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Experimental Investigation on Synthesis and Characterization of Chitosan/Silk Fibroin Polymer Composites

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Abstract: Chitosan is the most important derivative obtained from Chitin, a natural polysaccharide constitute of crustaceous shells. In this research work the polymer blends were prepared by mixing of nano chitosan with poly vinyl pyrrolidone (PVP) and silk fibroin in the ratio 1:1:1 with and without the inclusion of cross linking agent glutaraldehyde. The characterizations of prepared polymeric blends were carried out by advanced analytical tools FT-IR, TGA, DSC and XRD methods. From the FTIR study it can be revealed that there is intermolecular hydrogen bonding interaction. The XRD studies show the films of this study are of amorphous in character. By using DSC experiments the Glass Transition temperature and crystallization of the polymer blends were investigated.Further analysis of cytoxicity assay will provide information on the suitability of the material for tissue engineering applications.
Keywords: Nanochitosan, PVP, Silk fibroin, characterization

1. Introduction

The chitin and chitosan are renewable polymers considered to be abundantly available and also they are biodegradable, biocompatible, and non-toxic and being good adsorptants [1]. Chitosan has been regarded as a multifunctional polymer applicable to various purposes. Nanoparticles which are made of chitosan, a naturally occurring polymer is normally isolated from shrimp shells and the crab, used as carriers of anticancer drugs, antitumor genes, and other therapeutic agents and found to have high surface area.Nanochitosan are natural and ecofriendly materials because of its great physicochemical properties and also they are harmless to the human[2]. The chitosan can be modified to improve its properties; one of the modifications will be adding silk fibroin with it. Silk fibroin films find application in the immobilization matrices for enzymes[3]. Chitosan is a promising natural substances used in biomaterials research as it has several essential properties that can be applied in tissue engineering. This polymer can be easily combined with other biomaterials and it can be rapidly and economically processed to deliver growth factors and drugs [4]. Ternary blend is obtained by mixing the polymers Nano chitosan, Poly vinyl pyrolidone, Silk fibroin. In this work PVP added as a supporting material. The two composites were prepared Nanochitosan/PVP (1:1), NC/PVP/SF (1:1:1) with and without adding cross linking agent.

1.1 Materials and Methods

50 gram of Chitosan with an average molecular weight of 1,00,000 and degree of deacetylation of 79.1% was taken and slowly added to 1litre of 1:1 formic acid with constant stirring. The mixture was then heated and a whitish viscous gel of chitosan– formic acid solution is formed.1 gram of chitosan in 200 ml of 2% acetaldehyde prepared in the form of homogeneous solution. 0.8g of sodium hexameta phosphate [TPP] was then added and dissolved in 107 ml of water. Now the nanochitosan suspension was formed. The silk was boiled in 0.5% wt of Na_2CO_3 aqueous solution for 40 minutes to remove the sericin, then rinsed three times successively and soaked in distilled water over night. The degummed silk was dried in an oven at 600°C for 7 hrs[5]. 4gram of nanochitosan was taken in a clean beaker, 4g of PVP was dissolve in water and added with nanochitosan.0.5g of Silk fibroin and 4ml of glutaraldehyde was added. The solution was stirred well with moderate agitation for 20 minute films of nanochitosan, Silkfibroin, PVP composites were obtained by casting the solutions on to tissue culture and then dried under atmospheric temperature.

