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Optimisation of Dyeing and Mordanting Parameters on Cotton Fabrics Treated With *Allium Cepa* as a Natural Dye Source

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Abstract

Onion peel is having rich source of colorant and found to be waste in large quantities. This can be used for dyeing of textile materials as a sustainable eco-friendly approach. In the present study cotton fabrics were dyed with onion peel dye extracted by using Soxhlet apparatus. Four different mordants like Amla, Myrobalan, Pomegranate Rind and Chitosan were used in different concentrations in order to optimize the right combination. Design of experiment was used for selection of various combinations in order to carry out dyeing process. The color strength was measured using spectrophotometer and also measured the fastness properties of dyed samples. Out of four different mordants chitosan was found to be showing highest K/S values in pre-mordanting method. Fastness properties for almost all the samples were found to be average.

Keywords

Allium Cepa, Amla, Chitosan, Myrobalan, Pomegranate Rind

1. Introduction

Natural dyes are extracted from naturally available sources such as plant, animal, and mineral extracts can be conveniently applied to various textile substrates which are safe because of their nontoxic, and biodegradable nature.[1]Thenatural dyes derived from different sources are given in fig. 1. Among the different sources of natural dyes onion peel considered to be waste and found in large quantities. The skin of onions is inedible however it contains a dyestuff called "Pelargonidin".[2][3]The dye was extracted from onion skin using boiling process and dyed to cotton fabric by using natural and synthetic mordants. Lemon and aloe vera were used as natural mordants and potassium dichromate, iron sulphate, copper sulphate and alum were used as synthetic mordants. The aloe vera under simultaneous mordanting technique and lemon under post mordanting and premeditating technique gave the good result. The iron sulphate under post mordanting technique gave best result in terms of color fastness.[2]Dye was extracted from onion skin by boiling process and dyed to cotton, wool and silk fabrics. In

this the fabric was pre-treated with 2% metal mordant and using 5% of plant extracts. Alum, Copper sulphate, Ferrous sulphate, potassium dichromate, Stannous chloride, Stannic chloride were used as metal mordants to obtain different shades. Colour values such as L*, a, b, c and H and K/S were measured by Premier Colour scan using D65 and 10 standard observer. Fastness properties were also measured.[4]

Dyeing of silk and wool fabrics were carried out with the colorant extracted from *Allium cepa*. Dyeing experiments were carried out varying temperature and pH. The color uptake and color fastness was investigated. Potassium alum was used as the mordant. It was seen that pH of 3 and temperatures of 80 and 90°C produced the best results in wool and silk, respectively. Tests of fastness to washing showed satisfactory results.[5] The experiments on the denim swatches and dyeing using onion skin extracted by using natural mordants like Tannic acid, tartaric acid and harda. In this pre mordanting, simultaneous mordanting and post mordanting techniques were used by taking different combinations such as harda-tartaric acid, tartaric acid - tannic acid, tannic acid -harda as mordants. The mordant combination of harda - tartaric acid in simultaneous mordanting and pre mordanting gave best results.[6]Studies on the application of natural dyes on textiles and its characterization and chemical/biochemical analysis of natural dyes, extraction of

