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# Effect of drying on tensile properties and structure of tasar and muga silk

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Tasar and muga silk yarns have been dried at 80ºC, 100ºC and 140ºC and the effect of temperature on tensile and structural properties is investigated. No significant effect of drying on tenacity of tasar and muga silk yarn is observed. However, elongation decreases with drying due to increase in moisture loss with temperature. Crystallinity has no effect even when dried up to 140˚C. Scanning electron micrograph study reveals that there is no significant change in surface structure with different temperature of drying.

**Keywords:** Crystallinity, Muga, Silk, Tasar, Tensile properties

The non-mulberry silks (tasar, muga and eri) are now being popularized as vanya silk. These are wild in nature, user-friendly and healthy because of their inherent nature[1]. Two species of *Antheraea* are exploited for production of silk known as tropical tasar (*Antheraea mylitta*) and temperate oak tasar (*Antheraea proyeli*); these are cultured in India. Muga silk is the product of the silkworm *Antheraea assamensis* endemic to Assam. The stress-strain behavior of mulberry silk filament is different from that of tasar silk.

Tasar has a very high elongation compared to mulberry filament[2]. The structural changes of silk fibres induced by heat treatment were studied[3] and the physical properties of these were compared using the silk from two different species, viz. mulberry (*Bombyx mori*) and wild silk (*Antheraea pernyi*). The original crystalline structure of the mulberry and tasar silk did not show any significant changes in spite of the heat treatment. Investigations were done[4] on stress and strain curves and the corresponding structural parameters in mulberry and non-mulberry silk using wide angle x-ray scattering. Exclusive crystal structure studies of tasar show interesting feature of transformation from anti-parallel chains to parallel chains. These studies show that the characteristic behavior of tasar silk is quite different from mulberry silk. With increase in percentage of the load, the crystallite area per volume decreases, which is quite contrary to the results obtained for mulberry. Studies on strength properties of different types of silk fabrics / garment in wet condition have been reported[5]. The elongation of mulberry silk fabrics in wet condition increases by 12% and even more than in tasar silk fabrics. Divakara *et al*.[6] has studied changes in stacking faults and micro structural parameters of dry and wet bivoltine silk using whole powder pattern fitting technique to understand the basic physics involved in the absorption of water by silk. The stacking and twin faults are low in dry bivoltine silk fibre than in wet condition. The stacking fault density decreases with increase in external strains, whereas twin fault density increases with increase in weight. The correlation between structure and property within different varieties of silk was also investigated[7]. The tenacity values increased substantially along the filament length within a cocoon, as it moves from outer layer to inner layer. All the micro-structural parameters showed significant increase from outer layer to inner layer. It is observed that the silk filaments from the same cocoon have considerable differences in linear density. Mulberry is the finest, followed by Eri, Tasar and Muga. Muga silk is the coarsest of all varieties[8]. The cross-section of mulberry is triangle and for non-mulberry it is nearly rectangle. Mulberry silk has higher density and more compact structure as compared to non-mulberry silk. It has been observed that mulberry silk is finer as compared to non-mulberry silk and it has crystallites of smaller size, high molecular orientation, and a more compact overall packing of molecules. Elastic recovery behavior is superior in mulberry silk. Muga silk has the highest toughness followed by tasar silk, while the toughness is low for mulberry and eri silk[9,10]. The study on macro-characterisation and analysis of amino acid revealed that denier of the filament decreased from outer layer to inner layer. In mulberry silk, glycine, alanine and serine constitute about 82% of amino acids present and in non-mulberry it is 73% with high proportion of alanine. Amino acid analysis shows no difference in the

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chemical architecture between the outer and the inner layers of the cocoon. Density showed increasing trend in all the varieties[11].

Silk fibres undergo several kinds of heat treatments either in dry or wet state during the silk yarn production process. A study was conducted to examine the impact of different treatment temperatures, like 100°C, 110°C, 120°C, 130°C and 140°C on technological properties of tasar and muga silk during cocoon drying and subsequent reeling on a prototype multi-end reeling cum twisting machine[12]. It has been found that the heat treatment removes the moisture in the cocoon shell and pupa and alters the crystal size of raw silk. The heat treatments sometimes result in changes in the structure of amorphous and crystalline regions. However, little attention has been paid to study the structural changes induced by drying. Comparative investigation of the changes in fine structure of various kinds of silk filaments induced by heat treatment entails either fundamental or applied interests in relation to the technological problems of silk production. Tensile properties give important indications of structural variants of silk and are used widely for assessing degradation in textile substances. The main purpose of this work is to investigate tensile properties and crystallinity of tasar and muga silk at different temperatures of drying. This study is also undertaken in order to elucidate the fine structural changes, i.e. crystallinity on drying at different temperature for two varieties of wild silks, viz. tasar and muga filaments and to study the surface morphology of different dried samples.

