Indian Journal of Fibre & Textile Research
Vol. 45, September 2020, pp. 362-365

Bio-polishing of mulberry silk

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Received 29 October 2018; revised received and accepted 6 March 2019

A proteolytic enzyme, papain, has been used for bio-polishing of mulberry silk. Improvement in lustre, with no significant change in absorbency, flexural rigidity and crease recovery, due to the cleaning of the fibre surface by the enzyme is reported. A chemometric experimental design is used with two variables and five levels.

Keywords: Absorbency, Bio-polish, Crease recovery, Flexural rigidity, Mulberry silk, Papain

The bulk of the commercial silk produced in the world comes from mulberry silk. Mulberry silk is produced from the silkworm, *Bombyx mori L.* which solely feeds on the leaves of mulberry plant. These silkworms are completely domesticated and reared indoors. Silk emitted by the silkworm consists of two main proteins, (sericin and fibroin), fibroin being the structural center of the silk, and sericin being the sticky material surrounding it. Fibroin is made up of the amino acids Gly-Ser-Gly-Ala-Gly-Ala and forms beta pleated sheets. Hydrogen bonds form between chains, and side chains form above and below the plane of the hydrogen bond network. A substantial part of sericin is removed in degumming in order to make the silk soft, lustrous and highly absorbent for dyes and chemicals. Degumming of mulberry silk[1] has been the subject of many recent investigations. Among different degumming processes, enzymatic degumming has been proved to be a better process[2], because of its mild action on fibres and it produces uniformly degummed silk with soft handle. The term bio-polishing is increasingly being used to define the enhancement of luster and improvement in the feel of the cotton fabric after its treatment with enzymes, Such a study is limited in case of different varieties of silk.

A proteolytic enzyme (degummase) was used for bio-polishing of tasar silk[3] with improvement in luster and no change in mechanical properties. Bio-polishing of different varieties of tasar silk with degummase and Protease A "Amano" 2 was also reported[4] for the improvement of luster. Normally, mulberry silk looses its luster and feel due to degumming process depending on the removal of silk gum to different extent. Bio-polishing of mulberry silk is expected to enhance such properties. However, there is no reported literature on bio-polishing of mulberry silk fabric. In the present work, enzymatic treatment has been used to enhance the various properties of mulberry silk fabric by removing the residual sericin.

Experimental

Degummed mulberry silk fabric of 85 g/m^2 was purchased from the local market of Sathyamangalam, Tamil Nadu and used in this study. Papain, a proteolytic enzyme was purchased from the chemical market of Erode, Tamil Nadu. All other reagents used were of analytical grade. Before the fabric was subjected to any further treatment, the degummed mulberry silk fabric was washed at 60^{o}C for 30 min The fabric was then dried and stored.

Treatment with Papain Enzyme

To optimise the experimental conditions vis-à-vis enzyme concentration (X_1) and time (X_2), while keeping the temperature constant at 60^{0}C, a chemometric experimental design[5] for two variables and five levels was used. The levels of variables investigated are shown in Table 1. A total set of 13 experiments was performed.

Washed fabric samples of the size 90 cm × 40 cm was conditioned and weighed accurately. The treatment was carried out keeping the material – to – liquor ratio at 1:20. The recipe for the enzyme treatment was as follows:

Parameters		Value
Enzyme concentration, %	:	Variable
$NaHCO_3$, g / L	:	5
M: L ratio	:	1:30
Temperature, ^{o}C	:	60
Time, min	:	Variable

After the enzyme treatment, the mulberry silk was washed, dried and conditioned.

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Preparation of Control Sample

The control sample in comparison with enzyme treated sample was prepared by subjecting the washed samples to the treatment with non-ionic detergent (Hostapal MRN Liquid, Clariant Chemicals). Treatment with 1 g/L detergent was given at 60^0C for 30 min, keeping the water level constant.

The alkali treated fabric was prepared by boiling mulberry silk sample in a buffer of Na_2CO_3 and $NaHCO_3$ (0.05M; *p*H 10.3) for 60 min. The parameters evaluated were: Y_1 = weight loss, Y_2 = absorbency, Y_3 = flexural rigidity, Y_4 = crease recovery and Y_5 = lustre.