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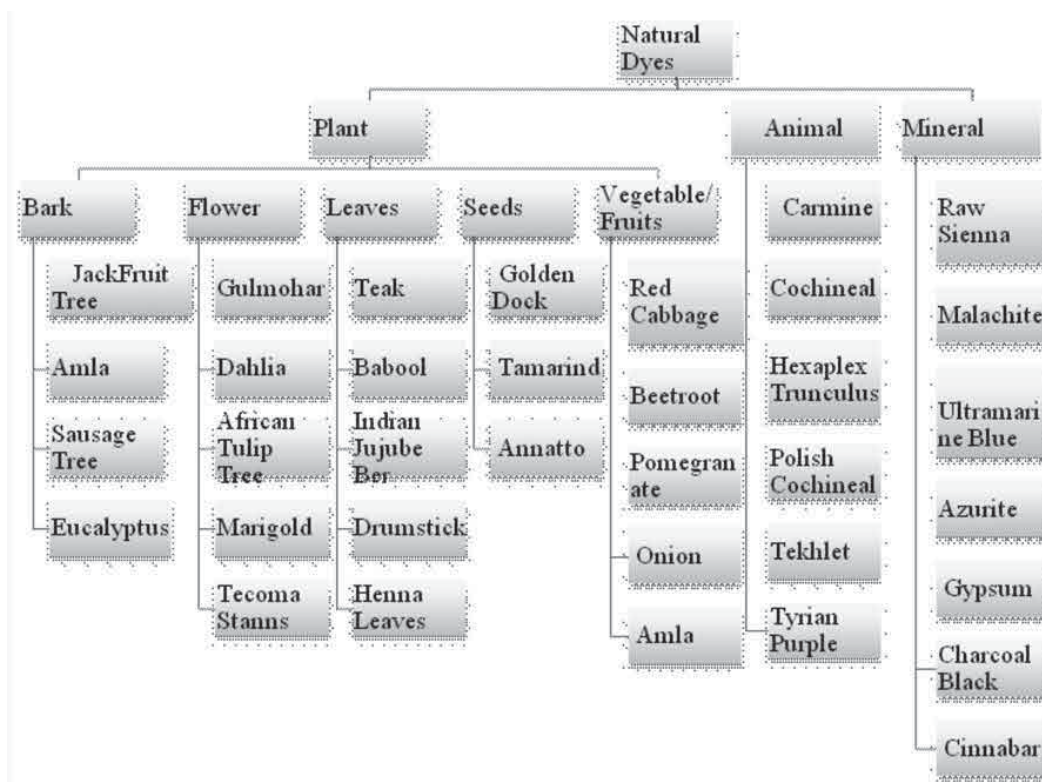


Figure1.1: Classification of Natural Dyes

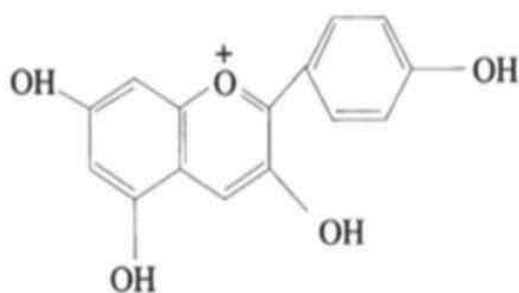


Figure1.2: Structure of "Pelargonidin" (3, 5, 7, 4 tetra hydroxyl antocyanidin)

colorants from different natural sources and effects of different mordants and mordanting methods and also studied about the conventional and non-conventional methods of natural dyeing; physico-chemical studies on dyeing process variables and dyeing kinetics.[7]The dye was extracted from the leaves of *Symplocos racemosa* and dyed to wool by using combination of mordants such as lemon juice + copper sulphate, lemon juice + potassium dichromate, lemon juice + ferrous sulphate and lemon juice + stannous chloride in the ratio of 3:1, 1:2 and 1:3 separately with the help of pre mordanting, simultaneous mordanting and post mordanting technique and fastness properties were evaluated.[8] The colorant

was extracted from the flower of *Spathodea campanulata* and dyed to the cotton fabric with various mordants, such as myrobolan:nickel sulphate, myrobolan: aluminium sulphate, myrobolan: potassium dichromate, myrobolan: ferrous sulphate and myrobolan:stannous chloride, at 1:3,1:1 and 3:1 ratios.[9][10]

Most of the previous works done on dyeing of cotton fabric with onion peel was focused on the fastness and color strength properties by using synthetic mordants. Our interest is to optimize the dyeing and mordanting parameters on cotton fabrics treated with allium cepausing natural mordants. In this present work natural mordants used were amla, myrobolan, pomegranate rind and chitosan by using different mordanting methods with various combinations applied on to cotton fabrics. Color strength and fastness properties such as rubbing (dry/wet) and washing were tested as per the AATCC standards.[11]

2. Materials & Methodology

2.1. Materials

100% Cotton bleached plain fabric was purchased from local market and the specifications were as follows:

- ◆ Weave :plain
- ◆ EPI :102
- ◆ PPI : 90
- ◆ Warp count :80 s Ne
- ◆ Weft count :80 s Ne
- ◆ GSM :120

Onion peel was collected from the local market and the dye was extracted from the peel by Soxhlet apparatus using water as a solvent and the mordants like Amla, harda, chitosan, pomegranate rind powder were purchased from the local suppliers to dye the cotton fabric.

