DYEING OF COTTON WITH NATURAL COLORANTS EXTRACTED FROM RED ROSE FLOWER

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ABSTRACT: Cotton substrate was dyed with an extract of red rose petals. The dyeing was carried out with and without metallic mordants (alum, copper sulphate, nickel sulphate, ferric chloride and ferrous sulphate), using two different dyeing systems: pre-mordanting system, simultaneous mordanting system. The colour of the cotton substrate was measured by Data color in terms of K/S, and CIELAB values. In both mordanting system ferrous sulphate produced dark shade, the K/S value for simultaneous system is 16.76 and for pre-mordant K/S values is 12.05. It is also observed the higher %fix is of simultaneous system for ferrous sulphate is 76.80% and for pre-moderating the % fix is 71.50%. But statistical analysis of both techniques tells us the reproducibility of copper sulphate is better to other metallic salts the S.D of K/S values for copper sulphate in simultaneous method is 0.2112 and for pre-mordant method the S.D is 0.1132.

Key words: Cotton, Extract, Metallic salts, Mordanting system, Red Rose flower.

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INTRODUCTION

Natural dyes, dyestuff, and dyeing date back to the dawn of the time. Colors have always piqued man's curiosity, and the technique of dyeing has a lengthy history, with many dyes dating back to prehistory. It was used in Europe during the Bronze Age. The earliest recorded mention of the usage of natural dyes is from 2600 BC in China [1]. Natural dyes were the primary colorants for textiles until the late 1800s. Because synthetic dyes have better application qualities than natural colors, synthetic dyes have nearly completely replaced natural dyes. High repeatability and enhanced dyeing quality might be accomplished at a lower particular cost, in addition to a wide range of accessible colors. Demand for more in the future [2,3]. Natural dyes are made up of natural coloring materials that are neither carcinogenic nor harmful to the environment. The hues are relaxing to the eyes, earthy, warm, and aesthetically pleasing. Natural dyes are environmentally benign and aid in the maintenance of ecological equilibrium.

They are non-allergenic, non-toxic to the human body, and they continue an old tradition [4]. Many natural dyestuffs and stains were derived mostly from plants, which prevailed as natural dye sources, generating a variety of colors such as red, yellow, blue, black, brown, and a mixture of these. Plants create dyes in almost every part of their bodies, including the root, bark, leaf, fruit, wood, seed, flower, and so on. It's worth noting that plants generate about 2000 pigments, but only about 150 have been economically utilized [5,6]. Despite the few advantages of natural dyes over synthetic, the use of the original is still very limited due to the unavailability of standard shade cards and standard application procedures [7]. Many natural dyes do not have the strength of fiber and need to be used in conjunction with mordants. Mordant, usually a salt of iron, is considered a chemical, made of dyestuff. The bond is a form between the substrate and the dye, allowing certain dyes with no cohesion to form a substrate [8]. This study aims to address the problems associated with natural dyes. As a type of mordants, system methods for adjusting and investigating CIE laboratory values and K / S of dyed samples and their speed characteristics.

MATERIAL AND METHODS

Flower Colour chosen: We have tried to take some advantage from the petals of red roses as you know in the wedding ceremonies there is a culture of using abundant amount of roses in Pakistan. Approximately in a single wedding 5-10 kg of roses are usually used. But after the ceremony is over, these flowers are spread on the floor as garbage. So in order to take advantage from that flower we did cotton dyeing by using extract of petals.

Studies on cotton substrate: Scoured and bleached, optical brightener free 100% woven cotton fabric (150g/m^2, 1/1 plain weave) was used for natural dyeing purpose.

Chemicals and auxiliaries used: Metallic salts such as Alum, Copper sulphate, Ferric chloride, Nickel sulphate, Ferric sulphate were used analytical grade, purchased

from Merck chemicals Pakistan, and soaping agent Sandopan DTC were used of commercial grades.

Equipment: Dyeing was carried out by dyeing machine (IR AHIBA). The data color (SF 650X) was used for the assessment of colorimetric data. Light fastness was determined on ATLAS Ci 3000^{+} xenon weather-O-meter. Samples were cured on curing machine of Rapid. Fastness rating was assessed on colour matching cabinet by comparing with grey- scale. Rubbing fastness was determined on ATLAS crock meter.

