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Eco-friendly Antibacterial Printing of Wool Using Natural Dyes

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Abstract

Natural dyes which are known to possess antibacterial properties can be safely used for the purpose of antibacterial finishing of natural fibres like wool. Chitosan is a functional biopolymer which can be utilized for various applications in textiles. In the present work bio-waste chitosan was utilized as a mordant in the printing of wool fabric with natural dyes making it eco-friendly. The efficacy of natural mordant chitosan was studied in comparison with alum as a standard mordant. Both the mordants gave prints of similar color values. The printed fabric showed excellent antibacterial properties against both *S. aureus* and *E. coli.* This method hence can be suitable for eco-friendly printing and antibacterial finishing of wool fabrics.

Keywords: Natural dyeing; Antibacterial; Wool; Chitosan

Introduction

Natural fibres are an excellent medium for the growth of microorganisms when the basic requirements for their growth such as nutrients, moisture, oxygen, and appropriate temperature are present. The large surface area and ability to retain moisture of textiles also assist the growth of microorganisms on the fabric [1]. The growth of microorganisms on textiles inflicts a range of unwanted effects not only on the textile itself (such as tendering and its degradation) but also on the wearer (skin rashes, foul smell, etc.) [2]. In the last few decades, with the increase in new antimicrobial fibre technologies and the growing awareness about cleaner surroundings and healthy lifestyle, a range of textile products based on synthetic antimicrobial agents such as triclosan, metal and their salts, organometallics, phenols and quaternary ammonium compounds, have been developed and quite a few are also available commercially [3]. Even though the excellent antimicrobials are available, their user ecology and safety is always a question. Some of the natural dyes, on the other hand such as turmeric, catechu, marigold etc. are reported to possess antimicrobial properties. This is also substantiated by the work reported from our laboratory [4-8].

Synthetic dyes offer the flexibility of selection of proper hue and substantively with reference to the fibre. The development of synthetic dyes in the second half of nineteenth century led to a high quality dyes with better reproducible techniques of application. As a result, a distinct lowering in the dyestuff costs per kg of dyed goods was achieved [9]. Also a full gamut of color is available for application on various types of fibres. However, during the last few decades, the use of synthetic dyes is gradually receding due to an increased environmental awareness and harmful effects, of either toxic degraded products of these dyes or their non-biodegradable nature. In addition to above, some serious health hazards like allergy and, carcinogenicity are associated with some of the synthetic dyes based on azochromospheres. As a result, a ban has been imposed all over the world including European Economic Community (EEC), Germany, USA and India on the use of some synthetic dyes (e.g. azodyes) which finally triggered active research and development to revive world heritage and traditional wisdom of employing safer natural dyes [10]. Even though natural colors cannot substitute the synthetic dyes completely, there is definitely increasing market for such complete eco-friendly dyed or printed materials, if not for common market, but for those who desire to have such products.

Natural dyes with a few exceptions are non-substantive and hence must be used in conjunction with mordants. Mordant is a chemical, which can fix itself on the fibre and also combines with the dyestuff. The challenge before the natural dyers in application of natural color is thus, the necessity to use metallic mordants which themselves are pollutant and harmful. Due to the environmental hazard caused by metallic mordant while dyeing of textile fabric, dyers are always looking for safe natural mordant for natural dyes. In this paper, this issue of screening a natural mordant and exploring its potential for printing of wool with natural dyes is reported.

The main objective of printing is to produce colored patterns with sharp boundaries on textile materials without any dye spreading beyond the boundaries of the motif of design [11]. Although a lot of research has been reported in the area of natural dyeing of textiles, the research in the area of natural dye printing is reported to a limited extent. Also the subject like obtaining antibacterial finish through natural dye printing has also remained unexplored. Under this backdrop chitosan which has been utilized for various applications in textiles ranging from fibres, dyeing auxiliary, printing thickener, antimicrobial finishing, etc. [12-27], becomes an obvious choice for study. There is very limited information available in the literature with regard to use of such functional biopolymer as a mordant in natural dyeing. Application of chitosan as an eco-friendly mordant in dyeing [28] and printing [8] of cotton with natural dyes was reported from our laboratory.

In the present work, chitosan extracted from waste shrimp shells [29] was utilized as a mordant for simultaneous natural dye printing and antibacterial finishing of wool and its performance is discussed in comparison with alum as a standard mordant. The efficacy of chitosan as eco-friendly mordant and antibacterial finish against both gram positive and gram negative bacteria has been investigated.

