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Environment Friendly Process of Dyeing Wool with Natural Dye Turmeric (*Curcuma domestica*) After Enzymatic Pre-Treatment

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Abstract

The study was undertaken to optimize the conditions of enzymatic pre-treatment for pure wool using protease enzyme before dyeing with natural dyes. The conditions for enzymatic pre-treatment were optimized after studying the change in the physical properties—weight loss, strength loss, loss in thickness, moisture absorption and flexural rigidity. The optimized conditions included 8.4 pH media, 1% enzyme concentration and 45 minutes treatment time. The treated and the untreated wool fabrics were dyed with turmeric dye along with four natural mordants- arjun, babool, eucalyptus and amla, after optimizing the dyeing and mordanting conditions. The effect of enzymatic pre-treatment on the dyeing properties was studied after comparing the CIE Lab values and the color fastness properties of both the pre-treated and the untreated dyed wool fabric samples. It was observed that the samples pre-treated with enzyme were darker in color and had improved fastness properties as compared to the untreated samples.
Key words : Enzyme, Natural dye, Natural mordant, Optimization, Fastness properties.

Natural dyes can be broadly classified as natural organic dyestuffs of vegetable and animal origin, mineral dyestuffs and inorganic pigments. The organic dyestuffs are obtained from roots, stems, leaves, berries and flowers of various plants and from certain insects and shellfish with a elaborate series of processes. Natural dyes are either substantive, needing no mordant, or adjective, requiring a mordant. The majority of the natural dyes need a chemical in the form of a metal salt to create an affinity between the fiber and the pigment. These chemicals are known as mordants. The mordant helps to produce faster shades by forming an insoluble compound of mordant and dyestuff within the fiber itself (1). Therefore, mordants are considered as an integral part of natural dyes, however the use of natural dyes with mordants alone does not help the environment much because some of the synthetic mordants are deadly poisonous. The use of most of synthetic mordants result in metal contamination in the waste liquor resulting from the dyeing process. In wool dyeing using alum tarter, potassium bromate, stannous chloride, ferrous sulfate and copper sulfate, the proportion remaining in the waste stream is in the range of 84 to 94% (2), hence, eschewing hazardous chemicals and using natural mordants is the need of the hour. Eco-friendly mordants however, help in the production of some pleasing colors without harming the eco-cycle. Still dyeing is a complex process that requires pre-treatment or modification of the raw material or semi-products to improve the dyeing effects. At present some reagents used for the pre-treatments are harmful and its effluents cause water pollution. But the problem can be solved by the use of bio-materials such as enzymes. Thus, taking into consideration the growing environmental consciousness and to make the process of natural dyeing truly eco-friendly, the present study was undertaken to standardize the process of enzymatic pre-treatment and dyeing conditions of turmeric dye using natural mordants for wool and to study the effect of enzyme pre-treatment on its dyeing and fastness properties.

Methods

Pure wool (off white) fabric was used for the research work. Protease enzyme—Greasenz PWE New liquid was used for the pre-treatment of wool. Rhizomes of turmeric (*Curcuma domestica*) dye were finely ground and powdered to extract the dye. The natural mordants used included arjun (*Terminalia arjuna*) bark, babool (*Acacia nilotica*) bark, eucalyp-

Table 1. Optimization of the conditions for enzymatic pre-treatment.

