STUDIES ON MODIFIED SEMI-IPN CHITOSAN POLYETHYLENE GLYCOL BLENDED HYDROGEL FILM FROM PINK TIGER PRAWN SHELL WASTE

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ABSTRACT

Highly deacetylated chitosan (CS) was prepared from chitin (CT) extracted by the exoskeleton of pink tiger prawn (*Metapenaeus Dobonii*), which is a by-product of sea food industries. A preparative method has been established to obtain highly deacetylated chitosan having the desired degree of deacetylation up to 98.6%. Effective deacetylation was readily attained by intermittent washing the intermediate product in water during the alkali treatment. The functional groups interaction between chitin and chitosan were analyzed using Fourier Transform Infrared (FT-IR) spectroscopy. The important parameters for production of highly deacelylated chitosan product are degree of deacetylation (DDA) and molecular weight. The DDA of chitosan product was determined by hydrogen chloride titrimetric method and also confirmed by cross polarization magic angle spinning technique (^{13}C CP–MAS NMR). From ^{13}C CP-MAS NMR, the prepared chitosan was found to be 98.6 %DDA. The molecular weight of prepared chitosan was measured by intrinsic viscosity method and also confirmed by Gel Permeation Chromatography (GPC). M_p of the prepared chitosan product was 6.57 x 10^5 Da (GPC). Based on the elemental data, the atomic ratios of carbon to nitroge were found to be 8:1 and 6:1 which are in agreement with the monomeric units of chitin ($C_8H_{13}NO_5$) and chitosan ($C_6H_{11}NO_4$). The macrofibril structure of prepared chitin and chitosan were recorded by Scanning Electron Microscopy. From SEM micrographs, the prepared both chitin and chitosan showed epicuticle and membraneous layer, macrofibrils which are arranged orderly in a row to form lamellar layers. From the XRD analysis, the prepared chitin exhibits the high crystallinity and prepared chitosan showed hydrated crystal characteristics. In this study, the film forming ability of chitosan was evaluated with modified Semi–IPN chitosan hydrogel film prepared by blending with polyethylene glycol (PEG). The criteria ratio was based on the performance

mechanical properties such as tensile strength, elongation at break % and tear strength. All prepared CS Film and CS – PEG blended hydrogel films are smooth surface, highly transparent and of pale yellow colour. Comparative characterization of CS hydrogel film and CS – PEG blended hydrogel films includes FT-IR analysis, SEM analysis and XRD analysis. The thermal properties of CS hydrogel film and CS- PEG blended hydrogel films were also studied by Thermogravimetric analysis (TG – DTG) and Differential Scanning Calorimetry (DSC). From FT-IR analysis, the characteristic absorption peaks of CS- PEG blended hydrogel film clearly showed that the two polymers are blended. SEM micrograph of CS- PEG blended hygrogel film showed layer by layer film formation. According to the XRD analysis, all of the prepared chitosan hydrogel films indicated semi–crystalline nature. From TG-DTG thermogram profile, the thermal decomposition of CS-PEG blended films were observed slightly increasing in temperature as compared to pure CS hydrogel film. DSC thermogram of all prepared hydrogel films exhibited exothermic peak at about 300°C, which indicated polymer decomposition, fragmentation and unzipping of the polymer chain. The property of various types of chitosan hydrogel films and CS-Cellulose composite membranes are tested on antibacterial activity using agar disc diffusion method. From these results, all of the prepared CS hydrogel film, CS-PEG blended hydrogel films and CS- Cellulose composite membranes showed effective antibacterial activities. SEM micrographs of CS-PEG-Cellulose composite membrane showed that an increase in evaporating time favours the decrease in pore size diameter on the surface of well–defined CS-PEG-Cellulose composite membrane.

Keywords: chitin, chitosan, %DDA, molecular weight, ^{13}C CP MAS NMR, FT-IR, CS- PEG hydrogel film, XRD, SEM, thermal analysis, antibacterial test, CS–Cellulose composite membrane.