



MECHANICAL PROPERTIES OF EPOXY / CHITOSAN BIOCOMPOSITES

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ABSTRACT

Completely biodegradable epoxy/ chitosan biocomposites were prepared. The structure of epoxy resin, chitosan and epoxy/chitosan biocomposite were confirmed by FT-IR spectroscopy. The effect of inclusion of different weight percentage of chitosan in epoxy on tensile properties and water absorption behavior were studied. Morphologies of the fractured surfaces of epoxy/chitosan biocomposites were observed by scanning electron microscope.

Key words: Epoxy/chitosan biocomposite, Tensile properties, Water absorption, Morphology.

INTRODUCTION

Epoxy resins have been widely used in many industrial applications, such as adhesives, construction materials, composites, laminates, and coatings due to their excellent mechanical properties, low cost, ease of processing, fine adhesion to many substrates, and good chemical resistance. However, the inherently brittle nature of epoxy resin has limited its applications^{1,2}. The integration of biodegradable materials into an epoxy resin has advantages for the development of environmentally friendly biocomposites³. Chitosan, (1-4)-2-amino-2-deoxy- β -d-glucose, is derived by the alkaline deacetylation of chitin, a polysaccharide found in the exoskeletons of shrimps and crabs^{4,5}. The main parameters influencing the characteristics of chitosan are molecular weight and the degree of deacetylation. As a nontoxic, renewable and biodegradable polymer, chitosan has been extensively studied over the last two decades⁶. Due to the presence of amino and hydroxyl groups, chitosan exhibits multiple functionality including antimicrobial activity and metal binding⁷. Many attempts have been made to develop functional materials from chitosan, such as films, sutures, beads, and hydrogels, and to apply them in the wound healing⁸ drug

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delivery⁹, metal removal¹⁰ and antimicrobial food packaging¹¹. In this work, epoxy/chitosan biocomposites were prepared. Subsequently, the effects of chitosan content on the tensile and water absorption for the epoxy biocomposites were investigated.

EXPERIMENTAL

Chemicals

Commercially available diglycidyl ether of bisphenol-A (DGEBA) based epoxy resin, LY556, (epoxy equivalent of about 180-190 and viscosity of 10,000 cPs), 4,4'-diaminodiphenylmethane (DDM) and epoxy curing agent were obtained from Ciba-Geigy Ltd., India. Chitosan (powder, $M_v = 1$, 80,000 Da, moisture content = 9.46%, viscosity = 229 cps, DD = 86.39) was supplied by Indian Sea Foods, Cochin, India and used without further purification.

Preparation of neat epoxy matrix

100 g of epoxy resin was heated to 70°C in an oil bath until the removal of trapped air bubbles and moisture. The DDM, amine hardener was also heated around 95°C, up to melting completely. Both the resin and hardener (27% in accordance with the epoxy resin) were mixed with continuous stirring. The mixture was then poured into moulds and cured in an air oven at 100°C for 4 hours and post cured at 140°C for 3 hours.

Preparation of epoxy/ chitosan biocomposite

Calculated amount of chitosan (1, 2, 4 and 8 wt %) was sonicated with epoxy resin for 2 hours. The mixture was then stirred for 10 minutes at 12000 rpm using high speed stirrer. A stoichiometric amount of curative, 4,4'-diaminodiphenylmethane was also added and subjected to vacuum to remove the trapped air. The matrix mixture was casted in the moulds, cured and then post-cured.

Measurements

FT-IR spectra

The FT-IR spectra were recorded on a Shimadzu FT-IR spectrometer, with KBr pellets for solid samples. For viscous liquid samples, the spectra were carried out by placing the sample between two KBr pellets.

Tensile properties

Tensile strength was determined as per ASTM-D3039 using a universal testing

machine (Instron, Model 6025 UK), at a cross-head speed of 10 mm/min. with specimens of width 25 mm, length 200 mm, and thickness 3 mm. Five specimens were tested for each sample.

Water absorption

Test specimen were immersed in water for 24 hours at 30°C and the percentage of water absorbed by the specimen was calculated using Equation :

$$\% \text{ Increase in weight} = (w_2 - w_1) \times 100/w_1 \quad \dots(1)$$

where, w_1 is the initial weight of the sample and w_2 is weight of sample after immersion in water for 24 hours at 30°C.