Procedure:

Extraction method: 200 gm of dried red flowers are dissolved in 75% water and 25% ethanol in 1 liter and burned at 100 ^{o}C in a circular bottom bottle stored over a water bath for immediate removal for 80 minutes. All colors are removed from the flowers at the end of the 1 hour. After the extraction was filtered through standard filter paper, filtrate was collected, and the solvent evaporated from a rotating evaporator and returned to dry (5.86gm). 100ml of distilled water is added to this extract. The absorpsor is restored to determine the concentration of the aqueous extract. The visible UV spectrum of the extract showed a high color yield. This was also used for dyeing cloth.

Preparation of cotton substrate: Cotton fabric was washed with a solution containing 0.5g/l of sodium carbonate and 2g/l of non-ionic detergent (Sandopan DTC) at 60 ^{o}C for 30 min, keeping the material to ratio at 1:20. Then washed with distilled water twice then dry the bath substrate for dyeing purpose.

Dyeing without a mordant (control Sample): Dyeing of rose extract is done by exhaust dyeing process. Cotton substrates were dyed in a ratio of 1:20; the dye was 1g/l of soda ash. The temperature was raised to 80 ^{o}C over 30 min and maintained at this temperature for 1 Hr. The dyed cloth was then rinsed with hot water at 60 ^{o}C, followed by a non-ionic soaping agent 2g/l Sandopan DTC. Finally the fabric samples are thoroughly rinsed with cold water, lightened and dried, and then cured to 155 ^{o}C for 60 seconds in a laboratory treatment machine instantly.

Dyeing with mordants: Two different dyeing methods used were pre-mordanting and simultaneous mordanting system. Mordant concentration is 2 and 5g/l used. In the process of recycling, the fabric was first immersed in an aqueous solution of alum, copper sulphate, Nickel sulphate, ferric chloride and ferrous sulphate. The mordant was dissolved in distill water to make an alcohol ratio of 40: 1. A wet sample was placed in a mordant solution and brought to a boil. The dye temperature was raised to 60 ^{o}C for more than 1 hour. The mordanted material was then thoroughly rinsed with water, concentrated, and then dried. Mordanted cotton substrate needed to be applied immediately for dyeing because some mordants were very sensitive to light [9] .The entire fabric was then dyed in the above manner. In the simultaneous merging process five different mordants are also used during the dyeing process above.

Fastness testing: The speed of washing of dyed samples under well-developed conditions was assessed in accordance with the ISO 105-CO3 method. Samples were washed with a standard soap solution at 60 ^{o}C for 30 minutes with an M: L ratio of 1:50.The speed of dry and wet dyeing was assessed in accordance with ISO 105-X12 standards. Light speed was tested according to ISO 105-BO2 methods. The dye was exposed to the xenon arc lamp for 24 hours in standard experimental conditions [10].

Measurement of colour strength: Percentage dye ratio was calculated first by determining the R-appearance of the dyed samples at the wavelength of the small display (high absorption) in the data color SF 650X spectrophotometer. Production value (K/S) was calculated using the Kubelka-Munk (equator 1) and dye adjustment% was analyzed using calculation 2.

$$K/S = (1-R^2)/ 2R \quad \text{---------- (1)}$$

$$\% \text{ Dye fixation} = \frac{K/S \text{ values of sample after soaping}}{K/S \text{ values of sample before soaping}} \times 100 \quad (2)$$

Statistical Analysis: For K / S, L, a, b, c, h, the values of each dyeing condition were assessed by analysis of variance and each treatment was multiplied five times. The most common errors of SED differences are calculated in each mordant with the washing process.

RESULTS AND DISCUSSION

Optimization of extraction conditions: The color density associated with the absorption of dye extracted under different extraction conditions is given in fig. 1. As can be seen in fig. 1, the density of the dye extracted at room temperature was lower, preferably slightly at 60 ^{o}C and above, when the base is made into boiling heat. Tired of room temperature, 60 ^{o}C and boiling temperatures, the moving effect was unusual. The maximum motivation time is 60min. After this time the color density decreases, this is because after such a long movement, certain impurities are removed along with the pigment components, thereby reducing the total color density of the dye extraction [11, 12]. The liquid extraction showed one large Peak, λmax at nm in the visible area. The visual spectrum of the release of new flowers is shown in the picture. The output shows significant changes in different pH as shown in fig 2. At pH 5, 7, 9, λmax was also changed in the case of pH 9, also showing the color of the discharge in the green on the spectrum, and indicating that the dyeing is good in an alkaline state and the effect is a shade of green or similar to green.

