



Research Article

# Influence of mordant application on the dyeing of nylon substrate with natural dyes extracted from flowers

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The manufacturing of synthetic dyes and their extensive utilization commercially for the coloration of various textiles has been disparaged due to introduction of contamination into the environment. Natural dyes, which were also used prior to the advent of synthetic dyes, in textile wet processing for dyeing and printing, may be considered as an environmental-friendly alternative for the preservation of valuable nature. Natural dyes, particularly those derived from various vegetable resources are considered safe owing to their biodegradable and non-carcinogenic characteristics. The present study deals with the application of natural dyes, extracted from floral parts of various plants, onto a synthetic substrate, nylon. The flowers selected for the dyeing of nylon are – China rose/Gurhal (*Hibiscus rosa-sinensis*), Marigold/Genda (*Tagetes petula*), Flame of the forest/Palas (*Butea monosperma*) and Yellow Bells/Piliya (*Tecoma stans*). The application of the dyes, extracted from respective flowers, has been performed on pre-mordanted samples using exhaust as well as padding techniques. The effect of mordant on the shade, tone, color strength (K/S) values and various fastness properties has been studied on natural color dyed nylon samples. Enhancement in depth of shade, tone/hue variation and improvement in fastness characteristics occur owing to better fixation of the dyestuff onto the mordanted substrate.

**Keywords:** color strength, fastness properties, flower extract, mordant, natural vegetable dye

## INTRODUCTION

Textile wet processing, as the name suggests, consumes a lot of water and causes air and water pollution. The criteria for selecting a textile product can be cost and strength, quality, competence to supply on schedule, ecological and toxicological processes. Nowadays, the ecological aspect are gaining ground for the simple reason that the customer's requirements today are not only influenced by "technical requirements" but also by "social deliberation". The processor must consider safety aspects, hygiene and environmental issues during chemical wet processing of textiles.

The consumption of textiles all over the world is projected to about 45 million tonnes. The estimated consumption of dyes for textile coloration is about 8-12 lacks tonnes, which is being satisfied by the usage of synthetic dyestuffs; however, the cognizance towards environment has modelled many limitations to their use. Synthetic dyes are not only detrimental to the environment but also carcinogenic to human. The use of synthetic dyestuff, during their application in the dyeing and printing industries, has been criticized due to introduction of contamination into the environment (Smith and Wagner,

1991; Glover and Pierce, 1993). To overcome the difficulties and harmful effects, Europe has taken steps by putting prohibition on the use of many dyes and chemicals which are red-listed and unsafe (Ramkrishna, 1999; Gulrajani, 1999). An increasing comprehension that the intermediates and chemicals used in synthetic dyes are toxic and hazardous to human health as well as to the environment, has directed to the resurgence of interest in the non-toxic, eco-friendly coloring materials (Uwe and Micheal, Private Circulation; Ali, 1993; Taylor 1998, Agrawal and Patel, 2000).

One alternative approach to overcome the harmful consequence of synthetic dyes may be the use of natural dyes. Natural dyes exist in nature and have been used for coloration of textiles since time immemorial. The utilization of natural dyes in lieu of synthetic dyes will undoubtedly save the environment and the processing may become harmless; but considering the massive consumption of dyes for textiles and commercial feasibility of natural dyes, it seems that the idea of using natural dyes is not a viable resolution for the commercial applications. Recently, interest in the use of natural dyes has been growing rapidly due to the result of rigorous environmental standards executed by many countries in response to toxic and allergic reactions linked with synthetic dyes. Until about 150 years ago, all dyes were natural substances, derived mainly from plants and animals. There are several plants/plant parts that provide natural dyes which are used in the textile industry. The natural dyes exist in plants as pigment molecules, which impart color to textiles and other materials (Sekar, 1999; Kamel et al., 2005; Vankar et al., 2008; Samanta and Agarwal, 2009). With the world becoming more conscious towards ecology and environment, there is greater need today to resuscitate the tradition of dyeing with natural dyes as an alternative of the hazardous synthetic dyes. However, the traditional method of dyeing is extremely crude. It is well known that the rural folk dye the yarn by heating chopped leaves or flowers of the plant in water. The process lacks proper shade calculation and reproducibility of shade for subsequent dyeing processes; it is also laborious and time-consuming. The ancient art and the written literature, related to the coloration of textiles with natural dyes, has lost with time due to several uncontrollable reasons; at present, many researchers have been working on the dyeing and printing of various textile substrates with different kind of natural dyes. The field may be limited as far as commercial utilization is concerned, but is surely gaining importance for art works, hobby and the nature lovers and environmentally conscious people (Mohanty et al., 1987; Schweppe, 1992; Dalby, 1993; Paul et al., 1996; Glover, 1998).

