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Development of Eco-Friendly Method for Degumming of Eri Cocoons

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Article

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ABSTRACT

In this study, a new eco-friendly degumming technique was developed for Eri cocoons without any chemicals. A newly developed eco-Eri degumming process was tried at 110 °C, 120 °C, 130 °C and 140 °C temperatures for 10, 20 and 30 min with soft and hard water. The novel eco-friendly degumming method was compared with the existing method of degumming viz. Soap-soda along with the enzymatic method. After degumming, weight loss per cent, changes in the fibre surface morphology concerning SEM imaging and fibre properties concerning tensile strength and elongation were studied. The eco-Eri degumming method at 130 °C for 30 min using soft water removed the maximum sericin content (degumming loss 15.7%) from Eri cocoon shells and had optimum degumming efficiency. In the eco-Eri degumming method, the degummed Eri cocoons were uniformly softened. The soap soda method of degumming resulted in 14.5% weight loss whereas enzymatic degumming resulted in a maximum of 13% weight loss (12% dosage). Enzymatic degumming significantly reduced the tensile strength but no results found in the tensile properties of Eri silk fibres were observed in new eco-friendly and soap-soda methods. SEM images indicated optimal fibre surface with the smoother surface until 130 °C, but fibre damage and fibrillation occurred at 140 °C and also with the Soap-soda method. An optimal condition for degumming Eri cocoons with the new eco-Eri degumming process is 130 °C for 30 min using soft water. The eco-Eri degumming method is better compared to other methods used presently in the industry.

KEYWORDS

degumming, Eri cocoons enzyme, chemical-free device, soap-soda, Eri silk

INTRODUCTION

Eri culture is a rural occupation in India. Eri silkworm rearing, Eri silk yarn spinning and weaving activities are passed on from one generation to another in Eri-culture clusters of India. It is carried out traditionally during leisure hours of villagers using mainly the leaves of castor plants. Eri silk has gained a great deal of popularity among the average and below-average-income population across the Northeast Regions (India). India produces four varieties of silks i.e. Mulberry, Eri, Tasar and Muga. The second largest variety is Eri silk being produced. About 80% of the total non-mulberry (Vanya) silk contributes to the production and about 21% of the total raw silk production of India for the year 2021-2022 [1].

The Eri cocoons are open-mouthed at one end and due to the discontinuity of filament length, these cannot be utilized for yarn production through the reeling process [2]. Eri silk cocoons can only be spun like cotton fibres by the textile spinning process [3]. The Eri spun yarn is also called “Poor man’s silk”. It acquires a unique dual characteristic of having the softness of silk and the warmth of wool [4].

Two silk fibres (brin) extruded through the spinneret of the Eri silkworm (*Philosamia ricini*) after joining by sericin gum to form a single fibrous strand (bave) during cocoon spinning. The presence of sericin over fibre makes it hard to open for further processing [5]. Before spinning, the degumming process has to be carried out on the Eri cocoon shells to remove the sericin gum from the silk. During the degumming process, the splitting of peptide bonds of sericin occurs either by enzymatic or hydrolytic technique and subsequently, the sericin removal from the silk fibre is damaged. Enzymatic, alkaline and acidic degumming methods have been reported as different options to remove sericin from Eri silk. A chemical reaction in which a compound reacts with water, causing the decomposition of sericin can be carried out under neutral, alkaline conditions to obtain four tiny grades of sericin each having different properties [6,7]. Reports on silk fibres degummed with enzymes and soap and soda solutions were dipped in an acetic acid solution [8]. The process parameters for the silk fibres for degumming with enzymes were also optimized. Swelling of fibres during soap and soda solution was reported to assist effective degumming compared to the enzymatic method. The alkaline soap-soda method employs degumming of Eri silk in a solution of soap (neutral/non-ionic) in the presence of mild alkali such as sodium carbonate and is predominately used in the silk industry. This method of sericin hydrolysis is not only harsh on silk, but also it is not easy to control [9].

The present research study is an attempt to develop an eco-friendly degumming process for Eri cocoons without using any chemicals through optimized process parameters by utilization of appropriate equipment in comparison with soap-soda and enzymatic techniques.

