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Effect of degumming with sodium carbonate on properties of oak tasar silk filament

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Abstract
Degumming of cocoon is one of the major processes in post cocoon technology. Silk cocoon is degummed for the reeling of silk filaments, oak tasar cocoon is commonly degummed with sodium carbonate. Under the present study degumming of oak tasar cocoon was done with various concentrations of sodium carbonate. Effect of different concentration on fibre properties was assessed by testing the degummed silk fibre samples for its tensile strength, elongation and fineness. SEM images of each sample were also recorded to analyze the surface morphology. It was noticed that higher concentrations of sodium carbonate adversely affected the fibre properties. Present study shows that lower concentrations i.e. 0.3% to 0.5% on weight of material, of sodium carbonate can be used for the degumming of oak tasar cocoon without affecting fibre properties.

Keywords: Oak tasar, degumming, sodium carbonate, fibre properties

Introduction
Sericulture is one of the largest industries in textile sector. Currently India is the second largest producer of raw silk and has an important place in global market. India is the only country which is producing all the four varieties of silk namely mulberry silk, tasar silk, eri silk and muga silk. Tasar silk is found in two varieties one is temperate tasar produced mainly in Jharkhand and Bihar region and tropical tasar found mainly in Uttarakhand, India. Tropical tasar is also known as oak tasar. Oak tasar cocoon feeds upon oak tree leaves thus named as oak tasar cocoon. According to Central Silk Board in the year 2017-18 total production of raw silk was 31,906 metric ton and 2,988 metric ton of tasar silk was produced [1].
Silk industry is agriculture based and can be divided majorly into two stages. First is rearing of silk worm along with cocoon production and second is post processing technology which includes all the processes regarding production of fibre to fabric for various end uses. Silk filament is mainly composed of two types of protein namely fibroin and sericin. Both the protein are different in chemical nature. Fibroin is a true silk filament which is insoluble in water and sericin is a gum which hydrolyses and get solubilize in water during degumming process. Under the degumming process cocoons are cooked by various methods to remove sericin gum from the cocoons which facilitate in reeling of silk filament. Degumming of silk cocoon results in hydrolysis of sericin and can be carried out in neutral, alkaline and acidic condition with varying time and temperature [2]. The present study is focused on the degumming of oak tasar silk cocoon which is produced majorly in the sub Himalayan range of India**.** Degumming is commonly carried out by sodium carbonate [3] thus an attempt was made to assess the effect of sodium carbonate concentration on various properties of silk fibre.

Material and Methods: Double distilled water and lab grade chemicals were used for the entire study. 10g cocoons were degummed with different concentrations of sodium carbonate which is a commonly used chemical for silk cocoon degumming. Treated and control fibre samples were tested for various parameters to study the effect of degumming process on fibre properties.

Degumming of cocoon: Cocoons were cooked at 95-100°C for 45 minutes at MLR of 1:30 with 11 concentrations of sodium carbonate i.e., 0.05%, 0.1%, 0.2%, 0.3%, 0.4%, 0.5%, 0.6%, 0.7%, 0.8%, 0.9% and 1.0% on the weight of material and control sample was degummed in distilled water. After completion of the degumming, cocoons were washed and reeled to obtain silk filament.

Reeling of silk filament: Reeling of silk filament was done manually and fibres were dried in open air. Finally fibres were kept in the hot air oven at 60 °C for 30 minutes for complete removal of moisture and weight of each sample was recorded.

Testing of silk filaments: To assess the effect of sodium carbonate on the fibre properties. the physical properties, surface morphology of fibre and weight loss percent after degumming of cocoons were analysed.

Physical properties of silk fibres: Physical properties namely i.e. tensile strength, elongation and fineness of fibre were assessed. Tensile strength and elongation was measured using Fafegraph M instrument as per ASTM test standard-D3822:07. Fibre fineness was measured using Vibromat M instrument and test method used was ASTM D1577:07. Thirty fibres of each sample were taken and tested for the physical properties and average value was taken as final result.