2.2. Methodology

Four different concentrations of dyes were prepared by using Soxhlet apparatus at 70°C up to 10 cycles.[12] The extracted dyes were treated with pre-treated cotton fabrics with different mordants and mordanting types. In order to carry out the dyeing of cotton fabrics, all the four different concentration of dyes were treated on to cotton fabrics with different mordants and three mordanting techniques at a temperature of 80°C in IR beaker dyeing machine for a period of one hour. After that, the dyed samples were taken out from the bath and subjected to soaping in order to remove unfixed dye from the fabric surface.

2.3. Design of experiment

In this present study a total of three factors were taken such as dye concentration, mordanting methods and mordant types with different levels as shown in the table 2.1.

Table 2.1: Factors and Levels Table

Factors & Levels	concentration Dye	Mordanting methods	Mordant Type
1	3	Pre	Amla
2	6	Sim	Myrobolan
3	9	Post	Pomegranate
4	12		Chitosan

A multilevel factorial design was used to optimize the dyeing parameters and table 2.2 shows the particulars of the design. Different combination of treatments selected for the present study as per the design shown in table 4. Therefore, a total of 48 samples were prepared and measured for its color strength and fastness properties.

Table 2.2 : Multilevel factorial particulars

Particulars	Levels
Factors	3
Replicates	1
Base runs	48
Total runs	48
Base blocks	1
Total blocks	1
Number of levels	4,3,4

2.4. Tests for color strength and Fastness

For the dyed samples, the followings tests were carried out to understand the effect of different mordants and their combinations on color strength and fastness properties. Colour strength of dyed samples measured using premier colour scan SS5100HSpectrophotometer. Colour fastness of dyed samples to washing was measured using Laundro-meter according to AATCC-61-2010. Colour fastness of dyed samples to rubbing was measured using Crock meter according to AATCC -08.

3. Results and Discussion

Dyeing was carried out on pre-treated cotton fabric samples as per the multilevel factorial design. CIEL*a*b* values,color strength and fastness properties with respect to wash, and rub/crocking (dry and wet) have been studied on the dyed fabric samples.

3.1. CIE L*a*b*values

Initially the mordanted samples were assessed for their CIE L*a*b* and K/S values by using Premier color scan Spectrophotometer and the results were illustrated in table 3.1. The effect of mordants on the above parameters has been studied.The lightness values has been found to be highest (61.51) in case of pomegranate mordanted cotton fabrics which corresponds to the lighter shades and the lowest (52.92) in case of Chitosan mordanted cotton fabricscorresponding to darker shades. The a* and b* values of amla, harda & pomegranate follows in the range of 3.062 to 6.723 and 13.671 to 31.394, it indicates that mordanted samples are in yellowish red color. In case chitosan mordant samples a* value lies in the range of other mordants, but b* value falls in light blue range. Therefore these samples are in light reddish blue color. Out of four mordants pomegranate treated sample shows highest K/S value of 18.806 and chitosan treated sample shows lowest K/S value of 1.089. These measurements have

been carried out to understand the effect of L* a* b* and K/S values on cotton fabrics dyed with onion peel in combination with different mordants and mordanting methods.

Table 3.1 : CIE L*a*b*and K/S, Reflectance values for mordanted samples

	L*	a*	b*	K/S
Amla	53.146	3.062	13.671	8.087
Harda	57.177	3.655	22.019	10.622
Chitosan	52.924	3.979	-12.675	1.089
Pomegranate	61.513	6.723	31.394	18.806

The effect of different combinations of natural mordants and mordanting types with 3%, 6%, 9%, 12% of onion peel dye on L*, a*, b* and color strength(K/S) values is shown in table 3.2. For the Amla mordanting combinations the lightness values were found to be highest (57.44) which corresponding to be lighter shades and lowest (49.58) which corresponding to darker

shades. K/S values were observed to be highest (43.55) in case of pre-mordanted sample dyed with 9% concentration of onion peel dye powder. The lowest K/S value (14.23) found in the case of post mordanted samples with 3% concentration of dye.