	Weight loss (%)	Strength loss (%) Warp	Weft	Loss in thick-ness (%)	Moisture absorp-tion (% regain)	Flex-tural rigidity
pH						
8.0	9.13	26.63	48.52	0.15	57.31	0.562
8.5	7.59	24.02	42.13	1.56	60.13	0.205
9.0	14.62	39.58	56.29	1.97	68.48	0.166
Enzyme Concentration (o.w.f, %)						
0.25	0.76	17.35	29.44	0.61	50.2	0.544
0.5	4.10	20.43	35.76	0.84	56.92	0.266
1.0	7.81	23.13	40.72	1.25	60.00	0.207
1.25	11.06	31.41	46.85	2.0	60.27	0.208
1.5	13.98	48.63	52.04	3.2	64.47	0.122
Treatments Time (min)						
30	5.12	20.13	36.50	0.83	58.63	38
45	7.32	23.72	41.84	1.39	60.21	211
60	11.24	31.97	48.32	1.42	63.98	207
75	17.81	46.23	54.95	2.13	67.63	198
90	21.12	59.18	61.78	2.76	69.84	167

tus (*Eucalyptus* sp.) bark and amla (*Embilica officinalis*) fruit. These were selected because of their high tannin content.

To remove impurities from the fabric, the samples were soaked in a warm 0.5% solution of a mild non-ionic detergent for 30 minutes, keeping M:L ratio of 1:20. After kneading and squeezing, the samples were rinsed in tap water and dried in shade.

Optimization of Enzymatic Pre-Treatment

Experiments were conducted to determine the optimum values of three variables, namely pH of the enzyme solution, treatment time and the enzyme concentration after preparing a buffer solution of tris (hydroxymethyl) amino methane (tris HCl) by mixing 0.2 M solution of tris (hydroxymethyl) amino methane, 0.2N HCl solution and distilled water. The wool fabric was treated with protease enzyme at different pH levels—8, 8.5 and 9; at different enzyme concentrations, viz., 0.25, 0.5, 1.0, 1.25 and 1.5% (o.w.f.) and at different treatment times—30, 45, 60, 75 and 90 minutes in a launderometer. The recipe for enzymatic treatment was as follows : Buffer solution – 1: 30 (M:L), enzyme concentration – variable, non-ionic surfactant – 1g/liter, pH –variable, temperature – 50—55 C, time – variable.

To destroy the activity of enzyme, the temperature of the liquor was raised to 80—90 C. The fabric was thoroughly washed in water and dried (3). The physical properties—weight loss, strength loss, loss in thickness, moisture absorption and fluctural rigidity of wool samples treated with protease enzyme at different pH levels, enzyme concentrations and treatment durations were tested using standard test methods (4). On the basis of change in the physical properties, the variables for the enzymatic pre-treatment were selected for the final treatment.

Table 2. Optimized dyeing conditions.

Dyeing conditions	Optimum value
Dye extraction pH	9
Dye extraction time (min)	60
Dye concentration (g/g)	2
Dyeing pH	4
Dyeing time (min)	75

Optimization of Dyeing Variables

Experiments were conducted for optimizing the dyeing and mordanting conditions of wool with turmeric dye using four different natural mordants. The conditions namely—dye extraction pH, dye extraction time, dyeing pH, dye material concentration, dyeing time, mordant extraction pH, mordant extraction time, mordant concentration and mordanting method

Table 3. Optimized mordanting conditions.

Mordant	Mordant extrac-tion pH	Mordant extrac-tion time, min	Mordanting method	Mordant concen-tration (% o.w.f)
Arjun	10	60	Simultaneous-mordanting	0.6
Babool	10	90	Pre-mordanting	0.4
Eucalyptus	10	60	Pre-mordanting	0.5
Amla	7	90	Simultaneous-mordanting	0.5

Table 4. Effect of enzymatic pre-treatment on CIE LAB values of samples dyed with turmeric dye using four mordants. ET=Enzymatically treated, EUC=Enzymatically untreated control.

	L*		a*		b*		C*	
Mordants	EUC	ET	EUC	ET	EUC	ET	EUC	ET
Control	61.860	63.053	9.910	9.038	61.616	66.932	62.407	67.407
Arjun	58.405	60.395	7.868	6.649	29.969	32.230	30.984	32.908
Babool	54.303	58.276	9.835	7.711	33.396	32.068	34.814	32.982
Eucalyptus	56.432	61.385	9.659	6.274	33.787	37.654	35.140	38.199
Amla	59.746	63.519	7.821	5.298	52.880	56.860	53.455	57.106

were optimized (5).