Surface morphology: Surface morphology of degummed fibre was studied using Scanning Electron Microscope (SEM) model- JEOL-JSM-6610LV. Prior to SEM analysis fibres were fixed on the aluminum holder called 'stub' with the help of two sided carbon tape and then gold coating was done using auto fine coater machine JEOL JFC-1600. Then samples were analyzed and images were recorded at 50kv under 1500x and 1100x magnification.

Weight loss percent of cocoon: weight loss of cocoon was also assessed which shows the effect of degumming process. Weight loss percent was assessed with the given expression.

$$\text{weight loss percent} = \frac{FW - IW}{IW} \text{ X } 100$$

Where,
FW= Final weight of cocoon after degumming
IW= Initial weight of cocoon before degumming
Statistical analysis: Data was statistically analyzed using SPSS software with one way ANOVA.

Results and Discussions
Result of the present study was studied under the following subheadings

Degumming of tasar silk cocoon
Degumming of ten grams of each cocoon was done with various concentrations of sodium carbonate and weight loss of cocoon was calculated. 10 gm of cocoon was degummed with various concentrations of sodium carbonate. After degumming cocoon were washed, dried and then their final weight and weight loss percent was calculated.
Weight loss of cocoon was directly proportional to concentrations of sodium carbonate (Fig.1). Weight loss shows the removal of sericin from cocoon which is desirable for the reeling of silk filament but higher concentration of chemical may damage the fibroin which is the real silk filament.

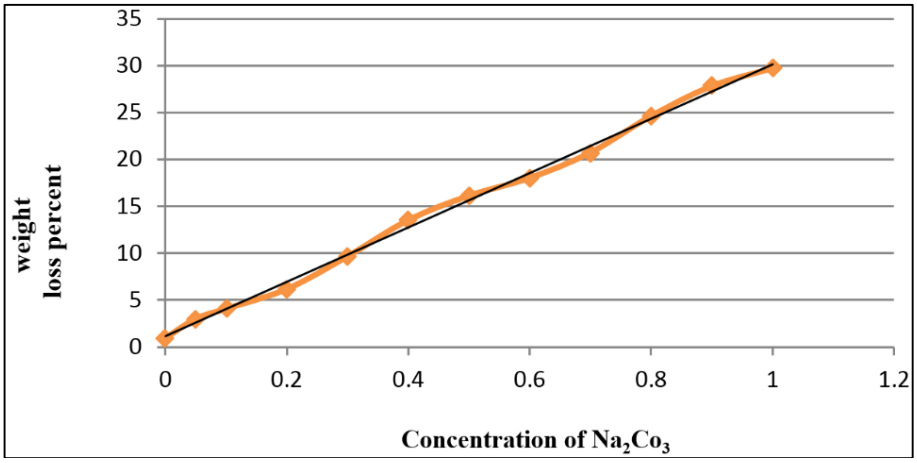

Fig 1: Weight loss of cocoon at various concentration of Na_2Co_3

SEM image of each samples are given in Fig.2. Weight loss of cocoon during degumming is also related with the mechanical properties of resulting filaments. Alkali ions react with the peptide bonds and hydrolyze the sericin protein. During degumming with alkali, Na^+ ion react with the COOH and OH groups of sericin and form a new molecule COO-Na^+ and O^- Na^+, thus get solubilize in the degumming liquor. Fibres tensile strength and fineness were found inversely proportional to the concentration of sodium carbonate. Highest weight loss (29.7 percent) in cocoon was seen, in the sample treated with highest percentage of Na_2Co_3 i.e. 1.0 percent.
Statistically no significant difference for weight loss was found at 5% level of significance using lower concentration of sodium carbonate whereas at higher concentration of sodium carbonate, significant difference was noticed at 5% level of significance between the percent weight loss of cocoon and sodium carbonate concentration. Similar results were also reported in few studies where higher concentration of alkali damaged the fibroin thus degumming must be done at lower concentration of alkali [4, 5].

