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Synthesis of chitosan based gel and study of swelling characteristics

Navneet Bhullar*[,1], Kamlesh Kumari[2] & Dhiraj Sud[3]

[1,2]Department of Chemical Engineering,
[3]Department of Chemistry, Sant Longowal Institute of Engineering and Technology, (Deemed University), Longowal - 148 106, Sangrur, Punjab, India

E-mail: navneetphd2010@gmail.com

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Synthesis of chitosan based grafted gels and their swelling characteristics have been recorded in terms of (P_s) percent swelling. Initiator potassium per sulphate is used for polymerization of chitosan gels under microwave radiations. Synthesized hydrogels are characterized by various physico-chemical techniques viz. Scanning electron microscopy (SEM), Fourier transform infrared spectra analysis (FTIR), X-ray diffraction (XRD) and Thermal gravimetric analysis (TGA). The swelling behavior of chitosan based synthesized gels has been observed to determine the stability of gels in aqueous medium. The dependence of swelling behavior of chitosan gel on monomer, initiator concentration and reaction time has been investigated. The optimization of monomer concentration for getting samples with maximum percent swelling (311.53%) observed is 35 mL of 5 molL^{-1}. Further decreased percent swelling (P_s) is observed when monomer concentration increase which could be due to the more compactness of the samples. The maximum water uptake capacity has been observed under *p*H 7 at 25°C.

Keywords: Chitosan, Swelling Characteristics, Hydrogels

A renewable polymer, Chitosan obtained from deacetylation of chitin has remarkable properties like; non toxic, biocompatible, biodegradable, and antibacterial. These unique properties of natural polymer make it promising material in the field of effluent treatment, clinical analysis, cosmetic preparation and food industries. These and many other applications of chitosan gels are principally dependent on water sorption behavior which provides insights into the network structure of the polymer and mechanism of water transport processes. Chitosan, a natural abundant renewable biopolymer, is a linear polymer of 2-acetamido-2-deoxy-β-D-glucopyranose linked through β (1→4) linkage to 2-amino 2-deoxy-β-D- glucopyranose[1]. The unique properties of chitosan make it the promising material for inclusion in waste water, clinical analysis, cosmetic preparation and food industries[2-4]. The modification of natural polymer to form grafted polymer, semi-IPN and IPN, introduced the sensitivity of the polymeric materials to the conditions of surrounding environment and they act as smart materials[5-9]. These smart materials have diversified application in biomedical fields such as membrane dialysis, drug delivery systems and artificial implants[10-12]. A number of applications of chitosan based smart materials are principally related to the water uptake behavior of smart polymeric materials. The present work reports the synthesis of chitosan grafted gels and their swelling characteristics were investigated as a function of swelling percentage.

Experimental Section

Chemicals and Reagents

Chitosan (Hi-media), acetic acid (Hi-media), acrylic acid (S.D fine) and thiourea (Merck) were used. Millipore water was used for the preparations of the various solutions. Analytical grade chemicals were used without further purification.

Preparation of Chitosan solution

Chitosan flakes (0.5 g) in 10 mL of 3% acetic acid were used to prepare chitosan solution. The reaction mixture was kept through the whole night at room temperature and the viscid solution was obtained for the synthesis of grafted chitosan gels.

Synthesis of chitosan grafted gels

The requisite amount of acrylic acid (5 molL^{-1}) was added drop by drop to the filtered homogenized viscous chitosan solution under continuous stirring. The solution was stirred for 10-20 min at room temperature, followed by the addition of appropriate amount of potassium persulphate to the homogeneous mixture to initiate the reaction under continuous stirring for 20 min. The mixture was poured into the reaction flask and kept under the microwave radiations operating at 100 W for 45 min where an aqueous environment was generated within the microwave system using water containing glass bowl. The obtained synthesized gel was washed with millipore water to remove homopolymer and dried in the oven at 60°C till a constant weight was obtained. Various reaction parameters such as effect of solvent, initiator concentration and reaction time, monomer and

crosslinker concentrations were optimized with respect to percent swelling (Ps) using following equation:

$$Ps = \frac{Ws - wd}{wd} \times 100$$

where Ws and Wd are the swelled weight and dry weight, respectively.

