

Cotton *Khadi* fabric dyeing with natural dye extracted from the petals of *Butea monosperma* Flower using different mordants

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■ **ABSTRACT** : Natural dyes are eco-friendly, biodegradable, non-toxic, and having no side effects on skin as compared to synthetic dyes. These dyes obtained from different natural sources which may be animal and plants sources (like stem, bark, flowers, leaves and roots of the plants). In the present work, dye was extracted from the petals of *Butea monosperma* flowers were used for natural colouring to cotton *Khadi* fabric with different concentrations of dye *i.e.* 10%, 20% and 30% using five different types of mordant *i.e.* *P. granatum*, *P. emblika* *T. bellirica*, *A. catechu* and *F. sulfate* (Iron (II) sulfate heptahydrate) at three different concentration (5%, 10% and 15%). Dyed *Khadi* fabric was evaluated for colour fastness towards washing, rubbing, and sunlight. Cotton *Khadi* fabric samples dyed with *B. monosperma* with 30% concentration of dye and 15 per cent mordants concentration using pre-mordanting method gave good to excellent wash and rub fastness ratings with all mordants. *P. granatum* and *T. bellirica* mordants with pre-mordanting method using different mordant's concentrations *i.e.* 5%, 10% and 15% as compare to all other mordants produced moderate fading to no fading against blue dyed standards of 1-8 rating scale. The dyed samples showed very good colour strength (K/S) and colourimetric appearance using CIE L* a* b* colour space in terms of colour co-ordinates. The fabric samples mordanted with *P. granatum* gave very good colour strength with all concentrations of dye and mordants as compare to other mordants, respectively. Large ranges of shades were obtained using different types of mordants and their concentrations with petals of *Butea monosperma* dye.

■ **KEY WORDS**: Natural dye, *Butea monosperma*, Colour fastness, Colour strength, Cotton *Khadi* fabric

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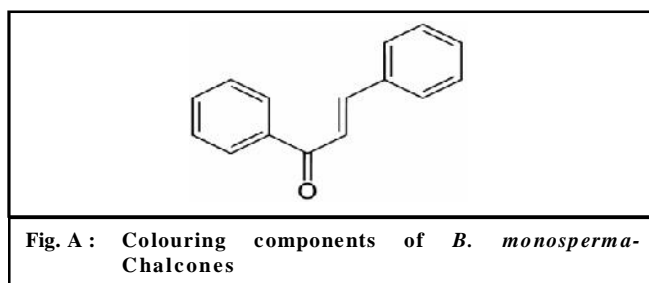
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Colours add beauty to our fascinating world. The beauty led to the discovery of colouring matter from natural sources such as plants and animals. The use of natural dyes to colour textiles declined rapidly

after the discovery of synthetic dyes in 1856. The main reason for the replacement of natural dyes by their synthetic counterparts is that most of the natural dyes have poor to moderate colour fastness properties, while

synthetic dyes represent the full range of fastness properties at moderate costs. The widely and commercially used synthetic dyes impart strong colours but causes carcinogenicity and inhibition of benthic photosynthesis (Husain, 2006, Adeel *et al.*, 2012 and Farid 2015). Natural dyes and colourants derived from flora and fauna are believed to be safe because of its nontoxic, non-carcinogenic and biodegradable in nature (Kumar *et al.*, 2009 and Yusuf *et al.*, 2016). Natural dyes are among the promising options for developing a greener textile dyeing process. Textiles composed of proteinous materials such as wool and silk as well as cellulosic materials such as cotton, jute, flax and other fibres come in contact with the body, which provide an ideal environment for the growth (Khan *et al.*, 2011).

Flame of forest belongs to the family Fabaceae, with botanical name is *Butea monosperma*, and many other common names like Kesula, Palas, Palash, Dhak, Khakara, Chichra, Tesu and bastard teak, and is a species of *Butea* native to tropical and sub-tropical parts of the Indian Subcontinent and Southeast Asia, ranging across India, Nepal and western Indonesia. The Flame of forest is a medium sized dry season-deciduous tree, straight growing 12 to 15 m tall (Sinha *et al.*, 2012 and Krolkiewicz-Renimel *et al.*, 2013). The leaves are pinnate, with 8-16 cm pedicel and three leaflets, each leaflets 10-20 cm long. These flowers start to come in February and stay until the end of April. The flowers are around 2-4 cm in diameter, bright orange red in colour, and produced in racemes upto 15 cm long (Firdaus and Mazumder, 2012 and Sinha *et al.*, 2012).