In case of chitosan as a mordant, the pre-mordanted sample with 3% of dye observed to have highest K/S values of 118.72 and lowest value of 13.76 found with simultaneous mordanted samples with 6% concentration of dye. The lowest lightness value of 41.22 found with 12% of dye with simultaneous mordanted samples and highest value of 66.94 found with simultaneous mordanted sample dyed with 6% of dye. The lightness values are in the range of 41.22 to 66.94 which corresponds to darker and lighter shades respectively. From a* and b* values of dyed samples it is clear that the color of dyed samples are in light reddish blue.

Myrobolan as a mordant used to improve the dye uptake by the cotton fabric and the effect of Myrobolan on lightness and color strength has been studied. From the table it is found that, highest K/S value of 54.03

Table 3.2 : CIE L*a*b*and K/S, Reflectance values of Onion peel dyed cotton fabric with different combinations of natural mordants and mordanting types

Run Order	Dye conc. %	Mordanting methods	Mordant Type	Sample Code	L*	a*	b*	K/S
1	6	Post	Myrobolan	M6POST	57.368	10.662	20.889	13.418
2	3	Sim	Amla	A3SIM	52.185	13.767	18.336	16.612
3	6	Pre	Amla	A6PRE	50.783	15.337	16.015	33.651
4	6	Sim	Amla	A6SIM	53.334	10.945	20.45	25.628
5	3	Pre	Pomegranate	P3PRE	46.193	15.855	21.64	28.463
6	3	Post	Chitosan	CH3POST	62.695	9.436	11.88	22.341
7	12	Sim	Pomegranate	P12SIM	71.056	10.477	20.988	21.879
8	3	Pre	Chitosan	CH3PRE	62.65	6.6	11.933	118.722
9	9	Sim	Chitosan	CH9SIM	48.808	16.443	25.492	17.544
10	6	Post	Amla	A6POS	49.582	7.088	12.728	17.821
11	9	Sim	Amla	A9SIM	52.536	8.794	18.895	16.085
12	3	Sim	Chitosan	CH3SIM	66.12	10.53	20.383	17.283
13	9	Post	Amla	A9POST	49.798	6.586	13.633	20.244
14	3	Post	Amla	A3POS	50.061	7.478	14.285	14.233
15	12	Post	Amla	A12POST	54.281	9.069	14.378	19.577
16	3	Sim	Pomegranate	P3SIM	49.19	12.447	26.476	17.173
17	12	Sim	Amla	A12SIM	57.44	12.568	20.14	30.534
18	3	Post	Pomegranate	P3POST	42.372	12.127	12.055	21.753
19	9	Pre	Pomegranate	P9PRE	68.624	10.938	16.081	27.068

20	9	Post	Myrobolan	M9POST	66.144	9.095	20.14	16.181
21	12	Pre	Amla	A12PRE	50.014	11.82	14.28	35.69
22	3	Pre	Myrobolan	M3PRE	55.941	15.38	18.333	44.174
23	9	Sim	Pomegranate	P9SIM	70.291	8.088	19.373	12.536
24	6	Sim	Pomegranate	P6SIM	70.31	7.461	19.39	11.587
25	6	Post	Chitosan	CH6POST	63.92	11.549	14.972	40.776
26	9	Post	Chitosan	CH9POST	43.264	15.862	14.191	28.547
27	6	Post	Pomegranate	P6POST	70.574	6.491	20.086	19.905
28	9	Pre	Myrobolan	M9PRE	56.224	14.021	18.831	49.906
29	12	Sim	Myrobolan	M12SIM	67.516	9.752	21.909	22.034
30	9	Sim	Myrobolan	M9SIM	57.207	12.67	19.744	24.25
31	3	Pre	Amla	A3PRE	50.243	16.483	14.304	40.579
32	6	Pre	Myrobolan	M6PRE	55.927	15.158	18.475	38.376
33	6	Pre	Pomegranate	P6PRE	44.673	16.268	18.205	22.116
34	9	Pre	Chitosan	CH9PRE	45.829	18.396	20.965	64.508
35	12	Post	Pomegranate	P12POST	69.749	9.206	18.542	22.086
36	9	Post	Pomegranate	P9POST	71.368	6.32	21.459	15.508
37	9	Pre	Amla	A9PRE	50.83	14.104	15.813	43.555
38	12	Sim	Chitosan	CH12SIM	45.105	6.605	18.257	16.189
39	12	Pre	Myrobolan	M12PRE	64.138	9.832	16.058	54.033
40	12	Pre	Chitosan	CH12PRE	43.548	14.827	15.573	49.465
41	12	Post	Chitosan	CH12POST	41.226	5.984	8.432	20.527
42	6	Pre	Chitosan	CH6PRE	63.22	13.276	14.003	101.567
43	6	Sim	Chitosan	CH6SIM	66.944	9.892	21.807	13.768
44	12	Pre	Pomegranate	P12PRE	69.59	11.95	18.335	36.851
45	6	Sim	Myrobolan	M6SIM	59.601	12.33	24.227	16.454
46	12	Post	Myrobolan	M12POST	66.064	6.934	20.295	13.459
47	3	Post	Myrobolan	M3POST	58.191	9.483	22.857	14.62
48	3	Sim	Myrobolan	M3SIM	57.831	13.482	21.523	12.803