Results and Discussion

A smart polymeric material was synthesized by grafted copolymerization of acrylic acid onto the chitosan viscous solution in the presence of potassium persulphate initiator under microwave radiations. Synthesized material was characterized by various physicochemical techniques. The SEM images showed clear demarcation in surface morphology of chitosan and grafted gel. FTIR spectra of synthesized smart gels were recorded on Perkin Elmer-RXI FT-IR. The absorption band at 3426 cm^{-1} was observed which corresponds to the hydroxyl group and NH stretching of chitosan and acrylic acid polymer. In Fig. 1, peaks around 1662, 1598 and 1540 cm^{-1} correspond to CO and NH_2 stretching vibrations of amide groups. Thermal analysis was carried out using TG/DTA 6300, SII EXSTAR 6000 under the temperature range 50 to 700°C. The TGA and DTG curves of grafted gel showed well differentiated wt. loss steps. Decomposition at higher temperature corresponds to breakdown of grafted chains shown in Fig. 2. XRD was recorded using Philips X part diffractometer

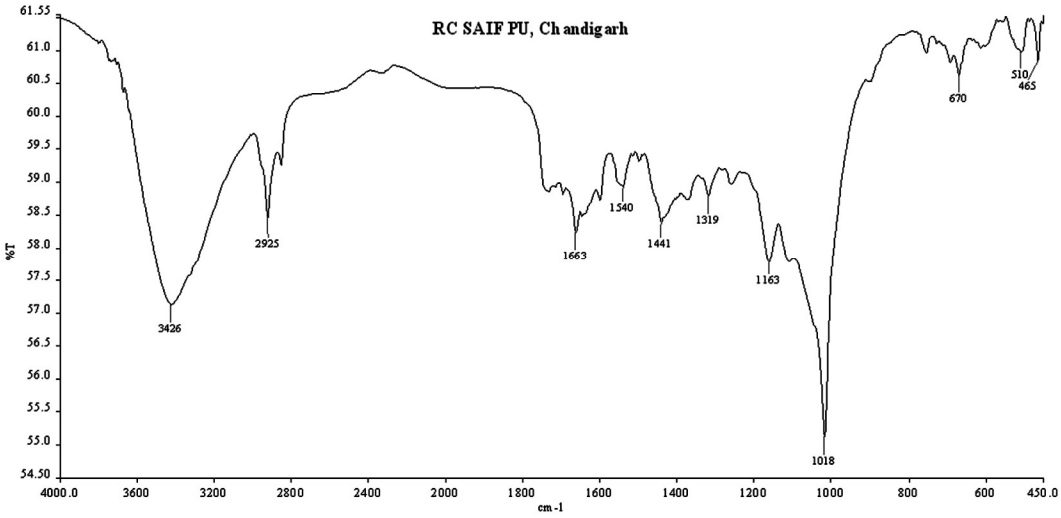

Fig. 1 — FTIR spectra of synthesized chitosan based grafted hydrogel

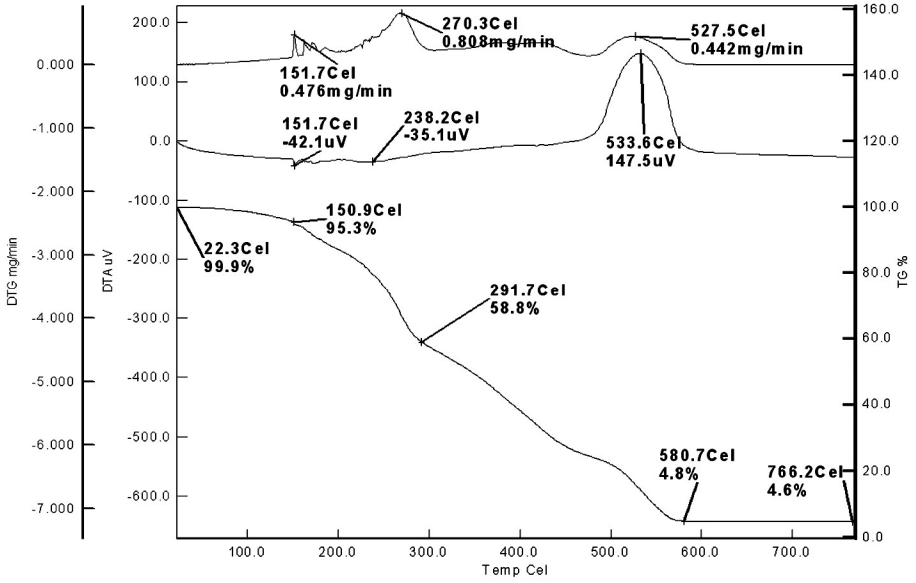

Fig. 2 — TG/DTA/DTG spectra of synthesized chitosan based grafted gel

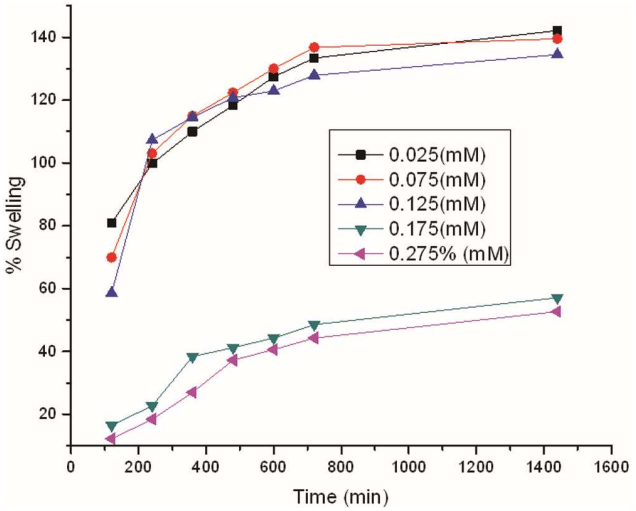

Fig. 3 — Effect of Monomer concentration (mM) on % swelling of chitosan based grafted

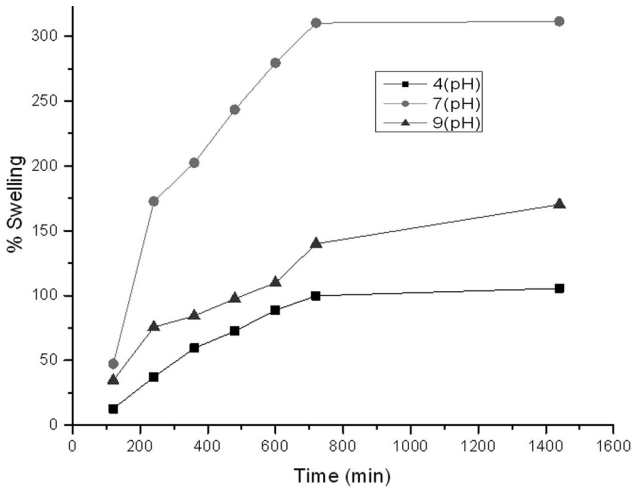

Fig. 4 — Effect of *p*H on % swelling of chitosan based grafted gel

(Almelo, Netherlands) with monochromatic CuKα radiation operating at 40 kV and 20 mA. Broad peaks indicating the amorphous nature of synthesized smart material.

