CHAPTER - 3

Tensile Properties of Short Waste Silk Fibers/Wheat Protein Isolate Green Composites
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ABSTRACT

Green composites of short waste silk fibers/wheat protein isolate were prepared with different fiber loadings. Wheat protein isolate was cross linked with glutaraldehyde in the presence of glycerol plasticizer. Surface of the waste silk fibers was modified with alkali treatment. The effect of fiber loading on the tensile properties of the composite was studied. The tensile strength and modulus of the composites increased with fiber content whereas the %elongation at break decreased. The interface between the fibers and the matrix was found to be narrow. The improvement in tensile properties of the composites was attributed to the high tenacity and surface modification of the fibers.

3.1. INTRODUCTION

Owing to their outstanding properties such as high specific strength, low cost, ease and rapid processing etc., polymers in general and polymer composites in particular are finding many applications in every field. As these are generally non-degradable, unfortunately, the environment problems posed by them are also simultaneously increasing [1]. To overcome these problems, more attention is paid towards developing more environmentally benign green composites utilizing natural materials. In this direction, many natural and renewable materials such as soy protein [2,3], wheat protein [4], polybutylene succinate [5] etc., were used to make biodegradable films. As these matrices are brittle, often they are reinforced with natural fibers/fabrics such as hemp [6-8], sisal [9], Alfa [10], kenaf [11], Hildegardia [12,13] etc. Protein matrices are preferred due to their abundance and economics. However formation of protein films is a complex process involving three steps - first, rupture of low-energy intermolecular bonds, secondly, rearrangement and orientation of polymer chains (shaping) and finally,
formation of a three dimensional network structure [14,15]. Cohesiveness in wheat protein isolate (WPI) facilitates film formation [16]. As wheat is abundantly available and renewable, the author selected WPI as the matrix. Silk is a proteinous fiber which is compatible with WPI. Silk fabrics are produced mainly in China and India. Annually, thousands of tons of waste silk fibers (WSFs) are generated in silk industries in these two countries. The author chose WSFs inorder to add value to the waste generated. The effect of fiber loading on the tensile properties of WSF/WPI composite films was studied. The interface in these composites was examined using a scanning electron microscope.

3.2. MATERIALS

WPI powder was obtained from Honeyville Food Products, Salt Lake City, Utah, USA. According to the manufacturer, WPI supplied consisted of 90% protein, 4% fat, about 5% ash and 1% remaining unknown constituents. Glutaraldehyde solution 25% Laboratory reagent grade was purchased from SD fine chemicals limited, Mumbai, India. Glycerol about 98% purified and Analytical grade sodium hydroxide (NaOH) were purchased from MERCK chemicals, Ahmedabad, India. The WSFs were obtained from silk industries in Dharmavaram town of Ananthapuramu district, Andhra Pradesh state, India.

3.3. METHODS

3.3.1. Modification of Waste Silk Fibers (WSFs)

The WSFs were treated with 5% aqueous NaOH solution for 1 hour to remove the sercin on the surface of the fibers. The alkali treated WSFs were washed with diluted mild soap for 2-3 minutes and later rinsed thoroughly with distilled water to remove grease and dirt. The dried fibers were cut to a size of ~1mm length.

3.3.2. Preparation of Waste Silk Fibers (WSFs)/Wheat Protein Isolate (WPI) Composite Films
The green composites of WSFs/WPI were prepared using a casting method [17]. The desired amounts of WPI powder and glycerol (10% by wt. of WPI) were mixed with 20 times (by wt. of WPI) distilled water. Proteins are soluble in high alkali medium [16,18,19]. When the alkalinity of protein isolate solution is raised in the pH range of 8-12, the protein dissociates and unfolds. Such changes enhance the formation of near s-s bonds between the aligned protein polypeptide chains. In the present work, the pH of the solution was optimized and adjusted to 10 by adding desired amount of 1N NaOH solution. To this mixture, glutaraldehyde (20% by wt. of WPI) was added as the crosslinker [20-22]. Glutaraldehyde reacts with the amine groups in protein particularly in an alkaline pH to form intermolecular crosslinks [20-22]. To this solution, different amounts of WSFs (1 to maximum 5 wt. % of WPI) were added and stirred well and degassed. The mixtures were poured separately on Teflon coated glass plates and dried for 48 hours at room temperature. The sheets were then hot pressed (cured) at 120 °C and 2 MPa pressure for 20 min. The cured dark brown composite films with ~0.4mm thickness were removed from the press and allowed to cool.

3.3.3. Tensile Testing

The tensile properties of WSF/WPI composites were studied employing an INSTRON 3365 Universal Testing Machine according to ASTMD 3039 procedure using 10mm wide specimens maintaining a gauge length of 50mm and crosshead speed of 5 mm/min.

3.3.4. Fourier Transformer Infrared (FTIR) Spectra

The FTIR Spectra of virgin WPI and crosslinked WPI powders were recorded in the range of 4000 to 500 cm$^{-1}$ on an Analect RFX-65A spectrometer using KBr pellet method after diluting the powders to 2 wt. % in KBr.
3.3.5. Morphology (SEM)

The brittle fractured surface of the gold coated specimens was observed using a JSM 6700F scanning electron microscope (SEM).

3.4. RESULTS AND DISCUSSION

The FTIR Spectra of virgin WPI and crosslinked WPI are shown in Fig.3.1. From Fig.3.1, it is evident that the intensity of bands corresponding to crosslinked WPI were lower than those of virgin WPI confirming the crosslinking. Further the films were found to swell in water.

Fig.3.1. FTIR Spectra of (a) virgin WPI and (b) crosslinked WPI

The tensile parameters of the matrix and composites under study are
presented in Fig.3.2. It is evident that the tensile strength (Fig.3.2a) and modulus (Fig.3.2b) were higher than the matrix and increased with fiber content. Two factors – tensile properties of the fibers and interfacial bonding may be responsible for this improvement. The cocoon silk fibers have the tensile strength, modulus and % elongation at break in the range of 300 – 600 MPa, 9 – 17 GPa and 0.1 – 0.2 respectively [23]. However, the % elongation at break of the composites decreased with fiber content. This is understandable as the silk fibers have lower % elongation at break [23].
Fig. 3.2. Tensile properties of WSFs/WPI composites – (a) Tensile strength, (b) Tensile modulus and (c) % Elongation at break
Fig.3.3. Fractographs of WSFs/WPI composite films for different fiber contents—
(a) 1.5% (b) 2.5% (c) 5%

Fig.3.3. presents the fractographs of the composites with 1.5, 2.5 and 5.0 wt% fiber loadings. These fractographs indicate narrow interface with matrix skin formation on the surface of the fibers. Because of the chemical treatment, the fiber surface became rough resulting in better interfacial bonding. Based on the tensile properties and the dark brown color, these more environmentally benign composite films can be considered for packaging applications especially for photosensitive medicines.

3.5. CONCLUSIONS

Green composite WSF/WPI films were prepared using glycerol as plasticizer and glutaraldehyde as crosslinking agent. The effect of fiber loading on the tensile properties was studied. The tensile strength and modulus were improved with increasing fiber content whereas the % elongation at break decreased. Better interfacial bonding between the fibers and the matrix was observed. These studies were aimed at adding value to the waste silk fibers. The WSF/WPI composite films can be considered for packaging food and medicines.
REFERENCES