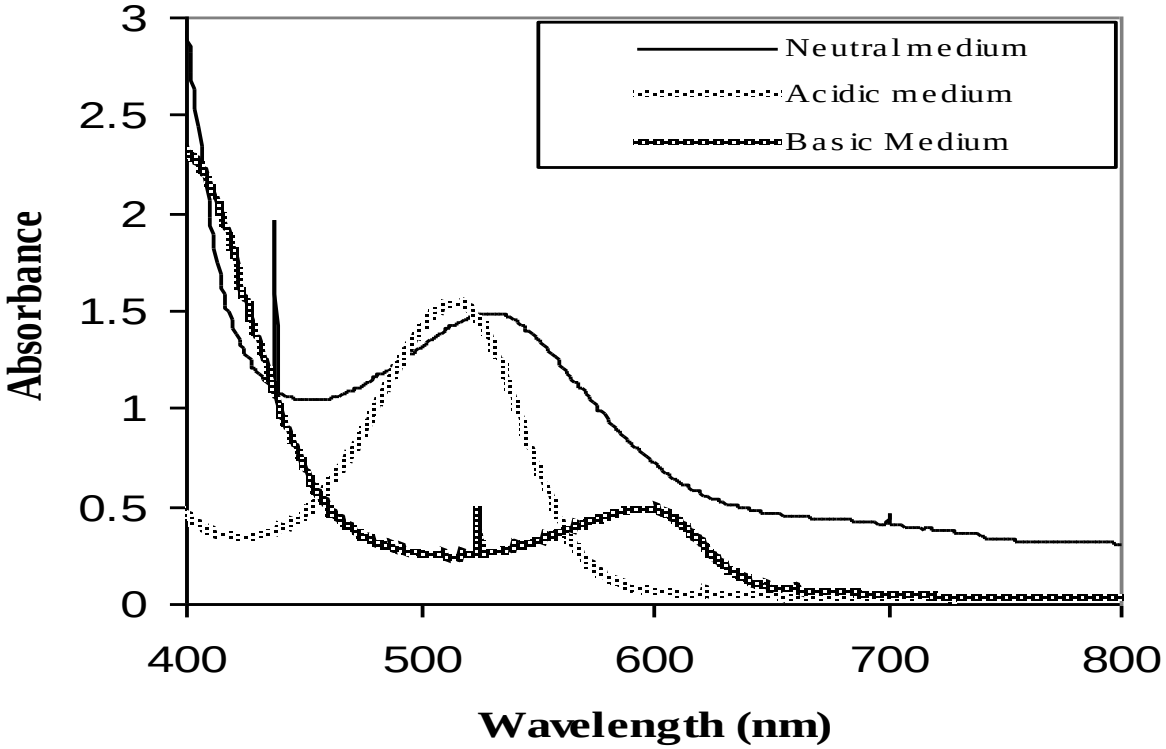

Fig-1.

