J. Environ. Nanotechnol.
Volume 2, No.3 pp. 10-16
ISSN (Print) : 2279-0748
ISSN (Online) : 2319-5541
doi : 10.13074/jent 2013.09.132024

Degumming of Silk using Papaya Skin

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Received: 08.07.2013 Revised : 18.08.2013 Accecpted: 13.09.2013

Abstract

Silk the exotic, elegant, beauty and luxury fabric has fascinated man for many decades. Degumming of silk is one of the most important studies in silk manufacturing process. In this study an attempt has been made to replace the traditional soap soda method with enzymes. The enzyme protease is being extracted from the skin of papaya which is a rich source of protease enzyme papain. The yarns are degummed with the enzyme extracted from the papaya skin. The performance of enzyme treated yarn has also been compared with the yarns degummed by the conventional method. The degummed yarns were further dyed using natural dye turmeric in the presence and absence of mordants. The degummed samples were subjected to subjective evaluation like colour, luster and hand, dyed yarns for general appearance, depth of colour, evenness in dyeing and luster. Objective evaluation like Weight loss, Tensile strength, Elongation, Degumming efficiency, Colour fastness to washing, rubbing and to light were also carried out.

Keywords: *Degumming; Dyeing; Enzyme; Protease; Silk.*

1. INTRODUCTION

Silk, the Queen of Textiles is a splendid gift of nature to the mankind known for its elegance, refinement, beauty and luxury. The story of silk is fascinating, romantic and adventurous too. In India no religious ritual is completed without the use of silk cloth. Textile is a vast field ever growing with the improvement in the field of science and technology. Though varieties of fabrics are available in the market, silk continues to be the queen of fabrics (Krishnaveni *et al*. 2007).

Natural silk is a continuous protein-filament spun by the silk worm (Ibrahim *et al*. 2007). The fibroin filaments of cocoon silk are naturally gummed with the protein sericin and also small amounts of non proteinaceous impurities like dust, minerals, pigments and waxy matter. Degumming is the process of cleavage of peptide bonds of sericin either by hydrolytic or enzymatic methods and its subsequent removal from silk fibroin (Trotman, 1984).

Enzymes are now being considered as alternative degumming agents for the processing of silk. The action of enzyme can be controlled to avoid strength loss but at the same time obtain a uniformly degummed silk (Gulrajani *et al*. 2000). The application of enzymes for silk degumming not only provides better control over the degumming process but also reduces the load on effluent. The proteolytic enzymes are soft on fibre and they hydrolyze the peptide bonds formed by amino acids (Gokarneshan, 2003). Protease is a class of enzymes that converts the complex proteins into simple

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proteins, amino acids and other non protein parts by catalyzing the hydrolysis processes. A number of such enzymes have been found in animals, plants and microorganisms. Proteases represent one of the three largest groups of industrial enzymes and find application in detergents, leather industry, food industry, pharmaceutical industry and bioremediation processes (Gupta, 2002). Papain enzyme is a protein with papain proteinase, chymopapain and lysozyme. Enzymes accelerate reactions within body cells. The latex of the papaya plant and its green fruits contains two proteolytic enzymes, papain and chymopapain.

The biggest damage to the environment is supposed to be done by the dyeing units (Amsamani, 2007). The term 'Natural dye' refers to the dyes obtained from insects, plants and mineral substances, and used for dyeing to textile material. The main natural dye substances used in India have been extracted from the roots, barks, flowers and fruits of various dye producing plants (Mondal *et al*. 2004).

2.METHODOLOGY

Silk is prized for its lustre, sheen and hand. The popularity of this "Queen of Fibres" is mainly because of the finishing it is subjected to denotes. Mulberry silk the popular variety of silk was taken for the study.

2.1. Selection of source and extraction

The waste skin of different fruits like pineapple, apple, guava, orange and papaya were taken for the study. The refrigerated fruit skin was taken and kept in room temperature before proceeding to the process. 15 gms of each of the fruit skins were crushed using a cleaned and dried motor and pestle with 100 ml of phosphate buffer. The obtained solution was filtered. Inorder to obtain a clear solution it was further centrifuged using Rotar 4r plasto craft and the supernatant served as the enzyme source. The protease activity was determined by the method of Taraporewala and (Shah, 1996). The protease activity was found in all the samples and also the degumming efficiency was found out by calculating the weight loss after degumming. Based on the results the best source was obtained.

2.2. Optimization for Degumming

Inorder to obtain the best results for the silk yarn degumming various physical parameters which play an important role in degumming were optimized.

