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Discharge printing on turmeric dyed cotton and Silk fabrics

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Abstract
The impact of the textile industry on the environment and the consumption of raw materials and natural resources are becoming prime concerns. It is no longer adequate to have a finished product to be safe only to human beings, but the product has to be environmentally safe during its entire life cycle. This study aims to get environment friendly discharge print on natural dyed cotton and silk fabrics. The ecological impact was assessed on the basis of waste water emission and applicability of the prints by whiteness and tensile strength test. Fabric samples were dyed with natural turmeric in combination with myrobalan and mordants – pomegranate rind (natural), alum and copper sulphate (metallic). Four discharging agents were explored, two reducing i.e. Safolite and Safolin and Hydrogen peroxide and Potassium permanganate from oxidising category. Oxidising discharge pastes gave better results as compared to reducing agents. Discharge results were better on silk fabric than on cotton.

Keywords: Natural Dye, Mordants, Discharge Printing, Effluent Emission

1. Introduction
The various processes used in the textile processing industry contribute to a major portion to the environmental pollution. Textile wet processing industry usually generates large volume of effluents, which are complex in nature and variable both in regards to quantity and characteristics. The wastewater from the textile industry is known to be strongly coloured presenting large amount of suspended solids, pH broadly fluctuating, high temperature, besides high chemical oxygen demand (COD).
Colour also interferes with the transmission of light and upsets the biological processes which may then cause the direct destruction of aquatic life present in the receiving stream [3, 4, 5].
Various attempts are made to reduce the pollution load with the use of natural dyes. Natural dyes refer to those colourants which are obtained from plant, animal and mineral resources [7].
From time to time fashion calls for large patterns or dark grounds to which fine lines, decorative motifs; flowers are to be added in colours or white. Technically it is difficult to meet this requirement with direct printing and hence the popularity of discharge printing [8].
In 1868, German chemists Carl Graebe and Carl Theodore Liebermann observed that coloured organic compounds could be converted into colourless compounds and the original colour could be restored by removing the hydrogen atoms by oxidation. Otto Witt, another German chemist proposed a theory in 1857, according to him; colour usually appears in an organic compound if it contains certain unsaturated groups i.e. groups with multiple bonds. For example, simplest organic compound glyoxal (O=HC-CH=O) is coloured due to double bonds and its reduction product is colourless. This is the basic working principle of discharge printing [3].
Technically, the printing styles may be broadly categorized as direct style, resist style and discharge style. Earlier, discharge method was used to bleach the natural colour of fabric to be printed with direct or resist style [2].
So, the main aim of this work was to combine both the traditional method of natural dyeing and creating prints with discharge style of printing to craft fashionable eco-friendly prints.

2. Methodology
2.1. Material
100% cotton (lawn) and 100% silk fabrics were selected for present study.

Before dyeing the fabrics were scoured in 2g/l of sodium carbonate and 2g/l of commercial detergent were used for cotton and 2g/l of non-ionic liquid detergent for silk at a MLR of 1:40.

2.2. Dye and Chemicals

Locally available Turmeric powder as a natural dye source was selected, which was then extracted by soaking in water for 15 minutes and boiling for 30 minutes with dye conc. of 2% o.w.f. and MLR 1:40. Solution was then filtered through fine fabric and subsequently used for dyeing process.

2.3 Myrobalan: was extracted in water at 3% o.w.f. by the same method used for dye; where the filtrate was used for pre-treating the cotton fabric at boil and silk fabric at 60 ^{0}C for 30 minutes, squeezed evenly, dried and entered in the mordanting bath.

2.4 Mordants: pomegranate rind (powder) as natural mordant and alum and copper sulphate as metallic mordants were selected for the study. Pre-mordanting was selected in which the concentration of the mordants was kept 5% for pomegranate rind and copper sulphate and 10% for alum o.w.f. Natural mordant was extracted by the method used for dye extraction and metallic mordants were dissolved in water before application. Samples were pre-mordanted for 30 minutes at boil for cotton and at 60 ^{0}C for silk, squeezed evenly, dried and entered in the dye bath without intermediate washing.

2.3. Dyeing: The exhaust dye baths were prepared with the requisite amount of dye filtrate o.w.f. Mordanted samples were introduced in the dye bath at room temperature and the temperature was raised to boiling for cotton and till 60 ^{0}C for silk gradually and dyeing was carried out for 45 minutes. After dyeing the samples were rinsed thoroughly and dried.

To understand whether elements of natural dyeing are easily dischargeable or not and which elements are dischargeable by which discharging agent; dyeing was carried out in following combinations from simplest to complex.

