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Comparative Study of the Degumming of Mulberry, Muga, Tasar and Ericream Silk

Abstract
The degumming of Mulberry, Muga, Tasar and Ericream silk substrates was investigated and compared. Experiments were carried out according to the Box-Behnken response surface design. On the basis of weight loss, absorbency, bending length, breaking load, elongation at break and crease recovery, optimum conditions for degumming with soap-soda were determined.

Key words: *Box-Behnken model, degumming, Ericream, Muga, Mulberry, Tasar.*

Introduction

Silk fibre belongs to the family of protein fibres. It is a strong, soft, lustrous fibre and the longest of all natural fibres. This "Queen of Textiles" has all characteristic properties such as coolness, elasticity, strength, etc. India is the second largest producer of raw silk in the world [1], after China. There are two types of silk: cultivated and wild. Bombyx mori belongs to the cultivated silk category, whereas Muga, Tasar and Ericream silks are categorised as wild silk. Cultivated and wild silk differ not only in appearance, filament structure and self colour but also in their different amounts of sericin (gum), which is 20 - 30% in the case of cultivated silk and 8 - 15% in the case of wild silk [2].

Silk mainly consists of fibroin and sericin along with very small amounts of waxy substances, mineral salts and colouring matter. Sericin is also referred to as silk gum, and its removal is, therefore, called degumming [3 - 6]. Degumming is the process of the cleavage of peptide bonds of sericin by hydrolytic or enzymatic methods, and it is removed by solubilisation or dispersion in water [7, 8]. It is very important that the method of degumming is well standardised. To obtain optimum parameters of degumming and the relation between different variables, the Box and Behnken statistical method can be used to minimise the number of trials. The Box Behnken design allows us to reduce the number of samples and also enables the incorporation of most of the main effects, as well as first order interaction and quadratic relationships [9, 10]. In the present investigation, an attempt was made to optimise the soap-soda degumming process using the Box Behnken method for varieties of silk such as Mulberry, Muga, Tasar and Ericream. The comparative study of optimised process parameters will throw light on the nature of silk.

Materials and methods

Materials

Silk fabrics (Mulberry, Muga, Tasar and Ericream) were supplied by Vrijesh Corporation, a government recognised export house.

Chemicals, auxiliaries and dye

Sodium carbonate, 501-bar soap [11], and Acid dye: Solar Brilliant Red BA, supplied by Clarient India Pvt. Ltd, were used.

Methods

Sample preparations and experimental design

Samples were cut and conditioned at 65% RH and 27 °C for 72 hrs. The samples were weighed, and degumming treatment was carried out at a 1:30 for M : L ratio. The degummed samples were washed thoroughly, dried, conditioned and weighed.

The experiments were planned according to the Box and Behnken response surface design. The second order response surface design was used to ascertain the most favourable processing conditions and relationship between the influencing factors and dependent variables. The parameters selected as independent variables for the experimental design were the temperature in ºC, the time in min, the concentration of soap in g/l and the concentration of soda in g/l. The variables and their levels are given in ***Tables 1*** and ***2***, respectively [9, 12, 13]. A quadratic polynomial was used to analyse the

Table 1. *Box-Behnken design for degumming at three levels.*

Variables	-1	0	1
Temp in °C	60	80	100
Time in mins	30	52.5	75
Conc of soap, g/l	1	3	5
Conc of soda, g/l	0	0.75	1.5

Table 2. *Experiments with various parameters of degumming.*

Variables	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27
Temp in °C	60	60	60	60	60	60	80	80	80	80	80	80	80	80	80	80	80	80	80	80	80	100	100	100	100	100	100
Time in mins	53	30	53	53	53	75	53	30	30	53	53	30	75	53	53	75	30	53	75	53	75	53	30	53	75	53	53
Conc of soap, g/l	3	3	5	5	3	3	1	3	1	5	3	5	5	3	3	3	3	1	1	3	3	3	3	5	3	1	3
Conc of soda, g/l	0	0.8	0.8	0.8	1.5	0.8	0	0	0.8	0	0.8	0.8	0.8	0.8	0.8	0	2	1.5	0.8	1.5	2	0	0.8	0.8	0.8	0.8	1.5

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data, with four independent variables (X).Where Y is the response measured, b_0, b_i, b_{ii} & b_{ij} are the coefficients of the regression equation, and i & j are integers with i < j [14 - 16].

$$Y = b_0 + \sum_{i=1}^{4} b_i X_i + \sum_{i=1}^{4} b_{ii} X_i^2 + \sum_{i=1}^{4} b_{ij} X_i X_j$$

The Responses of raw-silk (control sample) of different varieties before degumming are presented in ***Table 3***.