Table 1: Optimising Parameters

Parameters	Variants	Optimum variant
Extraction method	Hard surface, Mixie, Motor& pestle	Motor & pestle
Incubation time	3,6,9,12 and 24	6
Source concentration	5, 10, 15, 20 and 25 g	15
pH	3, 4, 5, 6, 7, 8 and 9	6
Temperature	normal, 37°C, 45°C and 90°C	45

2.2.1. Large scale production of protease

The 600 g of papaya skin was weighed using a Shimadzu electronic weighing balance and collected in a clean vessel. The skin was crushed in a motor and pestle using 1000 ml 0.1 m phosphate buffer. The pH of the extract was adjusted to 6.0 using 0.1N HCl or 0.1N NaOH. The extract was filtered and centrifuged at 4 °C at 5000 rpm using Rota 4R (Plastocraft) refrigerated centrifuge and the supernatant served as the enzyme source.

2.3. Degumming using Papaya Skin

Based on the standardised procedure, 35.15 g of silk yarn skin was degummed by the process as follows. The enzyme was taken in a clean beaker and into it 35.15 g of raw silk yarn washed with cold water and immersed. The beaker was placed in a hot air oven and the temperature was maintained at 45 °C for duration of 6 hours. After the time duration the degummed silk

yarn was removed and washed thoroughly with hot and cold water. The yarn was further dried and the weight noted.

2.4. Conventional Degumming of Silk

Degumming of silk involves the cleavage of peptide bonds of sericin. The soap which is used for degumming purpose has to be neutral, readily soluble in water and free from any colouring matters (Sankar, 2004). The conventional degumming was carried out by the method of (Anandan *et al.* 2006) taking a clean stainless steel vessel of 100 ml capacity and to it Marseille's soap 5 gpl, soda 0.5 gpl and H_2O_2 of about 20 cc was added. The liquor ratio was maintained as 1:20. 35 g of silk yarn washed with soft water was immersed into the solution. The above solution was boiled for a period of 90 mins and the temperature was adjusted to 80 - 85 °C using a water bath. After 90 mins the yarn was removed and washed with hot and cold water and then the yarn was dried.

2.5. Degumming using Commercial Enzyme

The commercial enzyme used for degumming of silk yarn was bacterial enzyme Anilozyme P marketed by Anil products limited, Ahmedabad. A pilot study was conducted for silk yarn degumming followed by bulk production. Enzyme bath was prepared by adding the following ingredients in g/l – 2 g Anilozyme-P, 0.5 g sodium bicarbonate, 1 g non-ionic detergent with M :L ratio 1:20. 35 g of silk yarn skein washed with soft water was immersed into the prepared enzyme bath. The temperature was maintained at 55 °C to 60 °C using incubator for a period of 100 mins. The degummed silk yarn was removed and followed by washing, rinsing and drying.

2.6. Dyeing of the Yarn

Colour brings to the fore front why silk is labeled as the queen of textile fibres. Dyeing brings out the special quality of this fibre into full bloom while at the same time offering flexibility to fashion designers (Iyer, 2005). Dyeing with natural herbs is an ancient art kept alive by some very persistent folk. Natural dyes exhibit lower toxicity and allergic reaction than the synthetic dye (Padhyay *et al*. 1997). The dye selected for the study was turmeric. The turmeric was used to obtain brilliant yellow shades and also turmeric has an antimicrobial property.

2.6.1. Extraction of natural dye

Dye from natural sources can be extracted in alkaline, acidic and aqueous medium. As aqueous medium does not involve the use of harmful chemicals, to extract the dye, aqueous medium was selected for the study. 100 g of the material was taken in a beaker containing 2 litre of water and boiled for 1 hour. The solution was allowed to stand for sometimes until cooled and then filtered.

2.6.2. Optimization of dyeing variable

Dye materials were entered and heated at 90-100 °C. Dye solutions were filtered after it gets cooled; the extract was used for dyeing. The dyeing material to liquor ratio was finalized as 1:50 for dyeing and the dyeing time was selected as 45 minutes. To optimize the dye concentration the silk samples were dyed in 2%, and 5%.

2.6.3. Dyeing of the silk yarn in the absence of mordants

The degummed silk yarn was dyed with extracted dye solutions keeping the material to liquor ratio of 1:50 in absence of mordants. The dyeing was carried out at 100 °C for 45 minutes in an open dye bath. After dyeing the dyed material was washed and finally dried.