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Materials and Methods

Materials

Chitosan (mol. wt. 124999.8, degree of deacetylation, 89.06%, nitrogen content, 7.1%) was extracted from waste shrimp shells [29] and used for the study. Wool fabric as well as catechu, turmeric and marigold were purchased from the market. All other chemicals used were of laboratory grade.

Methods

Preparation of mordant: Stock solution of alum was made by dissolving 1 g alum in 100 ml double distilled water. Chitosan was dissolved in aqueous acetic acid (20 g/l). Chitosan powder 1 g, was added to 100 ml acetic acid solution followed by warming it at 60°C for 2 h with continuous stirring using mechanical stirrer.

Preparation of dye for printing: All the dyes used were first converted to powder form. For this purpose, catechu, turmeric and marigold were first dried in an oven at 50°C for 24 h and later grinded in mixer grinder. The powder so obtained was filtered through the 60 mesh nylon fabric. The fine powder of natural dye was used for printing.

Mordanting of wool fabric: Mordanting of wool was carried out with different mordants at two different concentrations on the weight of fabric (alum 10% and 20%; and chitosan 10% and 20%) at 90°C for 60 min with material to liquor ratio as 1:30. The mordanting of wool was carried out in rota dyer (Rota Dyer machine, Rossari® Labtech, Mumbai). The mordanted fabric was squeezed and dried in open width form at 100°C for 5 min and then used for printing.

Printing: Guar gum being nonionic is best suited for textile printing. The stock paste of guar gum was prepared using 2% of guar gum powder. The guar gum powder was sprinkled slowly in water with continuous stirring in order to prevent lump formation. The paste was stirred continuously at 90°C for 1 h.

The print paste was prepared using different dye concentrations (1%, 5% and 10%) on the basis of weight of print paste. The 1% of print paste was prepared using 1 g of dye powder which was first pasted with small quantity of water followed by addition of 99 g of guar gum stock paste. The print paste was continuously stirred for 30 min. The mordanted wool fabrics were then printed with two strokes of squeeze and steamed at 102°C for 10 min. The samples were washed with water and dried in air.

Analysis of printed fabrics

Color value by reflectance method: The printed samples were evaluated for the depth of color by reflectance method using 10 degree observer using Spectraflash SF 300 (Datacolor International, U.S.A.). The K/S values were determined using equation

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

where, R is the reflectance at complete opacity, K is the Absorption coefficient and S is the Scattering coefficient.

Printed fabrics were simultaneously evaluated in terms of CIELAB color space (L*, a* and b*) values using the RayscanSpectrascan 5100+. In general, the higher the K/S value, the higher the depth of the color on the fabric. L* corresponding to the lightness (100=white, 0=black), a* to the red-green coordinate (+ve=red, -ve=green) and b* to the yellow-blue coordinate (+ve=yellow, -ve=blue). As a whole, a combination of all these parameters enables one to understand the tonal variations.

Washing fastness: The printed wool fabric was sandwiched between cotton and wool fabric and then subjected to washing as per the ISO II test method. Evaluation of color fastness to washing was carried out using ISO II method [30].

Light fastness: The light fastness was determined using artificial illumination with Xenon arc light source, Q-Sun Xenon Testing Chamber. One half of each sample was covered with an opaque black paper sheet leaving the other half exposed. The color fastness to light was evaluated as per ISO 105/B02 standards [31].

Determination of antibacterial activities of printed fabrics: The antibacterial properties of the printed fabrics against *S. aureus* and *E. coli* were estimated by AATCC Test Method 100-2004 [32]. The fabric samples were sterilized before actual antibacterial testing using autoclave followed by exposure to UV light.

Results and Discussion

Color values of natural dye printed wool

The color values of the printed fabrics using different concentrations of mordants and dyes are summarized in Tables 1-3.

When the printing was carried out without any mordant, the prints obtained were very light in shade and color bled heavily after washing. However, when alum or chitosan pre-treated fabric was printed, it gave deeper and fast prints. In case of chitosan, the color values of the prints were higher than the respective alum pretreated prints. They also were washing fast and this trend was valid for all the three dyes studied.

As the concentration of mordant increased, even with the fixed concentration of dye in the paste, the K/S values and hence depth of the prints progressively increased which may be attributed to increase in the extent of fixation of the dye. Similarly at fixed mordant concentration, with increase in dye concentration K/S values of the prints obtained increased. This clearly indicates that chitosan played significant role as a mordant and was marginally, but distinctly superior to alum with regard to its efficacy as a mordant. In case of all the three dyes studied, this trend of chitosan having a distinct edge as a mordant over alum was confirmed.

The role of chitosan as a mordant may be attributed to the presence of amino groups which get protonated and offer the sites for attachment of the dye which is mainly anionic in nature. The mechanism of dye attachment to the fibre is similar to that in case of metal mordants.