Table 3. Responses of raw-silk (control sample) of different varieties before degumming.

Silk	Absorbency time, s	Bending length, cm	Breaking load, g	Elongation at break, %	Crease recovery angle, degree
Mulberry	92	4.1	98.27	16.5	110
Muga	79	6.0	82.65	18.0	105
Tasar	41	4.2	60.90	23.3	94
Ericream	12	5.3	54.12	17.9	115

Tests for evaluation of samples

The weight loss WL in % was calculated using the formula,

$$WL \text{ in } \% = [(W_1 - W_2)/ W_1] \times 100$$

where W_1 and W_2 are the weights in grams of the raw and degummed samples, respectively [17]. The breaking load of the degummed and bleached fabrics was measured on an Instron Tensile Tester. The bending length of the degummed samples was measured using a 'Shirley' Stiffness Tester, and the crease recovery angle was tested using a 'Shirley' Crease recovery angle tester. The absorbency of the degummed samples was checked according to the AATCC Test Method 79-2000. The raw and degummed samples were viewed under a scanning electron microscope (Philips XL 30 SEM).

Results and discussion

Degumming with soap-soda

Values of the various responses of the 27 experiments are given in ***Table 4*** for Mulberry and Muga, and in ***Table 5*** (see page 12) for Tasar and Ericream silk.

A qualitative test of gum removal was performed by dyeing [2]. The different varieties of silk were dyed with direct dye Solar Brilliant Red BA at a neutral pH at boiling point for two minutes with a 2% shade. It is suggested that since sericin gives a pink colour with Solar Brilliant Red BA, a light pink colour represents the presence of a little amount of sericin on the fabric, whereas for fibroin, which does not give any color, the absence of any color represents a complete removal of sericin. These results are reported in ***Tables 4 and 5*** (see page 12).

Effect of independent variables on different responses

The results from ***Tables 4 and 5*** and representative response surface diagrams shown in ***Figure 1*** (see page 12) for mulberry silk only indicate the following trends:

Weight loss

In general, with an increase in the parameters of degumming, such as the temperature, time, and concentration of soap and soda, the weight loss in % increased due to the degumming of the silk material (Refer ***Figure 1.a***). In the case of Mulberry silk, such weight loss was as high as 25.48%, followed by 16.11% for Muga, 14.25% for Tasar and 9.59%

Table 4. Values observed for respective degumming experiments on Mulberry and Muga Silk.