The flowers of this tree have been used traditionally for dyeing and a coloured powder used during the Indian festival Holi (Choedon *et al.*, 2010). Sinha *et al.* (2012) and Yadava and Tiwari (2007) reported that the main colouring pigment of the *B. monosperma* is butrin. Besides, butein and isobutrin are also present in trace amounts and also contains flavonoids and steroids

(Choedon *et al.*, 2010 and Rasheed *et al.*, 2010). Studies have shown that isobutrin slowly changes to butrin on drying. The bright orange red colour of the flower is attributed to the presence of chalcones, pterocarpans and aurones but other anthochlor pigments are also present (Krolkiewicz-Renimel *et al.*, 2013).

Khadi is an Indian handspun and hand-woven cloth and known as ecofriendly textile. The raw material may be cotton, silk, or wool, which is spun into threads on a spinning wheel called a charkha, was introduced in 1920 by "Mahatma Gandhi" during the freedom struggle (Pant and Sharma, 2009). Cotton *Khadi* fabric is known as "Khaddar". It prevents skin rashes and imparts very elegant and sober look. It is the symbol of Indian's identity and *Khadi* has specific physical and chemical properties such as high water absorption, high comfort, and good dyeability. The aim of this study was to evaluate the performance of dyes and extracting natural dye from the petals of Flame of forest flower using aqueous extraction method of the dyeing, to analyze the colour strength and assess the colour fastness properties of dyed cotton *Khadi* fabric with dye concentrations and different mordants with their concentrations.

■ RESEARCH METHODS

Experimental :

Materials :

Khadi fabric was purchased from *Khadi* Bhandar, Udaipur. Scouring was done on the fabric (Tiwari and Jain, 2017). Petals of Flame of forest flower was used as dye source, collected from the road side open ground, near the university campus of Udaipur city and all mordants were also purchased from the local market of Udaipur (Rajasthan).

Method :

Aqueous extraction of dye from the petals of flame of forest (*Butea monosperma*) flower :

Firstly, the petals of Flame of forest flower were dried in shade at room temperature to make powdered. In the view of reported good colour yield in case of extraction of colourant from required quantity (o.w.f.) of powdered petals of Flame of forest were taken using 1:40 M:L ratio and kept for 12 hour and boiled for one hour, then cooled at room temperature and filtered through a clean cotton cloth. The remaining residue was percolated until the entire colour had been extracted. In

this study, crude extracts of *Butea monosperma* dye were used.

Mordanting :

Mordanting can be achieved by either pre-mordanting, simultaneously mordanting or post-mordanting (Teli *et al.*, 2013 and Prabhu *et al.*, 2014). Samanta *et al.* (2009) reported that different types and selective mordants or their combination can be applied on the textile fabrics to obtain varying colour or shade, to increase the dye uptake and improve the colour fastness behavior of any natural dye. Kumaresan *et al.* (2012) found that pre-mordanting is the best method among all methods of mordanting. The researcher selected pre mordanting method for mordanting of cotton *Khadi* fabric.

Mordanting process :

After scouring, firstly myrobalan treatment was given to the cotton *Khadi* fabric with 2% of myrobalan for 1hr at room temperature. Mordants were added to water in separate baths at optimized concentrations, and the temperature of each mordant solutions were raised from 30°C to make them completely soluble. Pre-soaked fabric samples were added to the mordant baths and then the temperature of the mordanting baths was carried out at 90°C for 45 minutes with constant stirring (Teli *et al.*, 2013 and Tiwari and Srivastava, 2017). After mordanting, the baths were cooled and samples were removed from the mordanting baths and rinsed with tap water.

Mordanting of cotton *Khadi* fabric :

Fabric samples were soaked in water for 5 minutes and all mordants (*P. granatum*, *P. emblika*, *A. Catechu*, *T. bellirica* and F. sulfate) with optimized conce. *i.e.* 5%, 10% and 15% o.w.f. (on weight of fabric), were dissolved in water keeping MLR 1:40 and filtered with a clean cotton cloth. Pre-soaked cotton *Khadi* fabric samples were added in to mordant solution at 91° to 94°C and this process was continued for 45 minutes with constant stirring (Mahangade and Jain, 2009; Guesmi *et al.*, 2012 and Tiwari and Jain, 2017). After mordanting, the samples were removed from the bath and washed with tap water in order to remove extra mordant particles.