EXPERIMENTAL

Materials and Methods

White Eri cocoons (dry) were procured from the Eri silkworm seed production centre, in Azara, Guwahati, India. A total of 35 kg (single lot) of Eri cocoon shells were procured for the study. About 1 kg of Eri cocoons (400 no.s) were tested for the cocoon characteristics, the characteristics of Eri cocoons are given in Table 1. The dried Eri shells have a long shelf life and no quality deterioration.

Table 1. Characteristics of Eri cocoon

Particulars	Average value	CV%
Cocoon Shell Length, mm	61	2.6
Cocoon Shell Diameter, mm	24	3.1
Cocoon weight, gm	2.6	1.2
Shell weight, gm	0.42	1.9
Shell Ratio,%	16.2	2.6

The water used in this study to degum Eri cocoon shells was tested for both hardness and softness as per the standard method [10,11]. The groundwater (bore-well) collected was hard with 6.8 pH, total hardness of 425 ppm (parts per million) and alkalinity of 360 ppm whereas the soft water (water supply from corporation/municipality) was neutral with pH 7.0, total hardness of 60 ppm and alkalinity of 62 ppm, respectively.

Degumming of Eri cocoons

Eri cocoon degumming can be carried-out using soap-soda chemicals at boiling temperature for 90 minute duration. The degumming process for Eri silk to be used for spun silk was developed by CSTRI, Central Silk Board, Bengaluru, and this method was found to be most effective for degumming Eri cocoons, which is now commonly practiced in the industry [12,13]. It is reported that sericin content in raw Eri silk is in the range of 13-17%. The weight loss % of cocoons during degumming in mulberry silk is 25-30% [12] and for muga, tasar and Eri silk ranges between 8-15% [14,15,16,17,18]

Many literatures have reported studies of enzymatic methods of degumming silk. One of the literatures cites different methods of Eri silk (cocoon) degumming using enzymes to find the efficacy of degumming [19].

Three methods of degumming Eri silk cocoons have been attempted in this study, namely, soap-soda, enzymatic and eco-friendly Eri cocoon degumming. The soap-soda degumming is the conventional method trailed in Ericulture, and this study of degumming by soap-soda method has been marked as a control sample for comparison purposes [20,21] The efficiency of eco-friendly degumming with only water using Eco Eri cocoon degumming device with various combinations has been attempted.

Soap-soda method

In this experiment, 200 g of Eri cocoon shells were kept with porous polyester net (bolting) fabric, thereafter Eri cocoon shell bundle was immersed in a degumming bath in a vessel comprising 10 g/L, neutral soap, 5.0 g/L, soda and treated at boiling point for 90 min. The material-to-water liquor ratio was 1:40. Degummed Eri cocoons were washed thoroughly to remove excess water through squeezing. Post degumming process, the Eri cocoon shell bundle was again hot-washed for five minutes

in simple water to reduce the content of alkaline in the material. Further, the degummed Eri silk fibres were washed twice in plain water, squeezed and dried. A total of five degumming trials were taken. This soap-soda method was considered as a control for comparison purposes in this study.

Enzymatic method

200 grams of Eri cocoon shells were taken for individual study and kept in a porous polyester net (bolting) fabric. The enzymatic degumming using Protease enzyme (serine protease) was carried out in two stages by dipping the cocoon in the vessel containing the ingredients mentioned in Table 2. In the first step, the cocoon shells were pre-treated with 1gpl wetting agent (turkey red oil) at 40 °C for 30 min. In the 2nd step, the different dosages of protease enzymes (with concentrations of 6%, 8%, 10% and 12% to study the effect) were added along with a small quantity of sodium carbonate solution to maintain pH of 8.5 – 9 and the degumming process was carried-out at 55-60 °C for 50 min. After the enzymatic degumming process, the temperature was raised to boiling point for 10 min to denature the enzyme, and lastly, the degummed material was washed with water and dried. A total of five enzymatic degumming trials were taken.