Run order	Mulberry							Muga						
	Weight loss, %	Absorbency time, s	Bending length, cm	Breaking load, g	Elongation at break, %	Crease recovery angle, degree	Colour	Weight loss, %	Absorbency time, s	Bending length, cm	Breaking load, g	Elongation at break, %	Crease recovery angle, degree	Colour
1	3.3	78	3.72	89.8	15.72	119	Pink	7.88	69	5.34	80.13	17.89	109	Pink
2	3.5	77	3.56	88.7	15.37	120	Pink	8.00	68	5.22	79.14	17.12	112	Pink
3	3.5	75	3.26	87.3	14.92	123	Pink	8.15	67	5.15	78.56	16.78	115	Pink
4	4.0	75	3.43	88.0	15.12	126	Pink	8.52	67	5.07	78.00	16.50	115	Pink
5	4.1	73	3.00	86.1	14.53	126	Pink	8.91	66	4.99	77.64	16.00	116	Pink
6	4.4	71	3.12	87.0	14.76	127	Pink	9.24	65	4.93	76.82	15.96	117	Pink
7	6.5	51	2.70	66.2	13.09	135	Pink	9.87	47	4.63	73.86	15.01	121	Pink
8	6.5	49	2.75	66.8	13.24	137	Pink	10.0	46	4.57	73.00	14.78	122	Pink
9	7.7	48	2.63	65.9	12.95	138	Pink	10.2	44	4.50	72.56	14.33	125	Pink
10	8.3	46	2.8.0	65.7	13.59	140	Pink	10.9	44	4.34	72.10	14.00	126	Pink
11	8.6	45	2.25	65.4	12.10	141	Pink	11.3	43	4.27	71.84	13.88	127	Pink
12	9.2	43	2.34	63.8	12.38	142	Pink	11.6	43	4.14	71.52	13.52	128	Pink
13	9.2	42	2.19	63.4	11.93	143	Pink	11.9	40	4.07	71.00	13.30	130	Pink
14	9.4	40	2.51	63.6	12.70	144	Pink	12.0	39	4.00	70.50	13.00	131	Pink
15	10	40	2.45	60.3	12.58	146	Pink	12.1	38	3.94	70.24	12.72	132	Pink
16	11	39	2.86	59.4	13.94	147	Pink	12.4	38	3.83	70.00	12.13	133	Pink
17	12	37	2.11	59.0	11.78	148	Pink	12.6	38	3.72	69.78	12.00	134	Pink
18	14	36	1.83	58.8	11.07	150	Pink	12.6	37	3.60	69.42	11.90	136	Pink
19	15	35	1.99	58.5	11.59	151	Pink	12.8	35	3.47	69.00	11.68	137	Pink
20	15	35	1.92	57.2	11.26	153	Pink	12.9	34	3.29	68.84	11.34	137	Pink
21	19	33	1.78	56.3	11.00	153	Pink	13.7	33	3.15	68.11	11.07	138	Pink
22	23	11	0.69	45.0	10.24	233	Pink	14.0	8.0	0.92	62.71	10.39	234	Pink
23	24	9.7	0.55	44.4	10.04	238	Light Pink	14.4	7.3	0.87	61.90	10.03	240	Pink
24	24	8.4	0.48	44.0	9.97	243	Light Pink	15.0	6.4	0.75	61.23	9.87	245	Light Pink
25	25	6.8	0.43	43.5	9.87	250	Light Pink	15.0	5.7	0.72	60.24	9.84	253	White
26	26	5.9	0.50	43.0	9.70	259	White	15.0	4.3	0.69	59.87	9.69	259	White
27	26	4.6	0.46	40.8	8.68	267	White	16.1	4.0	0.62	59.00	9.46	263	White

Table 5. *Values observed for respective degumming experiments on Tasar and Ericream Silk.*

Run order	Tasar							Ericream						
	Weight loss, %	Absorbency time, s	Bending length, cm	Breaking load, g	Elongation at break, %	Crease recovery angle, degree	Colour	Weight loss, %	Absorbency time, s	Bending length, cm	Breaking load, g	Elongation at break, %	Crease recovery angle, degree	Colour
1	5.14	21	3.70	57.34	21.00	96	Pink	2.48	7	4.78	52.1	17.30	119	Pink
2	5.62	20	3.59	57,00	20.17	97	Pink	3.18	7	4.69	51.5	17.00	120	Pink
3	5.82	20	3.40	56.39	20.49	98	Pink	3.73	6	4.54	51.1	16.80	123	Pink
4	6.12	19	3.24	56.00	20.89	100	Pink	4.14	6	4.23	51.0	16.40	126	Pink
5	6.31	19	3.17	55.65	21.32	101	Pink	4.31	5	4.15	50.0	16.00	126	Pink
6	6.47	18	3.10	55.12	21.73	103	Pink	4.71	5	4.00	49.5	15.70	127	Pink
7	6.94	14	3.04	50.13	18.00	111	Pink	5.61	5	3.74	47.7	14.80	135	Pink
8	7.46	14	2.93	50.00	17.76	112	Pink	5.68	5	3.70	47.2	14.30	137	Pink
9	8.23	13	2.85	49.55	18.27	113	Pink	5.78	4	3.63	46.7	14.00	138	Pink
10	8.41	13	2.76	48.87	17.50	115	Pink	5.95	4	3.60	46.0	13.70	140	Pink
11	8.70	13	2.69	48.21	18.79	117	Pink	6.33	4	3.52	45.0	13.10	141	Pink
12	8.87	13	2.60	47.37	16.31	118	Pink	6.45	4	3.48	44.1	12.70	142	Pink
13	8.95	13	2.54	47.00	19.01	119	Pink	6.57	4	3.37	44.0	12.30	143	Pink
14	9.00	13	2.49	46.82	17.14	119	Pink	6.57	4	3.25	44.0	12.00	144	Pink
15	9.54	13	2.42	46.07	14.82	120	Pink	6.57	4	3.2	42.8	11.40	146	Pink
16	9.68	12	2.36	45.98	15.56	122	Pink	6.85	4	3.15	42.3	11.01	147	Pink
17	9.85	12	2.29	45.62	15.14	122	Pink	6.91	4	3.10	40.4	10.92	148	Pink
18	10.00	12	2.21	45.00	17.00	123	Pink	7.17	3	3.03	40.0	10.80	150	Pink
19	10.23	12	2.13	44.54	16.23	125	Pink	7.23	3	2.97	39.5	10.60	151	Pink
20	10.58	11	2.09	44.00	15.89	125	Pink	7.54	3	2.92	38.2	10.43	153	Pink
21	11.24	11	2.05	43.87	16.00	128	Pink	7.66	3	2.85	38.0	10.24	153	Pink
22	11.30	2	0.56	39.65	12.75	202	Pink	**7.69**	**1**	**1.15**	**37.8**	**10.00**	**233**	**Light pink**
23	12.41	2	0.53	38.78	13.07	206	Pink	8.63	1	1.10	37.6	9.87	238	Light pink
24	12.54	2	0.51	38.14	12.93	209	Pink	8.73	0	1.05	36.9	9.35	243	White
25	13.57	1	0.49	38.00	11.40	212	Pink	8.83	0	1.00	36.1	9.12	250	White
26	14.05	1	0.47	37.85	12.30	221	Pink	8.89	0	0.92	36.0	9.00	259	White
27	**14.25**	**0**	**0.45**	**37.23**	**12.01**	**235**	**Light pink**	9.59	0	0.80	34.5	7.97	267	White