F+D
F+M+D
F+H+D
F+H+M+D

Where,
F – Fabric (Cotton/Silk)
H – Pre-treatment (Myrobalan)
M – Pre-mordanting (Pomegranate Rind/Alum/Copper Sulphate)
D – Dyeing (Turmeric Dye)

2.4. Application of standardised discharge recipes on natural dyed samples

Before application of discharge paste on the dyed samples, CIELAB values were measured on spectrophotometer.

For recipe standardization of discharge print, two discharging agents were selected from each oxidising and reducing category. Hydrogen peroxide 35% and Potassium Permanganate were the two oxidising agents and Safolite (Sodium Formaldehyde Sulphoxylate) and Safolin (Zinc Formaldehyde Sulphoxylate) were the two reducing agents selected for discharge printing on natural dyed samples.

2.5. Test Methods

2.5.1. Measurement of Whiteness Index

After completion of discharge printing on combinations of natural dyed samples with standardized discharge recipes, the samples were assessed for whiteness index on Premier 5100 spectrophotometer. Scoured, undyed fabrics were kept as controlled sample.

2.5.2. Measurement of Tensile Strength

Samples with good whiteness index were tested for strength on an Instron Tensile Strength Tester, Model 1121, (Principle – Constant Rate of Extension). Undyed scoured fabrics were kept as controlled sample.

2.5.3. Measurement of Effluent Emission in Waste Water

The effluent emission of the standardized discharge recipes was determined by the Test Method: IS: 14563-1998, ISO: 14184-1-1998 & DIN EN ISO 11885-1998 for magnesium and potassium release as metal from potassium permanganate and formaldehyde release from reducing agents. Hydrogen peroxide was not tested because, it ionises into hydrogen and oxygen ions on reaction.

3. Results and Discussion

3.1. Application of standardised discharge recipes on natural dyed samples

3.1.1. Assessment of CIELAB values of natural dyed samples using spectrophotometer

Table 1 and Table 2 shows the CIELAB value of the shades of turmeric dye produced on cotton and silk respectively by various combinations.

Table 1: CIELAB values of Turmeric on Cotton

	L*	**a***	**b***	**c***	**DL***	**dE***
Controlled	83.249	1.272	-3.52	3.743		
C+T	68.726	6.318	61.073	61.399	-14.526	66.398
C+H+T	59.46	7.176	53.478	53.957	-23.789	62.045
C+PR+T	69.155	7.764	66.949	67.398	-14.094	72.157
C+Al+T	58.066	17.808	41.796	45.432	-25.183	54.417
C+Cu+T	62.037	6.31	55.878	56.233	-21.212	63.273
C+H+PR+T	58.428	7.63	51.028	51.595	-24.821	60.266
C+H+Al+T	63.864	8.47	62.75	63.319	-19.385	69.421
C+H+Cu+T	39.574	11.622	30.821	32.939	-43.648	56.382

Key: C= Cotton, H= Myrobalan, PR= Pomegranate Rind, Al= Alum, Cu= Copper Sulphate, T= Turmeric

Table 2: CIE LAB values of Turmeric on Silk

	L*	a*	b*	c*	DL*	dE*
Controlled	79.914	1.038	10.35	10.402		
S+H	64.443	1.508	25.196	25.241	-15.471	21.447
S+T	67.837	8.895	75.084	75.609	-12.077	66.318
S+PR+T	63.028	5.7	65.352	65.6	-16.886	57.724
S+Al+T	71.49	4.211	76.249	76.365	-8.424	66.511
S+Cu+T	66.438	-2.78	59.957	60.021	-13.476	51.546
S+H+T	61.188	5.132	64.746	64.949	-18.726	57.675
S+H+PR+T	59.065	5.941	59.832	60.126	-20.849	53.918
S+H+Al+T	62.252	5.964	64.087	64.364	-17.662	56.779
S+H+Cu+T	36.625	10.901	30.24	32.145	-43.289	48.65

Key: S= Silk, H= Myrobalan, PR= Pomegranate Rind, Al= Alum, Cu= Copper Sulphate, T= Turmeric

Similar results were seen on both cotton and silk by the various combinations of Turmeric dye. Tables show that there was shade variation in the Turmeric dye by various mordants and myrobalan individually and in combination. With the complexity of the combinations the hue turned darker as seen in the L* values. DL* values show that the darkness in colour was brought by alum and similar trend of colour change was seen in dE* values. Since turmeric dye produces yellow colour in acidic pH, the b* values were higher and positive. Little redness was also seen in the dye. Mordant alum made the hue more yellow in shade and gave maximum b* values. On the contrary, copper sulphate gave redness to turmeric.

