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Indian
Journal of Advances in
Chemical Science

Indian Journal of Advances in Chemical Science S1 (2016) 183-189

Waste Silk Fiber Reinforced Polymer Matrix Composites: A Review

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Received 14th March 2016; Revised 14th May 2016; Accepted 16th May 2016

ABSTRACT

Natural fibers have recently become attractive to researchers, engineers and scientists as an alternative reinforcement for fiber reinforced polymer composites (FRPCs). Due to their low cost, fairly good mechanical properties, high specific strength/modulus, nonabrasive, eco-friendly, and biodegradability characteristics, they are exploited as a replacement for the synthetic fiber, such as glass, aramid, and carbon. Silk is one of the most precious, fibers among all textile fibers and it has a very wide range of uses such as sewing threads, clothes, home/technical textiles, and ornamental uses. The cocoons which are damaged during the cocoon-forming process are called waste silk, and because of the damage their trading value is decreased. These wastes can be reused in textiles as well as an effective reinforcement with various polymers in the fabrication of composite parts for automotive applications. This paper reviews the literature reports base on waste silk FRPCs using various silk waste fibers. Various processing methods and conditions; liquid molding, compression molding process, and injection molding are used in the polymer matrix composites productions. The mechanical properties of these waste fiber reinforced polymers are mainly influenced by the interfacial adhesion between the matrix and the fibers. Several chemical modifications are employed to improve the interfacial matrix–fiber bonding resulting in the enhancement of mechanical properties of the composites. In general, the tensile strengths of the natural FRPCs increase with fiber content, up to a maximum or optimum value, the value will then drop. However, the Young's modulus of the natural FRPCs increase with increasing fiber loading. Characterization challenges associated with the waste silk fiber reinforced thermoplastic and thermoset composites productions were also examined. Thus, the findings of this research review can be used as a data base for further inquiring into the waste silk FRPCs in a view to enhance the development of the automotive sector.

Key words: *Waste silk fiber, Silk fiber reinforced polymer composites, Processing, Structure, Mechanical properties.*

1. INTRODUCTION

In the past decades, Greenpeace groups and NGOs in various countries have increasingly addressed the environmental impact of the chemical substances, fully recognized as a global issue. As a consequence, this awareness is pushing governments toward more stringent legislation, which promotes the preservation and protection of the quality of the environment for future generations. Natural fibers which have advantages of being economical to manufacture, eco-friendly, harmless to health, lightweight, high stiffness, and specific strength provide a possible alternative to the synthetic fibers [1-3]. Biocomposites reinforced with plant fibers such as flax, jute, and hemp have been widely investigated in literature as potential eco-friendly alternatives to synthetic fiber reinforced composites [4-7]. It will be at used in some applications where a short life of the product is advantageous. In recent times, the natural fiber composites have had huge growth in the automobile industry due to the advantages of renewability, reduced emission of pollutants, and improved fuel efficiency because of reduced weight of the components [8-10].

Table 1 provides the comparison of the economic, technical, and ecological properties of silk, plant, and glass fibers**.** Plant-based natural fibers such as flax, jute, hemp, kenaf, and sisal have been more frequently used and extensively studied because of their natural abundance, cost effectiveness, high annual production, and a wide range of properties depending on the

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Table 1: Comparison of the economic, technical, and ecological properties of silk, plant, and glass fibers.

Properties	Silk fibers		Plant fibers		Glass fibers
Economy					
Annual global production of fibers (tons)	150,000		31,000,000		4,000,000
Distribution of fibers for FRPs in EU (tons)	0		60,000		600,000
Cost of commercial raw fiber (≤/kg)	2.0-30.0		0.5-1.5		1.3-20.0
Chemical nature	Proteinaceous		Lignocellulosic		Silica-based
Fiber length	Continuous		Discrete		Continuous
Fiber diameter (apparent) (Lm)	1-15	(8-15)	15-600	(15-30)	5-25
Density (g cm^{-3})	1.25-1.35		1.35-1.55		2.40-2.70
Moisture absorption (%)	5-35	(20-35)	7-25	(7-10)	0-1
Tensile stiffness (GPa)	5-25	(5-15)	30-80	(50-80)	70-85
Tensile strength (GPa)	0.2-1.8	(0.3-0.6)	0.4-1.5	(0.5-0.9)	2.0-3.7
Specific tensile stiffness (GPa/g cm^{-3})	4-20	(4-12)	20-60	(30-60)	27-34
Specific tensile strength (GPa/g cm^{-3})	0.1-1.5	(0.3-0.7)	0.3-1.1	(0.3-0.7)	0.7-1.5
Tensile failure strain (%)	15-60	(15-25)	2-30	(2-4)	2.5-5.3
Toughness (MJ m^{-3})	25-250	(70)	5-35	(7-14)	40-50
Specific toughness (MJ m^{-3}/g cm^{-3})	20-185	(50-55)	3-26	(4-10)	16-19
Abrasive to machines	No		No		Yes
Ecological					
Renewable source	Yes		Yes		No
Recyclable	Yes		Yes		Partly
Biodegradable	Yes		Yes		No
Hazardous/toxic (upon inhalation)	No		No		Yes