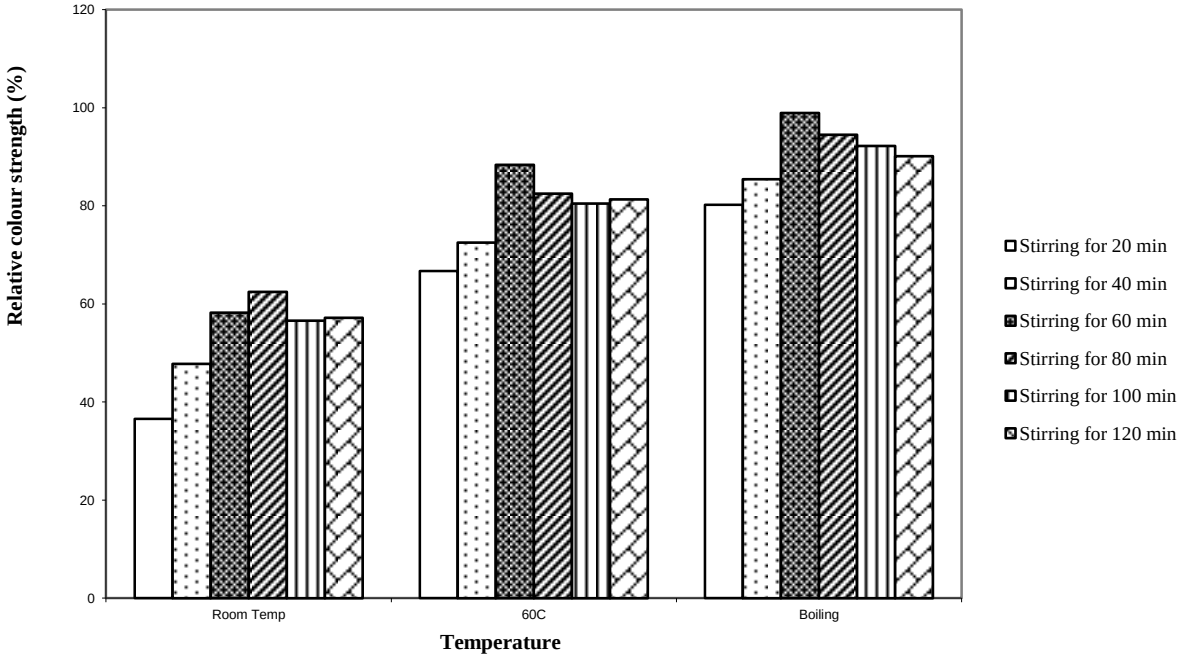

Fig-2.

Effect of Mordants on K / S values: Metallic mordants play a major role in the natural dyeing of the textile substrate [13]. As the K / S value is only 2.41. K / S values increase while dyeing with different mordants and a different mordent input system. In fact most of the extracts of natural dye have a poor affinity for cotton fibers; their speed is often enhanced by metals, which form an insoluble complex with dye molecules [14]. **Tables 1a, 1b, 1c** show that the color strength of the combined and dyed sample is much higher than that of

the control sample. When mordant is added, the increasing amount of K / S indicates the increasing consistency of the dye on the cotton yarn. Medium K / S average values such as mordant sequence simultaneously Ferrous sulphate> Ferric chloride> Copper sulphate> Nickel sulphate> Alum of 16.76, 14.388, 6.746, 5.018 and 3.154 respectively. While in the pre-mordant system the sequence has been Ferrous sulphate> Ferric chloride> Copper sulphate> Alum> Nickel sulphate 12,058, 8.704, 7.822, 5.448 and 3.484 respectively. An increase in the K / S value from the 2.7 controlled sample to the maximum K / S 16.76 of ferrous sulfate, it may be noted that metal mordants, such as polyvalent iron ions, form composite compounds containing specific dyes. During dyeing two types of bonds are involved in the basic reaction between mordants dye and morden. One is a harmonious bond that is usually composed of hydroxyl oxygen and a metal atom, while the other is a bond that binds to metal with double-stranded oxygen called chelation. By comparing the two mordanting techniques, the simultaneous mordanting system makes it look better in terms of high color power values. It may be argued that in the simultaneous mordanting system the metal ion has an equal chance of forming a chelate mixture between dye and cotton fiber, while in the pre-mordant system the mordant first forms the chelate compound with the dye molecule and may have a choice limited repair on the cotton substrate.

Fastness properties of dyed substrate: Washing and quick dyeing of the dyed sample is shown in **table 2**. Samples dyed without re-insertion i.e. the controlled sample showed poor performance properties compared to the mordanting system. The average washing speed of a controlled sample is 2-3. The addition of mordant, quickness of the bath improves the quality by 4-5, 4 of the mordanting system simultaneously and pre-mordants respectively. In the case of light acceleration, the controlled sample has 3-4 dimensions. When the pre-mordanting system is used the light intensity increases dramatically, the average is 6-7, but in the simultaneous mordant, the average is 4-5. In natural dyeing the measuring agent is directly influenced by their dyeing properties and speed structures. The higher light fastness properties of reactive dyes can be attributed to the strong intermolecular H-bonding which exists in the form of 06 membered rings. This enhances the stability of the compound by a decrease in electron density at the chromophore. As a result sensitivity of dye towards photochemical oxidation becomes reduced. But such strong interaction is not present in the rose extract. In fact, speed structures can be updated during repairs. The controlled sample has the worst speed as it has the least adjustment which is only 48.21%. While using mordant, the adjustment increases which increases the properties of the acceleration. Minor differences in adjustment can be seen in **table 2,** between simultaneous mordanting system and pre-mordant system. Stainless steel has high strength in both mordant systems. The order of adjustment in the Pre-mordanting system is Ferrous sulphate> Copper sulphate> Ferric chloride> Alum> Nickle sulphate 71.50, 68.45, 68.22, 64.35 and 59.17 respectively. In the case of the mordant system simultaneously the preparation process is Ferrous sulfate> Ferric chloride> Copper sulfate> Nickle sulphate> Alum 76.80, 70.24, 66.43, 62.55 and 58.06 respectively.