3.1.2. Standardisation of discharge recipes

Standardised recipes were formulated and printed on the combinations of natural dye with the help of a stencil. Time, temperature and pH were kept as constant variables. The standardised discharge printing recipes are given below:

Table 4: Standardised discharge recipe of Hydrogen peroxide

Concentration	1:2	1:1
Sodium silicate	13g	17g
Sodium hydroxide	1g	2g
Sodium carbonate	5g	5g
Hydrogen peroxide	14g	21g
Alginate	6g	5g
Water	63g	50g
Total	100g	100g

Table 5: Standardised discharge recipe of Potassium permanganate

Concentration	3%	4%
Potassium permanganate	3g	4g
Alginate	9g	8g
Water	88g	88g
Total	100g	100g

Table 6: Standardised discharge recipe of Safolite

Concentration	10%	15%	20%
Safolite	10g	15g	20g
Glycerine	4g	4g	3g
Alginate	8g	7g	7g
Water	78g	74g	70g
Total	100g	100g	100g

Table 7: Standardised discharge recipe of Safolin

Concentration	10%	15%	20%
Safolin	10g	15g	20g
Glycerine	3g	3g	3g
Gum Arabic	30g	26g	25g
Water	57g	56g	52g
Total	100g	100g	100g

3.2. Assessment of Whiteness/Yellowness Index of selected discharge printed samples

When the combinations of natural dye were printed with standardised recipes, a visual assessment was done and the samples giving results of 4 to 5 rating on the discharge scale were selected and tested on spectrophotometer for whiteness/yellowness index.

Table 8: Whiteness/Yellowness index of selected discharge printed samples on cotton without the pre-treatment of myrobalan

	Whiteness	Yellowness	Brightness
	Hunter	ASTM D 1925	TAPPI 452/ ISO 2470
Controlled	79.935	-5.403	67.013
C+T HP1	66.847	50.752	30.95
C+T PP3	66.846	46.8	32.162
C+T PP4	59.199	63.879	22.471
C+PR+T HP1	71.176	35.899	40.311
C+PR+T PP3	57.304	68.542	19.851
C+PR+T PP4	54.536	70.28	18.092
C+Al+T HP1	56.958	75.284	18.211
C+Al+T PP4	51.536	85.289	13.573
C+Cu+T HP1	51.821	80.829	14.898
C+Cu+T PP4	67.557	37.676	36.044

Key: C= Cotton, PR= Pomegranate Rind, Al= Alum, Cu= Copper Sulphate, T= Turmeric, Sa= Safolite, Z= Safolin, HP= Hydrogen peroxide, PP= Potassium permanganate.

Note: The numeric values represent the concentration of the discharge print recipe.

The wash fastness of turmeric rates 2-3. Turmeric dye is fugitive and bleeds in water. The result in Table 8 on the other hand was completely opposite. Reducing agents, even of higher concentrations, could not discharge C+T samples. Hydrogen peroxide in the oxidising category could discharge better than potassium permanganate. C+PR+T HP1 gave the highest whiteness index of this section. The yellowness index of turmeric dyed samples was found to be more, the reason being turmeric produces yellow colour, so if the dye did not discharge, it accounts into the yellowness index of the sample. The brightness of C+Al+T and C+Cu+T had also dropped. Similar results were also obtained by Arora, G. in their study (1996) [1], the brightness value of the samples dropped down considerably showing that the fabric was affected by the action of discharging agents. Table 8 also shows that it was easier to discharge simple combination of only dyed samples than mordanted and dyed samples, proving that mordants improve the fastness properties of natural dyes by creating stronger bonds with fabric which require more energy to break.

Table 9: Whiteness/Yellowness index of selected discharge printed samples on cotton pre-treated with myrobalan

	Whiteness	**Yellowness**	**Brightness**
	Hunter	ASTM D 1925	TAPPI 452/ ISO 2470
Cotton Control	79.935	-5.403	67.013
C+H+T HP1	65.416	52.288	29.144
C+H+T PP4	62.431	55.264	26.793
C+H+PR+T HP1	66.922	45.826	32.986
C+H+PR+T PP4	56.013	55.291	21.727
C+H+Al+T HP1	53.705	79.968	15.718
C+H+Al+T PP4	58.248	50.584	24.351
C+H+Cu+T HP1	58.917	65.889	21.546

Key: C= Cotton, H= Myrobalan, PR= Pomegranate Rind, Al= Alum, Cu= Copper Sulphate, T= Turmeric, SA= Safolite, Z= Safolin, HP= Hydrogen peroxide, PP= Potassium permanganate.
Note: The numeric values represent the concentration of the discharge print recipe.