FRPs=Fiber reinforced polymer

plant source. Most of the published articles about biocomposites have been focused on these plant-based natural fibers. On the other hand, only a few papers on biocomposites utilizing animal-based natural fibers like silk have been reported.

Most recently, some fundamental results on a variety of properties of novel silk-reinforced biodegradable polymer matrix biocomposites were investigated. It has been found that raw silk fibers (*Bombyx mori*) play an effective role as reinforcement in greatly improving the mechanical and thermomechanical properties of unreinforced polymer biocomposites. The literature stressed that the tensile and flexural properties of the biocomposites fabricated using the raw silk fibers are comparable to those of the biocomposites with plant-based natural fibers, suggesting that the use of silk fibers as reinforcement may be a potential candidate for enhancing the properties and performances of biodegradable polymer matrix resins [11].

Silk fibers have advantages over plant-based natural fibers, such as uniform fiber properties, continuous fiber type, high toughness, high crystallinity, and high tensile strength. However, *B. mori* raw silk fibers spun out from silkworm cocoons are relatively expensive. Therefore, they would not be cost-effective compared with plant-based natural fibers. However, waste silk fibers or scrap silk fibers, originated from *B. mori*, can be industrially available at extremely low cost. A large volume of the fibers may be discarded as scrap at the later stage of manufacturing process of silk fabrics. Such scrap fibers may be considered as industrial waste. Therefore, they have also been increasingly challenged as recyclable resource.

The overall objective of this review paper is ultimately to elucidate the performance, to explore the effect of waste silk fiber content and length on the mechanical and thermal properties of the biocomposites and potential application of waste silk reinforced polymer biocomposites.

2. COMPOSITION, PROCESSING, AND PROPERTIES OF SILK FIBER AND ITS WASTES

Composition, structure, and material properties of silk fiber produced by spiders, silkworms, scorpions, mites, and flies may differ widely depending on the specific source and the uncontrollable reeling conditions of those insects. Silk fibroin film has good dissolved oxygen permeability in wet state but it is too brittle to be used on its own when in dry state; whereas for chitosan, it is a biocompatible and biodegradable material which can be easily shaped into films and fibers. Park *et al*. and Kweon *et al*. [12,13] introduced an idea of silk fibroin/chitosan blends as potential biomedical composites as the crystallinity and mechanical properties of silk fibroin are greatly enhanced with increasing chitosan content.

Cocoons are natural polymeric composite shells made of a single continuous silk strand with length in the range of 1000-1500 m and conglutinated by sericin. This protein layer resists oxidation, is antibacterial, ultraviolet resistant, and absorbs and releases moisture easily. Since this protein layer can be cross-linked, copolymerized, and blended with other macromolecular materials, especially artificial polymers, to produce materials with improved properties. In average, the cocoon production is about 1 million tons worldwide, and this is equivalent to 400,000 tons of dry cocoon. Raw cocoon silk and side view of the silk fiber is shown in Figure 1.

Normal compact cocoon exhibits a high ability of elastic deformation with an elastic strain limit higher than 20% in both longitudinal and transverse directions. Anisotropic properties mainly due to the nonuniform distribution and orientations of silk segments and the inner layer of cocoon has low porosity (higher silk density) and smaller average diameter of silk, therefore, there is an increase in elastic modulus and strength from outside to inside layers. When fabricating silk-based composites, the amount of resin gained by fiber is strongly related to the degree of swelling of the noncrystalline regions, that is, the amorphous regions and the microvoids inside the fiber. Hence, the composite made from silk fibers are have high elastic modulus, tensile strength, and furthermore, it is cost effective as it is a waste material of silk industry.

