CHAPTER – 1

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Food, shelter and clothing are the three basic needs of human. Based on these three radical needs, over the centuries the human civilization has acquired varieties of other essential commodities so as to avail a secure, comfortable and enriched life. The development of science and technology and simultaneous enhancement of knowledge based techniques have paved the paths towards production of qualitatively and quantitatively improved materials. Over the centuries, the advancement in clothing materials is as significant as the development in nutrition and accommodation. Besides providing physical support around the body, clothing has another unique role of maintaining the cultural domain of a civilization. Clothing undertakes a range of socio-cultural roles, such as individual, occupational and sexual differentiation. Moreover, the modesty and beauty that is provided by clothing is worth mentioning.

Fibres are the basic structural components of clothes. Fibres are woven or knitted together to form fabrics. There are two types of fibres, natural and man-made. Natural fibres are obtained from natural resources such as animals and plants, while man-made or synthetic fibres come from chemical resources. The natural fibres include natural cellulosic fibres (Seed fibres: cotton, kapok; Bast fibres: jute, hemp, ramie, flax, bamboo; Leaf fibres: agave, pineapple,
abaca and Nut fibres: coir), *protein fibres* (Animal secretion: wool, silkworm silk, spider silk and Animal-hair fibres: hair, fur fibres) and *mineral fibres* (Asbestos). Man-made fibres are either based on natural polymers (e.g. viscose rayon or cellulose acetate) or synthetic polymers (e.g. polyolefines, polyacrylonitrile, polyamide or polyester fibres).

Fibres come in one of the two forms based on its length; a *filament* is a fibre of continuous length of several hundred meters, while a *staple* is a fibre of limited length ranging from about one-quarter of an inch to many inches in length (Miller, 1969). In general, filaments are used to make very fine and thin fabrics while staples are used to make rough and thick fabrics.

Silk is a kind of natural protein fibre prevalent in the human civilization for a long time primarily as a prestigious textile material. Silk is produced by some spiders and insects, like silkworms. Silkworms produce silk in the form of cocoons. Silk cocoons have been used as a source of silk for producing exquisite textiles and dress materials. According to Confucius (551 B.C. - 479 B.C.), it was in 2640 B.C. that the Chinese princess Xi Ling Shi was the first to reel a cocoon of silk which had dropped accidentally into her cup of tea. From that historic moment, the Chinese discovered and continued the use of silk and silkworm. The Chinese realized the value of the beautiful material they were producing and kept that secret very safely from the rest of the world. As a result, demand for this exotic fabric eventually increased and created a lucrative trade route for its export to other central Asian and European countries known
as the 'Silk Road'. Remarkably, for the next 3000 years China successfully kept their monopoly of silk production over the world. But finally in the 3rd Century B.C. sericulture (i.e. the cultivation of silkworms) began to find their way into Asia and then to the rest of the world. Over the past centuries silk has attained a reputation of luxury and beauty unmatched by other textile fibres. Despite the great advances made by the synthetic fibres in terms of quality and quantity, silk still retains its supremacy among the textile fibres because of its unique characteristics.

Biologically, silk is a very fine strand of animal fibre i.e. a solidified protein secretion produced by certain Arthropodan invertebrates such as silkworms, spiders, scorpions, mites, flies and aquatic insects of caddisfly (Kaplan et al., 1992; 1994; 1998; Altman et al., 2003). These invertebrates use it for a variety of functions, including formation of protective shelters (cocoons), capturing food (webs), structural support, reproduction, foraging etc. (Craig, 1997).

Although many insects produce silk, traditionally, silk fibers have been extracted from cocoons produced by Bombyx mori silkworms and to a relatively small extent from the wild silkworms belonging to the Saturniidae family. It was the cocoons of Bombyx mori silkworm that were used by the Chinese to produce silk in ancient times. Silk from silkworms has been used for thousands of years in textiles and for almost a century as suture material. Although silkworm silk has impressive mechanical properties, in recent years, spider silk
has attracted considerable attention due to its excellent resilience and mechanical properties, like high strength, high elasticity and high Young’s modulus. It has long been recognized that spider silks exhibit unique combinations of strength, stiffness and toughness which are unrivaled by all other silks and synthetic fibrous polymers. Spider silk has been claimed to be the toughest natural material (O’Brien et al., 1998; Craig, 2003). The most extensively investigated spider silk is dragline silk and it is found to possess the highest tensile strength among natural fibres.