for Ericream. This was in the range of the gum content of cultivated and wild silk, as reported in literature.

Absorbency

It was found that with the severity of degumming conditions, the time taken for the absorption of a water drop by degummed fabric was found to decrease, for example the control Mulberry fabric required 92 s of absorption, which decreased to 78 s at the mildest conditions of degumming, giving only a 3.31% weight loss. However, the absorbency time was found to drastically decrease to about 4.6 s at the maximum weight loss of 25.48% as a result of the severe conditions of degumming. The trend of the improvement in the absorbency of degummed silk of different varieties remained same for all the samples, although the extent of the improvement in absorbency did vary depending upon the variety of silk. (Refer ***Figure 1.b***).

Bending length

As a result of degumming, wherein sericin is removed, weight loss was observed, thus the silk material was rendered soft. In order to scale this softness, the bending length of the degummed fabric was measured, in which a decreased bending length was indicative of increased softness. The results indicate that bending length values for the control (raw silk) decreased from 4.1 cm to 0.46 cm for Mulberry, from 6.00 cm to 0.62 cm for Muga, from 4.2 cm to 0.45 cm for Tasar and from 5.3 cm to 0.8 cm for Ericream (Refer ***Figure 1.c***).

Breaking load

The breaking load or tensile strength of the degummed samples in the warp direction was measured, the results of which indicate that with an increase in the severity of the parameters of the degumming, the tensile strength was found to decrease progressively. The silk gum or sericin acts as cementing material, adding to the strength of the yarns which constitute the fabric. When degumming is carried out, due to the loss of cementing material, the strength of the fabrics is likely to be reduced, making the delicate nature of the silk fabric more apparent, which is evident in the results from the tables and ***Figure 4***, clearly indicating a reduction in the breaking load, from the mildest to severe conditions of degumming, of the order of 89.8 g to 40.8 g for Mulberry, from 80.13 g to 59 g for Muga, from 57.34 g to 37.23 for Tasar and from 52.1 g to 34.53 g for Ericream.

Elongation at break

The elongation at break was found to decrease with an increase in weight loss, which may be attributed to the removal of sericin and wax, however little, acting as a lubricant. The elongation at break values for the control sample (raw silk) decreased from 16.5% to 8.68% for Mulberry. The same trend was observed for the other three varieties of silk.

Crease recover angle

With an rise in weight loss, the crease recovery angle of the fabric increased. That of Mulberry rose from 119° to 267°. Once fibroin is freed from sericin, being relatively more crystalline [18], it gives a high degree of the crease recovery angle. A good degree of the crease recovery property leads to good characteristics of the wrinkle resistance of the material.