Swelling behavior of smart material was investigated by varying different reaction parameters like reaction time, *p*H, initiator, and monomer concentration as a function of maximum swelling so as to get a device with maximum water uptake capacity. The swelling percent get increased by varying the concentration of initiator in the range 2.5×10^{-3} -3.5×10^{-3} mM (Fig. 3), beyond the 3.5×10^{-3} mM concentration of potassium persulphate, the swelling percent get decreased. The increase in swelling percent at lower concentrations is due to large free voids between the polymeric chains in the polymer network. The increase of acrylic acid in the reaction mixture of grafted gel in the range 0.1 - 0.25 mM resulted in decrease in percentage swelling. It is due to high density of the gel network. Further, the study was carried out to observe the *p*H effect on swelling behavior as a function of swelling percent. Three buffer solutions with *p*H 4, 7, and 9 were used. Figure 4 shows the effect of the swelling media on swelling percentage .The chitosan hydrogel, in water, showed about 311.53%, while in a basic medium just 170.2%, and in an acid medium, 105.56% was observed. This is due to the presence of functional groups (like -COOH, $-NH_2$) which get ionized and acquire a charge (+/) at specific *p*H.

Conclusion

The swelling behavior of synthesized gels was observed to determine the stability of gels in aqueous medium. The monomer concentration for getting samples with maximum percent swelling (311.53%) observed was found to be 35 mL of 5 $molL^{-1}$. The maximum water uptake capacity was observed under *p*H 7 at 25°C.

Acknowledgement

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