



Contents lists available at ScienceDirect

International Journal of Biological Macromolecules

journal homepage: www.elsevier.com/locate/ijbiomac



Evaluation of selected properties of biocompatible chitosan/poly(vinyl alcohol) blends

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ARTICLE INFO

Article history:

Received 15 July 2015

Received in revised form

18 September 2015

Accepted 30 September 2015

Available online xxx

Keywords:

Biocompatible

Chitosan

Blends

ABSTRACT

Selected properties of chitosan (CS) and poly(vinyl alcohol) (PVA) blends crosslinked by tetraethoxysilane (TEOS) were studied. XRD analysis showed characteristic peak at 22.5° attributed to the crystalline structure of CS and PVA. DSC thermograms unveiled the quantitative determination of free, intermediate and bound water in the blends. Tensile strength and fracture strain of blends were observed due to the combined effect of physically and chemically crosslinked network structures. The decrease in water contact angle endorsed the hydrophilic performance while the storage modulus G' and loss modulus G'' was decreased as the temperature was increased exhibited the viscoelastic property of the blends. The fabricated blends can be employed for drug delivery systems, tissue engineering and other biomedical applications.

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1. Introduction

Natural and synthetic polymeric materials are employed frequently in medical field. Natural polymers being attributable to their explicit biomedical applications and non-toxicity, biodegradability and biocompatibility are required with synthetic polymers [1–3]. Polymeric blends can be prepared by blending both with natural and synthetic polymers useful for diverse biomedical usage. Therefore, blending is a very practical and reliable method to merge natural and synthetic polymers and modify their required physicochemical properties [4,5]. Blending technology can make a new class of materials with enhanced thermal, mechanical, viscoelastic and structural properties. Biodegradability, biocompatibility and other biomedical properties of the blended materials are also improved. This class of materials have been entitled as biosynthetic or bioartificial polymeric materials [5]. Natural and synthetic polymer blends have been fabricated for different scientific and other applications with exceptional properties [6–13].

Chitosan [poly-β(1-4)-D-glucosamine] (CS) is a polysaccharide [6,7,14–18] have been reported for its biomedical properties and used to form biodegradable and biocompatible nontoxic blends.

Based on these characteristics, chitosan can be used for drug delivery systems, pharmaceutical industry, biotechnology etc. PVA is a water soluble polyhydroxy polymer and consumed extensively in medical applications due to its exceptional physical and chemical characteristics [4,6,7,19,20]. CS/PVA blends have been reported to have excellent structural, biomechanical, viscoelastic and surface properties [4,7,11,21–24]. These characteristic properties are attributable to inter-molecular interactions between polymer chains [7,24]. Surface properties of the blends can be evaluated using contact angle method as wetting behavior of solid surfaces is an essential characteristic of many procedures. The contact angle is a low cost, easy and fast method for determining the solid surface wettability [5].

Blend hydrogels are employed for drug delivery systems, tissue engineering, actuators for optics, and as extracellular matrices for genetic research. The choice of hydrogel, though, is sternly restricted a lot by their poor mechanical performance. Most of the hydrogels are brittle and do not show high tensile strength and stretchability. Many efforts have been assigned to fabricate blend hydrogels with advanced mechanical characteristics [25,26].

In this work, we explored some of the properties of the CS/PVA blend hydrogels from natural polymers blended with biocompatible synthetic polymers forming physically and chemically crosslinked blend hydrogel networks. The purpose of this work was to evaluate the characteristics of our already synthesized blend for drug delivery application [6]. The manufactured blends

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Table 1
Composition and codes of CS/PVA blends.

	Sample code		
	CP4	CP8	CP10
Crosslinker (%)	2	2	2
CS/PVA (wt%)	96:4	92:8	90:10

were chemically crosslinked with novel nontoxic silane crosslinker (TEOS) [7,27,28]. The tensile strength, fracture strain and crack bridging by the network growth of blend hydrogel was attributed to the physically and chemically crosslinked structures by TEOS. The structural, thermal, surface, and viscoelastic properties of blend hydrogels were also studied. The surface properties were determined by the contact angle analysis. Based upon these characteristics properties, the blend hydrogels are useful for drug delivery and other biomedical applications as well as explore other significant areas related to this research.

2. Experimental materials and preparation

Acid soluble crab shell source chitosan (product number C3646 having degree of deacetylation (DDA) ca. 75% with viscosity >200 cP), (PVA) (M_w : 146,000–186,000; 98–99% hydrolyzed), TEOS, methanol (99.7%), acetic acid, and sodium hydroxide were purchased from Sigma–Aldrich (Milwaukee, WI). All chemicals were of analytical grade and were used as received. The thickness of the films at different positions was determined by thickness meter and the average value was assessed.