Surface morphology of degummed fibre
Each sample was analyzed under Scanning Electron Microscope to assess the effect of degumming process. Sericin was seen on the surface of fibre degummed with water (fig. 2.a) which showed that less sericin was removed and degumming was not effective with water. Sericin was also present on the fibre degummed with 0.05 (fig. 2.b) and 0.1 (fig 2.c) percent of sodium carbonate. Cocoon degummed with 0.2 percent of sodium carbonate and above, exhibited removal of sericin in a higher amounts whereas higher concentration i.e.0.9 and 1.0 percent of sodium carbonate showed damage in the silk filament (fig. 2.k and fig. 2.l).

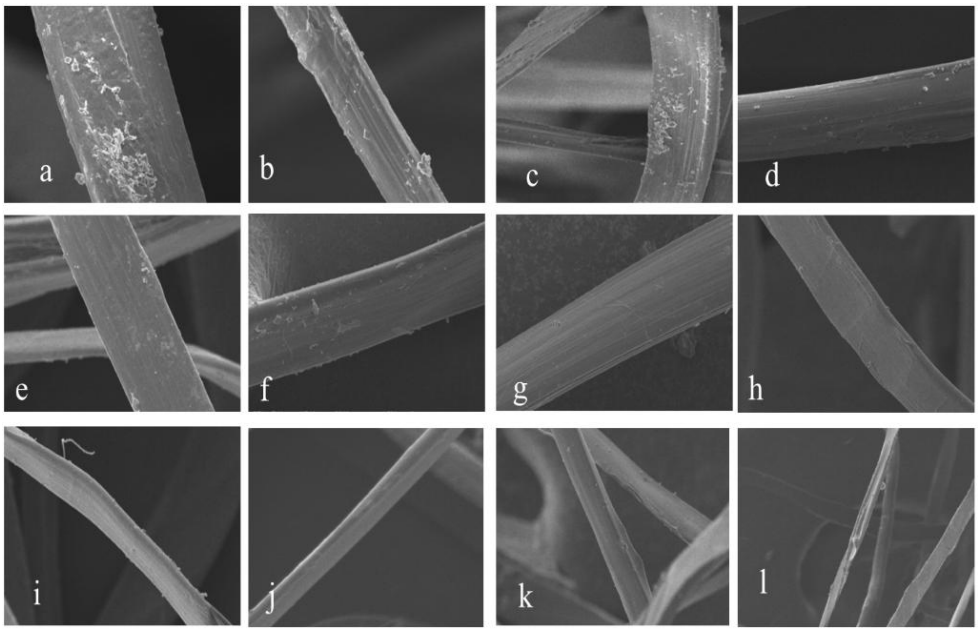

Fig 2: SEM images of degummed samples treated with various concentrations of Na_2Co_3 a) Fibre degummed with water b) 0.05 % of Na_2Co_3 c) 0.1 % of Na_2Co_3 d) 0.2 % of Na_2Co_3 e) 0.3 % of Na_2Co_3 f) 0.4 % of Na_2Co_3 g) 0.5 % of Na_2Co_3 h) 0.6 % of Na_2Co_3 i) 0.7 % of Na_2Co_3 j) 0.8 % of Na_2Co_3 k) 0.9 % of Na_2Co_3 l) 1.0 % of Na_2Co_3

Effect of degumming on the physical properties of silk filaments

Physical properties of fibres were tested and it was found that with the increase in concentration of sodium carbonate, tensile strength was decreased.