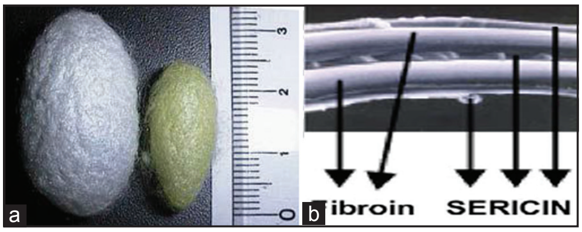

Figure 1: (a) Raw cocoon silk, (b) side view of the silk fiber.

Silk is primarily made up of proteins, and it is in close composition to human skin (Hyuarinen). Silk is least wasteful and more natural than any of its manmade substitutions. However, the waste products sometimes can be numerous when compared to the final product. Most of these products can be reused or can provide another commercial value. The byproduct of manufacturing silk includes the unusable parts of the pupa and cocoon. The real source of silk waste is from the cocoons and manufacturing processes. Silk fiber is a valuable resource with good properties and it can be used in a great many value added products. Using silk fiber for composite has many advantages. Silk is renewable versatile nonabrasive, porous, hydroscopic, viscoelastic, biodegradable, combustible, computable, and reactive. The fiber has a high aspect ratio, high strength to weight ratio, is low in energy conversion, and has good insulation properties. Table 2 depicts the India's export earnings (Source: Indian silk, August - 2002).

3. RECYCLABILITY AND SUSTAINABILITY OF WASTE SILK FIBER BIOCOMPOSITES

The composite materials consisted of high strength fibers such as carbon, glass and aramid, and low strength polymeric matrix, now have dominated the aerospace, leisure, automotive, construction, and sporting industries. Unfortunately, these fibers have serious drawbacks such as (i) Nonrenewable, (ii) nonrecyclable, (iii) high energy consumption in the manufacturing process, (iv) health risk when inhaled, and (v) nonbiodegradable. Although glass fiber reinforced composites have been widely used due to its advantages of low cost and moderate strength, for many years to provide solutions to many structural problems, the use of these materials, in turn would induce a serious environmental problem that most Western countries are now concerning.

Recently, due to a strong emphasis on environmental awareness worldwide, it has brought much attention in the development of recyclable and environmentally

Table 2: India's export earnings.

Item wise exports	2000-01		2001-02		% Increase/decrease	
	Rs. crores	US $ million	Rs. crores	US $ million	Rs.	US$
Natural silk yarn fabrics, made ups	1622.79	355.25	1309.47	274.87	−19.3	−22.6
Ready-made garments	642.82	140.72	720.88	151.32	12.1	7.5
Silk carpet	110.88	24.27	168.45	35.36	51.9	45.7
Silk waste	45.49	9.96	36.58	7.68	−19.6	−22.9
Total	2421.98	530.21	2238.38	469.22	−7.7	−11.5

sustainable composite materials. Environmental legislation as well as consumer demand in many countries is increasing the pressure on manufacturers of materials and end-products to consider the environmental impact of their products at all stages of their life cycle, including recycling and ultimate disposal. In the United States, it encourages manufacturers to produce materials and products by practicing the 4 Rs, which are (i) reduce the amount and toxicity of trash to be discard (sourced reduction); (ii) reuse containers and products; (iii) repair what is broken, and (iv) recycle as much as possible, which includes buying products with recycled content. After these processes are gone, the materials finally are entitled to be disposed to the landfill [14].

Considering the increasing renewed interest in engineering materials of natural origin, silks seem to be a strong natural fiber candidate for reinforcements in polymer composites. From an environmental perspective, however, as raw silk cocoons are produced in a two-step process namely mulberry plant production and silkworm farming, the cumulative energy demand of silk cocoon production is (i) Much higher than that of plant fiber production, which relies on a single-step agricultural process and (ii) comparable to that of glass fiber productions. Processed silk textiles, like processed plant fiber textiles, have much higher production energy demands than glass fiber textile productions. Therefore, a detailed life cycle assessment should be carried out to examine the sustainability of such natural fiber composites in comparison to glass composites. However, it is important to mention that socioeconomic aspects, such as job creation, also need to be considered as natural fibers are agricultural fibers with notable benefit for the fiber crop growers and their communities [15,16].