Weight Loss

Conditioned samples were weighed accurately before and after the enzyme treatment and the weight loss was calculated as follows:

$$\text{Weight loss percentage (\%)} = [(W_1 - W_2) / W_1] \times 100$$

where W_1 is the weight of the fabric before treatment; and W_2 , the weight of the fabric after treatment.

Measurement of Properties

Absorbency was evaluated using AATCC/ASTM test method TS-018. The fabric to be tested was placed over the beaker and water was allowed to drop on the fabric from a height of 1 cm. Time taken for water absorbency was observed. For flexural rigidity, bending length was determined. Eight strips of (6 × I) inch each were cut warp - and weft - wise and the bending length was determined on Shirley Stiffness tester. The following formula was used to convert bending length to flexural rigidity:

$$\text{Flexural rigidity (G), micro Nm} = M \times C^3 \times 9.807 \times 10^3$$

where C is the bending length in mm; and M the mass per unit area (g/m^2). For crease recovery, strips of (2 × 1) inch were cut and tested on the Shirley crease recovery tester. The lustre of the fabric was measured as reflectance on a reflectance spectrophotometer (*Gretag Macbeth*) with D65 light using Color i-control software interfaced with IBM PC. Lustre was then calculated using the following equation:

$$\text{Lustre} = \{(L_1 - L_2) / L_2\} \times 100$$

where L_1 and L_2 are the area under the reflectance curves of the enzyme treated and untreated samples. The scanning electron micrographs were recorded on a Cambridge SEM.

Table 1 — Variables and their levels used in the experiments

Variables	Levels				
	-1.137	-1	0	1	1.137
Enzyme conc. (X_1), %	0.03	0.15	0.5	0.85	0.97
Time (X_2), min	18	30	60	90	102

Results and Discussion

The results of various properties of papain treated samples are given in Tables 2 and 3.

The spatial diagram of the response surface (Fig.1) shows a mixed effect of concentration and time on weight loss. A maximum weight loss of 0.80% is achieved at higher level of temperature (85^0C) with minimum treatment time (30 min). The weight loss which occurred could be due to the removal of sericin and some part of the fibroin. Treatment with non-ionic detergent does not result in any significant

Table 2 — Treatment conditions and observed responses

Expt. No.	Enzyme concentration, %	Time, min	Absorbency, s	Flexural rigidity, μNm		Crease recovery, deg	
				Warp	Weft	Warp	Weft
1	0.15	30	30	0.000136	0.000210	130	134
2	0.85	30	32	0.000517	0.000217	114	107
3	0.15	90	33	0.001567	0.000315	113	104
4	0.85	90	29	0.001462	0.00146	116	105
5	0.03	60	34	0.000751	0.000360	108	102
6	0.97	60	33	0.005994	0.000651	112	109
7	0.5	18	31	0.000437	0.000215	121	115
8	0.5	102	32	0.000901	0.000447	109	105
9	0.5	60	27	0.000383	0.000180	117	117
10	0.5	60	36	0.000557	0.000152	126	119
11	0.5	60	32	0.006814	0.000860	115	108
12	0.5	60	31	0.000144	0.000113	127	121
13	0.5	60	35	0.000513	0.000315	130	123
Control	-	-	20	0.001216	0.000443	113	104

Table 3 — Weight loss of enzyme treated samples

Expt. No.	Enzyme concentration, %	Time min	Weight loss, %
1	0.15	30	0.52
2	0.85	30	0.80
3	0.15	90	0.52
4	0.85	90	0.25
5	0.03	60	0.53
6	0.97	60	0.55
7	0.5	18	0.76
8	0.5	102	0.75
9	0.5	60	0.25
10	0.5	60	0.26
11	0.5	60	0.51
12	0.5	60	0.52
13	0.5	60	0.530
Control	-	-	NIL
Alkali treated	-	-	0.07