Colour Values: The effect of the pre-repair and simultaneous repair system on L^*, a^*, b^*, c^* and h with different metal modes in the improved conditions is given in table1a, b, c. L^* represents the values of light, the height of light represents the decrease in color output, a^* and b^* represent the color tones, the positive numbers a^* and b^* represent the red and yellow tones while the opposite values show the green and blue tones. C^* represents chroma or purity of color, h represents hue, color shade [15]. Thus the tones, color and chroma of the different experiments translated from **table 1 & 2** are. The L values of the dyed sample showed that the extracted dyes produced only light shades on the cotton substrate. The addition of mordants increases the color blackness by changing the nature of the mordants. The L values of the pre-moderent system and simultaneous moderation, both methods show ferrous sulphate produced a dark shade. The total L values of the ferrous sulphate system are 33.726, but in the pre-moderent system i -sulfate sulphate produces a dark shade total L. 49.936. The values a^* and b^* indicate the tone of the shadow. Alum and nickel sulphate produce a lemon-yellow shade in both dyeing systems but in the case of copper sulphate it produces a green tone. In the case of ferrous sulphate in the simultaneous mordanting system the value of b^* is 14.4, showing a blue tone the overall shade is Grayish violet, in the pre-mordent system they produce a small gray violet with a b^* value of -6.68 . Numbers for all dye tests show a red tone.

Table-1a: Colour coordinates values of Red Rose petal extracts.

Mordent	K/S	Mean	S.D	L	Mean	S.D	a	Mean	S.D	b	Mean	S.D	C	Mean	S.D	h	Mean	S.D
Blank	1.82			69.56			-3.12			3.15			2.75			263.5		
	2.45	2.41	0.3374	68.67	68.606	0.5711	-3.24	-3.306	0.1310	3.18	3.3	0.1251	3.42	3.356	0.3534	265.46	269.37	4.83094
	2.64			68.42			-3.45			3.37			3.64			275.23		
	2.6			68.26			-3.4			3.42			3.57			272.22		
	2.54			68.12			-3.32			3.38			3.4			270.44		

Table-1b: Colour coordinates values of Red Rose petal extracts by pre-moderating system.

S. No	Mordent	K/S	Mean	S.D	L	Mean	S.D	a	Mean	S.D	b	Mean	S.D	c	Mean	S.D	h	Mean	S.D
1	Alum	5.63			67.44			-3.91			2.62			3.92			136.66		
		5.46	5.448	0.1434	67.52	67.636	0.3986	-3.37	-4.008	0.5579	3.18	3.332	0.4413	4.63	4.074	0.3112	136.78	137.368	0.5931
		5.52			67.21			-3.9			3.62			3.94			137.79		
		5.25			68.26			-4.91			3.61			3.92			137.8		
		5.38			67.75			-3.95			3.63			3.96			137.81		
2	Copper sulphate	8.62			50.16			-3.21			14.23			15.14			140.21		
		8.8	8.704	0.1132	50.25	49.936	0.2759	-4.6	-3.54	0.8768	9.82	10.984	1.9166	14.33	13.586	1.1613	128.24	119.75	13.8968
		8.75			49.88			-4.11			9.86			12.41			109.73		
		8.8			49.56			-3.47			11.24			13.48			111.34		
		8.55			49.83			-2.31			9.77			12.57			109.23		

Table-1c: Colour coordinates values of Red Rose petal extracts by Simultaneous moderating system.