The application of natural dyes in textile wet processing is a step towards environmentally friendly process. Today, all over the world, people have come to accept the fact that

the natural dyes are more traditional and hygienic (Robertson, 1973).

Beautiful flower resources are available throughout the world and they contain wide range of coloring pigments which can be extracted from the flowers and applied on the textiles. Floral dye sources are more significant for dyeing and printing of textile materials as they offer both color pigment as well as fragrance (Robertson, 1973; Iqbal and Ansari, 2014). A flower's pigment not only help to attract possible pollinators, such as honeybees, butterflies and hummingbirds but it can also be used for the colouration of textile materials. There are two major classes of flower pigments: carotenoids and flavonoids. Carotenoids include carotene pigments (which produce yellow, orange and red colors) whereas flavonoids include anthocyanin pigments (which produce red, purple, magenta and blue colors). Usually, the color a flower appears depends on the color of the pigments in the flower, but this can be affected by other factors. For example, blue cornflowers have the same pigments as red roses, but the pigments in the cornflower petals are bound to other pigments and metal ions, making cornflowers look blue (Mongkhorrattanasit, et al., 2006; Padma et al., 2008; Haddar et al., 2014). Dyes obtained from flowers of Kapila or Kamala, Golden red safflower, Marigold, Parijataka, Tessu, Dahlia gives yellow colour; Balsam flower gives brown dyes; Gold Mohar gives orange dye. Dyes from Rosa-indica gives dull yellow colour on cotton and yellow colour on silk with good fastness properties (Bechtold et al., 2003; Mongkhorrattanasit et al., 2011; Mansour and Gamel, 2011; Nanthini et al., 2013).

In the present investigation, dyes extracted from various well-known flowers have been used for the dyeing of nylon fabric. Apart from traditional dyeing methods, other dyeing techniques, such as pad-dry-steam and pad-dry-cure dyeing have also been adopted to deal with the energy conservation and several ecological aspects.

## MATERIALS AND EXPERIMENTAL METHODS





### Materials

**Fabrics:** Pure nylon fabric (weight: 0.041 g/in<sup>2</sup>; 81 picks/inch and 67 reeds/inch), used for the present investigation, was procured from the local market of Vadodara (India).

**Natural Dyestuffs:** Four natural vegetable dyes, extracted from flowers, were selected for the study. Various characteristics and photographs of these dyes/flowers are given in Table 1.

**Chemicals and Auxiliaries:** Three metallic salts, namely aluminium sulphate (M1), copper sulphate (M2), and

**Table 1:** Various characteristics of natural flower dyes

Common name (Indian name)	Botanical name	Color Index number	Family	Coloring pigment	Photograph
<b>D I</b> China rose (Gurhal)	<i>Hibiscus rosa-sinensis</i>	--	Malvaceae	Anthocyanin	
<b>D II</b> Marigold (Genda)	<i>Tagetes petula</i>	CI Natural Yellow 27	Asteraceae	Rubixanthin, Violaxanthin	
<b>D III</b> Flame of the forest (Palas, Tesu)	<i>Butea monosperma</i>	CI Natural Yellow 28	Fabaceae	Butrin, butein	
<b>D IV</b> Yellow Bells (Piliya)	<i>Tecoma stans</i>	CI Natural Yellow 16	Bignoniaceae	Flavanoid, Zeaxanthin	

potassium dichromate (M3) were used for mordanting purpose. These mordants were procured from Durga Traders, Vadodara and were of Laboratory Reagent grade. All other chemicals used during the work were also of Laboratory Reagent grade.

### Experimental Methods

**Preparation of the Dye Solution:** The solutions for natural dyes from flowers were prepared as follows (Singh and Srivastava, 2015; Vankar, 2016):

In case of *Tagetes petula*, *Butea monosperma* and *Tecoma stans*, 10 g dried flowers of Marigold, Tesu and Piliya were individually soaked in 100 cc cold water overnight and then boiled for 2 hrs and filtered. The volume was made 100 cc and then 2 g/l caustic soda was added and the liquor kept overnight. Finally, the solution was neutralized with dilute acetic acid, and used directly after filtration.