Table 2. Enzymatic degumming Recipe

Particulars	Enzyme dosage for degumming			
	6%	8%	10%	12%
Material-to-liquor ratio	1:40	1:40	1:40	1:40
1st bath				
Turkey Red Oil, ml l^{-1}	1	1	1	1
Temperature, °C	40	40	40	40
Time (min)	30	30	30	30
2nd bath				
Protease enzyme, ml	3.85	6.42	8.02	9.65
Soda, g/l	1	1	1	1
Temperature °C	55-60	55-60	55-60	55-60
pH	8.5 -9.5	8.5 -9.5	8.5 -9.5	8.5 -9.5
Time, minute	60	60	60	60

New Eco-friendly method

Eco-degumming equipment has been designed and developed by CSTRI for degumming Eri cocoons with optimized degumming methods without any chemicals. The degumming equipment was used to

degum the Eri cocoon shells. In this degumming device, only plain water is used at high temperatures and pressure for degumming Eri cocoons [22,23]. The equipment in Figure 1 has a control unit to maintain the water temperature and pressure as desired.
The duration of degumming is controlled manually.

Figure 1. Eco Eri degumming equipment

Degumming of Eri cocoons by Eco-friendly method

Eri cocoons were degummed at four temperatures, i.e.,110 °C, 120 °C, 130 °C and 140 °C for 10, 20 and 30-minute durations using hard and soft water. For each experiment, 1kg of Eri cocoon shell was used for degumming. The Eri cocoon shells were filled in cages and introduced in the eco-friendly degumming (Eco Eri degumming) machine at 40 °C, and then the door was closed. The temperature was raised slowly to the desired temperature. After treatment for the desired duration, the material was soaked in hot water and cold-washed in water. It was observed that 12 PSI pressure was developed at 110 °C, 18 PSI at 120 °C, 28 PSI at 130 °C, and 38 PSI at 140 °C, irrespective of treatment time.

Testing methods

Degum loss

Based on the initial weight of cocoon shells, the degumming loss % was calculated by the formula.1:

$$\text{Degumming Loss, \%} = \{(W1 - W2) / W1\} \times 100 \ (1) \qquad (1)$$

Where W1 is the initial weight of the material and W2 is the weight of the material after degumming.

Tensile strength

Eri silk fibres before and after the degumming process were subjected to a fibre bundle test as per the Indian standard IS 3675 using steel-meter equipment at zero-gauge length.

Fibre surface morphology

The surface morphology of Eri silk fibres was studied after the degumming process to understand the effectiveness of the degumming process and also to assess the damage caused to the fibres in case of excessive degumming. Scanning Electron Microscopy (SEM) images were obtained for the without degummed (raw) and degummed Eri silk fibres to understand fibre surface morphology. The morphologies of the degummed Eri fibres were examined with a Hitachi SU3500 N scanning electron microscopy (SEM) at 15 kV of acceleration voltage. Before placing the samples in the SEM chamber, the samples were mounted onto an aluminum stud and sputter-coated with gold/palladium for 180 s (E-1010 ION SPUTTER, Hitachi, Japan) to prevent charging. The SEM images at 15 KV 9.1 mm x 1.00 k 50.0 μm were used.

RESULTS AND DISCUSSION

The comprehensive results and discussion are elucidated for degumming of Eri cocoons with conventional soap-soda, enzymatic and eco-Eri cocoon degumming methods regarding degumming and other properties of Eri degummed fibres.

Degumming loss

Soap and soda method

Table 3. Shows the results of the degumming loss of soap and soda Eri degummed cocoons.

Table 3. Degumming loss of soap and soda

Method	Avg. Degumming loss (%)	CV (%)
Soap and Soda	13.50	3.7

The degumming weight loss of 13.5% with a minimum of 12.50 to a maximum of 14.50% observed in this study by soap-soda method agrees with the range of degumming loss observed for Eri silk reported in previous studies [17]. The report observes that degumming loss of Eri silk shells with more than 10% for the white variety, 13% for the brick red variety no significant deterioration of tensile characteristics of single fibre.

Enzymatic Eri cocoon degumming

Table 4 shows the results of enzymatic degumming trials in terms of average degumming loss.