The life of the spiders is dominated by silk (Craig, 2003). Virtually every aspect of their existence involves silk in one way or another. They use silk webs for capturing food, silk in their nest construction, silk egg-sacs in their reproduction and silk threads to carry them on the wind. Despite promising properties, spider silk has not been commercialized owing to the relatively low level of production. Spider farming is not viable due to the highly territorial and cannibalistic nature of these insects; there arises difficulty in breeding spiders, reeling of the dragline silks as well as obtaining the silk from the egg sac. Spider silks have been in applications for more than thousands of years. It has effectively made its use as fishing nets as well as in useful devices such as telescopes and guns. It is now successfully used in biomedical fields such as in making bandages and non-allergenic sutures (Colgin et al., 1995; Mayes et al., 1998) and making artificial tendons and ligaments for supporting weak blood vessels. It is also tried in ropes and parachutes due to its high tensile strength.
Silk produced by silkworms may be classified into two main types—mulberry silk or cultivated silk produced by *Bombyx mori* silkworm and non-mulberry or wild silk produced by the silkworms of ‘tasar’, ‘muga’, ‘eri’ etc. The mulberry silkworm is monophagous i.e., it solely feeds on the leaves of mulberry plant (*Morus* spp.) while the non-mulberry silkworms feed on the leaves of different types of plants. Mulberry silk is by far the most known silk in the world, constituting about 90% of all silk produced worldwide. India holds a unique distinction in producing three commercially important varieties of non-mulberry silks, viz. ‘eri’, ‘tasar’ and ‘muga’ silks along with mulberry silk.

Functionally, silk cocoons are the silken container that the larvae spin around its body during morphogenesis to protect themselves from microbial degradation and predators (Zhao *et al*., 2005). In most cases, the cocoons have been exploited for production of silk by interrupting this metamorphosis stage.

The silk fibre is synthesized in the silk glands of the silkworm. Silk glands are the modified paired labial glands. When the silkworm secretes the liquid silk during spinning, it is passed through the anterior gland and expelled out through the spinneret opening situated at the mouth of the insect (Shimizu, 2000).

Silk fibre consists of two types of proteins, *fibroin* and *sericin*. The fibroin is the highly crystalline core component of silk fibre produced from the posterior section of the silk gland. It contains two fibroin proteins, light-chain (25 kDa) and heavy-chain (325 kDa) (Horan *et al*., 2005). The principally
amorphous protein sericin is a minor component produced in the middle section of the silk-gland (Sehnal and Akai, 1990; Fedic et al., 2002) that serves as a glue-like coating on the two fibroin cores thus concealing the unique luster of fibroin. Both fibroin and sericin contain same 18 amino acids, although these exist in different amounts (Sonthisombat and Speakman, 2004). Fibroin is insoluble even in hot alkaline solution whereas hydrophilic sericin is easily hydrolyzed in hot water or alkaline soap solution (Sadov et al., 1978; Gulrajani, 1988).

Although, the amounts of sericin and fibroin in the silk fibre exist roughly as 25% and 75% respectively, these amounts vary from season to season, place to place and also with the races of the silkworm. Percentage of sericin and fibroin reflect the quality of the silk fibre (Basavaraja et al., 2000). Silk having more sericin percentage are not considered economical as these result in the lower yield of fabric (Choudhury, 1981). The sericin percentage has relationship with the silk manufacturing industry and raw silk products (Rui, 1998). Sericin also plays an important role in cocoon processing (Kannan, 1986).