For *Hibiscus rosa-sinensis* flowers, 10 g dried China rose flowers were digested for 1 hour in boiling water and then filtered and the volume adjusted to 100 cc with plain water and the solution was used directly.

### Mordanting Procedures:

The mordants used for the study are aluminium sulphate, copper sulphate, and potassium dichromate. Mordanting was done by using pre-mordanting technique. The mordanting process was done in a bath containing 2% mordant (*owf*) using liquor ratio of 30:1 for 30 minutes at

60° C. After mordanting, the samples were washed thoroughly and air-dried.

### Dyeing Procedures:

Various dyeing techniques utilized for dyeing of nylon fabric are-

- ❖ High temperature exhaust dyeing
- ❖ Pad – dry – steam
- ❖ Pad – dry – cure

**Exhaust dyeing:** The dyeing was performed on a laboratory High temperature water bath (Model, Paramount). The dye solution was prepared as follows

	3 %	Natural dye ( <i>owf</i> )
	10 %	Acetic acid (1:10)
Liquor to material ratio	50:1	
pH	4.5	
Temperature	115 °C	
Total dyeing time	1 hour	

The fabric sample was entered in the above bath at room temperature and worked in cold for about 5-10 min. The temperature of the bath was gradually raised to 115°C and dyeing continued at this temperature for about 45 min. After dyeing, the samples were taken out from the dyebath.

**Pad-dry-steam technique:** This dyeing technique was performed by padding the fabric through a solution containing requisite amount of dye, followed by drying and steaming. The dye bath was prepared using

30 g/l	Natural dye
10 - 15 g/l	Glacial acetic acid

The solution was heated slightly to 40 - 45°C and the fabric was impregnated in this bath and kept under slightly warm

condition for about 5 – 10 min. The fabric was then passed through a padding mangle to squeeze out excess dye solution, using 65 % padding expression by 2-dip-2-nip technique. The padded samples were dried at ambient temperature and then steamed at 105° C for 15 min in a laboratory steam ager.

**Pad-dry-cure technique:** This dyeing technique was performed by padding the fabric through a solution containing requisite amount of dye, followed by drying and curing. The dye bath was prepared using

30 g/l	Natural dye
10 - 15 g/l	Glacial acetic acid

The solution was heated slightly to 40 - 45° C and the fabric was impregnated in this bath and kept under slightly warm condition for about 5 – 10 min. The fabric was then passed through a padding mangle to squeeze out excess dye solution, using 65 % padding expression by 2-dip-2-nip technique. The padded samples were dried at ambient temperature and then cured at 150° C for 5 min.

**Washing procedures:** All dyed sample were thoroughly rinsed with cold water, soaped at 60° C for 10 min and finally washed with cold water and dried.

### Testing and Analysis

**Evaluation of Dyed Samples:** Dyeing performance of various dyed samples was evaluated by computing the relative color strength (*K/S* value) spectrophotometrically. These values are computer calculated from reflectance data according to Kubelka-Munk equation (Billmeyer and Saltzman, 1981).

**1. Assessment of Fastness Properties:** All the dyed samples were assessed for fastness to several agencies like washing, light and rubbing using standard procedures (Standard Methods for the determination of colour fastness of Textiles, *The Society of Dyers & Colourists*, 1962) as mentioned below –

❖ **Fastness to washing:** Wash fastness of different dyed samples was assessed on Launder-o-meter using ISO standard Test No. 3. The change in shade was visualized using Grey scale and graded from 1 to 5; where 1 indicates poor and 5 excellent fastness to washing.

❖ **Fastness to light:** Color fastness to light was evaluated by exposing the dyed samples to sunlight according to AATCC test method 16B-1977. They were graded from 1 to 8; where 1 indicates poor and 8 excellent fastness to light.