S.No	Mordent	K/S	Mean	S.D	L	Mean	S.D	a	Mean	S.D	b	Mean	S.D	c	Mean	S.D	H	Mean	S.D
1	Alum s	3.4			65.32			-4.82			2.23			3.94			122.41		
		2.85	3.154	0.3005	65.54	65.548	0.2773	-4.62	-4.102	0.9484	2.42	2.762	0.4517	3.68	3.5	0.4632	120.46	120.184	1.5763
		3.45			65.38			-3.92			2.92			3.72			120.72		
		2.82			66.02			-2.52			2.86			3.42			118.61		
		3.25			65.48			-4.63			3.38			2.74			118.72		
2	Copper sulphate	6.61			58.45			-4.01			17.85			18.29			102.66		
		6.88	6.746	0.2112	59.26	59.22	0.8796	-4.12	-4.746	0.6230	16.98	17.198	0.5701	17.46	17.748	0.4310	102.43	102.634	0.2173
		6.55			58.26			-5.21			17.44			17.38			102.47		
		7.05			59.79			-5.18			17.38			18.14			102.63		
		6.64			60.34			-5.21			16.34			17.47			102.98		
4	Ferric chloride	14.4			38.72			4.85			7.64			3.48			293.24		
		13.88	14.388	0.2938	45.38	40.858	2.7498	4.76	4.628	0.3808	8.52	8.384	0.4396	3.49	3.574	0.1260	293.26	293.298	0.7524
		14.56			41.44			4.54			8.68			3.48			292.15		
		14.5			39.87			4.98			8.72			3.68			294.21		
		14.6			38.88			4.01			8.36			3.74			293.63		
5	Nickel Sulphate	5.32			69.51			2.3			15.6			15.96			78.06		
		4.62	5.018	0.4206	69.48	69.238	0.7618	2.17	2.224	0.0577	15.24	15.402	0.1997	15.47	15.614	0.7556	78.63	78.38	0.4905
		5.35			68.76			2.18			15.27			14.48			78.84		
		4.5			70.21			2.2			15.26			16.54			78.69		
		5.3			68.23			2.27			15.64			15.62			77.68		
6	Ferrous sulphate	17.05			32.48			6.72			-14.33			5.28			296.76		
		16.45	16.76	0.2247	35.58	33.726	1.3132	6.44	6.548	0.1688	-14.4	-13.786	0.5964	5.26	5.136	0.1774	297.23	297.09	0.2931
		16.85			32.51			6.38			-13.84			5.18			296.81		
		16.65			34.38			6.46			-13.1			4.84			297.21		
		16.8			33.68			6.74			-13.26			5.12			297.44		

Table 2: Fastness properties of Pre or Simultaneous mordranting system.

	Pre-Moderating System					Simultaneous mordranting system				
Metal Salts	**% Fix**	**Washing Fastness ISO-CO3**	**Rubbing Fastness Dry**	**Wet**	**Light Fastness**	**% Fix**	**Washing Fastness ISO-CO3**	**Rubbing Fastness Dry**	**Wet**	**Light Fastness**
Controlled sample	48.21	2-3	4	3-4	3-4	-	-	-	-	-
Alum	64.35	3-4	4	3-4	4-5	58.06	4	4	4	4-5
Copper sulphate	68.45	3-4	4-5	3-4	5-6	66.43	4	4-5	4	5-6
Zinc Chloride		4-5	4-5	3-4	6-7		3-4	4-5	4	4-5
Ferric chloride	68.22	4	4	3	5-6	70.24	4-5	4-5	3-4	5-6
Nickel sulphate	59.17	4-5	4-5	4	6-7	62.55	4-5	4	4-5	5-6
Ferrous sulphate	71.50	4	4	3	5-6	76.80	4-5	4-5	3-4	5-6

Conclusion: The conditions for extraction and dyeing of natural dyes from red rose petal were improved. The resulting dye indicates a good saturation of the cotton substrate. They also have the ability to replace other toxic, sensitive, and cancerous dyes with the medium. Rose leaf as a natural dye is very powerful because it is widely grown due to its commercial and medicinal value. These do not cause damage to the environment during extraction and may be used effectively with mordants. These dyes also improve the depth of the shade. Lighting, washing and rubbing materials of rose petal fabric can be enhanced by a variety of mordants systems. Immediate results have shown that the rose petal dye on the cotton substrate is good enough. The above results strongly indicate that a fabric dyed with rose petal dye can get a huge amount in the color of the fabric and the export market.

Conflict of interest: The authors declare that they have no competing interests.

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