The sericin envelops the fibroin fibre with successive sticky layers to help in the formation of a cocoon. It is the silk sericin and other impurities that mask the luster of the silk fibroin and cause hardness and coarseness of raw-silk texture. Therefore ‘degumming’, the process of removing sericin and impurities (e.g. waxes, fats and mineral salts) is applied for improving the sheen, colour
and texture of silk fibre. Another important purpose of degumming is to make the fibre highly absorbent for dyes and chemicals (Saligram et al., 1993). In some cases, the fabric is woven to completion and then degummed to protect the yarn from abrasion on the loom.

The word 'degumming' has been derived from the French word 'degommage'. Sericin contains the same amino acid residues as fibroin but the proportions contained in both components are quite different. So sericin and fibroin differ considerably in their chemical composition as well as accessibility. Sericin, being a resinous polymer present on the surface of silk fibroin, is comparatively easily accessible to degumming agents. However, the degumming process must be carefully carried out in appropriate conditions so that the fibroin is not damaged.

Various degumming methods provide different conditions of pH, temperature and duration of treatment. But alkaline conditions are more preferred than the acidic conditions. Moreover, as silk is not damaged around 100 °C, boiling of silk fibres is common in most of the degumming processes.

All the commercial and traditional degumming methods are theoretically based on the ability of degumming agents to attack on the specific peptide bonds formed by the major amino acids of sericin. However, except the commercial degumming methods, the traditional degumming methods have not been studied in systematic way and so the mechanisms involved in those
processes are not well established (Gulrajani, 1992). Moreover, there is lack of standardization of the traditional degumming methods.

Different types of chemicals including alkali and acid and other natural materials are used as degumming agents for silk (Choudhury, 1981). These include sodium carbonate, ‘kolakhar’, ‘ritha’, cowdung, papain etc.

Degumming with sodium carbonate (washing soda, Na$_2$CO$_3$) is the most conventional method used to degum non-mulberry silks. Its low price and easy availability has made it a preferred degumming agent. ‘Kolakhar’, a traditional food additive derived from banana plants, is used as a degumming agent for silk, particularly ‘muga’ silk in Assam of North Eastern region of India. Dried fruits of ‘ritha’ produced by the soapnut (Sapindus mukorossi) plants are traditionally used to wash silk fabrics. Cowdung has been tried as a degumming agent by some earlier workers as the microbial cluster of cowdung is known to produce proteolytic enzymes. Papain, a protease enzyme which degrades proteins by hydrolysis of peptide linkage has been tried successfully as a degumming agent in mulberry as well as non-mulberry silks. It is reported that papain hydrolyzes only sericin but does not damage fibroin.

The feasibility of different degumming agents and methods on silk is very important. Non-standardized degumming processes may result not only in sericin dissolution but also in more or less pronounced decomposition of fibroin. So it should be taken care that the silk (particularly fibroin) does not lose its strength after degumming. On the other hand, good degumming
practices may retain the physical properties of the silk intact. So it is likely that different degumming agents and methods affect differently on the properties of the silk fibre.

The quality of the degummed silk fibres can be measured with the variation in the physical and biochemical properties possessed by these. The physical properties include density, fineness, water sorbency, dynamic contact angle, tensile strength, thermal behaviour, crystallinity etc. The biochemical properties include the sericin percent, different amino acids in the protein structure etc.

The density of silk fibres tends to be relatively low as compared to other textile fibres, making fabrics woven from yams of these fibres feel comparatively light. Density and denier of the silk fibres are related to each other, which influences the other mechanical properties and fibre quality of silk. As the denier or linear density of a fibre is inversely proportional to its tensile properties, silk fibres with low denier have high tensile strength. Water sorbency helps to draw an idea about the water relation of the fibre on treatment with water. Dynamic contact angle indicates the hydrophilic or hydrophobic nature of the fibre, as high contact angle is seen in case of hydrophobic fibres. The cross-sectional view as well as longitudinal view by SEM helps to study the morphological structure of the fibres in greater detail. Different types of fibres may have different shapes in longitudinal view and cross-sectional view under SEM.
The tensile properties are the most important mechanical properties of fibres which include their behaviour under forces and deformations applied along the fibre axis. The tensile properties include breaking strain, breaking tenacity, Young's modulus and toughness. The strain of a fibre is the ratio of its elongation under applied force to the initial length. The stress is the ratio of force applied to the cross-sectional area of the material. Tenacity is actually the mass stress at break and it is the ratio of the force applied at break to the linear density (denier) of the fibre. The slope of the initial linear portion of a stress-strain curve is called Young's modulus. Toughness (or work of rupture) is the energy needed to break the fibre (Booth, 1996).