when it was pre-mordanted with 12% of dye and lowest K/S value of 12.803 observed in simultaneous mordanting with 3% dye. Almost all the dyed samples the lightness values found to be in the range of 55.9 to 67.5 and from a* and b* values it is clear that the color of the dyed samples are in light reddish blue.

Pomegranate rind powder was also used as a mordant and the effect of it in three different mordanting methods on color strength and lightness values has been studied. Pomegranate in pre-mordanting with 12% concentration of dye shows highest K/S value of 36.85 and lowest value 11.58 in the case of simultaneous mordanting with 6% concentration of dye. The lightness values are in the range of 42.37 to 71.36. From a* and b* values of dyed samples it is clear that the dyed samples were in light reddish blue in color.

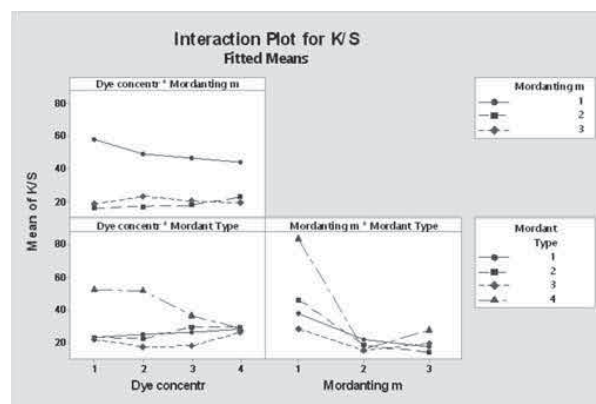


Figure 3.1 : Interaction plots for K/S of fitted means

Interaction plots of K/S have shown in fig. 3.1. The

Table 3.3 : Effect of different natural mordants and its combination on Colour Fastness of onion peel dyed fabric