Dyeing of mordanted cotton *Khadi* fabric :

Firstly mordanted *Khadi* samples were soaked in

water for 5 min then drenched in to dye bath containing optimized dye concentrations *i.e.* 10%, 20% and 30%, with 1:40 MLR and the temperature of the dye bath was raised till simmering point (91° to 94°C) and left for 1 hour and stirred regularly. The dyed *Khadi* fabric samples were washed and dried in shade.

Colour fastness tests :

The dyed samples were assessed for colour fastness with respect to light, washing and rubbing. The light fastness of the dyed *Khadi* fabric samples were conducted on digital light NxTM having water cooled Mercury Blended Tungsten lamp, according to the test method AATCC 16e-2004 similar to ISO 105-B02:1994 (Amd.2:2000) (Mahangade *et al.*, 2009 and Bukhari *et al.*, 2017). The wash fastness were assessed using Launder-o-meter as per the ISO 105-C06:1994 (2010) specifications. A measured peace of dyed fabric was sewed between the two pieces of adjacent cotton fabrics (Tiwari and Srivastava, 2017, Bukhari *et al.*, 2017 and Ganesan and Karthik, 2017). The specimen was treated with 5 g/L non-ionic detergent at 50°C for 45 min in Launder- o-meter. The samples were assessed for colour change on washing and staining on adjacent fabrics. The rub fastness were tested with Crock-o-meter as per Indian standard IS 766:1988 (Reaffirmed, 2004) which is based on ISO 105-X12:2001 by mounting the fabric on the panel, giving 20 strokes for both wet and dry. The samples were assessed for staining on white cotton fabrics which were used as adjacent (Tiwari *et al.*, 2010 and Ganesan and Karthik, 2017).

Colour measurement :

Colour values of the samples were analyzed on the basis of L a* b* values using reflectance spectra through Macbeth- colour Eye 5100A UV spectrophotometer. L* value is measure of lightness and darkness. Positive 'a' is for red; negative 'a' is for green. Positive 'b' is for yellow and negative 'b' is for blue. The average colour values for the samples were recorded. Three replicates were taken for all individual parameters. This method also involves colour strength (K/S), is calculated by the Kubelka Munk K/S equation, which indicates that the production of reflected light involves absorption and scattering (Guesmi *et al.*, 2012 and Ganesan and Karthik, 2017). K/S can be calculated by the following formula:

$$\frac{K}{S} = \frac{(1-R)^2}{2R}$$

where R is the reflectance, K is the sorption coefficient, and S is the scattering co-efficient.

RESEARCH FINDINGS AND DISCUSSION

In this work, natural dye extracted from the petals of *Beauteamonosperma* flowers using aqueous extraction procedure. The dyeing process was carried out by optimized dyeing concentrations i.e. 10%, 20% and 30%, with different types of mordants (*P.granatum*, *P.emblika*, *A. Catechu*, *T.bellirica* and F. sulfate) and their concentrations i.e. 5%, 10% and 15% o.w.f. As the concentrations of dyes and mordants were increased the colour of dyeing were also increased with optimized time (90 min) and temperature (91°C to 95°C) and the

different shades were obtained.

Colour fastness of dyed fabric with *B. monosperma* dye using different mordants :

The table 1 and 2 contains the data of colour fastness to rubbing, washing and light with selected mordants and colour change and colour staining was determined. Table 1 depicted that the wash fastness of *P. emblica* and *P. granatum* mordanted samples with 10% and 15% mordant concentration and 20% and 30% dyeing concentration shows very good (4) to excellent (5) colour change (cc) properties and good to very good (4-5) wash staining on wash staining grey scale and no staining on adjacent fabric were observed. Ferrous sulfate also gave fair to excellent wash fastness ratings with all the dyeing and mordant concentrations, respectively.