## Experimental

Indian tasar (*Antheraea mylitta D*) and muga (*Antheraea assamensis*) degummed silk filament yarn collected from Central Silk Board, India were used for the experiments. Denier of tasar and muga silk yarn was 54.6 and 80.1 respectively. The experimental samples of tasar and muga silk yarns were conditioned at a standard atmosphere of 65 ± 2% RH and 27 ± 2°C temperature for 24 h. Tasar and muga silk filament length was measured and weighed on an electronic balance to calculate denier. Drying of silk samples was done in a drying oven with forced ventilation and positive valve control which was capable of drying the samples at required temperature. The drying temperatures for this experiment were 80°C, 100°C and 140°C. Duration of treatment was one hour. Moisture loss was calculated by finding the weight before and after drying of the samples. The samples were weighed using electronic balance immediately after drying. Specimens were conditioned at standard atmosphere of 65±2% relative humidity and 27±2°C temperature. An Instron tensile tester was used to study the tensile strength of tasar and muga filaments. The clamps of the testing machine was set in such a way so that the distance between nips of clamps along the specimen axis (including any portion in contact with snubbing surfaces) is 200±2mm with the help of preliminary specimens. The machine was set so that the specimen breaks within 20±3s. The test specimen was mounted using pre-tension of 0.50±0.05 cN/tex. The machine was operated and the test was carried out till rupture. The breaking load and elongation-at-breaks were recorded. Rigaku Miniflexıı Desktop X-ray diffraction instrument was used to study the diffraction pattern of different samples of silk. Crystallinity area was taken by tracing out the smooth curve of the graph and cutting the area under two peaks and taking weight; then whole area starting from 0º to 60º of the curve was taken out which included both crystalline area and amorphous area. The weight was taken under electronic balance and percentage crystallinity[13] was estimated. ZEISS EVO Series Scanning Electron Microscope EVO 50 was used to study the surface morphology of tasar and muga silk filament yarn.

## Results and Discussion

### Moisture Loss after Drying

Moisture loss values of tasar and muga silk after drying at 80ºC, 100ºC and 140 ºC are given in Table 1. It has been observed that the moisture loss percentage increases with the increase in drying temperature. This is due to the evaporation of moisture from the silk because of thermal energy in the form of dry heat.

### Tensile Strength and Elongation

The tenacity and elongation of tasar and muga silk samples dried at 80ºC, 100ºC, 140ºC are shown in Table 2.

Table 1 — Moisture loss of tasar and muga silks

| Temperature ºC | Weight before drying, g | | Weight after drying, g | | Moisture loss % | |
|---|---|---|---|---|---|---|
| | Tasar | Muga | Tasar | Muga | Tasar | Muga |
| 80 | 0.2790 | 0.5191 | 0.2583 | 0.4785 | 7.52 | 7.82 |
| 100 | 0.2660 | 0.6030 | 0.2450 | 0.5522 | 7.81 | 8.42 |
| 140 | 0.2300 | 0.6353 | 0.2112 | 0.5791 | 8.26 | 8.84 |

Table 2 — Tenacity and elongation of tasar and muga silks

| Temp., °C | Tenacity, g/den | | C.V., % | | Elongation, % | | C.V., % | |
|---|---|---|---|---|---|---|---|---|
| | Tasar | Muga | Tasar | Muga | Tasar | Muga | Tasar | Muga |
| Nil (control) | 3.1±0.08 | 3.7±0.08 | 11.9 | 10.8 | 20.9±0.44 | 26.1±0.44 | 9.5 | 9.5 |
| 80 | 3.0±0.08 | 3.6±0.08 | 12.9 | 17.0 | 0.1±0.60 | 223.1±0.60 | 13.5 | 13.5 |
| 100 | 3.0±0.08 | 3.5±0.08 | 13.9 | 9.4 | 20.2±0.69 | 24.3±0.69 | 15.1 | 15.1 |
| 140 | 3.0±0.08 | 3.5±0.08 | 12.7 | 8.6 | 19.3±0.64 | 24.4±0.64 | 15.2 | 15.2 |

Table 3 ─ ANOVA for tenacity and elongation values of tasar and muga silk

| Source of variation | SS | | df | | MS | | F-value | | P-value | | F critical | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Tenacity | Elongation | Tenacity | Elongation | Tenacity | Elongation | Tenacity | Elongation | Tenacity | Elongation | Tenacity | Elongation |
| Sample | 12.996 | 771.7623 | 1 | 1 | 12.996 | 771.7623 | 76.19053 | 126.9577 | 4.29E-15 | 8.65E-22 | 3.903366 | 3.903366 |
| Temp. | 0.3545 | 85.6625 | 3 | 3 | 0.118167 | 28.55417 | 0.692766 | 4.697263 | 0.557806 | 0.003637 | 2.664107 | 2.664107 |
| error | 25.927 | 923.992 | 152 | 152 | 0.170572 | 6.078895 | | | | | | |
| Total | 39.519 | 1813.335 | 159 | 159 | | | | | | | | |

Tenacity values do not change significantly with the increase in temperature of drying for both tasar and muga silk. It appears that tasar and muga silk do not degrade with the increase in temperature and even at 140°C tenacity remains unaffected. The ANOVA (Table 3) for tenacity values of tasar and muga silk shows that there is no significant difference within the samples at different temperatures of drying.

