CHAPTER 10

SUMMARY AND CONCLUSIONS

10.1 SUMMARY

The major objective of this study is to investigate the morphological and mechanical properties of silk fibres subjected to various physical and chemical treatments. The literature pertaining to silk fibre emphasizes the use of chemical modifications in overcoming some of their deficiencies. Unlike other fibres, not much work has been reported on the effect of physical treatments on silk fibres. Although a considerable amount of work has been carried out involving chemical treatments of silk fibres, there is a need to continue the studies in some of the areas. The areas considered in this study were partial degumming, treatments with different types of degumming agents and common reagents used on silk fibres. Also, fixation or insolubilization of sericin present in raw silk, which is gaining importance in these days, was included in this study. The effect of physical modification of silk fibre brought out by stretching the yarn was also considered. The stretching was carried out in the presence of water followed by drying both in the slack and taut conditions, at room temperature. The effect of these treatments on the properties of mulberry silk yarn was investigated. Some aspects of these treatments on the properties of tasar silk yarn were also considered.

The properties studied for the treated mulberry silk yarns include linear density, tensile properties, breaking twist angle, fatigue and abrasion resistance. Microcrystalline parameters of yarns subjected to stretching and reagent treatments was determined using wide angle x-ray diffraction
technique. The study on partial degumming was supplemented with infrared spectroscopy to evaluate the effect of degumming on the chemical nature of fibroin and sericin present in the silk. Fracture analysis of fatigue broken specimens carried out using scanning electron microscopy was included.

For tasar silk yarn, only the stretching followed by drying treatments were carried out and the effect of these agents on microcrystalline parameters alone was investigated. Due to the non-uniformity present in this yarn (it being available as a hand reeled yarn), other treatments and their effect on the yarn properties were not considered.

The results obtained on the various properties as a result of the treatments have been discussed in the earlier chapters. The conclusions drawn from these studies are presented in the following section. Unless otherwise stated, the term silk refers to mulberry silk.

10.2 CONCLUSIONS

a) In respect of the effect of partial degumming on the silk yarn properties, the following conclusions are drawn:

1) The excellent correlation that exists between the gum loss and degumming duration shows that the rate of gum removal is constant. This indicates that the different layers of sericin present around the raw silk behave alike during degumming.

2) The sericin gum plays an important role on the tensile properties of silk yarn. The tenacity, breaking extension and initial modulus continue to drop with increasing gum loss levels.
3) The infra-red spectroscopy reveals that the degumming operation does not affect the chemical nature of sericin and fibroin up to 16% gum loss studied.

4) The study on breaking twist angle indicates that the removal of gum results in lowering the yarn brittleness. The reduction in brittleness does not improve with increasing gum loss.

5) The fatigue life of the partially degummed yarns is higher than that of the raw silk yarn; however, among the partially degummed yarns, fatigue life drops with increasing gum loss.

6) The abrasion resistance of the silk yarn improves on gum removal but yarns with different gum loss levels behave alike.

b) Following are the conclusions drawn from the study on the effect of different types of degumming agents on the properties of silk yarn:

1) Treatments with sodium silicate, sodium carbonate and sodium hydroxide have resulted in an almost equal weight loss (19.5 ± 0.5%), when treated at a pH of 10.5 for 15, 60 and 120 minutes respectively.

2) The effect of all these agents on yarn properties such as tensile, breaking twist angle, fatigue life and abrasion resistance is similar. It reveals that for degumming of silk, any of these can be used, provided the process conditions mentioned above are followed. Among the three agents, sodium silicate requires the least degumming duration.
c) The conclusions drawn from the study on the effect of reagents namely, formic acid and zinc chloride on silk yarn are listed below:

1) Treatment with these agents results in a weight loss. The loss in weight increases with increasing concentration but the increase is only marginal.

2) The lower concentration (5% w/v) of formic acid and zinc chloride does not affect the tenacity and breaking extension.

3) At higher concentration (20% w/v), formic acid does not affect the tenacity and breaking extension while zinc chloride decreases the tenacity and breaking extension.

4) Both the agents increase the initial modulus of the silk yarn.

5) Brittleness increases when treated with higher concentration of these reagents, while a lower concentration does not have an effect. In this respect, zinc chloride has a more pronounced effect than formic acid.

6) The fatigue life is reduced due to the action of these reagents. The reduction is greater at a higher concentration, with zinc chloride having a greater effect than formic acid.

7) The abrasion resistance improves with a lower concentration of these agents. However, at a higher concentration the trend is reversed.
d) The effect of stretching treatment followed by drying in both the slack and taut conditions gives rise to the following conclusions:

1) The increase in length caused by stretching is found to have a good correlation with stretch levels in both the methods of drying.

2) The tenacity of the stretched yarn increases with increasing stretch levels.

3) The improvement in tenacity due to stretching is quite substantial in tension dried than in slack dried samples.