Table 4. Degumming loss of enzymatic degumming method

Enzyme Concentration (%)	Degumming loss (%)	CV (%)
6	10.5	3.9
8	10.5	4.1
10	12.7	0.9
12	13.5	0.8

Table 4a. Single-factor ANOVA for Enzymatic degumming

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	36.02212	3	12.007	114.99	4.8759E-11	3.239
Within Groups	1.67076	16	0.104			
Total	37.69288	19				

The results presented in Table 4 and 4a reveals that degumming loss increased in a dose-dependent manner and at 12% enzyme concentration a marginal degumming loss per cent was noted as compared to the soap and soda method. Less CV% indicates good experimental uniformity. The degumming weight loss was comparatively lower, which is in line with the reports of [18] who inferred that the enzymatic degumming method using protease enzyme is not very effective for Eri cocoons. Although the higher dosage of the enzyme resulted from higher degumming weight loss (12.7% loss for 10% and 13.5% loss for 12% dosage)and uniform degumming loss (with low CV%), a higher dosage of the enzyme has also resulted in higher weight loss which is not acceptable. This has been reasoned that whenever higher enzyme dosage or process parameters are changed to achieve optimal degumming in Eri silk, it results in the deterioration of fibre tensile properties due to the damaging action of enzymes on the fibre surface [19]. The results in Table 4a Single factor ANOVA for Enzymatic degumming show that higher enzyme concentration has a significant effect on loss of degumming.

Degumming loss of eco-friendly degumming

The Eri silk degumming loss of Eri cocoons using hard and soft water at different temperatures and durations is given in Table 5.

Table 5.Degumming loss per cent of Eco-Eri cocoon degumming

Temp, °C	110 °C			120 °C			130 °C			140 °C		
Time, minute	10	20	30	10	20	30	10	20	30	10	20	30
Hard water	10.2	11.5	12.2	9.6	10.8	11.7	9.9	10.4	12.7	9.3	12.4	13.6
Coefficient of variation (%)	2.8	1.4	1.4	3.7	5.0	2.8	5.6	6.0	1.6	1.4	2.5	1.7
Soft water	10.0	10.6	12.3	10.3	11.2	11.7	11.4	13.6	15.6	12.3	14.5	18.7
Coefficient of variation (%)	2.2	2.4	3.1	6.5	2.1	2.7	6.2	1.9	1.7	2.8	1.4	1.1

Water has an important role in textile wet processing also it has a manner on the processing of cost. The water quality affects the different processing operations. The effect of soft and hard water on textile material during processing has been investigated [25]. The soft water hardness (total dissolved solids-TDS) of less than 50 ppm is preferred for textile processing as per [26,27], but many of the silk processing sectors have natural / sourced water that has TDS above 200 (hard water). The hard water can create many problems during wet processing (degumming/desizing to finishing [28]. Hard water interferes with catalysts, causes resins and other additives to become non-reactive, breaks emulsion and deactivates soap [24].

Sericin gum in silk starts to dissolve in water at 80 °C and above. In this study, under high temperature and high-pressure systems, degumming was carried out on Eri cocoon shells for 10, 20 and 30 minutes. Higher temperature and pressure result in higher sericin removal. At enhanced treatment time, sericin swelling is more and thus results in further dissolution and effective removal. Soft water enhances the sericin dissolution while hard water retards sericin dissolution in water. Hence the results observed in this study are in tune with the general sericin dissolution characteristics with respect to temperature, water hardness and treatment duration.

Table. 5a Two-factor ANOVA for hard water degumming loss,%

Source of Variation	SS	df	MS	F	P-value	F crit
Sample	8.912591667	3	2.970864	7.232544	0.000423	2.798061
Columns	80.44761333	2	40.22381	97.92453	1.15E-16	3.190727
Interaction	14.50585333	6	2.417642	5.88573	0.000112	2.294601
Within	19.71664	48	0.410763			
Total	123.5826983	59				

Table. 5a Two-factor ANOVA p-value was 0.00 for temperature, 1.15E-16 for the duration and 0.00012 for the interaction of temperature and duration, respectively. The degumming loss per cent was maximum at 140 °C and 30 minutes. It is clear that degumming with hard water at temperatures

ranging from 110 °C to 140 °C in different durations of treatment, the degumming loss of 13.6 % is less as compared to the control (Soap-soda) sample having a degumming loss of 14.5%.