After giving the enzymatic pre-treatment, wool was dyed at the optimized dyeing and mordanting conditions.

Evaluation of CIE LAB Values

The color strength of the dyed samples (enzymatically treated and untreated) was measured numerically through computerized color matching system. The L*, a*, b* and c* for all the dyed samples were calculated and those of the enzymatically treated samples were compared with untreated samples to see the effect of enzyme treatment on the color obtained.

Evaluation of Color Fastness Properties

Dyed samples were evaluated for their color fastness to washing, light, rubbing and perspiration using standard test methods (6) to study the effect of enzyme pre-treatment on dyed samples.

Results and Discussion

The change in the physical properties of the treated wool samples at different pH levels of the enzyme solution is shown in Table 1. It was observed that weight loss and strength loss (both warp and weft) were minimum at the pH 8.5. A negligible change in loss of thickness, moisture regain and flextural rigidity was observed. Since weight and strength are comparatively more important physical properties to be considered, therefore, the pH 8.5 was selected as the optimum pH for enzyme treatment.

On the other hand, as the concentration of enzyme increased, there was an increase in the weight and strength loss. A slight decrease in thickness was also seen with an increase in the concentration. Further, the moisture absorption increased and flextural rigidity decreased. Out of the five concentrations, the middle concentration i.e. 1.0% concentration was considered optimum because beyond this concentration the weight and strength loss increased which is not recommended as it reduces the durability of the fabric. With regard to treatment time, an increase in the time of enzyme pre-treatment led to an increase in the weight and strength loss. These was also a negligible increase in the loss of thickness. An increase in the treatment time also resulted in an increase in the moisture absorption but the flextural rigidity decreased. To maintain the quality of the fabric in terms of its weight and strength, 45 minutes of pre-treatment was selected for the final treatment.

The optimum dyeing conditions of turmeric dye on wool were given in Table 2. It was envisaged that the maximum percentage of dye was absorbed at dye extraction pH 9, dye extraction time 60 minutes, dyeing pH 4, dye material concentration 2 g/g of fabric and dyeing time 75 minutes. Therefore, these conditions were considered to be optimum and were used for carrying the research forward. The optimum mordanting conditions have been presented in Table 3. The mordant extraction pH and mordant extraction time were optimized after measuring the optical density of the dye solution with uv-vis spectrophotometer whereas the optimum mordanting method and the mordant concentration were selected on the basis of visual inspection. The optimized mordant extraction pH for the mordants was 10 except alum for which it was 7. The optimum extraction time was 60 min for arjun and eucalyptus but it was 90 min in babool and amla. In mordanting method, simultaneous mordanting was optimum for arjun and amla, whereas

Table 5. Color fastness grades for enzymatically untreated and treated wool samples dyed with turmeric dye using four mordants. CC–Color change, CS–Color staining, W–Wool, C–Cotton, EU–Enzymatically untreated, ET–Enzymatically untreated.

Samples	Light fastness grades	Washing fastness grades			Rubbing fastness grades				Perspiration fastness grades					
		CC	CS		Dry		Wet		Acidic			Alkaline		
			W	C	CC	CS	CC	CS	CC	CS		CC	CS	
										W	C		W	C
Unmordanted														
EU	2	5	2	3	5	5	5	4	5	4	4	5	3	3
ET	2	5	3	4	5	5	5	4/5	5	4	4	5	3	3
Arjun														
EU	2	3	5	5	5	5	5	5	5	5	5	5	5	5
ET	2/3	5	5	5	5	5	5	5	5	5	5	5	5	5
Babool														
EU	2/3	5	5	5	5	5	5	3	5	5	5	5	5	4/5
ET	3	5	5	5	5	5	5	4	5	5	4/5	5	5	4/5
Eucalyptus														
EU	2	5	5	5	5	5	5	4	5	5	4/5	4	5	4
ET	2/3	5	5	5	5	5	5	4/5	5	5	5	5	5	4/5
Amla														
EU	2/3	4	4	4	5	5	5	3	5	4/5	4	4/5	4	4
ET	3	4/5	4/5	4/5	5	5	5	4	5	4/5	4	5	4/5	4