The varying combinations of mordant and dye resulted in different shades and tones of printed wool fabric. This is evident from the CIE colour co-ordinate results shown in Tables 1-3. The prints obtained using catechu dye, in case of chitosan were more reddish yellow (distinctly higher a* values for almost similar b* values) in tone as compared to the respective prints obtained using alum. In case of turmeric dye, it was alum which gave the prints more yellowish-green (distinctly higher b* values and lower a* values) as compared to those obtained using chitosan as a mordant. For marigold however, no distinct trend in the tonal variation was observed, although the K/S values were slightly but distinctly higher for the chitosan than that for alum mordanted prints.

The results in Tables 1-3 indicate the comparative fastness properties in the case of both the mordants. The printed samples without mordanting showed much inferior fastness properties to those obtained with mordanting (irrespective of the type) which indicates the positive role of a mordant in holding of the dye on the fabric. It is well

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Mordant	Mordant (%)	Catechu (%)	Colour value	CIE colour coordinates			Washing fastness$	Light Fastness#
			K/S@	L*	a*	b*		
None	0	10	0.83 ± 0.04	73.35	7.52	15.39	3	3
Alum	10	1	0.96 ± 0.04	70.32	0.04	10.19	4-5	6
	10	5	1.52 ± 0.03	71.69	2.93	15.15	4-5	6
	10	10	1.63 ± 0.04	72.02	3.95	16.65	4-5	6
	20	1	1.04 ± 0.03	69.55	-0.18	8.60	4-5	6
	20	5	1.41 ± 0.04	70.22	1.96	11.92	4-5	6
	20	10	1.64 ± 0.03	72.10	3.96	16.70	4	7
Chitosan	10	1	1.20 ± 0.02	68.01	2.53	12.65	4-5	6
	10	5	1.61 ± 0.03	67.87	4.74	13.98	4-5	6
	10	10	1.75 ± 0.04	68.31	6.05	15.96	4-5	6
	20	1	1.09 ± 0.04	66.87	0.91	10.21	4-5	6
	20	5	1.68 ± 0.02	67.39	4.69	13.37	4-5	7
	20	10	1.75 ± 0.03	68.14	5.43	15.29	4-5	7

$Rating 1-5, where 1-poor, 2- fair, 3-good, 4-very good and 5-excellent.
#Ratings 1-8, where 1-poor, 2-fair, 3-moderate, 4-good, 5-better, 6-very good, 7-best and 8-excellent.
@Represents average values of three determinations.

Table 1: Effect of mordants and catechu concentration on color strength of printed wool.

Mordant	Mordant (%)	Turmeric (%)	Colour value	CIE colour coordinates			Washing fastness$	Light Fastness#
			K/S@	L*	a*	b*		
None	0	10	1.60 ± 0.02	85.14	-1.61	11.32	2	2
Alum	10	1	1.97 ± 0.03	72.39	-5.70	34.03	4-5	4
	10	5	4.45 ± 0.02	79.54	-5.41	47.18	4-5	5
	10	10	8.68 ± 0.04	84.08	-6.19	55.34	4-5	5
	20	1	2.55 ± 0.03	76.88	-7.97	42.05	4-5	4
	20	5	6.62 ± 0.02	83.12	-7.29	53.38	4-5	5
	20	10	9.32 ± 0.04	85.12	-7.17	57.05	4	5
Chitosan	10	1	2.42 ± 0.03	71.24	-3.44	34.48	5	4
	10	5	5.37 ± 0.04	77.24	-3.94	44.77	5	5
	10	10	11.31 ± 0.04	78.18	0.53	48.07	4-5	5
	20	1	2.89 ± 0.02	70.24	-1.47	33.98	4-5	4
	20	5	7.86 ± 0.03	76.59	0.30	45.11	4-5	5
	20	10	11.95 ± 0.04	82.25	-3.95	52.64	4-5	5

$Rating 1-5, where 1-poor, 2- fair, 3-good, 4-very good and 5-excellent.
#Ratings, 1-8, where 1-poor, 2-fair, 3-moderate, 4-good, 5-better, 6-very good, 7-best and 8-excellent.
@Represents average values of three determinations.

Table 2: Effect of mordants and turmeric concentration on color strength of printed wool.