4. FABRICATION AND CHARACTERIZATION OF WASTE SILK FIBER REINFORCED BIOCOMPOSITES

Seong *et al*. [11] have investigated the mechanical and thermal properties of polybutylene succinate (PBS) biocomposites reinforced with industrially available waste silk fibers, fabricated with varying fiber contents and lengths. Figure 2 shows the microstructure of single filaments in waste silk fibers. To investigate the waste silk fiber length effect, the as-separated waste silk fibers were chopped to approximately 25.4 mm (1 in.), 12.7 mm (1/2 in.), 6.4 mm (1/4 in.), and 3.2 mm (1/8 in.) in average length. The fiber contents used are 0, 20, 30, 40, and 50% by weight. The biocomposite molding was performed by compression method. The tensile strength was gradually increased with the increase in the waste silk fiber loading up to 40 wt.%, compared with that of the unreinforced PBS control. The decrease of the tensile strength in 50 wt.% waste silk/PBS biocomposite was due to insufficient filling of melted PBS matrix resin into waste silk fibers during biocomposite processing at such a high fiber loading. The flexural properties were gradually increased with the fiber loading up to 40 wt.%. With the addition of 50 wt.% waste silk fibers, the flexural properties were somewhat decreased. This is due to the insufficient filling of the melted PBS resin into the reinforcing natural fibers during composite processing. The flexural result also indicated that the strength of the biocomposites does not significantly depend on the variation of chopped waste silk fiber length, whereas the modulus was more or less greater than that reinforced with as-separated waste silk without chopping. The glass transition temperature, the storage modulus of all the waste silk fiber-reinforced PBS biocomposites studied was significantly greater than that of PBS resin, especially in the higher temperature region. The storage modulus of the biocomposites was increased with increase in the loading of waste silk fibers, and the greatest value was obtained with the chopped waste silk fibers of 12.7 mm length. The thermal stability of waste silk/PBS biocomposites was observed to be intermediate between the PBS resin and the waste silk fiber depending on the fiber content.

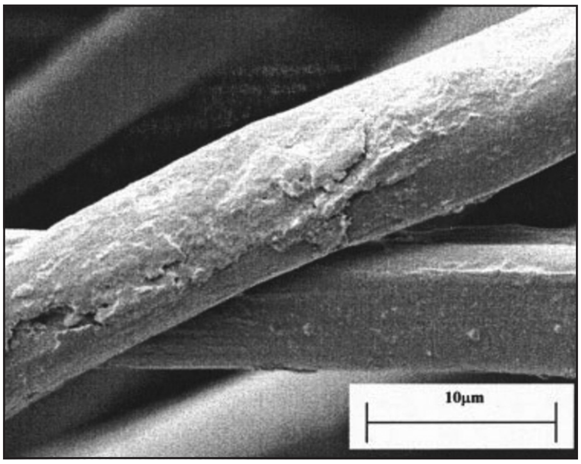

Figure 2: Scanning electron micrograph of single filaments in waste silk fibers [11].

Taşdemir *et al*. [17] studied the composite structures produced using high-density polyethylene (HDPE) polymer with silk and cotton waste as reinforcement fibers in different ratios. Cotton and silk wastes were mixed in the ratios of HDPE/silk or cotton waste 100/0, 97/3, and 94/6. This mixture was prepared with double-screwed extruder. Addition of 3% and 6% cotton and silk fibers to HDPE polymer increased the elasticity module values of mechanical characteristics of the composite. When the waste textile fibers are compared to each other, the elongation values of the composite formed from the addition of cotton fibers are lower than those of silk fibers. When the morphological structure of the composite showed that the segments of silk fibers were closer to each other. Izod impact values seem to decrease. Breaking faces were seen to have big gaps and polar fibers hold nonpolar fibers weakly, as a result of which fibers are pulled out of the matrix.

When differential scanning calorimetry thermal analysis results are considered, the melting behaviors of the composites differed due to the use of two different waste fibers added to the HDPE polymer matrix. When scanning electron microscope (SEM) photograph were analyzed, it was clearly seen that fibers were not well dispersed within the structure. As a result, the existence of the waste fibers does not contribute to durability. Elongation characteristic of the composite made from textile waste is suitable for the material with little elongation values and can be considered to lead to a decrease in production costs.