Table 5b Two-factor ANOVA for soft water degumming loss,%

Source of Variation	SS	df	MS	F	P-value	F crit
Sample	166.2916	3	55.43053	186.6599	1.86E-26	2.798061
Columns	138.263	2	69.13151	232.7974	1.97E-25	3.190727
Interaction	55.51714	6	9.252856	31.15859	5.87E-15	2.294601
Within	14.25408	48	0.29696			
Total	374.3258	59				

The Eri cocoon degumming loss percent with soft water as a function of temperature and treatment duration was compared with hard water, but the sericin removal was high in soft water than hard water. ANOVA Two-factor is indicated that together temperature and duration had considerable effects on degumming loss. Also, the temperature-period interface does affect of percentage of degumming. The Table 5b Two-factor ANOVA p-value was 1.86E-26 for temperature, 5.87E-15 for duration and 5.87E for interaction of temperature and duration, respectively. The degumming loss was maximum at 130 °C and 30 minutes. It is clear that degumming with soft water at temperatures ranging from 110 °C to 130 °C in different durations of treatment, the degumming loss was 15.6% is more as compared to the control (Soap-soda) sample having a degumming loss of 14.5%.

The degumming loss using soft water at 140 °C and 30 min was 18.9%, which is beyond the range specified by researchers [29,30]. Degumming loss using the new eco-friendly process with soft water was higher than the control soap-soda method, enzyme method and hard water treatment even at 130 °C and 30 min duration. Low CV indicates good experimental consistency.

Tensile strength and elongation

Soap-soda method

Table 6 shows the tenacity and elongation of soap-soda degummed Eri silk fibre. Undegummed silk showed 24.1 g per tex tenacity, while the control sample (soap-soda) showed 18.0 g per tex tenacity. Similarly, undegummed silk showed a higher elongation percentage (17.8%) as compared to the soap-soda sample (16.4%). The raw (undegummed) Eri fibres showed better tenacity and elongation as compared to the soap and soda technique due to the presence of sericin gum content which supports the fibre.

Table 6. Eri fibre tenacity (grams per tex) and elongation per cent

Characteristics	Undegummed	Soap-soda
Tenacity	24.21 ±0.62	17.81 ± 1.52
Elongation	17.67 ±1.64	16.42± 1.63

Enzymatic method

The enzyme degumming on Eri silk results in Table 7 show that it has a significant difference in tenacity and elongation. The tenacity was higher than the Soap soda method at 6% enzyme dosage due to incomplete degumming. Breaking elongation was also observed to be higher with an increase in enzyme concentration and dropped at 12% concentration.

Table 7. Enzymatic degummed Eri silk fibre tenacity and elongation

Characteristic	6%	8%	10%	12%
Tenacity	20.90 ± 2.11	16.12 ± 1.03	15.51 ± 0.82	14.14 ± 0.61
Elongation	21.22 ± 0.43	22.60 ± 2.23	20.36 ± 0.97	16.03 ± 1.05

Table 8. Single-factor ANOVA for Tenacity g/tex

Source of Variation (Groups)	SS	Df	MS	F	P-Value	F crit
Between	252.7726	3	84.25418	25.40043	5.27E-08	2.866254
Within	119.3205	36	3.306124			
Total	372.153	39				

Single factor (Enzyme concentration) Anova p-value is 5.27-8 for tenacity and 1.9E-11 for breaking elongation.

Table 9. Single-factor ANOVA for Elongation per cent

Source of Variation (Groups)	SS	Df	MS	F	P-Value	F crit
Between	402.374	3	134.1473	39.3076	1.9E-11	2.85765
Within	122.9	36	3.4012789			
Total	525.275	39				

The ANOVA results are shown in Tables 8 and 9, in which the Eri silk degummed with enzymes, had a significant effect on tenacity and elongation. Tenacity was more than that of the soap and soda method at 6% and 10% enzyme concentration. Breaking elongation was higher with an increase in enzyme concentration and dropped at 12% concentration. Single-factor (Enzyme concentration) ANOVA p-value was 5.27E-08 for tenacity and 1.9E-11 for breaking elongation. There is a direct relation between degumming loss and tenacity. The higher the degumming loss, the lower the tenacity observed in the enzymatic degummed Eri silk fibres.