❖ **Fastness to rubbing:** The rub fastness of dyed samples was tested on Crockmeter. The specimen to be tested was rubbed against perfectly scoured and bleached

cloth of dimension not less than 22 cm x 5 cm. The white rubbing cloth was placed over the end of the finger of the testing device. In the dry rubbing test, the cloth to be tested was rubbed 10 times in 10 sec in dry state; while in the wet rubbing test, the procedure was same, except that the rubbing cloth was wetted out and squeezed to 100 % expression. The grading was given by taking into consideration the intensity of stain obtained on white fabric as well as lowering in the depth of the rubbed sample. The staining on the rubbing cloth was assessed with the Grey Scale and grades awarded from 1 to 5, where 1 stands for poor and 5 for excellent fastness to rubbing.

### Determination of Shades/Hues of the Dyed Samples:

The shades / hues of various dyed samples were judged by visual assessments from various shade cards (Pantone color code, Asian paint, Dulux, Berger, and Nerolac) available in the Indian market. The final shade was given from the judgment of 5 different viewers.

## RESULTS AND DISCUSSION

In the present investigation, various natural dyes, viz. china rose, marigold, fire of the forest, yellow bells – all obtained from respective flowers, were used for the coloration of nylon substrate. Before dyeing, various metallic mordants were applied by using pre-mordanting technique. The mordants selected were aluminium sulphate, copper sulphate and potassium dichromate. The mordanted nylon samples were then dyed with various extracted flower dyes using different dyeing techniques, such as exhaust, pad-dry-steam and pad-dry-cure dyeing methods. All these dyeings were compared with those obtained on unmordanted nylon samples dyed by analogous dyeing procedure. The dyed samples were evaluated for their color strength, shades/hues obtained with different mordants and fastness properties.

### Dyeing Performances of Various Natural Dyes Using Different Dyeing Methods

Table 2 exemplifies the comparison of dyeing performance of various natural flower dyes applied on nylon substrates using exhaust, pad-dry-steam and pad-dry-cure dyeing techniques respectively. In cases of high temperature exhaust dyeing method, all flower dyes gave good performance in the presence of mordants. The results obtained with china rose and marigold dyes are much better on the mordanted samples than the unmordanted samples. The nylon fabric mordanted with copper sulphate mordant and subsequently dyed with the dye extracted from china rose (*Hibiscus rosa-sinensis*) and marigold (*Tagetes petula*) flower respectively showed 115.2 % and 20.3 % enhancement in the color yield of the dyed sample as compared with the unmordanted dyed sample. It is possible that during exhaust dyeing, maximum fixation of

**Table 2:** Color strength (in terms of *K/S* values) for nylon fabric dyed with various natural flower dyes using different dyeing techniques

Dye	Mordant	K/S values for shades		
		Exhaust	Pad-dry-steam	Pad-dry-cure
D1	Control	8.41	4.02	0.89
	M1	8.99 (+6.80)	1.01 (-2.8)	0.85 (-4.4)
	M2	18.10 (+115.2)	1.31 (+25.9)	0.87 (-2.2)
	M3	8.66 (+2.90)	1.14 (+9.6)	0.93 (+4.4)
D2	Control	17.25	11.71	11.01
	M1	18.39 (+6.6)	12.95 (+10.8)	10.89 (-1.1)
	M2	20.76 (+20.3)	7.95 (-32.1)	8.47 (-23.0)
	M3	21.78 (+26.2)	10.40 (-11.1)	10.67 (-3.1)
D3	Control	21.67	19.88	17.66
	M1	22.69 (+4.7)	15.84 (-20.3)	18.96 (+7.3)
	M2	20.69 (-4.5)	13.56 (-31.7)	12.38 (-21.8)
	M3	24.43 (+12.7)	17.16 (-13.6)	14.32 (+18.9)
D4	Control	3.33	1.21	1.04
	M1	3.90 (+17.1)	1.10 (-9.1)	0.82 (-21.1)
	M2	3.58 (+7.5)	1.35 (+11.5)	1.03 (-0.9)
	M3	3.63 (+9.0)	1.10 (-9.1)	1.05 (+0.9)

D1: China rose D2: Marigold D3: Palas D4: Tecoma stans M1: Aluminium sulphate M2: Copper sulphate M3: Potassium dichromate