SEM Analysis

Surface morphology of fractured surface of the biocomposites was recorded using scanning electron microscope (JEOL JSM Model 6360).

RESULTS AND DISCUSSION

FT-IR Analysis

Fig. 1a shows FT-IR spectra of epoxy resin. The peak at 3504 cm^{-1} indicates the presence of hydroxyl group of epoxy resin. The peaks between 920 and 770 cm^{-1} suggests the asymmetric stretching of epoxy. The peaks at 1600 cm^{-1} and 2960.8 cm^{-1} corresponds to C=O stretching and C-H stretching vibration, respectively¹².

Fig. 1b shows FT-IR spectra for chitosan. The broad peak at 3430 cm^{-1} indicates that the stretching vibration -OH, which is superimposed onto -NH stretching vibrations and broadening of band is due to the inter molecular hydrogen bonds of polysaccharides. The peaks at 1640 cm^{-1} , 1586 cm^{-1} and 1383 cm^{-1} corresponds to -C=O stretching of acetyl unit, N-H bending and -CH₃ symmetrical angular deformation, respectively. The peaks at 1151 cm^{-1} and 1041 cm^{-1} indicates β -(1,4) glycosidic bonds and C-O-C stretching vibration, respectively¹³.

Fig.1c shows FT-IR spectra for epoxy/chitosan biocomposite, the β -(1,4) glycosidic bonds and -CH₃ symmetrical angular deformation are indicated at 1151 cm^{-1} and 1383 cm^{-1} , respectively. However, it is evidenced that the blending of chitosan with epoxy causes a considerable decrease in the intensity of epoxy peak at 912.12 cm^{-1} .

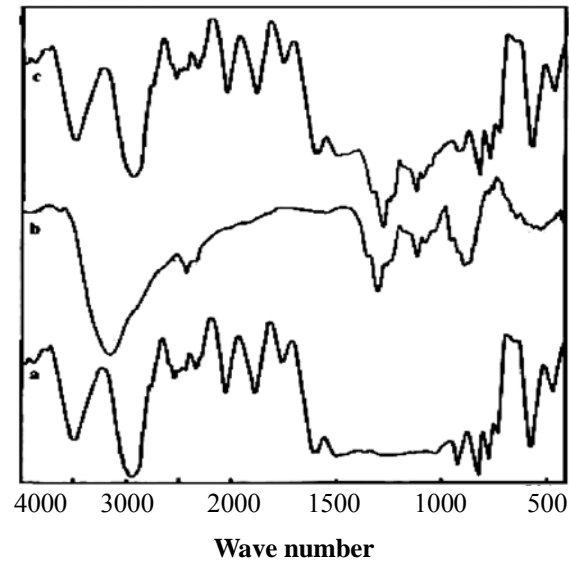


Fig. 1: FT-IR spectra of (a) Epoxy resin (b) Chitosan (c) Epoxy/chitosan biocomposite

Mechanical properties

Fig. 2 indicates that the tensile strength of epoxy/chitosan biocomposites decreases with increase of inclusion of chitosan in epoxy.

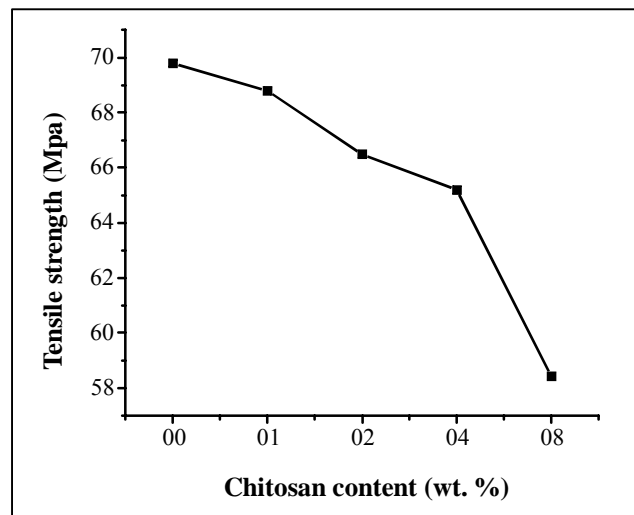


Fig. 2: Effect of chitosan content on tensile strength of epoxy/chitosan biocomposites