Rub fastness of *Khadi* samples mordanted with *P.*

Table 1 : Rub and wash fastness properties of dyed cotton *Khadi* fabric samples

Mordants	Dye %	Rub fastness								Wash fastness							
		Dry				Wet				CC				CS			
		Control	5%	10%	15%	Control	5%	10%	15%	Control	5%	10%	15%	Control	5%	10%	15%
PE	10	3	2	3	4	3	3	3	4.5	2.5	2	2.5	3	3	3	4	4
	20	4	2.5	5	5	3	3	4	5	3	2.5	3	4	3	3	4	4
	30	4	4	5	5	3.5	4	4	5	3	4	4	4.5	4	5	4.5	5
AC	10	3	3	3	4	3	3	2.5	4	2.5	2	3	4	3	2	3	4
	20	4	3	4	5	3	3	3.5	4.5	3	3	3	3.5	3	3	4	4.5
	30	4	4	5	5	3.5	3.5	4.5	4.5	3	3.5	4	4	4	5	4	5
PG	10	3	2	3	4	3	3	3	3.5	2.5	3	3	3.5	3	3	4	4
	20	3	3	5	5	3	3.5	3.5	4.5	3	3	4	4.5	3	4	4	5
	30	4	4	5	5	3.5	3.5	5	5	3	4	5	5	4	5	5	5
TB	10	3	2	3	4	3	3	3	4	2.5	3	3	4	3	3	3.5	4
	20	4	3.5	5	5	3	2.5	3.5	4.5	3	3	3.5	4	3	4	4	4.5
	30	4	4	5	5	3.5	3.5	4.5	5	3	3.5	4	4.5	4	5	5	5
FS	10	3	3	4	4	3	3	4	4	2.5	3	4	4	3	4	4	4
	20	4	4	5	5	3	4	5	5	3	4	4.5	5	3	4	4.5	4.5
	30	4	5	5	5	3.5	4.5	4.5	5	3	4	4.5	5	4	5	5	5

PE= *P.emblica*, AC= *A. Catechu*, TB= *T.bellirica*, PG= *P.granatum*, FS= F. sulfate, CC=Colour change, CS=Colour staining,

Table 2 : Light fastness properties of dyed cotton *Khadi* fabric samples

Mordants	D 10%			D 20%			D 30%					
	Control	Mordants		Control	Mordants		Control	Mordants				
		5%	10%	15%		5%	10%	15%		5%	10%	15%
PE	3	4	4	6	4	4	5	6	5	5	6	7
AC	3	4	5	5	4	4	6	5	5	5	6	7
PG	3	3	4	5	4	3	5	6	5	6	7	8
TB	3	4	4	5	4	4	5	6	5	6	7	8
FS	3	4	5	5	4	5	4	5	5	5	6	7

D= dye, PE= *P.emblica*, AC= *A. Catechu*, PG= *P.granatum*, TB= *T.bellirica*, FS= F. sulfate,

Table 3 : CIE Lab colour co-ordinates of dyed cotton Khadi fabric using *B. monosperma*

Mordants	Dye	Mordant %	L*	a*	b*	C*
PE	Control					
	10%		82.69	11.91	36.36	38.18
	20%		78.84	16.39	45.72	48.94
	30%		71.23	19.03	49.01	52.14
		5	64.64	9.60	32.10	33.81
	10%	10	63.46	10.13	31.34	33.19
		15	63.89	10.49	32.64	34.27
	20%	5	63.97	10.98	32.24	34.06
		10	65.02	10.44	33.98	33.55
AC		15	57.94	12.28	29.99	31.27
	30%	5	60.66	11.68	30.75	32.89
		10	61.91	12.59	31.17	32.68
		15	51.76	14.83	25.92	27.38
	10%	5	64.34	11.66	32.83	34.84
		10	60.53	11.51	29.69	31.32
		15	58.62	11.87	31.03	33.25
	20%	5	58.85	12.15	29.66	31.99
		10	59.20	12.64	29.30	31.91
PG		15	51.46	12.15	27.66	30.17
	30%	5	57.38	13.27	28.86	31.76
		10	56.92	13.11	31.12	33.49
		15	49.23	12.73	26.65	29.53
	10%	5	66.27	10.68	33.11	34.79
		10	62.76	11.15	33.58	35.33
		15	55.37	11.28	34.54	36.34
	20%	5	65.10	10.10	33.36	34.87
		10	62.54	11.41	35.44	37.23
TB		15	50.59	11.84	34.92	36.87
	30%	5	60.95	11.82	34.03	36.03
		10	61.08	11.94	34.21	36.25
		15	48.75	13.26	37.54	39.81
	10%	5	64.87	9.51	32.86	33.49
		10	64.33	10.15	29.95	31.49
		15	64.56	9.34	30.13	31.54
	20%	5	61.97	10.10	31.97	33.53
		10	60.19	10.26	34.43	35.92
FS		15	60.73	10.69	34.53	34.64
	30%	5	60.99	10.21	33.19	34.82
		10	56.33	10.05	33.10	34.64
		15	52.00	11.58	35.83	37.66
	10%	5	60.88	8.30	29.36	30.51
		10	44.92	10.42	27.11	29.45
		15	43.10	11.70	29.94	31.45
	20%	5	58.62	7.98	26.49	28.02
		10	47.29	10.32	32.09	33.03
	15	45.83	11.94	32.97	35.92	
	5	52.09	9.87	28.33	29.86	
	10	45.87	11.73	30.68	32.86	
	15	45.32	10.77	25.39	28.06	