2.6.4. Dyeing of the silk yarn in the presence of mordants

Natural dyes may require some mordanting agent's inorder to produce affinity between the fibre

Table 2: Visual Evaluation of Degummed Silk yarn

Sample name	General appearance			Whiteness			Texture			Lustre		
	Rating in %											
	G	F	P	B	M	D	S	M	R	H	M	L
CS	56	44	0	40	56	4	88	12	0	72	28	0
OS	32	52	16	44	56	0	32	60	8	36	52	12
ES	76	24	0	96	4	0	84	16	0	72	28	0

G-Good, F-Fair, P-Poor, B-Bright, M-Medium, D- Dull, S-Smooth, M-Medium, R-Rough, H-High, M-Medium and L-Low. CS-Conventional sample, OS- Commerical Sample, ES-Enzyme treated sample.

Table 3: Visual Evaluation of Dyed Silk Yarn

Sample name	General appearance			Evenness in dyeing			Depth of colour			Lustre		
	Rating in %											
	G	F	P	E	F	U	H	M	L	H	M	L
CSM1	56	44	-	76	24	-	56	44	-	72	16	-
OSM1	-	48	52	44	56	-	28	36	36	-	40	60
ESM1	52	32	16	64	36	-	48	52	-	40	60	-
CSM2	64	36	-	96	4	-	84	16	-	84	16	-
OSM2	20	64	16	-	68	32	44	36	20	12	68	20
ESM2	60	40	-	96	4	-	64	36	-	72	20	8
CSC1	48	40	12	80	20	-	48	48	4	76	24	-
OSC1	48	32	24	24	76	-	36	36	28	28	56	8
ESC1	60	40	-	76	24	-	52	48	-	84	16	-
CSC2	48	40	12	80	20	-	76	24	-	92	8	-
OSC2	48	32	24	24	76	-	36	56	8	24	76	-
ESC2	60	40	-	88	12	-	80	20	-	76	24	-

G-Good, E-Even, H-High-Fair, M-Medium, P-Poor, U-Uneven, L-Low, CSM -Dyeing of Conventional sample with mordant, OSM- Dyeing of Commercial sample with mordant, ESM- Dyeing of Enzyme treated sample with mordant, CSC- Dyeing of Conventional sample without mordant, OSC- Dyeing of Commercial sample without mordant, ESC- Dyeing of Enzyme treated sample without mordant.

and the dye. The degummed silk yarn was simultaneously mordanted with 2% alum and dyed with extracted dye solutions keeping the material to liquor ratio of 1:50. The dyeing was carried out at 100 °C for 45 minutes in an open dye bath. After dyeing the dyed material was washed and finally dried.

3. RESULTS & DISCUSSION

Silk degumming is the heart of the chemical processing. Enzymes were extracted from plant sources and with these enzymes the silk was degummed and the results attained are being discussed below.

3.1. Evaluation of Degummed Silk Yarn

3.1.1. Subjective Evaluation

The traditional method of evaluating silk fabrics is the subjective assessment of fabric handle by experienced people. The subjective evaluation of silk yarn was done through visual inspection and the findings are given in the Table 2. From the Table 2 the results conclude that ES was rated to be good by 76% of the judges for the general appearance, followed by CS and OS of 56 and 32% respectively. While looking on to the overall rating of the judges ES were given the maximum rating followed by CS and finally OS which lacked in all the four aspects of visual evaluation

From the table 3. the results conclude that the samples CSM2, ESM2, ESC1 and ESC2 were rated high. Colour depth CSM2, ESM2 and CSC2 were rated high by 84, 80 and 76 percent of the judges. With regard to lustre CSC2, ESC1 and CSM2 were rated high by 92 and 84 percent of the judges

3.1.2. Objective Evaluation

3.1.2.1. Weight loss

The weight loss in fabrics on enzyme treatment is attributed to the hydrolytic removal of silk gum, sericin. The weight of the sample was weighed before and after enzyme treatment and the weight loss calculated and tabulated in Table 4

Table 4: Weight Loss of the Silk Yarn

S.no	Sample name	Weight before treatment (grams)	Weight after treatment (grams)	Weight loss (grams)	Weight loss % ($[(W_1 - W_2)/ W_1] \times 100$)
1.	CS	35.15	24.61	10.54	29.93
2.	OS	35.15	30.79	4.36	12.40
3.	ES	35.15	27.12	8.03	22.84

CS-Conventional sample, OS-Commerical Sample, ES-Enzyme treated sample.