The decrease in tensile strength is partially due to the thermodynamic immiscibility and inherent incompatibility between chitosan and epoxy¹⁴. The mechanical performance of a polymer composite depends on the strength of the filler. But, the chitosan samples used for preparing epoxy/chitosan biocomposites have a moisture content of 9.4% on dry weight basis. However, the addition of chitosan beyond 4 wt.% indicates a sharp decrease in tensile strength. Epoxy matrix having higher chitosan content form aggregates, which leads to failure even at lower stresses. The bonds between the chitosan particles are weaker than those between chitosan and the polymer matrix, cavities would be formed, resulting in failure at lower stresses.

Water absorption

Water absorption of the epoxy/chitosan biocomposites are presented in Table 1. The chitosan incorporation into epoxy system increases the water absorption behavior with increasing its content. The water absorption of pure epoxy system is 0.1201%, whereas for 1, 2, 4 and 8 wt.% chitosan incorporation shows 4.6%, 10.3%, 16.4% and 23.1% increase of water uptake. The increase in percentage water uptake for chitosan incorporated epoxy system is due to the inherent hydrophilic nature of the chitosan. Presence of moisture in the matrix materials affects the mechanical properties also.

Table 1: Water absorption in epoxy/chitosan biocomposites

Percentage water absorption					
Percentage of chitosan in epoxy system	0%	1%	2%	4%	8%
Chitosan	0.1201	0.1256	0.1325	0.1398	0.1479

SEM analysis

Scanning electron microscope was used to investigate the morphology of epoxy and epoxy/chitosan biocomposite systems. (Fig. 3) SEM micrographs of pure epoxy system shows smooth, glassy and homogeneous microstructure without any plastic deformation. In contrast (Fig. 4), SEM micrograph of epoxy/chitosan biocomposite shows heterogeneous morphology, which reveals that the chitosan aggregate is embedded in the epoxy matrix.

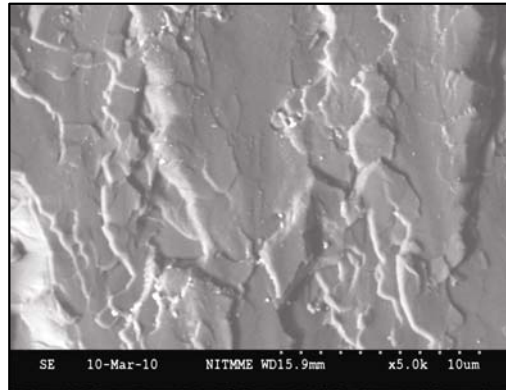


Fig. 3: SEM image of pure epoxy system

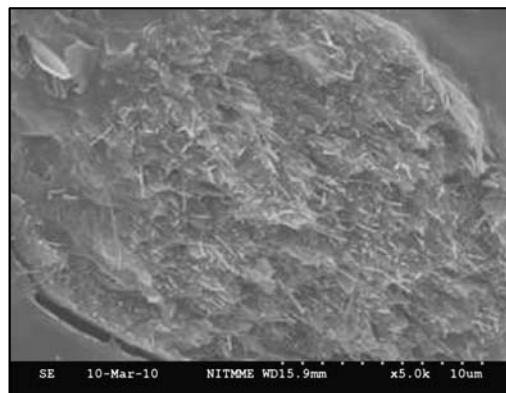


Fig. 4: SEM image of epoxy/chitosan (wt. 8%) biocomposite

CONCLUSION

Epoxy/chitosan biocomposites were prepared. Chitosan particles are not homogeneously dispersed in epoxy resin even by sonication method. Tensile strength decreases by the addition of chitosan due to the immiscibility. Water absorption increases according to the weight % of chitosan. The SEM results confirm that chitosan aggregates are embedded in epoxy matrix. Chitosan in epoxy biocomposites are completely biodegradable.

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