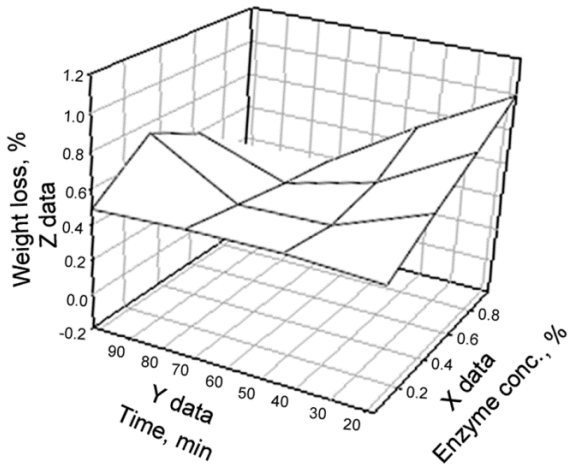

Fig. 1 — Spatial diagram of weight loss, enzyme concentration and time

weight loss. Thus, the enzyme could be removing some stubborn resin residue, sericin impurities, oligopeptides or even part of fibroin which are not removed easily by using conventional procedures such as treatment with non-ionic detergent.

Absorbency, flexural rigidity and crease recovery of the treated fabric samples show poor correlation with concentration of enzyme and time of treatment. Therefore, no best fitted model is obtained. Nevertheless it can be seen from data in Table 2 that there is no significant change in dependent variables of the treated and control fabrics. The enzyme treated fabric exhibits noticeable improvement in lustre. This is due to the cleaning of the surface of fibres by the action of enzyme.

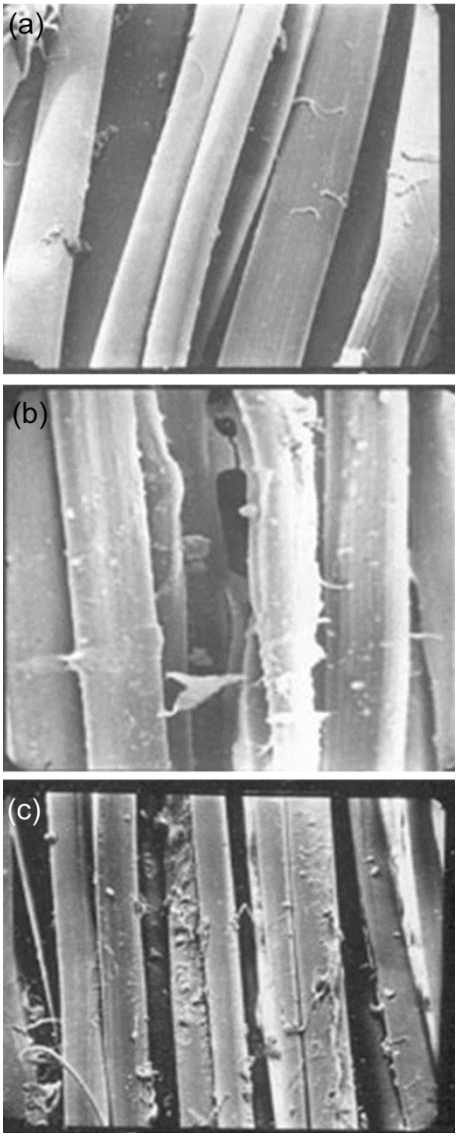

Fig. 2 — Scanning electron micrographs of (a) enzyme treated silk (b) detergent treated silk and (c) alkali treated silk (×860)

The scanning electron micrographs [Fig 2(a)] show that mulberry silk has some deposits on the surface, which are then completely removed on treatment with papain enzyme. The cleaning of the surface by the enzyme is responsible for the improvement in lustre of the treated samples. A comparison of the enzyme treated and detergent samples indicates that the enzyme treated fabric has a smoother and cleaner finish [Fig 2(a)], while the detergent treated fabric shows removal of sericin to a lesser extent [Fig 2(b)], which was, however, not perceptible as weight loss (Table 3). The alkali treated mulberry also shows incomplete removal of the sericin residue [Fig. 2(c)].

It has been reported that the treatment with stronger alkali causes weakening of the fabric[5].

The scanning electron micrographs of the enzyme-treated mulberry samples show the complete removal of the deposits on the surface of the fabric, which results in bio-polishing as indicated by the smooth and clean surface of the fabric. Hence, the treatment of the mulberry silk with proteolytic enzyme results in a completely clean fabric with a better bio-polished finish.

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