Taşdemir *et al*. [18] reported on the composite structure obtained by mixing silk and cotton waste and recycled PA6 polymer was mixed. Silk and cotton wastes were in fiber lengths of 1 mm, 2.5 mm, and 5 mm. The recycled PA6/silk and cotton wastes were mixed in the rates of 97/3, respectively. There was an increase in the elasticity modulus of the composites made up of PA6 polymer/waste silk fibers. The amount of the increase for the composite made up with the addition of 1 mm length waste silk fibers, especially can be explained in terms of attachment achieved by the dimensional volume increase. When tensile strength values were evaluated, there was no strength increase in either fiber added to PA6 polymer. As silk fiber is stronger than cotton fiber, tensile values were protected as before. It was known that tensile strength of a composition mainly depends on the intermediate structure. When hardness and izod impact strength values are considered; SEM pictures taken from breaking surfaces showed interestingly big gap. When mechanical, thermal, and morphological characteristics are considered, with the addition of waste cotton fibers to the PA6 polymer and elasticity modulus values of composite formed with silk fibers increase and the composite formed in both fibers meet the demanded characteristics in themselves.

The mechanical and thermal properties of a silk fiber/polylactic acid (PLA) biocomposites were studied Hoi-Yan Cheung *et al*. [19] through different experimental approaches. Optimal values, in terms of fiber length and weight content of 5 mm and 5 wt.%, respectively, to achieve the maximum microhardness of the biocomposites were obtained. The tensile property test revealed that the modulus of elasticity and ductility of the biocomposites were substantially increased to 53% and 39%, respectively, as compared with pure PLA. Besides, the glass transition temperature of the biocomposite increased approximately by 10%. SEM images also showed that good interfacial bonding between the silk fibers and PLA matrix was achieved, which reflected a good wettability of the resin during injection and extrusion process was resulted. This biocomposite, made by biodegradable silk fibers and PLA together, to produce its strength and thermal stability is better than the host polymeric material for tissue engineering applications.

Waste silk fibers which are obtained during the cocoon forming or the silk filaments production were studied by Taşdemir *et al*. [20]. They were cut with guillotine in between 1, 2.5 and 5 mm fiber lengths. Waste cotton fibers were spilled down from grids during the opening, carding and combing process. The waste cotton obtained from this process is gathered into fiber dimensions in between 1, 2.5 and 5 mm. The mixing of polycarbonate (PC) with the silk and cotton waste was carried out at 85-230°C temperature, 9 bar pressure, and mix rate of 249 rpm, using a double-screw extruder. The addition of 1 mm length silk waste to recycled PC polymer decreased the composites' elasticity modulus value, while increasing the length of silk increases the elasticity modulus. With the addition of waste silk, the Izod impact strength decreased but increasing the length of silk fiber increases this value. Izod impact strength increased, and increasing the length of cotton fiber increased this value gradually. The addition of waste silk to PC decreases the T_g value. However, in the case of waste silk fiber length of 2.5 mm, there was an increase in T_g value of the composite. On the other hand, the addition of cotton waste to PC decreases the T_g value of the composite. When the PC-silk and PC-cotton polymer composites' microstructure were examined, it was observed that silk and cotton fibers did not orientate in a clear direction but there was a better adhesion of the cotton to the matrix. This was due to the absence of compatibilizer or to differences in surface energy of the two kinds of fibers.

5. CONCLUSIONS

- As silk fiber is a waste material of textile industry so it is cheaply available and it shows the perfect utilization of waste product.
- Silk fibers bio-degradable and highly crystalline with well aligned structure. Hence, it has been known that they also have higher tensile strength than glass fiber or synthetic organic fibers, good elasticity, excellent resilience and in turn it would not induce a serious environmental problem like in glass fibers.
- As in glass fibers have serious drawbacks such as nonrenewable, nonrecyclable, nonbiodegradable, and high energy consumption in manufacturing process; therefore it is a perfect replacement for glass fibers composites.
- The optimum results in the properties were obtained with the incorporation of waste silk fibers (ideally 40-60%) to the matrix material as the fibers ensured to carry large fraction of loads.
- The waste silk fibers may be a potential as reinforcement for effectively improving the static and dynamic mechanical properties of a biodegradable polymer matrix resin, depending on the waste silk fiber content and length in the present biocomposite system.
- Waste silk fiber reinforced composites can be used for structural applications (building and construction industry-panels, doors and window panels, false

ceilings, partition boards etc.), packing, automobile and railway coach interiors, and storage devices as they are very cost-effective material.

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***Bibliographical Sketch**

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