubbing in terms of both color staining and color change was due to the formation of chemical bonding between dye molecules and leather. Comparing present results of colour fastness to rubbing with reported earlier study (Agarwal *et al.*, 2007; Musa *et al.*, 2009; Onem *et al.*, 2011; Erisen *et al.*, 2012; Selvi *et al.*, 2013; Sundari, 2015), it was found that selected dyes showed commercially viable output.

Table 5. Colour fastness to rubbing with pre-mordanting and post-mordanting.

Mordants and Without Mordants	Pre-mordanting Method				Post-mordanting Method			
	Staining		Change in Colour		Staining		Change in Colour	
	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
Without mordant	5	5	5	5	5	5	5	5
Acetic acid	5	4-5	5	4-5	5	5	5	5
Aluminium sulphate	5	5	5	5	5	4	4-5	4
Copper acetate	5	5	5	5	5	5	5	5
Copper chloride	5	5	5	5	5	5	5	5
Copper sulphate	5	5	5	5	5	5	5	5
Ferric chloride	5	4-5	5	4-5	5	5	5	5
Ferrous sulphate	5	5	5	5	5	4-5	5	4-5
Potash alum	5	5	5	5	5	5	5	5
Potassium dichromate	5	5	5	5	5	5	5	4-5
Tartaric acid	5	4-5	5	4-5	5	5	5	4-5

5= Excellent, 4= Good, 3= Average, 2= Poor, 1= Very Poor

Colour fastness to daylight with pre-mordanting and post-mordanting

Dyed leather specimens were analyzed with the daylight colour fastness test showed with fairly good to good results. Pre-mordanted and post-mordanted dyed leather specimens with tartaric acid exhibited very good performance by recording 6 Blue Wool Standard.

In addition, the post-mordanted leather specimens with ferrous sulphate recorded with good light fastness (Blue Wool Standard No. 5). Good fastness to daylight is one of the desirable commercial properties of dyed leather. Analyzing results of colour fastness to daylight of present study demonstrated that dyed specimens showed fair results of light as reported by (Sundari, 2015).

Table 6. Colour fastness to daylight with pre-mordanting and post-mordanting.

Mordants and Without Mordant	Pre-mordanting Method	Post-mordanting Method
	Change in colour according to blue wool scale	Change in colour according to blue wool scale
Without Mordant	2	2
Acetic acid	6	4
Aluminium sulphate	4	4
Copper acetate	3	2
Copper chloride	2	4
Copper sulphate	3	4
Ferric chloride	3	4
Ferrous sulphate	3	5
Potash alum	3	3
Potassium dichromate	4	4
Tartaric acid	6	6

8= Exceptional, 7= Excellent, 6= Very Good, 5= Good, 4= Fairly Good, 3= Average, 2= Poor, 1= Very Poor

Economic sustainability of produced T. erecta dye

Distilled water was used to extract dye from marigold flower petals (Islam *et al.*, 2016) as it is considered cost effective (Ali *et al.*, 2016). In addition, *T. erecta* waste garlands were purchased at low price. Therefore, the cumulative estimated cost of 1kg extracted dye from the *T. erecta* floral waste taking into consideration utilities, labor, transportation, overhead and miscellaneous was 1.78 USD which was found to be quite economical and commercially viable to dye 300g of leather.

Conclusion

The findings of the study showed that *T. erecta* flower petals possess significant yellow colour and developed wide array of soft shades on leather with and without mordants. In addition, use of natural dyes from stale petals was found inexpensive. *T. erecta* is readily available and most abundant commercial flower of Punjab. The shades produced from the *T. erecta* (marigold) petals were found uniform with and without mordants except ferrous sulphate mordant which produce fair dye evenness results. Owing to the growing trend of going “green” globally, eco- friendly practices like dyeing of leather with natural colourants should be adopted to achieve sustainable development goal.

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