Sample code	Rubbing Fastness				Washing Fastness	
	Dry		Wet			
	color change	color staining	color change	color staining	color change	color staining
A3PRE	4-5	2-3	4	2	1	3-4
A3SIM	4-5	2-3	1-2	2-3	1	4-5
A3POST	4-5	2-3	4-5	2-3	3	4
A6PRE	4-5	2-3	4	2-3	2	4
A6SIM	3-4	2-3	2-3	2	1	3-4
A6POST	3	2-3	3-4	2-3	3	4
A9PRE	3-4	2-3	2-3	1-2	1	3-4
A9SIM	4	2-3	3-4	2	1	4
A9POST	4-5	2	4-5	2-3	3	3-4
A12PRE	2	2-3	2-3	2	1	4
A12SIM	2-3	4	2	2	1	3-4
A12POST	2-3	2	2	2	1-2	4
M3PRE	4-5	2-3	2	4	1	4
M3SIM	4	3	2-3	3	2	2-3
M3POST	4	2-3	2	4-5	2-3	2-3
M6PRE	2-3	2-3	4	1-2	1-2	4
M6SIM	4-5	2-3	3-4	2	1-2	3
M6POST	4-5	3	3-4	2	2	3
M9PRE	4-5	2-3	4	2	1	3-4
M9SIM	4	3	3-4	2	2	2-3
M9POST	4	2-3	4-5	2	1-2	2
M12PRE	4	2-3	3-4	1-2	2	3-4
M12SIM	3-4	2	4	1-2	1-2	2-3
M12POST	3-4	2-3	4	2	3	2-3
P3PRE	4	3	3-4	2	1	4
P3SIM	3-4	2-3	4	1-2	3	3-4
P3POST	2	3	4	1-2	1	4
P6PRE	4	2-3	4-5	2	1	3-4
P6SIM	4-5	3	4-5	2	2-3	4
P6POST	4-5	3	4	2	3	3
P9PRE	4	3	4	2	1	4
P9SIM	3-4	3	3	1-2	1-2	4
P9POST	4-5	3	4	1-2	2-3	1-2
P12PRE	4	2-3	3-4	1-2	1	4-5
P12SIM	3-4	3	4	2	1	4-5
P12POST	4	2-3	4	2	1	4-5
C3PRE	2	3	2-3	1-2	2	3-4
C3SIM	4	3	3	2	1	4
C3POST	3-4	2-3	4-5	1-2	1	4
C6PRE	4	2-3	2-3	1-2	1-2	4
C6SIM	3-4	2-3	2-3	2	1	4-5
C6POST	3-4	3	3	1-2	1	4
C9PRE	4	1-2	4	3	1	3-4
C9SIM	4-5	1-2	3	1-2	1	4-5
C9POST	3-4	2	4	2	1	4
C12PRE	4	1-2	3-4	2-3	1	4-5
C12SIM	3-4	2	3	3	1	4-5
C12POST	3-5	2	3-4	2-3	1	4-5

interaction effect of dye concentration v/s mordanting method shows that pre-mordanting method with the dye concentration of 3% obtained more K/S values it is due to pre-mordanting. In the pre-mordanting process mordant also have dye compounds and it improves the dye fixation on to the fabric while dyeing. By comparing with mordants used in the study chitosan have shown good K/S values. Fig 3.1 shows the interaction plot for K/S of data means.

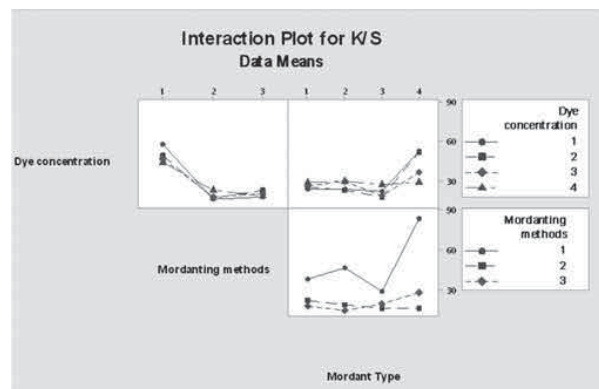


Fig 3.2: Interaction plot for K/S of data means

Fig 3.2, show the main effects plot for K/S of all three variables. The main effects plots confirm that the chitosan has the better colour strength than the Amla, Myrobolan and pomegranate.

3.2. Colour Fastness

The colour fastness measurements were carried out for all the dyed samples and the results were shown in table 3.3. Fastness properties like washing and rubbing have been studied on onion peel dyed cotton fabrics. It has been observed that the washing fastness of onion peel dyed samples for color change rating found to be in the range of 1-2 for almost all the samples. Staining rating on adjacent cotton fabric found to be in the range of 4-5 for all dyed samples. Dry rubbing fastness for color change was found to be in the range of 3-4 and color staining was about 2-3. Wet rubbing fastness for color change was 3-4 and color staining was observed to be 1-2.

4. Conclusion

Dyeing of cotton fabric was carried out by using onion peel as natural dyes and Amla, Myrobolan, pomegranate and chitosan as mordant. It was observed that the interaction effect of dye concentration v/s mordanting method shows that pre-mordanting method with the dye concentration of 3% obtained more K/S values it is due to pre-mordanting. In the pre-mordanting pro-

cess mordant also have dye compounds and it improves the dye fixation on to the fabric while dyeing. By comparing with Mordants used in the study chitosan have shown good K/S values. Average fastness rating for both washing and rubbing is found in almost all the dyed samples.

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