4) Irrespective of the method of drying, the breaking extension initially decreases and then increases with the increase in stretch levels. However, the breaking extension at the highest stretch level is lower than that of the unstretched yarn.

5) The breaking extension of tension dried samples is lower than that of the slack dried samples at all stretch levels.

6) With increase in stretch levels, the initial modulus of the stretched yarns increases initially and decreases thereafter, irrespective of the mode of drying. However, the initial modulus at the highest stretch level is still higher than that of the unstretched yarn.

7) The initial modulus of the tension dried samples is higher than that of the slack dried ones at all stretch levels.
8) The stretching treatment increases the brittleness of the silk yarn. The drying methods and the stretch levels have the same effect on brittleness.

9) The stretching of silk yarn and drying at both the slack and taut conditions does not affect the fatigue life. The drying methods have the same effect on the fatigue life at all stretch levels.

10) The abrasion resistance of the silk yarn improves at lower stretch levels, whereas, at higher stretch levels, the abrasion resistance becomes lower than that of the unstretched yarn.

e) The conclusions drawn from the sericin fixation treatments are as follows:

1) The sericin fixing agents namely, glutaraldehyde and tannic acid used in 1% and 3%, 3% and 1%, and 3% and 3% w/v combinations are capable of fixing sericin in the raw silk yarn.

2) Improvement over the weight of raw silk occurs when glutaraldehyde and tannic acid are used in 1% and 3% w/v combination.

3) The use of 3% w/v each of the fixing agents does not affect the tenacity, whereas, the other two combinations lower the tenacity.

4) The fixation treatment does not affect the breaking extension but decreases the initial modulus.
5) The sericin fixed yams have lower brittleness than raw silk.

6) The fixation of sericin in raw silk improves the fatigue life and abrasion resistance.

f) The paracrystalline parameters obtained using the Fourier analysis of the scattered x-ray reflections for the (201) reflection of the silk samples are quite reliable.

The stretching followed by drying in both the slack and taut conditions of mulberry and tasar silk yarns improves the crystal size. Treatment of the mulberry silk yarn with reagents also improves the crystal size, which is attributable to the leaching of amorphous content from the fibre.

The lattice distortion of all samples varies between 2-9% in the (201) direction.

Minimum enthalpy of treated samples varies from 0.03 to 0.14, which is also true in the case of man-made fibres.

g) The breaking twist angle and fatigue life of all the treated samples tested in water are lower than those in air.

h) The breaking twist angle exhibits a good correlation with the breaking extension (negative) and initial modulus (positive).

i) The fatigue life exhibits a good (negative) correlation with the bending rigidity and breaking twist angle except in the case of stretching treatment.

j) The mode of fatigue fracture as revealed by scanning electron micrographs resembles that of nylon 6.6 and polyester fibres.
10.3 TECHNOLOGICAL RELEVANCE OF THE PRESENT STUDY

The results obtained in the study have the following practical implications.

1. The study on partial degumming of silk clearly shows that the properties of yarns with lower gum loss levels are better than the yarns with higher gum loss levels. So, the advantages offered by the sericin gum can be made use of by appropriately altering the gum loss levels depending on the end use.

2. The reagents formic acid and zinc chloride are used in dyeing and finishing of silk textiles. This study reveals that higher concentrations of these agents have a detrimental effect on the properties of silk. So, it is recommended to have a close monitoring of the concentration of these agents during their applications.

3. Stretching in the presence of water followed by drying, either in slack or taut conditions, hampers the properties of silk studied. It emphasizes the need for careful monitoring of the tension in the yarns and fabrics during silk processing in the presence of water. Tensioning beyond the required level should be avoided under any circumstances.

4. The good relationship of BTA with breaking extension, initial modulus and fatigue life indicates that the BTA test can be included along with the other tests in evaluating the performance of silk fibres.
10.4 RECOMMENDATIONS FOR FUTURE WORK

1. It has been found that the degumming agents namely, sodium silicate, sodium carbonate and sodium hydroxide have the same effect on the properties studied. Among these three agents sodium silicate requires the least degumming duration. It will be worth extending the study on the effect of sodium silicate on other properties not included in this study, so that its usefulness to the industry can be assessed.

2. It would be worthwhile investigating the effect of the various treatments on properties like birefringence, density, modulus of rigidity, moisture absorption and friction.

3. Visco-elastic properties such as creep and stress relaxation and dynamic mechanical properties such as dynamic modulus and tan δ are to be investigated to have a better understanding of the structure/property correlation.

4. This study can be extended to other types of silks namely, tasar, eri and muga.

5. An in-depth study of the effect of temperature and relative humidity on the properties of various types of silk is warranted.

6. The chemical treatments used for improving the resistance to crease formation and photo degradation of silk is worth investigating for their effect on various properties.

7. The effect of various treatments on the properties of fabrics having different construction parameters will be worth studying.