Eco-friendly degumming method

Table 10.Fibre tenacity and Elongation

	Tenacity (g per tex)			Breaking Elongation (%)		
Duration→ Temp.↓	10 min	20 min	30 min	10 min	20 min	30 min
Control (Soap-soda)	18.0±1.65			16.2±1.65		
110°C	19.10±1.80	21.78±1.84	19.51±1.92	14.70±1.61	15.04±1.43	14.28±1.68
120°C	23.21±1.92	17.0±0.64	15.01±1.49	16.2±1.29	16.15±1.40	16.01±1.65
130°C	17.40±1.23	12.8±1.21	18.59±1.63	16.4±1.21	17.29±1.37	15.45±1.54
140°C	16.12±0.89	19.0±1.43	14.91±2.15	17.1±1.49	17.39±1.10	15.18±0.91
Two-factor ANOVA p-value	For Temperature 5.22E-10, Duration 0.00033 and Interaction between Temperature and Duration 5.34E-16			For Temperature 0.0028, Duration 0.0269 and Interaction between Temperature and Duration 0.6912		

As shown in Tables 11 and 11a, the ANOVA values indicated that both temperature and duration have a significant effect on tenacity and breaking elongation. Also, temperature and duration interaction do affect. The perusal of data in Table 10 revealed that generally at higher temperature tenacity is reduced. The breaking elongation increases with temperature and time from 110 °C to 120 °C and a duration of 10 and 20 minutes, while at 130 °C and 30 minutes, it falls. The tenacity of the control sample showed higher fibre tenacity compared to hard water-degummed Eri silk fibres.

Table 11.Fibre tenacity ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Sample	282.100	3	94.057	19.0784	5.22E-10	2.6787
Columns	85.180	2	42.489	8.5470	0.00033	3.0604
Interaction	610.602	6	101.579	20.6122	5.34E-16	2.1737
Within	531.927	108	4.915			
Total	1509.81	119				

Table 11a.Fibre Elongation (%) ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Sample	61.6	3	20.55657	4.977178	0.002802	2.688691
Columns	30.70667	2	15.40733	3.676192	0.026881	3.080387
Interaction	16.05	6	2.665	0.65763	0.691091	2.183657

Within	445.3	108	4.114074
Total	553.9467	119	

Table 12. Fibre tenacity and Elongation per cent of soft water degummed Eri silk

Duration → Temp.↓	Tenacity (g per tex)			Breaking Elongation %		
	10 min	20 min	30 min	10 min	20 min	30 min
Control (Soap-soda)		18.0 ±1.6			16.2 ±1.7	
110°C	23.12±1.73	22.59±1.61	22.31±2.48	16.47±1.14	18.44±1.23	16.46±1.04
120°C	21.7±1.25	18.21±0.64	18.56±1.79	12.63±0.62	11.94±1.15	16.45±1.30
130°C	19.20±1.34	18.89±1.77	18.38±2.90	14.01±1.13	16.03±0.79	17.21±0.62
140°C	17.31±1.48	16.3±2.10	11.80±0.63	14.64±1.33	17.11±1.19	15.63±0.67
Two-factor ANOVA p-value	For Temperature 1.39E 20; Duration 0.041772 and Interaction between Temperature and Duration 4.334E-08			For Temperature 3.58E-14; Duration1.16E 07 and Interaction between Temperatu re and Duration 8.32E-10		

ANOVA demonstrated (Table 12a and 12b) that both temperature and duration had a significant effect on tenacity and breaking elongation. Also, temperature and duration interaction do affect. It was observed that at higher temperatures, tenacity decreased. The breaking elongation did not have any trend. It is evident from the degumming study in the eco-Eri degumming machine that hard water was not suitable as it had a detrimental effect at high temperatures. Eri cocoon degumming with soft water at 130 °C for 30 minutes displayed the best results in terms of degum weight loss (15.7%), tenacity (18.5 g per tex) and elongation (17.1%) compared to the control soap-soda method (Table 12).

Table 12a. Fibre tenacity,g/Tex ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Sample	857.1327	3	285.6476	59.80518	7.36E-23	2.689691
Columns	130.5631	2	65.13176	13.66451	5.17E-06	3.081387
Interaction	124.2819	6	20.67865	4.342706	0.000589	2.182657
Within	515.9089	108	4.767304			
Total	1627.627	119				