Mordant	Mordant (%)	Marigold (%)	Colour value	CIE colour coordinates			Washing fastness$	Light Fastness#
			K/S@	L*	a*	b*		
None	0	10	1.8 ± 0.02	85.63	-1.57	12.2	2-3	3
Alum	10	1	2.69 ± 0.03	70.76	-3.44	33.72	4-5	5
	10	5	6.03 ± 0.02	68.78	1.83	32.49	4-5	7
	10	10	13.44 ± 0.04	73.31	4.29	39.94	4-5	7
	20	1	3.09 ± 0.03	70.24	-0.99	36.67	4-5	6
	20	5	7.59 ± 0.04	69.54	2.76	37.35	4-5	7
	20	10	11.85 ± 0.04	80.47	-0.86	50.09	4	7
Chitosan	10	1	3.52 ± 0.03	64.37	0.58	26.33	5	6
	10	5	8.67 ± 0.05	77.40	0.07	47.08	5	6
	10	10	14.0 ± 0.04	78.81	3.85	51.57	4-5	7
	20	1	3.75 ± 0.03	64.98	-0.79	25.96	4-5	7
	20	5	8.79 ± 0.01	78.27	-1.22	46.95	4-5	7
	20	10	14.09 ± 0.05	71.14	7.61	42.03	4-5	7

$Rating 1-5, where 1-poor, 2- fair, 3-good, 4-very good and 5-excellent.
#Ratings, 1-8, where 1-poor, 2-fair, 3-moderate, 4-good, 5-better, 6-very good, 7-best and 8-excellent.
@Represents average values of three determinations.

Table 3: Effect of mordants and marigold concentration on color strength of printed wool.

Mordant	Mordant conc.	Dye	Dye Conc. (in print paste)	Bacterial Reduction@ (%)	
				S. aureus	*E. coli*
None	0	None	0	0.53 ± 0.1	0.65 ± 0.1
Alum	20	-	0	74.32 ± 0.5	70.25 ± 0.6
Chitosan	20	-	0	78.10 ± 0.25	75.75 ± 0.30
-	0	Catechu	10	65.30 ± 0.3	65.80 ± 0.40
-	0	Turmeric	10	63.50 ± 0.3	61.75 ± 0.45
-	0	Marigold	10	67.75 ± 0.25	66.80 ± 0.20
Alum	20	Catechu	10	98.00 ± 0.1	97.50 ± 0.4
Chitosan	20		10	99.20 ± 0.2	99.45 ± 0.25
Alum	20	Turmeric	10	97.25 ± 0.15	97.10 ± 0.20
Chitosan	20		10	98.75 ± 0.25	99.10 ± 0.3
Alum	20	Marigold	10	98.50 ± 0.4	99.10 ± 0.25
Chitosan	20		10	99.75 ± 0.20	99.60 ± 0.3

@Represents average value of 3 determinations.

Table 4: Effect of mordant and dye combinations on antibacterial properties.

known that the use of mordants is essential to fix most of the natural dyes on the textile fabric.

The improvement in fastness properties with increase in mordant concentration clearly indicates the positive role of mordants in case of printing with natural dyes. The washing fastnesses obtained varied in the range of good to excellent grade. Light fastness was found to be improving with increase in K/S values, which is quite obvious.

Antibacterial activity of printed wool fabric

The results for quantitative antibacterial assessment of the prints are presented in Table 4. The wool fabric showed negligible antibacterial properties against both *S. aureus* and *E. coli*. The prints obtained only using the dyes such as catechu, turmeric and marigold in absence of any mordant showed significant antibacterial activity (in the range of 61-68%) indicating the inherent nature of these dyes in preventing the bacterial growth. These results are also supported in the literature [4-8]. In case of prints obtained using the mordant such as alum and chitosan, the extent of bacterial reduction was significantly increased to the range of 97-100%. This clearly indicates the positive contribution of alum and chitosan mordants in enhancing the antibacterial activity of the printed fabric. As far as antibacterial activity is concerned, the chitosan mordanted samples showed comparative antibacterial properties than that of alum in case of all the dyes studied. Chitosan is known for antibacterial activity against broad spectrum bacteria and mechanism of antibacterial activity was explained by researchers [16,33]. It is also to be noted that alum itself provides antibacterial activity which is mainly because of metal ions. The chitosan hence can be claimed as eco-friendly mordant-cum-antibacterial finishing agent for printing of wool using natural dyes.

Conclusion

The printing of wool fabric with natural dyes in fine powder form was successfully carried out using chitosan as an eco-friendly mordant and its performance was compared with alum mordant. The color values varied with the dye-mordant combination; however the chitosan mordant showed higher color values than those with alum mordant. The excellent antibacterial activity and fastness properties were displayed by the printed samples. The role of chitosan as a mordant in natural dye printing of wool fabric has been confirmed. The antibacterial printed wool fabrics can thus be obtained using eco-friendly method employing chitosan.

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