Ac= *Acacia catechu*, Pe= *Phyllanthus emblica*, Tb= *Terminallia bellirica*, Pg= *Punica granatum*, Fs= Ferrous sulfate.

emblica, *P. granatum* and *T. bellirica* were found between good to very good (4-5) ratings and wet rub fastness ratings were fairly good to very good ratings of 3-5 while *Acacia Catechu* showed very good dry rub fastness and fairly good to good wet rub fastness and ferrous sulfate showed 4-5 (good to very good) dry, and moderate to good wet rub fastness at 1-5 rating's Gray scale of with pre-mordanting method of mordanting using different concentrations of mordants.

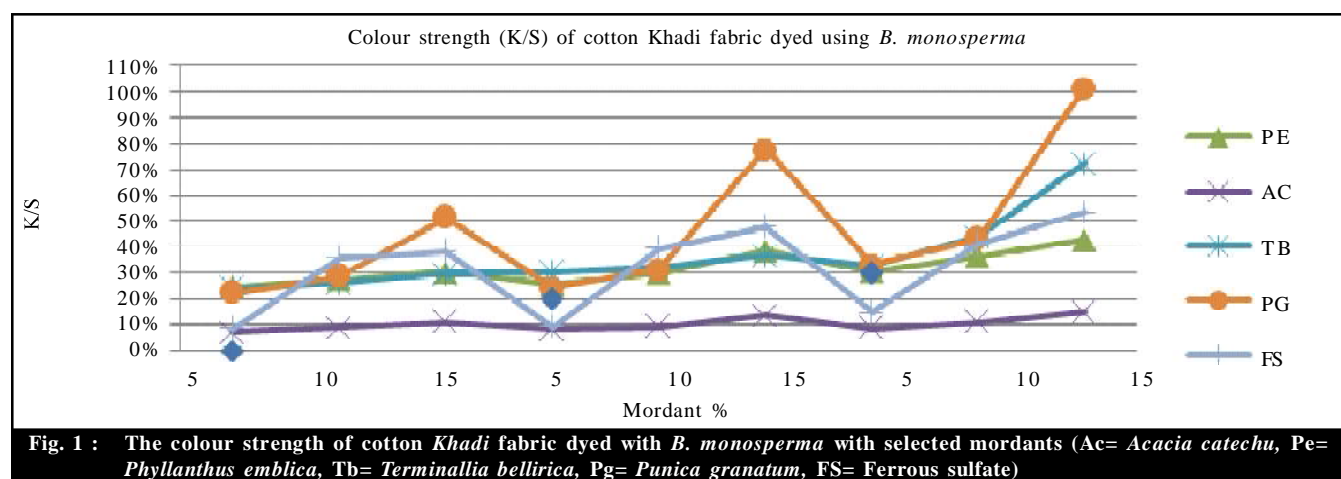
It can be noticed in Table 2, the light fastness of unmordanted dyed sample with 10%, 20% and 30% concentrations of *B. monosperma* dye showed; significant to moderate fading (3-5) against blue dyed standards of 1-8 rating scale. Light fastness properties of *P. granatum* and *T. bellirica* produced moderate fading to no fading. Application of *P. emblica*, f. sulfate and *A. Catechu* registered moderate to very slight fading (4-7) on the bases of light fastness grade scale (1-8). In comparison with un-mordanted and mordanted, the mordanted dyed samples gave best results of colour fastness properties of cotton *Khadi* fabric.