2.1. Preparation of blend hydrogels

CS/PVA blend membranes crosslinked by TEOS were synthesized using dissolution casting technique. CS (96, 92 and 90% for CP4, CP8 and CP10, respectively) was dissolved in 0.5 M acetic acid under magnetic stirring. 4, 8 and 10% PVA was dissolved in deionized water at 80 °C for 1 h till the appearance of clear solution. In case of CP4, 0.96 g of CS was dissolved in 40 mL acetic acid and added with 0.04 g of PVA in 20 g of water. pH of above CS/PVA solution was set at ~3.5 at ambient temperature. Under constant stirring, 2% TEOS was also mixed to this blend and stirred for further 1 h. This mixed solution was poured in Petri dishes, dried at room temperature and then in oven at 60 °C under vacuum. 4, 8 and 10% PVA was mixed in CS solutions and samples were designated as CP4, CP8 and CP10, respectively [6]. Systematic formulations of these blends are given in Table 1.

3. Characterization techniques

3.1. Equilibrium water content (EWC%)

Dried hydrogel sample (~50.0 mg) was taken in vial filled with distilled water (100 mL) in temperature controlled bath. The weight of the swollen sample was calculated at different time intervals after removing the excess solution from the surface. The sample was taken again in the same solvent and the equilibrium swelling content (EWC %) of the sample was calculated by using following equation [17]:

$$\text{EWC}(\%) = \left[\frac{W_e - W_d}{W_e} \right] \times 100$$

where W_e is the weight of swollen hydrogel at equilibrium and W_d is the initial dry weight of the sample. All results were the average of three readings.

3.2. Biomechanical analysis

Biomechanical tester (BOSE, Model ELF 3200) was used to determine the tensile strength (TS) and elongation at break (%E) of the hydrogel films. The specimen was cut of 20 mm length, 10 mm width and 12 mm distance was used between the two holders. The temperature was at 22 °C and 50% relative humidity was recorded during the experiment. Five samples were tested for each film and the average value was recorded.

3.3. Thermal analysis

Thermal analysis was performed using Perkin-Elmer Pyris 1 differential scanning calorimeter (DSC). Initially, the sample was cooled from room temperature to –90 °C and then subjected to heating scan up to 190 °C at a heating rate of 10 °C/min. The result of first scan was reported.

3.4. Surface analysis by contact angle measurements

The surface properties of the blend films were analyzed by determining the water contact angles of the films at room temperature. The contact angle was calculated by using drop method by contact angle Goniometer Model 100-00 (220) (Ramé-hart instrument company). The sample was located on the sample stage of the instrument. A steady drop of distilled water was poured on the sample surface by a micropipette needle. The drop and the contact angle visibility could be obtained by adjusting the instrument. The contact angle was calculated for a specific interval of time from 0 to 32 min until the drop was nowhere to be found and all the water was engaged in the film. Each measurement was performed for three times and the average value was recorded.

3.5. X-ray diffraction

The structural analysis of blends was done on STOE STADI P power diffractometer with (Cu) $K\alpha_1$ radiation with wavelength of 1.54 nm at 40 kV and 35 mA. The scanning range was from 2° to 50° (2θ) at a rate of 0.2°/min.

3.6. Rheological analysis

The viscoelastic behavior was carried out using ARG2 Rheometer (TA, Instruments, USA). The viscoelastic properties such as shear storage modulus (G') and loss modulus (G'') were determined as a function of temperature and angular frequency. The temperature sweep step was from 30 to 90 °C at the rate of 10 °C/min performed at 1 rad/s. The frequency ramp was from 0.1 to 630 rad/s at room temperature. Each frequency sweep was completed at about 275 s.

4. Results and discussion

4.1. Equilibrium water content (EWC %)

The equilibrium water content of CS/PVA blend in water is shown in Fig. 1. All the blends showed increase in water content and attained equilibrium condition around 7 h. It is clear from figure that EWC values were increased with increase in time and CP4 illustrated maximum EWC value of 99.987% after 7 h. The EWC of blends was decreased as the amount of PVA was increased. EWC % was changed or decreased with increase in the amount of PVA. The decrease in EWC % with rise in PVA amount was primarily owing to the increment in the hydrophobic moieties (as PVA having nonpolar backbone shows less hydrophilicity) and also the development of dense arrangement. The pore size of the CS/PVA blends was also

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