Table 1: Tensile strength, elongation and fineness of treated fibres

Sample	Treatment (Na_2Co_3)	Elongation (%)	Fineness (denier)	Tenacity (g/ denier)	Strength loss percent
Control	Blank water	$18.32^{a} \pm 0.015$	$1.97^{a} \pm 0.01$	$5.41^{a} \pm 0$	$00^{a} \pm 0$
1	0.05	$18.46^{a} \pm 0.036$	$2.48^{b} \pm 0.015$	$5.38^{ab} \pm 0.005$	$0.61^{ab} \pm 0.10$
2	0.1	$18.37^{a} \pm 0.026$	$2.53^{c} \pm 0.006$	$5.30^{b} \pm 0.006$	$1.97^{b} \pm 0.11$
3	0.2	$18.57^{b} \pm 0.01$	$2.55^{c} \pm 0.01$	$5.02^{c} \pm 0.005$	$7.33^{c} \pm 0.10$
4	0.3	$18.99^{c} \pm 0.01$	$2.73^{d} \pm 0.006$	$5.01^{c} \pm 0.006$	$7.39^{c} \pm 0.18$
5	0.4	$19.61^{c} \pm 0.02$	$2.77^{ef} \pm 0.01$	$4.97^{c} \pm 0.01$	$8.19^{c} \pm 0.43$
6	0.5	$21.35^{d} \pm 0.006$	$2.79^{f} \pm 0.01$	$4.68^{d} \pm 0.023$	$13.42^{d} \pm 0.49$
7	0.6	$21.42^{d} \pm 0.01$	$2.75^{de} \pm 0.006$	$4.63^{d} \pm 0.025$	$14.29^{d} \pm 0.28$
8	0.7	$21.78^{e} \pm 0.02$	$2.91^{g} \pm 0.01$	$4.43^{e} \pm 0.015$	$18.04^{e} \pm 0.28$
9	0.8	$21.15^{e} \pm 0.025$	$3.61^{h} \pm 0.02$	$3.90^{f} \pm 0.01$	$27.84^{f} \pm 0.185$
10	0.9	$21.81^{e} \pm 0.025$	$3.91^{i} \pm 0.015$	$3.92^{f} \pm 0.085$	$27.91^{f} \pm 1.58$
11	1.0	$21.76^{e} \pm 0.035$	$3.96^{j} \pm 0.006$	$3.66^{g} \pm 0.053$	$32.34^{g} \pm 0.98$

Data followed by same letter have no significant difference according to Duncan post hoc test ($p > 0.05$) at 5% level of significance

It was seen that the control fibre had highest tensile strength 5.41 (Table 1), it may be due to less amount removal of gum protein sericin as the control sample was degummed in distilled water only without adding sodium carbonate. Finding of the present study was similar with a study in which tensile strength, fineness and **e**longation of tasar silk fibre was found as 5.12, 19.46 and 2.62 respectively [6]**.** Sericin was not desirably removed in the control degummed liquor as in the absence of sodium carbonate no strong ionic reaction held and sericin was not hydrolyzed, therefore sericin was not removed properly.

Fineness of the fibres increased significantly with the increase in concentration of sodium carbonate up to 0.5 and further showed successive increase in the fineness. In contrast to fineness the tenacity of the fibres decreases with increase in the sodium carbonate concentration in the degumming recipe. It is clear from the table 1 that property i.e. Elongation, Tenacity and strength loss percent showed statistically non significant difference at 5% level of significance for the lower concentrations (0.05 and 0.1) of sodium carbonate in comparison with control sample.

Fibre strength was adversely affected at higher concentrations (0.8, 0.9 and 1.0 percent) of sodium carbonate with high strength loss of fibre i.e. 27.35, 26.24 and 33.45 percent respectively. Results were similar with the study[8], in which it was stated that higher concentration of alkali damages the structure of silk filament and thus the quality of silk fibre was degraded. Under the present study significant difference was observed between the properties of control and samples treated with higher concentrations of sodium carbonate.

Statistically non significant difference for strength loss was found at 5% level of significance using lower concentration of sodium carbonate whereas at higher concentration of sodium carbonate, significant difference at 5% level of significance was noticed between the percent strength loss of cocoon and sodium carbonate concentration.

Results of the study are in line with a study[7], where the fibre strength was reported 4.6 g/d when degumming was done with lower concentrations of sodium carbonate along with sodium hydroxide and surfactant.

Conclusion

As sodium carbonate is majorly used for the degumming of silk fibre thus it was important to investigate its affect on the properties of silk fibre. Concentration of sodium carbonate above 0.5 percent had adversely affected the fibre properties thus sodium carbonate should not be used above 0.5 percent. From the present study it can be concluded that lower concentration of sodium carbonate can be used for degumming of cocoon as it did not affect the fibre properties significantly.

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