Having studied six different responses, namely the weight loss, absorbency time, bending length, breaking load, elongation at break and crease recovery angle with respect to four different parameters of the degumming process, regression

equations were established relating all four independent variables: the temperature, time, and concentrations of soap and soda with the dependent performance responses. A total of 24 regression equations were created. In all the regression equations, the regression coefficient values for all responses were found to be as high as 99.3% i.e. very close to unity for all types of silk, indicating the perfect correlation of each response with the four independent process parameters of degumming. Some of them are listed below.

Substrate: Mulberry; Process: degumming, response

$$\text{Wt loss in \%} = 24.0443 - 0.997422\times T + \\ - 0.0713305\times t + 2.97747\times s + \\ - 4.33085\times S + 0.00956094\times T^2 + \\ + 0.00206496\times t^2 + 0.0658989\times s^2 + \\ + 2.08377\times S^2 + (0.000266227\times T\times t) + \\ - 0.0159962\times T\times s + 0.0342842\times T\times S + \\ - 0.0391729\times t\times s + 0.0408404\times t\times S + \\ - 0.0796788\times s\times S$$

Substrate: Tasar, process: degumming, response:

$$\text{Wt loss in \%} = -7.62196 + 0.116030\times T + \\ + 0.00294747\times t + 2.19356\times s + \\ -0.0149667\times S + 0.000522073\times T^2 + \\ + 0.000504267\times t^2 - 0.0374333\times s^2 + \\ + 0.139241\times S^2 + 0.000172000\times T\times t + \\ - 0.0157688\times T\times s + 0.0296600\times T\times S + \\ - 0.0106644\times t\times s - 0.0123185\times t\times S + \\ - 0.146783\times s\times S$$

Substrate: Ericream, process: degumming, response:

$$\text{Wt loss in \%} = -16.1880 + 0.326335\times T + \\ + 0.0710251\times t + 0.874041\times s + \\ + 1.10221\times S - 0.000910538\times T^2 + \\ + 0.000325824\times t^2 - 0.00114834\times s^2 + \\ + 0.0595287\times S^2 - 0.000737595\times T\times t + \\ - 0.00533902\times T\times s + 0.00130051\times T\times S + \\ - 0.00745285\times t\times s - 0.00609151\times t\times S + \\ + 0.00569056\times s\times S$$

Substrate: Muga, process: degumming, response:

$$\text{Wt loss in \%} = -11.5003 + 0.260122\times T + \\ + 0.0817166\times t + 1.63789\times s + \\ + 0.849228\times S - 0.000480445\times T^2 + \\ + 0.000246844\times t^2 - 0.0706080\times s^2 + \\ + 0.254220\times S^2 - 0.000342789\times T\times t + \\ - 0.00513477\times T\times s + 0.0183883\times T\times S + \\ - 0.0118128\times t\times s - 0.0192511\times t\times S + \\ - 0.126550\times s\times S$$

Independent variables were:
T - temp; t - time; s - concentration of soap; S - concentration of soda.

Table 6. *Optimised conditions and corresponding performance responses based on the cumulative ranking for degumming.*

Silk	Temperature, °C	Time, mins	Soap conc., g/l	Soda conc., g/l	Weight loss, %	Absorbency time, s	Bending length, cm	Breaking load, g	Elongation at break, %	Crease recovery angle, degree
Mulberry	100	30.0	3	0.75	24.02	9.70	0.55	44.40	10.04	238
Muga	100	52.5	5	0.75	14.75	6.37	0.75	61.23	9.87	245
Tasar	100	52.5	3	1.50	14.25	0.45	0.45	37.23	12.01	235
Ericream	100	52.5	3	0	7.69	0.63	1.15	37.80	10.00	233