Table 9 shows that oxidising agents discharged natural dye far better than the reducing agents. It became difficult to discharge the dye when harda was combined with various mordants. Hydrogen peroxide of concentration ratio 1:1 was found to be the best for this section and in oxidising category. C+H+PR+T HP1 gave better whiteness of 66.922 as compared to C+H+T HP1 with a whiteness of 65.416. Brightness of the samples was reduced considerably due to the number of treatment been performed on it.

Table 10: Whiteness/Yellowness index of selected discharge printed samples on silk without the treatment of myrobalan

	Whiteness	**Yellowness**	**Brightness**
	Hunter	ASTM D 1925	TAPPI 452/ ISO 2470
Silk Control	71.977	23.064	47.3
S+T HP1	72.314	32.797	42.914
S+T HP2	71.166	36.062	40.65
S+T Sa15	66.068	42.387	34.743
S+T Z20	61.496	49.639	28.081
S+PR+T HP1	61.9	53.093	28.043
S+PR+T HP2	57.059	57.603	22.853
S+Al+T HP1	71.131	36.158	40.344
S+Al+T HP2	71.19	34.585	41.383
S+Al+T Z15	60.336	50.624	26.53
S+Cu+T HP1	69.564	37.028	38.979
S+Cu+T HP2	63.856	53.562	27.565
S+Cu+T Z15	60.526	44.459	28.588

Key: S= Silk, PR= Pomegranate Rind, Al= Alum, Cu= Copper Sulphate, T= Turmeric, SA= Safolite, Z= Safolin, HP= Hydrogen peroxide, PP= Potassium permanganate.
Note: The numeric values represent the concentration of the discharge print recipe.

Table 10 shows that silk fabrics on discharging gave very good whiteness index which was very close to the controlled sample. Natural dyes dye silk easily with good fastness properties whereas they just stain the cellulosic fabric with poor fastness properties. This fact was been contradicted in the results seen in Table 8 and Table 10. Potassium permanganate could not discharge dyes on silk, not even as much the reducing agents could discharge. Whiteness of S+T HP1 is 72.314, which is even whiter than the controlled sample with whiteness of 71.977, showing that the dye was completely removed and the discharging agent had bleached the fabric. On a whole turmeric dye was easily discharged. This reading follows the facts of turmeric dye being fugitive and is easy to remove. But the yellowness index of the readings is higher since the dye itself is yellow in colour. Reducing agent, safolin, could also effectively discharge S+T, but hydrogen peroxide gave best results.

Table 11: Whiteness/Yellowness index of selected discharge printed samples on silk treated with myrobalan

	Whiteness	**Yellowness**	**Brightness**
	Hunter	ASTM D 1925	TAPPI 452/ ISO 2470
Silk Control	71.977	23.064	47.3
S+H+T HP1	68.419	39.273	37.251
S+H+T HP2	62.24	46.195	29.53
S+H+PR+T HP1	65.268	43.383	33.112
S+H+PR+T HP2	54.44	55.744	20.647

S+H+Al+T HP1	64.501	45.699	31.911
S+H+Al+T HP2	53.144	68.307	18.057
S+H+Cu+T HP1	52.391	65.302	17.322

Key: S= Silk, H= Myrobalan, PR= Pomegranate Rind, Al= Alum, Cu= Copper Sulphate, T= Turmeric, Sa= Safolite, Z= Safolin, HP= Hydrogen peroxide, PP= Potassium permanganate.
Note: The numeric values represent the concentration of the discharge print recipe.

Table 11 shows that reducing agents could not discharge the combinations. Potassium permanganate being non-compatible as discharging agent on silk substrates, both the concentrations of hydrogen peroxide was finally selected for whiteness/yellowness index under this section. S+H+T HP1 gave the highest whiteness index of 68.419, which is very near to the controlled sample reading of 71.977 whiteness. So we can say that harda could be easily removed from silk fabric with oxidation method giving good results. As metallic mordants make complexes with harda; hence they are difficult to discharge whereas natural mordant pomegranate rind does not make such complexes with harda and is easily dischargeable as seen in this section. These findings contradict the results of Sundrajan, M. & *et al*. (2009). Their study proved pomegranate rind as mordant improves the fastness of natural dyes much better than synthetic mordants [9].
The highlighted combinations from the whiteness/yellowness index values represent the best discharged samples of the study and are closest to the control sample. These were the samples selected for tensile test.
It could be seen that none of the combinations selected for tensile strength test had myrobalan in it and the standardised discharge recipes are from oxidising category.