Fourier Transform Infrared (FTIR) Spectroscopy, X-ray Diffraction (XRD) are used to study the structural properties of the fibres. FTIR spectroscopy provides a powerful way of examining protein secondary structure in the fibroin molecule of the fibre; it indicates the presence of different functional groups in the molecule. The position and intensity of the bands formed by these functional groups are sensitive to the molecular conformation (Magoshi, 1974; Tsukada, 1986; Freddi et al., 1997; Kweon et al., 1999, 2000; Kweon and Park, 2001). XRD is generally used to study the crystalline structure of the material. It gives a fair idea of the amount of crystalline and amorphous part in the fibre.

The thermal properties of the fibres can be studied with a lot of techniques, viz., differential scanning calorimetry (DSC), thermogravimetric
analysis (TGA) and differential thermogravimetric analysis (DTGA). The DSC thermograph indicates the change in enthalpy (heat released or absorbed by the sample) when heat is applied to the silk fibre. TGA and DTGA are used to evaluate the thermal stability (weight loss) of the fibre with increase in temperature up to its decomposition.

The silk fibres are constructed from amino acids that are cross-linked and generally oriented parallel to the fibre axis. This is referred to as a crystalline chain structure, and this structure is responsible for the strength of silk fibres. The variation in concentration of different amino acids present in the proteinous silk fibre is reported to affect the mechanical and thermal behaviour of the fibre. Some of the amino acids in silk fibre are chemically reactive and so silk fibre shows response when it comes in contact with strong organic solvents. The effects are noticeable with the change in its mechanical properties, especially the tensile properties.

North East (NE) India, a mega biodiversity hotspot, is incredibly rich in silkworm biodiversity. The forests of NE India, particularly Assam, provide home for a lot of commercially exploited non-mulberry silks along with the mulberry silk. These include ‘muga’ (*Antheraea assamensis* Hefler), ‘tropical tasar’ (*Antheraea mylitta*) and ‘eri’ (*Philosamia ricini*). Moreover, a number of wild silkworms, viz., *Antheraea frithi*, temperate tasar (*Antheraea proylei*), ‘kotkari muga’ (*Attacus atlas*), ‘amphutukoni muga’ (*Cricula trifenestrata*) are also available.
The semi-domesticated silkworm variety ‘muga’ silkworm, *Antheraea assamensis* Helfer (Lepidoptera: *Saturniidae*) is an economically important insect which produces golden yellow hued silk. Its habitat is strictly restricted to the NE region of India, especially the state of Assam and its neighboring regions (Rao, 1978; Choudhury, 1981). It is named after Assamese word "muga" which indicates the amber (brown) colour of cocoon. Being polyphagous in nature, it feeds on leaves of woody trees like som, sualu *Machilus bombycina* King, *Litsaea polyantha* A. Juss, *L. citrata* Roxy, *L. salicifolia* Roxy and many other host plants belonging to the family Lauraceae. It is multivoltine in nature having 5-6 generations in a year. Muga silk is highly durable and has high value and demand in the national and international market (Freddi *et al.*, 1994).

*Antheraea mylitta*, a Lepidopteron insect of the *Saturniidae* family produces silk of commercial importance which is known as tasar silk. This species is endemic and distributed in different geographical regions of India in the form of ecological races. They show variation in their phenotypic traits such as fecundity, voltinism, cocoon weight, silk ratio and also in their host plant preference (Sinha *et al.*, 1994).