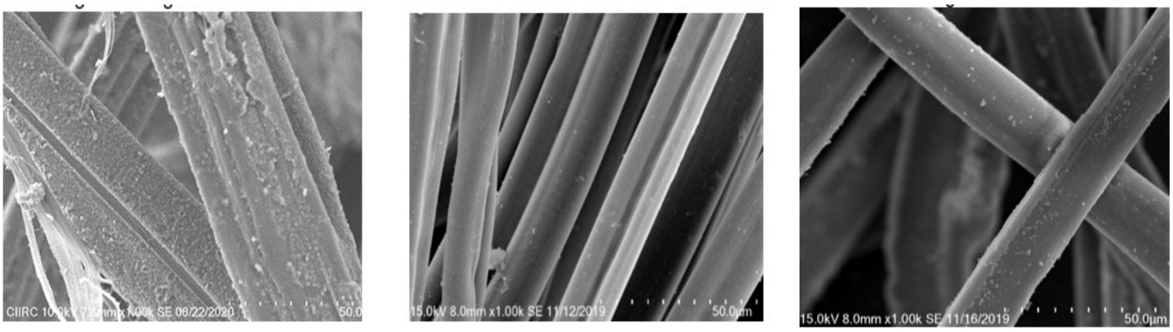
Table 12b. Fibre Elongation, percent ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Sample	190.537	3	63.4456	29.9453	3.58E-14	2.67869
Columns	78.7317	2	39.3158	18.5740	1.16E-07	3.07038

Source of Variation	SS	df	MS	F	P-value	F crit
Interaction	144.3624	6	24.0650	11.3414	8.32E-10	2.18465
Within	229.1449	108	2.13106			
Total	643.016	119				

Fibre Morphology

The SEM images of undegummed (raw) Eri silk fibre in Figure 2a clearly show the presence of sericin as depositions on the fibre surface. Figure 2b shows a soap-soda degummed Eri silk fibre surface with a smoother and clear surface without any fibre damage, indicating an effective degumming process.

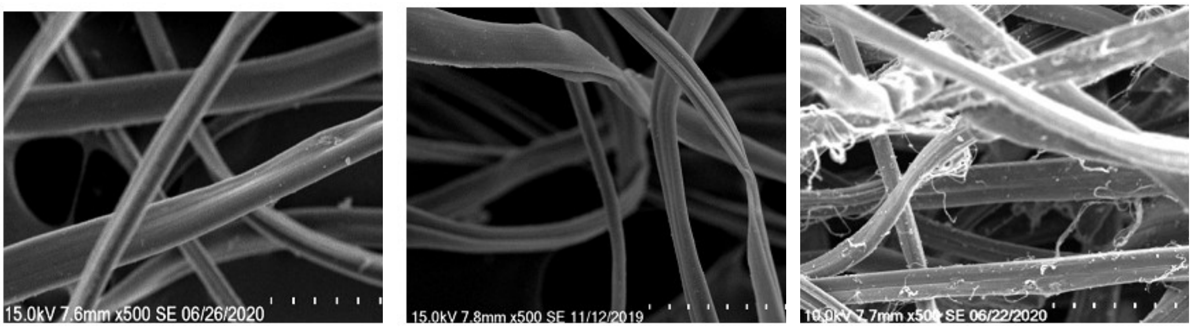


a) b) c)

Figure 2. SEM Images of Eri silk fibres: (a) Eri silk (undegummed) fibres; (b) Eri silk: soap-soda degummed fibres; (c) Eri silk: enzymatic degummed fibres

In 6%, 8% and 10% enzymatic degummed Eri silk fibres, it was noticed that the sericin deposition still appeared as moderately in homogeneous specs on the external fibres and several granular credits were visible in the spaces among filaments, which indicates incomplete degumming of Eri silk fibres. In 12% enzymatic degummed Eri fibre (Fig. 2c), the surface was much smoother with fewer sericin deposits compared to enzymatic degummed Eri fibres. Even then the sericin was not completely removed from degummed Eri silk, clearly observed as white dot spots in the images. In all chemical methods, it is very difficult to hold control of the final results like optimum degumming parentage and strength loo and elongation properties of the material. Whereas in the eco Eri degumming method, there will not be any type of adverse effect on the degummed material.