it was pre-mordanting for eucalyptus and babool. With regard to the mordant concentration, the optimum value for eucalyptus and amla was found to be 0.5, for arjun it was 0.6 and 0.4 for babool.

The CIE LAB values of both the enzymatically treated and untreated samples dyed with turmeric dye using four different mordants are given in Table 4. The Table reveals that the L* value of enzymatically treated sample was less than that of the untreated sample for unmordanted samples and samples mordanted with arjun, babool, eucalyptus and amla, which indicate that the enzymatically treated samples were darker in color. The results were similar to those of Riva et al. (7) who reported that the action of enzymes resulted in increased dye absorption. The authors were of the view that the enzymatic treatment resulted in better diffusion of the dye, giving darker shades and offered a possibility to dye wool under milder temperature.

The low a* value of enzymatically treated samples for unmordanted and those mordanted with all the four mordants as compared to the untreated samples indicated that the enzymatically treated samples were less red. Whereas, the high b* value of enzymatically treated samples for unmordanted, mordanted with arjun, eucalyptus and amla as compared to the untreated samples indicate that the enzymatically treated samples were more yellow except babool where the value was low indicating less yellow content.

The higher C* values for enzymatically pre-treated samples, unmordanted and those mordanted with arjun, eucalyptus and amla are indicative of the fact that the enzymatically pre-treated samples were more saturated and brighter as compared to the untreated samples. But on the other hand the lower C* values for enzymatically pre-treated samples, mordanted with babool show they were less saturated and dull than the untreated samples. Pankaj (6) and Banga and Patil (8) analyzed the CIE LAB values of dyes using natural mordants and concluded that the hue, value and intensity of the color is affected by natural mordants. Further, the mordants also improved dye uptake.

The color fastness grades of all the dyed samples, enzymatically treated and untreated are shown in Table 5. The light fastness grades were poor and fair for the control and they improved to fair and fairly good when these were enzymatically pre-treated. The washing fastness grades in terms of color change were fair to good for a few samples which improved to excellent because of enzymatic pre-treatment. In color staining the grades were fair and good for some samples and they improved due to enzymatic pre-treatment. The rubbing fastness grades regarding color change in both dry and wet condition were excellent for all the pre-treated and control samples. In wet and dry conditions, the color staining improved from good to excellent due to enzymatic pre-treatment. In case of perspiration fastness in both the acidic and alkaline media, there was no color change for all the pre-treated and control samples but the color staining grades were fair to good for most of the samples. The enzymatic pre-treatment did not show much improvement. Singh and Goel (9) treated wool with three proteolytic enzymes and then dyed it with natural dyes. The treatment led to significant increase in percent dye absorption and improved the color fastness properties.

It is evident that protease enzyme can be successfully used for the treatment of wool as it does not adversely effect the physical properties of the fabric. The results of Buschle-diller et al. (10) showed that the enzymatic treatment had a hydrolyzing effect on the cuticle of wool fiber, the correlation of enzymatic treatment with SEM (scanning electron microscope) of cuticle revealed that the hydrolysis was directly proportionate to the concentration of the enzyme that had an effect on the physical properties of the fiber. A decrease in weight, strength, thickness and flextural rigidity was observed in the present study but it was within an acceptable range. Moreover, the moisture absorption increased markedly because of enzymatic treatment (11). Hence, turmeric dye along with four different natural mordants can be successfully used for dyeing both the enzymatically pre-treated and untreated wool fabric with optimized dyeing and mordanting conditions.

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