From the table it is evident that when comparing the weight of the yarns before and after treatment the weight loss% is more in CS and ES. While the weight loss in OS is relatively low, indicating inefficient degumming process.

3.1.2.2. Degumming efficiency

The degumming efficiency between soap and enzyme treated samples were found out and tabulated in Table 4. Degumming efficiency of the enzymes was calculated according to (Gulrajani *et al*. 2000) using the formula:

Degumming efficiency = (% Wt. loss by enzyme treatment/ % Wt.loss by soap treatment) x 100

Table 5: Degumming Efficiency of Silk Yarn

S. no	Sample name	Weight loss over original	Weight loss % over original	Degumming efficiency
1.	CS	10.54	29.93	-
2.	OS	4.36	12.40	41.43
3.	ES	8.03	22.84	76.31

CS-Conventional sample, OS-Commerical Sample, ES-Enzyme treated sample.

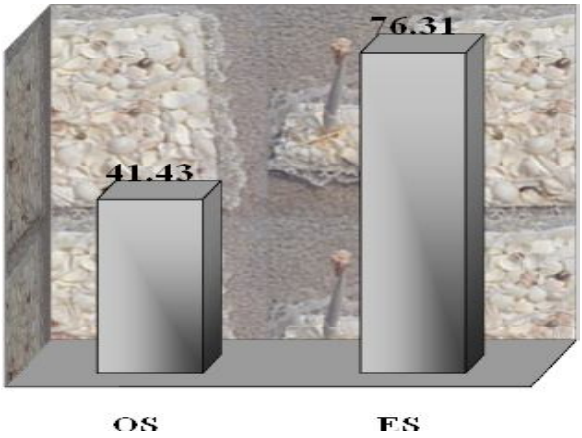

Fig. 1: Degumming Efficiency

From the Table and Figure it is obvious that when comparing the degumming efficiency of OS with CS and ES with CS, ES yielded the maximum result indicating efficient degumming.

3.1.2.3. Tensile strength & Elongation

3.1.2.3.1. Yarn Strength

The yarn strength and yarn elongation of the original and treated samples are shown in Table 6.

Table 6: Yarn Strength & Elongation of Degummed Silk Yarn

S. no	Sample name	Yarn strength In (Kg)	Yarn Elongation In (cms)	Loss or gain over Original (Yarn strength)		Loss or gain over Original (Elongation)	
		Mean	Mean	%	Actual	%	Actual
1	SS	1.515	3.420	-	-		
2	CS	0.560	1.850	1.57	45.9%	0.955	63%
3	OS	1.384	3.610	-0.19	-5.5%	0.131	8.6%
4	ES	1.305	2.520	0.9	26.3%	0.21	13.8%

SS-Standard Sample CS-Conventional sample, OS-Commerical Sample, ES-Enzyme treated sample.

Table 6. features a minimum the samples OS and SS where as a maximum difference between SS and CS. From the results it could be said that the treatment has reduced the strength. The strength was minimum in conventional sample and maximum in commercial method. The reduced strength showed that the enzyme has acted well in apaya enzyme treated than in commercial method.

3.2. Evaluation of Dyed yarn

The dyed yarns were evaluated for various colour fastness test like washing, rubbing and to sunlight, of all the samples evaluated conventional treated samples and enzymatic treated were rated from excellent to good. The sample dyed with mordant gave better results than the samples without mordant.

Table 7: Yarn Strength and Elongation of Dyed yarn

S no	Sample name	Yarn strength In (Kg)	Loss or gain over Original (Yarn strength)		Yarn Elongation In (cms)	Loss or gain over Original (Elongation)	
		Mean	%	Actual	Mean	%	Actual
1	SS	1.1515	-	-	3.420	-	-
2	CS	0.448	61.6%	0.7035	3.390	-0.87%	-0.03
3	OS	1.695	-35.9%	-0.544	4.350	-24.2%	-0.83
4	ES	1.710	-83.3%	-1.262	4.280	-25.1%	-0.86

SS-Standard Sample CS-Conventional sample, OS-Commerical Sample, ES-Enzyme treated sample.

4. CONCLUSION

There is a growing trend towards the application of highly specialized biocatalysts i.e., enzymes, for the chemical processing as well as for physio-chemical modifications of textile fibers. Enzymes being natural products and are completely bio-degradable and accomplish their work quietly and efficiently without leaving pollutants behind, so the enzyme concentration can be successfully used for carrying out the degumming of mulberry silk yarn.

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