Table 2 shows a decrease in elongation when drying of silk samples is done at 80ºC in comparison to control. However, not much difference is observed at 100ºC and 140ºC drying temperature. The decrease in elongation is faster upto 80ºC due to rapid loss of moisture and then it levels off when maximum moisture is removed from the experimental samples. That is why, not much difference is observed in elongation at 100ºC and 140ºC drying temperature. The ANOVA (Table 3) for elongation shows significant difference within the sample.

The tasar and muga silk samples show difference both in tenacity and elongation when dried at different experimental temperatures (80ºC, 100ºC and 140ºC). Due to inherent characteristics of different varieties of silk, tenacity and elongation of muga silk are higher than those of tasar.

**Degree of Crystallinity**

The x-ray diffraction patterns consisting of 2θ *vs*. intensity (counts / s) of tasar and muga samples have been analysed to determine the degree of crystallinity of the dried samples. The crystallinity percentage of tasar silk and muga silk is shown in Table 4.

The crystallinity of tasar and muga control samples and samples dried at 80ºC, 100ºC and 140ºC shows no significant difference. Tasar and muga silk still remain stable even when dried at 140˚C without any degradation and there is no significant difference in the crystallinity due to different temperature of drying. Silk is a thermally stable fibre, with a glass transition temperature of 175ºC and thermal degradation initiates at about 275 ºC (ref. 14). Hence, it is in line with the earlier study and there is no significant change in crystallinity at 140ºC.

Table 4 — Crystallinity percentage of tasar and muga silk

| Drying temperature, ºC | Crystallinity, % |
|---|---|
| **Tasar** | |
| Control sample | 42.6 |
| 80 | 39.4 |
| 100 | 40.7 |
| 140 | 38.0 |
| **Muga** | |
| Control sample | 43.2 |
| 80 | 37.3 |
| 100 | 40.7 |
| 140 | 38.5 |

**SEM Study**

Surface structure of tasar and muga silk has been investigated. Scanning electron micrographs of tasar and muga samples at different drying conditions are depicted in Fig. 1.

The change in surface structure for tasar and muga silk at 80ºC, 100ºC and 140ºC in comparison to control corroborates that there is no surface modification or degradation at different conditions of drying. Hence, both tasar and muga silk can be safely processed upto 140 ºC without any degradation in their structural morphology.

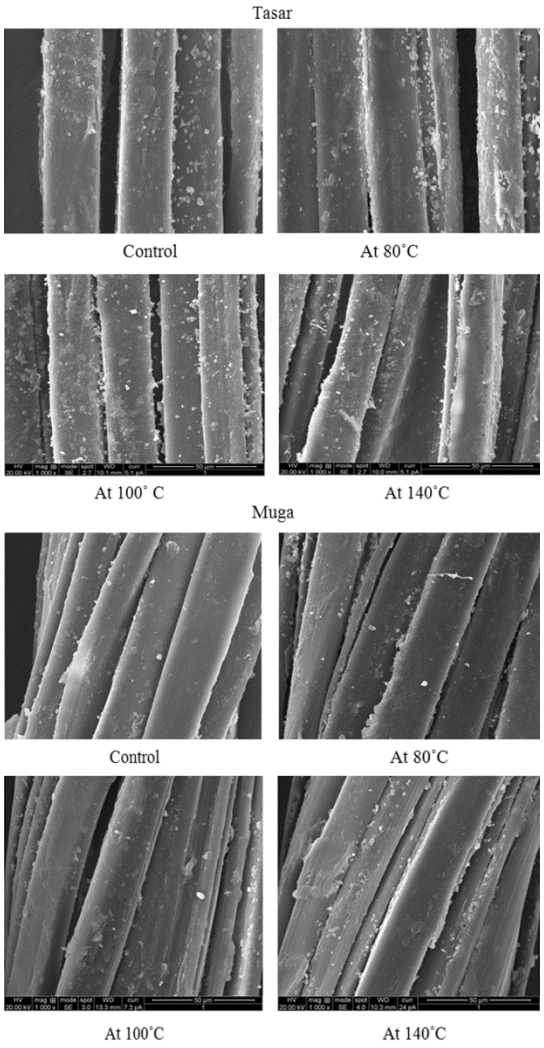


Fig. 1— SEM images of tasar and muga silk

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