The SEM images clearly show the presence of gum in un-degummed Eri silk, where the large whitish matter was visible compared to the soap-soda (control) sample. In degumming of Eri silk using enzymes at different concentrations showed that the removal of sericin was not effective as more whitish depositions of sericin were observed. Although, with the increase of enzyme concentration the surface was found to be smoother, still at the highest enzyme concentration degumming was incomplete as the images still showed the presence of sericin depositions on the fibres. Similar observations were also reported in a study carried out.



a) b) c)

Figure 3. SEM Images of Eri silk fibres: (a) Eri silk fibres degummed with hard water; (b) Eri silk fibres degummed with soft water at 130 °C; (c) Eri silk fibres degummed with soft water at 140 °C

The SEM images (Figure 3) show the presence of a low amount of sericin observed as white dotted spots in the degummed Eri fibre using hard water (Figure 3 (a)), which is an indication that the sericin was not completely removed in degummed fibres. If sericin is present in Eri fibres, it will affect all the preparatory processes and spinning, creating neps, thick and thin places in spun yarn leading to deterioration in quality. The SEM images of soft water degummed Eri silk at 130 °C for 30 min (Figure 3(b)) clearly show a smooth surface where effective degumming and smooth fibre surface are observed without any damage. Given this eco Eri cocoon degumming using soft water at 130 °C for 30 min is recommended for Eri cocoon degumming process. Further, it can be observed that degumming at 140 °C clearly showed degradation of fibres with many fibrillations due to excessive degumming as noticed in Figure 3(c).

Overall, it was observed that Eri silk fibres degummed with soft water at 130 °C -30 minutes displayed the finest results in terms of effective degumming (15.7 % degum weight loss) tenacity (18.5 g per tex) and elongation (17.1%) compared to the control soap-soda method, which showed 14.5% degum weight loss), tenacity (18.0 g per tex) and elongation (16.2%). Eco-friendly treatment beyond 130 ˚C resulted in lower tenacity, excessive degumming and fibre damage while degumming below 130 °C resulted in incomplete degumming. It clearly shows that the eco-friendly degumming process for Eri cocoons has countless possibilities to degum the Eri cocoons as the processing time taken was less compared to the soap-soda process. In eco Eri degumming technique evades the use of chemicals and takes one-third of the period of the soap-soda method. Also, the enzymatic degumming does not offer any advantage to degum the Eri silks.

This study revealed that a newly developed eco-friendly degumming process with an eco-Eri cocoon degumming machine is appropriate for degumming Eri cocoons competently. The quality of water plays a significant role in the degumming process and the soft water Eri cocoon degumming technique is recommended. Rise the temperature in degumming and while the outcome in higher degumming (removal of more amount of sericin). As no chemicals were used for degumming, hence, the new degumming was a 100% eco-friendly process and economical. In addition, the new processsaves water, energy, and chemicals and helps to reduce the effluent load. Also, as the process uses only water, sericin can be extracted from degummed wastewater, which in turn will enhance the value addition through by-product utilization.

Advantages of the Eco-friendly Eri degumming method

- Efficient degumming process for Eri silk shells.
- No chemicals are used for degumming and hence 100% eco-friendly process.
- Very less time-consuming (30 minutes) and hence a very productive machine.
- Very economical due to the no use of chemicals and high productivity.
- Less consumption of water and hence very little load on effluent.
- The process can be made zero discharge (effluent free) by extracting sericin.
- Saves water and energy.
- Sericin can be easily recovered after degumming and used for varied applications.

CONCLUSIONS

This study has revealed that the new eco-friendly method is suitable for degumming Eri cocoons efficiently. Good quality of water (soft water) plays an imperative role in the degumming process. Higher temperature, pressure and time result in an increased amount of sericin-dissolution during the eco-friendly degumming method. The optimum degumming treatment conditions are 28 PSI pressure at temperature of 130 °C for 30 minutes duration. Recommended Eco Eri cocoon degumming process parameters in the machine using soft water is at a temperature of 130 °C for 30 minutes for effective degumming. The newly developed degumming method could also be used for brick red Eri cocoons. Adoption of this new eco-friendly method results in low usage of water, and energy, low cost of chemicals and also reduces pollutant load in the effluent, which are the additional advantages making it more efficient and environmentally friendly.

Author Contributions

Conceptualization - Sreenivasa and Padaki NV; Methodology – Sreenivasa and Padaki NV; Formal analysis – Sreenivasa and Padaki NV; Investigation – Sreenivasa and Padaki NV; Writing-original draft preparation – Sreenivasa and Padaki NV; Writing-review and editing – Padaki NV; Visualization –

Padaki NV; Supervision – Sreenivasa. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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