**Note:** Figures in bracket indicate percent loss/gain over control sample

**Table 3:** Shades obtained for nylon fabric dyed with various natural flower dyes using different dyeing techniques

Dye	Mordant	Shades obtained		
		Exhaust dyeing	Pad-Dry-Steam	Pad-Dry-Cure
D1	Control	Cinnamon	Cornflower white	Cornflower white
	M <sub>1</sub>	Cinnamon	Cornflower white	Cornflower white
	M <sub>2</sub>	Sandstone	Cornflower white	Cornflower white
	M <sub>3</sub>	Cinnamon	Blue bell white	Blue bell white
D2	Control	Mustard	Vasant yellow	Vasant yellow
	M <sub>1</sub>	Buff	Vasant yellow	Vasant yellow
	M <sub>2</sub>	Sandstone	Bramble hedge	Bramble hedge
	M <sub>3</sub>	Sandstone	Canary yellow	Canary yellow
D3	Control	Mid buff	Lemon yellow	Lemon yellow
	M <sub>1</sub>	Daisy	Daisy	Daisy
	M <sub>2</sub>	Tata orange	Golden yellow	Golden yellow
	M <sub>3</sub>	Deep buff	Ripe mango	Ripe mango
D4	Control	Vista	Linen	Linen
	M <sub>1</sub>	Squire Hill Buff	Straw	Straw
	M <sub>2</sub>	Sandstone	Linen	Linen
	M <sub>3</sub>	Sandstone	Pasture	Pasture

D1: China rose D2: Marigold D3: Palas D4: Tecoma stans M1: Aluminium sulphate M2: Copper sulphate M3: Potassium dichromate

the natural dye onto the nylon substrate can be achieved during dyeing at higher temperature for 1 hour. The color of the dyebath at the end of the dyeing was almost negligible which confirms maximum uptake of the dye by the fibrous material. The Table 2 also indicate the dyeing behavior of various natural flower dyes using pad-dry-steam and pad-dry-cure dyeing techniques. It can be seen from the table that utilization of mordant virtually has no effect on the dyeing performance of the mordant used. Thus, for padding technique, the mordanting process can be ignored unless a desired shade, obtained with a specific mordant, is required. Padding techniques, however are quite helpful in the minimization of the pollution load since minimum amount of effluent is created during such techniques.

### Effect of mordant on shade/hue of dyed samples

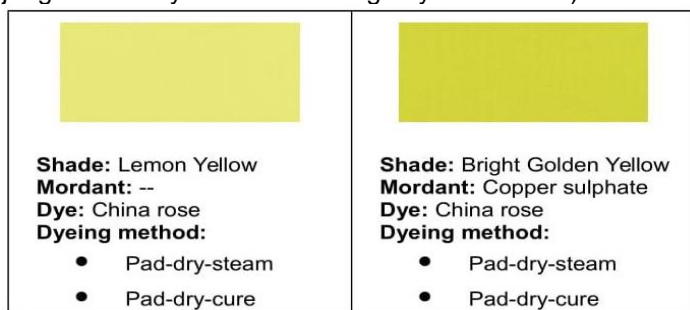
Mordants are substances which improve the affinity of a substrate for a particular dye. In case of natural dyes, various metallic mordants are usually employed to improve the fastness properties of the dyed materials as well as to obtain a variety of shades having different tone and hues. Table 3 represents the shade obtained for unmordanted as well as mordanted samples with various natural flower dyes applied on nylon substrate by various dyeing techniques. It can be clearly observed from the table that a wide range of shades can be obtained with different mordants.

Some of the shades with their fabric sample image have been demonstrated as follows

A variety of **Brown shades** are obtained when dyeing of china rose flower extract was performed by exhaust high temperature dyeing method on untreated as well as copper sulphate treated nylon substrate. Cinnamon shade has been obtained on unmordanted nylon fabric, whereas Sandstone variety of shade (adjudged from the Asian Paint color palette).



**Lemon yellow shade** has been obtained with Fire of the forest (palas) flower dye on unmordanted nylon by using both the padding (pad – dry – steam and pad – dry – cure) dyeing techniques. On the other hand, **Bright golden yellow** shades are being obtained with the same dye on copper sulphate mordanted nylon by using both padding methods (the shades are adjudged by visual judgement only without referring any shade card).



Selected range of **Buff** shades have been obtained on aluminium sulphate mordanted nylon using marigold and Tecoma stans dyes (by exhaust dyeing method) as well as on unmordanted nylon using palas dye (by exhaust dyeing method).