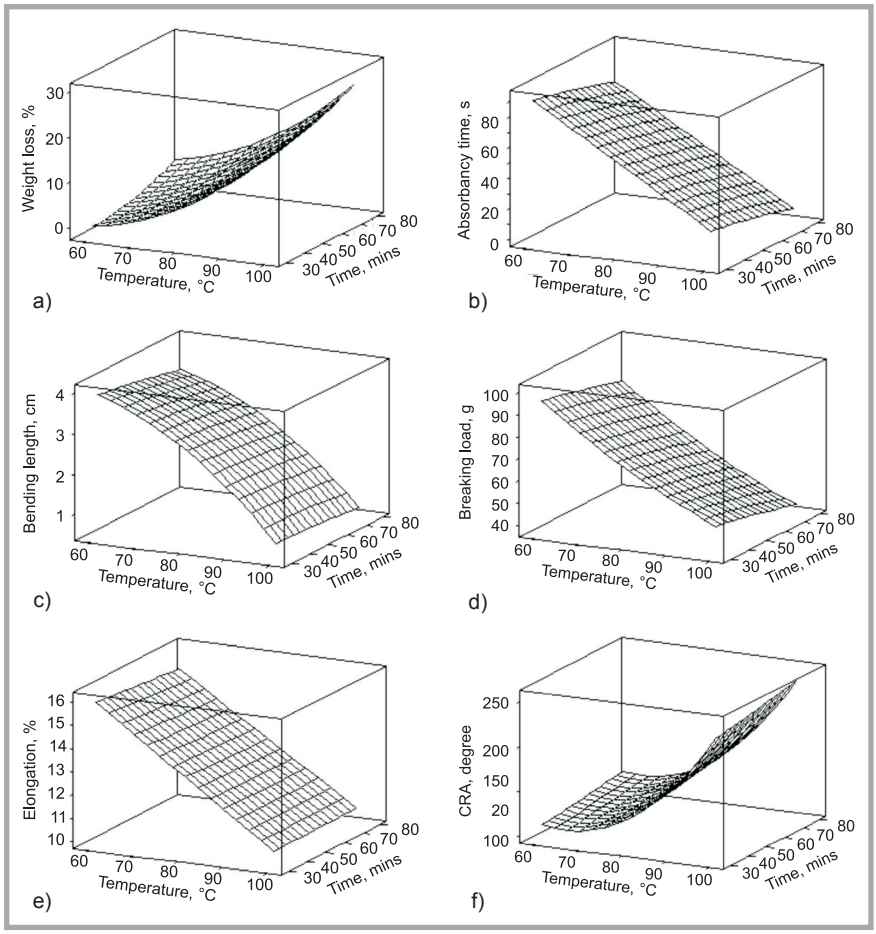

Figure 1. *Relations: weight loss - a), absorbency time - b), bending length - c), breaking load - d), elongation at break - e), and crease recovery angle (CRA) - f) vs. temperature and time.*

Optimisation

Having compared all six responses in terms of values observed and calculated in twenty seven different experiments based on various parameters of degumming process values, a ranking was created for individual responses and finally a cumulative ranking for every single experiment. Based on the cumulative ranking, the maximum of three experiments of the highest ranking were chosen to establish an optimised recipe for degumming. Considering the highest level of degumming as a prime factor (weight loss in %) and tolerable limit of tensile strength loss (indicated as the breaking load), maximum absorbency (least time for absorbing a water drop) and least of bending length the optimized conditions for Mulberry, Muga and Tasar silk were selected, as shown in ***Table 6***.

It should be noted that wild silk, such as Muga, Tasar and Ericream contain 8 - 15% of sericin as compared to 25 - 30% of sericin in Mulberry silk; this gum being very difficult to remove. Hence the optimum conditions indicate that the time and/or soap and soda concentration required in the case of degumming wild silk are higher than those required for the degumming of Mulberry silk. The weight loss levels observed were in the range of silk gum present in raw silk, which may also be the reason for the dif-