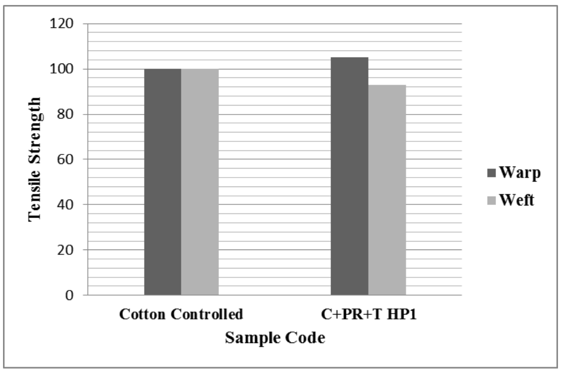
3.3. Measurement of Tensile Strength of the selected discharge printed samples

Generally when a fabric is mordanted or dyed the strength of the fabric increases since new bonds are formed with the fabric. When a discharge treatment is given to these dyed fabrics the deterioration of strength take place from this increased strength. The amount of whiteness obtained will be of no use if the fluidity of the fabric is non-acceptable. So, the strength of the samples giving optimum results in spectrophotometer was tested for their tensile strength.

Table 12: Tensile strength of selected discharge printed cotton samples

Sample Code	Maximum load (kgf)			
	Warp	%Warp	Weft	%Weft
Cotton Control	15.179	100	7.464	100
C+PR+T HP1	15.868	105	6.968	93

Key: C= Cotton, T= Turmeric, PR= Pomegranate Rind, HP= Hydrogen Peroxide.
Note: The numeric values represent the concentration of the discharge print recipe.



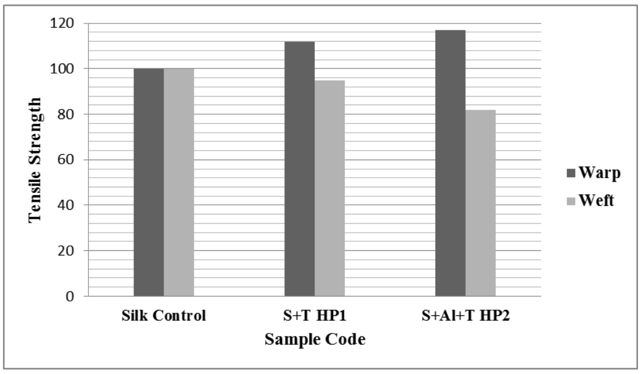
Graph 1: Tensile strength of selected discharge printed cotton samples

As seen in Table12 that warp of C+PR+T HP1 sample, even after discharge it retained more strength and weft had lost about 7% strength then the original fabric. So this standardized recipe could be considered effective for discharge printing, since overall strength loss was not more than 10% from the original fabric strength.

Table 13: Tensile strength of selected discharge printed silk samples

Sample Code	Maximum load (kgf)			
	Warp	%Warp	Weft	%Weft
Silk Controlled	7.034	100	16.941	100
S+T HP1	7.909	112	16.052	95
S+Al+T HP2	8.242	117	13.931	82

Key: S= Silk, T= Turmeric, Al= Alum, HP= Hydrogen Peroxide.
Note: The numeric values represent the concentration of the discharge print recipe.



Graph 2: Tensile strength of selected discharge printed silk samples

Table 13 gives the tensile strength of selected samples from discharge printed silk. From the preliminary data of silk it was seen that silk was weft faced rib fabric so, the wefts were more in number on the surface of the fabric and came in contact with the discharging agents. As a result, it was seen that wefts lost around 18% of its strength. Warps had a good strength after discharge. So, the standardised recipes for silk could be tried by room temperature drying for a longer period than oven drying, since hydrogen peroxide is not as much corrosive at room temperature.

3.4. Measurement of Effluent Emission

Hydrogen peroxide as discharging agent was found useful in discharging all combinations of natural dye with good results of whiteness index and tensile strength. Hydrogen peroxide ionises into hydrogen and oxygen ions on reaction emitting negligible effluent caused by the alkali used in the recipe.

4. Conclusion

- Discharge printing could be effectively put into practice as a new technique to commercialise eco-friendly natural dyed discharge printed products.
- Oxidising agents could discharge all combinations of natural dye effectively.
- Reducing agents could not discharge myrobalan and natural mordant pomegranate rind.
- Hydrogen peroxide from oxidising category gave the best whiteness index, tensile strength with negligible effluent

emission.

- Discharging natural dye from silk is easier but with considerable strength loss, whereas it is the opposite case with cotton.
- Lower concentrations of the discharging agents could be effectively used with simple combinations of natural dye to reduce the effluent emission and retain the strength of the fabric.

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