Colour characteristics based on CIE lab colour coordinates of dyed cotton *Khadi* fabric :

Colour characteristics of all the dyed *Khadi* samples were assessed in terms of CIE L^* , a^* , b^* Where L^* =Lightness, a^* =red-green, b^* = yellow-blue, and colour strength (K/S values). *B. monosperma* is a common plant in Indian continent and the flowers of this plant, flame of forest are very useful in traditional medicines and more particularly for inflammatory diseases etc. The colouring component is present in the petals of *B. monosperma*

flower is Chalcones (Firdaus and Mazumder, 2012). Dyed fabric samples gave low lightness with the increasing in the concentration of dye and mordants, unmordanted dyed samples gave higher to lower lightness values i.e. 82.69, 78.84, 71.23, respectively. As the concentration increased the shades of colour are going to be darker in case of mordanted dyed samples. The mordants, which presented darker to lighter colour shades are Ferrous sulfate > *P. granatum* > *A. Catechu* > *P. emblica* > *T. bellirica*. As the concentration was increased of dye and mordants, the lightness values of samples were decreased of in case of all mordants as compare to unmordanted samples. Lightness values of cotton *Khadi* fabric dyed with petals of *B. monosperma* reveals that darker shades were obtained with pre-mordanting method with different dye and mordant concentrations. Mordanting has significant effect on the lightness of shades.

In Table 3, a^*b^* plot of cotton *Khadi* fabric samples indicates that all the samples dyed with petals of *B. monosperma* extract with different concentrations were found in the red-yellow zone. Furthermore on the comparison of a^*b^* values of all mordanted samples with different concentrations in increasing order and it is observed from the above data in Table 3 that mordanting with, *P. granatum* and *T. bellirica*, is found to be helpful in increasing in both zone of a^*b^* plot, redness (higher a^* values) as well as yellowness (higher b^* values) of the samples. Dyeing with *B. momosperma* at 20% and 30% concentration and mordanting with *P. emblica* and *A. Catechu* with different concentrations are showing a very little towards green and blue colour and showing



dark shades of dyed samples in respect of a^* and b^* values. Table 3 depicts that a^* and b^* plot's values of dyed *Khadi* samples mordanted with F. sulfate showing, towards slight negative values as compare to un-mordanted dyed samples, that is why these samples are going towards green and blue colour in meager quantity.

Colour strength of cotton *Khadi* fabric with *B. monosperma* roots extract :

It is depicting from the Fig. 1 that the colour strength of dyed samples with *B. monosperma* that as soon as the dyeing and mordant's concentrations were increased, the colour strength of cotton *Khadi* fabric was increased. Mordanted with *P. granatum* gave excellent colour strength (K/S) with 30% dye concentration and 15% mordant concentration and it also gave very good colour strength with all the concentrations of dye and mordants. *T. bellirica* showed very good colour strength with all the concentrations of dye and mordants and was increasing with increase the concentrations of dye and mordant. *P. emblica* and Ferrous sulfate gave better colour strength than *Acacia Catechu*. The increase in the K/S values of dyed cotton *Khadi* fabric samples in the presence of different mordants may be due to the increase in fixation of the colour by the mordants. Dyeing with *B. monosperma* yielded dark shades, and generally darker shades showed little difference in numerical values of lightness, however considerable increase in colour strength (K/S) values after mordanting with different concentrations of all mordants indicates higher dye uptake in the case of mordanted samples.

Conclusion :

It is concluded from the results that *P. emblica* and *P. granatum* mordanted samples shows good to very good colour change (cc) properties and good to very good wash staining ratings on wash staining grey scale and no staining on adjacent fabric was observed as compared to un-mordanted and other mordanted samples. Ferrous sulfate also gave fair to excellent wash fastness. Dry rub fastness of cotton *Khadi* fabric samples mordanted with *P. emblica*, *P. granatum* and *T. bellirica* are found good to very good and wet rub fastness ratings were fairly good to very good on the bases of grey. Light fastness properties of *P. emblica*, *P. granatum* and *T. bellirica* mordants produced moderate fading to no fading. *P. granatum* and *Terminallia bellirica* gave

very good colour strength. The order of colour yield is *P. granatum* > *Phyllanthus emblica* > *Acacia catechu* > *Terminallia bellirica* > Ferrous sulfate. Other colour values were also found to be influenced due to mordanting. Although the petals of *B. monosperma* have very good scope for the application on cotton *Khadi* fabric and provided many colour shades.

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