**Effect of mordants on fastness properties of natural colour dyed samples**

Washing, light, and rub (dry as well as wet) fastness properties of various samples dyed with natural flower dyes on unmordanted as well as mordanted nylon substrates were examined and compared with each other. The fastness grades for various dyed samples at maximum concentration of dye (i.e. 3 % for exhaust and 30 g/l for pad-dry-steam and pad-dry-cure dyeing methods) are represented in Table 4. From the table, it is clearly seen that the fastness properties of various mordanted and dyed samples are better than those of unmordanted and dyed samples. The fastness grades particularly washing and light fastness as well as dry rubbing fastness for exhaust dyed samples are very good. For pad-dry-steam process the fastness properties are quite adequate. Out of various dyes used, palas flower dye showed overall excellent fastness properties.

**Table 4:** Fastness grades for nylon samples dyed by using various dyeing techniques

Dye	Mordant	Fastness property for 3 % / 30 gpl shade											
		Exhaust dyeing				Pad-Dry-Steam				Pad-dry-cure			
		W	L	R		W	L	R		W	L	R	
				Dry	Wet			Dry	Wet			Dry	Wet
D <sub>1</sub>	Control	4-5	6	4-5	4	4	6	4	4	4-5	6-7	5	4
	M <sub>1</sub>	5	7	5	4	5	7-8	4	4	4-5	7	4-5	4-5
	M <sub>2</sub>	5	7-8	5	4-5	5	7-8	4-5	4-5	5	7	5	4-5
	M <sub>3</sub>	4-5	7	5	4-5	5	7-8	5	4-5	5	7-8	5	4-5
D <sub>2</sub>	Control	4	7	4-5	4	4-5	6	4	4	5	6	4	4
	M <sub>1</sub>	4-5	7-8	4-5	4	5	6-7	4-5	4	5	6	4-5	4
	M <sub>2</sub>	5	8	4-5	4-5	4-5	7	4-5	4-5	4-5	6-7	4-5	4-5
	M <sub>3</sub>	5	8	4-5	4-5	4-5	7	4-5	4-5	5	7-8	4-5	4-5
D <sub>3</sub>	Control	4-5	6-7	4	4	4	7	4	4	4-5	7	4	4
	M <sub>1</sub>	5	7	4-5	4-5	4-5	8	5	4-5	5	7-8	4	4-5
	M <sub>2</sub>	5	7-8	4-5	4	5	8	5	4-5	5	8	5	4-5
	M <sub>3</sub>	5	8	4-5	4-5	5	8	5	4-5	5	8	5	4
D <sub>4</sub>	Control	4-5	6	4	4	4-5	6-7	4	3-4	4-5	6-7	4-5	3-4
	M <sub>1</sub>	4-5	7	5	4	4-5	7	4-5	4	4-5	7	4-5	3-4
	M <sub>2</sub>	4-5	7	5	4	4-5	6-7	4-5	4-5	5	7-8	4-5	4
	M <sub>3</sub>	4-5	7	5	4-5	5	6-7	4-5	4-5	5	7-8	4-5	4

D1: China rose    D2: Marigold    D3: Palas    D4: Tecoma stans    M1: Aluminium sulphate    M2: Copper sulphate    M3: Potassium dichromate  
 W: Wash fastness    L: Light Fastness    R: Rub Fastness

## CONCLUSIONS

'Eco-friendly', 'eco-conservation', and 'eco-protection' are the buzzwords today. Environmental related issues are freely debated and there is a growing concern over the depleting eco-system. The present study dealt with the replacement of synthetic dyes by natural dyes, derived from various vegetable sources such as flowers of trees and plants. These dyes are supposed to have no carcinogenic effect and are considered to be biodegradable. The flower dyes selected for the study, viz. china rose, marigold, palas and tecoma stans gave yellow to orange to reddish brown tones on nylon substrates. Mordants play an important role in obtaining a desired shade on a particular fabric. A variety of shades with these dyes were achieved on nylon with the help of different mordants. Padding technique was also utilized for the study because it is comparatively cheaper and wastage of dye on commercial application is minimal in such technique. The shades obtained were quite uniform. The fastness properties of the dyed samples were found to be quite good. Washing fastness was excellent for mordanted and dyed nylon fabrics. The rubbing fastness, particularly dry rub fastness was found to be good for all dyed fabrics.

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