2. Results and Discussion

FTIR and DSC Analysis

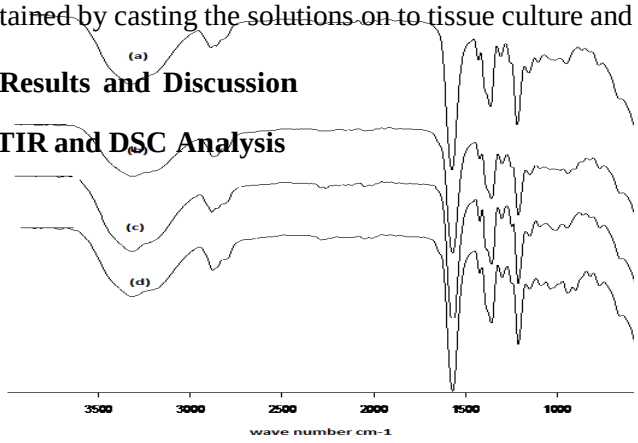

Figure 1. FTIR Spectra of polymer blends

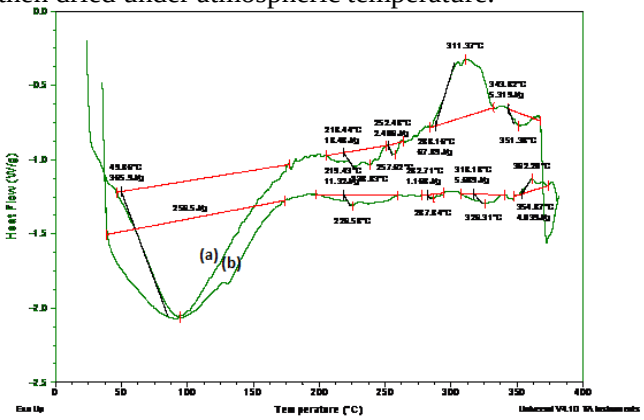

Figure 2. DSC curve

In Figure 1. Curve (a): The FT-IR Spectra of polymer blend of NC/PVP (1:1) shows the prominent peaks at 3392cm^{-1} confirms the intermolecular hydrogen bonding, N-H Stretching, O-H Stretching and strong polymerization. Peaks observed at wave numbers such as 2954.70cm^{-1}, 2925.40cm^{-1}, 2472.78cm^{-1},2329.22 cm^{-1}1639.95 cm^{-1}corresponds to asymmetrical and symmetrical C-H Stretching in CH_2 and CH_3,C=O and amide band –CO-NH.Similarly Curve (b): NC/PVP (1:1) in the presence of glutaraldehyde, Curve (c):NC/PVP/SF (1:1:1) and Curve (d): NC/PVP/SF (1:1:1) in the presence of glutaraldehyde shows the prominent peaks at 3389.80cm^{-1}, 2948.54 cm^{-1},2360.73cm^{-1}, 2223.09 cm^{-1},1372.82 cm^{-1} corresponds to intermolecular hydrogen bonding, N-HStretching and O-H stretching. C-H Stretching in methyl group O-H in plane bending in alcohols,3389.80cm^{-1}, 2948.54 cm^{1},2360.73cm^{-1},2223.09 cm^{-1},1372.82 cm^{-1}corresponds to aliphatic primary amine N-H Stretching, Methyl C-H asymmetric and symmetric stretching and 3388.16 cm^{-1}, 2950.29 cm^{1},2360.66cm^{-1},1642.18 cm^{-1},1494.95 cm^{-1}corresponds to aliphatic primary amine N-H Stretching, Methyl C-H asymmetric and symmetric stretching respectively .

In Figure 2. the curves a, b of NC/PVP/SF (1:1), NC/PVP/SF (1:1), reveals that NC/PVP/SF were mixed well and the molecules of individual polymer cannot crystallize to result in an increase of the amorphous regions with mixed molecules. The glass transition temperature (T_g) of NC/PVP (1:1) is 49.86°C and NC/PVP (1:1) in presence of glutaraldehyde is 50.5°C, the endothermic peaks changes from 351.38°C to 354.87°C.The endothermic peaks and the glass transition temperature (T_g) of NC/PVP in presence of cross linking agent glutaraldehyde are changed to higher values.

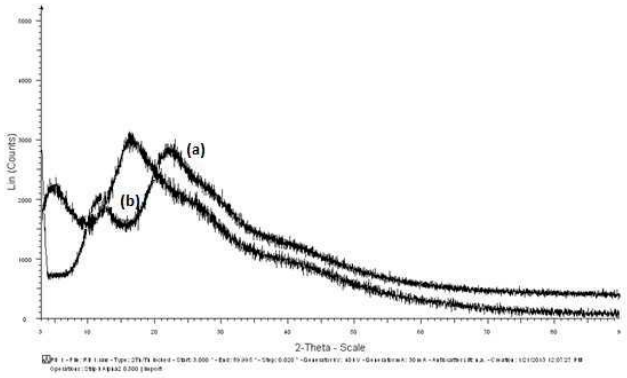

Figure 3. XRD

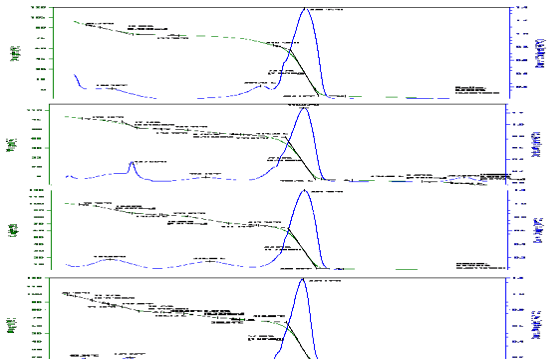

Figure 4.TGA

X-ray diffraction and Thermal Gravimetric analysis

X-Ray diffractograms of Nanochitosan and PVP (1:1) and NC/PVP/SF (1:1:1) composite films are shown in the Figure 3.The diffraction pattern with a broad amorphous peak indicated that there was a molecular miscibility and interaction between the components.The prepared films were subjected to TGA analysis. The TGA thermo grams of NC/PVP/ (2:1), Nanochitosan/PVP (1:1:1) + Glutaraldehyde, Nanochitosan/PVP (1:1) + Glutaraldehyde and Nanochitosan/PVP (1:1:1), Silk fibroin+ Glutaraldehyde are shown in the figures.The curve region above the temperatures 100°C indicates the decomposition of structure. The TGA of blended polymer shows different decomposition temperatures. Maximum decomposition was observed around 450°C which might be due to the decomposition of side chains. The Maximum weight loss occurs at temperature range of 250-400°C from this we can conclude that the blended films are thermally stable.

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