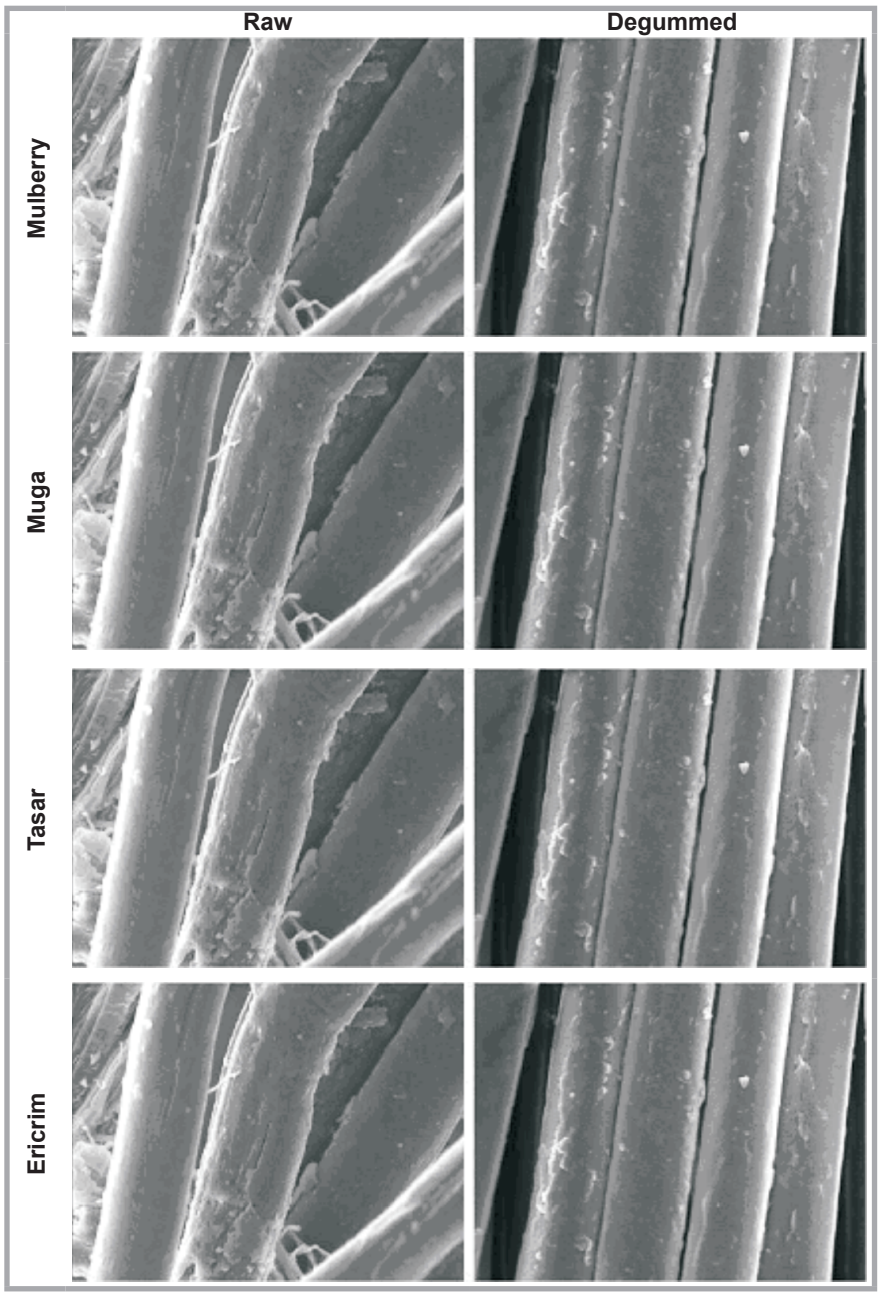

Figure 2. *SEM images.*

ferential response depicted by different varieties of silk.

Having obtained fitting regression equations, four experiments were randomly planned, in which four different independent variables were selected. Actual experiments were carried out for Mulberry, Muga, Tasar and Ericream silk. The responses observed were compared with the responses predicted and were found to in be in very good agreement, indicating that regression equations have the precision to give a realistic picture of the responses once the specific parameters of the degumming process are chosen. Scanning electron photomicrographs (SEM) of the raw and degummed silk samples are shown in ***Figure 2***. SEM results show a smooth surface after degumming, which confirms the removal of sericin from the surface of silk fibroin after the degumming process.

Conclusions

Relationships between the parameters of the degumming process such as the temperature, time, concentration of soap and soda were established with output responses such as weight loss, absorbency time, bending length, crease recovery angle, breaking load and elongation at break. These relationships were tested for their predictability. The regression coefficients between observed and calculated responses indicate the best fit. Using this statistical model, final optimised conditions for the degumming of Mulberry, Muga, Tasar and Ericream silk were established. Muga,Tasar and Ericraem silks, despite containing a reduced amount of sericin (8 - 15%) as compared to that of Mulberry (25 - 30%), required more time and severe conditions for its removal than those needed for Mulberry silk. This clearly indicates that sericin is more strongly embedded in wild silk as compared to that in mulberry silk.

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