Oak tasar *Antheraea proylei* (Lepidoptera: *Saturniidae*) is commonly known as a polyphagous insect which feeds on the oak plants, viz. *Quercus serrate*, *Q. incana*, *Q. semicarpifolia* etc. This semi-domestic insect is
multivoltine in nature. The colour of the silk produced by these silkworms are brownish yellow and as fine as the ‘muga’ silk.

*Antheraea frithi* (Lepidoptera: *Saturniidae*) is a multivoltine insect belonging to the tasar group. It feeds on the leaves of Sal (*Shorea robusta*), phutuka (*Melastoma malabathricum*) etc. It produces brownish yellow coloured cocoons. This is a wild tasar silk available mostly in Sal forest of NE region of India.

The name ‘eri’ is derived from the Assamese word ‘era’ which denotes the castor-oil plant (*Ricinus communis*) the main food plant of this silkworm. Eri silkworm, *Philosamia ricini* (Lepidoptera: *Saturniidae*) is multivoltine, polyphagous and feed on a wide range of host plants. It is found widely in various parts of NE India.

The *Attacus atlas* (Lepidoptera: *Saturniidae*) silkmoth is primarily found in tropical forests and surrounding lowlands in the vicinity of their host plants. The larvae feed on a variety of host plants including; Cinnamomum, Citrus, Salix, Annona, Clerodendrum and Mussaenda. It is bivoltine and produces large cocoons. The expanded winged moth is largest among the insects in Asia.

*Cricula trifenestrata* (Lepidoptera: *Saturniidae*) produces bright yellow golden cocoons with an open fibre network (Situmorang, 2002). The cocoons produced are small in size and have numerous perforations on the surface of the
cocoon. In Assam it is considered as a pest on the food plants of ‘muga’ silkworm, because it is voracious eater of Som plants.

In spite of a large number of man-made fibres overwhelming the textile arena, the natural silk still commands a considerable respect as its emotional and prestigious aura is incomparable. In Assam, the ‘muga’ silk has aesthetic value and is traditionally used in occasions and festivals, like ‘Bihu’, ‘puja’, marriage etc. Silk industries in this region are home-based and are livelihood for lakhs of people. Apart from ‘eri’, ‘muga’ and mulberry silk, other wild silks of tasar (A. mylitta, A. proylei and A. frithi), kotkari muga (A. atlas), amphutukoni muga (C. trifenesatra) are found in the forest in wild form throughout the NE region. Exploitation in commercial scale could be a support to the local farmers to boost their livelihood.

It was not until recently that scientific community realized the tremendous potential of non-mulberry or wild silks. Sporadic study has been done regarding the physical and mechanical properties of these silks and only a few reports are available till date (Jolly et al., 1979; Iizuka, 1985; Sonwalkar, et al., 1989; Baruah, 1991; Iizuka, 1993; Iizuka et al., 1993 a, b, c; Freddi et al., 1994; Iizuka 1994; Das, 1996; Iizuka and Itoh, 1997; Rajkhowa, 1998; Das et al., 2005). However the wild silks A. proylei, A. frithi, A. atlas and C. trifenesatra of NE India have not been studied systematically.
Attempt has been made to study the structural variants of non-mulberry silks of NE India with the following objectives:

- To apply various degumming methods on non-mulberry silks.
- To use different chemical agents on conventionally degummed silk fibres.
- To study the tensile properties of all the silks treated as above.

To achieve the above objectives, the morphological characters of the cocoon and physicochemical properties of fibres were also studied.

This effort will provide insight into the multidimensional aspects of silk i.e. cocoon characters, sericin content, degumming, mechanical properties, tensile properties, thermal behaviour, molecular conformation, crystallinity and change of tensile properties after treatment with chemical solvents. It is expected that this study will help to generate information on the unexplored silks of NE India other than ‘muga’ and ‘eri’ which could be effectively used in textile and as biomaterial.