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Synthesis and Characterization of poly(N- tert-amylacrylamide-co-Acrylamide/Sodium acrylate)$CaCO_3$ Nanocomposite Hydrogels

S.Nisha[1], P. Jeyanthi[1] and P. Pazhanisamy[2*]
[1]*Department of Chemistry, Queen Mary's College (Autonomous), Chennai-600 004, India*
[2]*Department of Chemistry, SirTheagaraya College, Chennai – 600 021, India*

ABSTRACT

In the present study, a series of poly (N-tert-amylacrylamide –co –acrylamide / Sodium acrylate) Calcium carbonate nanocompositehydrogels were synthesized by free-radical copolymerization in Water/Methanol medium using Ammonium persulfate(APS) as the initiator and N,N'-methylenebisacrylamide (MBA) as a crosslinker. The amount of N-tert-amylacrylamide (NTA)and Acrylamide (AM) monomers are fixed (50:50) and the amount of Sodium acrylate (AcNa) ionic monomer was variedfrom 0.1g. The swelling behavior of Nanocomposite Hydrogels studied by Gravimetric method and degree ofswelling was increased with respect to types water.

Keywords: Hydrogels Sodium acrylate, swelling behavior, Copolymerization

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I. INTRODUCTION

Hydrogels represent polymeric networks capable of absorbing large quantities of water, but remain insoluble due to chemicalor physical crosslink between individual polymeric chains. Recently, there is a great deal of interest concerning the productionof nanoparticles in the hydrogel networks since theyhave enormous valuable applications in bio-related fields.Indeed, the design and development of nanoparticles and nanostructurematerials have opened a new era for constructingwell designed nanostructures that have been considered as a novel class of materials for catalytic, optical, electronic and biomedical application.Polymer nanocomposite containing metal nanoparticles can be prepared by several methods. Methods used for preparationcomprise mechanical mixing of a polymer with metal nanoparticles, the in-situpolymerization of a monomer in the presence of metal nanoparticles and the in situ reduction of metal salts or in a polymer.
The $CaCO_3$ has become the focus due to its advantages including affordability, low toxicity, biocompatibility, cytocompatibility, pH sensitivity and sedate biodegradability and environment friendly materials. In this article, we will discuss the potential roles of $CaCO_3$-NPs in three major therapeutic applications; as antimicrobial, for drug delivery, and as gene delivery nanocarrier. Technology has introduced new methods in producing of newly codified products with improved efficacies, namely; nanoparticles (NPs). NPs can be prepared from various materials such as metals, metal oxides, synthetic polymers, proteins, polysaccharides or other organic based molecules [1] with various applications ranging from as contrast agents in medical imaging technology to drug delivery nano-systems [2-4]. $CaCO_3$ nanoparticles have attracted an interest among researchers nowadays especially for therapeutic applications. $CaCO_3$ present in three common polymorphs such as calcite, vaterite and aragonite. Calcite has high stability index and being studied in various sizes, shapes and structures [5,6] and naturally found in trigonal crystalline form. The other two polymorphs exist in metastable forms are vaterite usually colorless with hexagonal crystal system [7] and aragonite is also naturally occurring carbonate minerals [8].Calcium carbonate based material present the biodegradability and biocompatibility properties which is ideal as a smart carrier to deliver genes, enzymes, and drugs [9,10] In this work, we have synthesized $CaCO_3$ nanocomposite hydrogel by in situ polymerization of N-tert-amylacrylamide, Acrylamide and Sodium acrylate (AcNa) using ammonium persulphate as free radical initiator and N,N' – methylenebisacrylamide(MBA) as cross linker. Swelling behavior and swellingkinetics of the obtained hydrogels were evaluated.

2.MATERIALS AND METHODS

Materials

Acrylamide (AM,Merck) was recrystallized from acetone/ethanolmixture. Ammonium persulphate (APS), Acrylic acid andSodium hydroxide were supplied from Aldrich. The crosslinkerN,N'-methylene-bis-acrylamide(MBA) was used as received

Acrylonitrile

Acrylonitrile was first washed with 5% NaOH solution in waterto remove the inhibitor and then with 3% Orthophosphoricacid solution in water to remove basic impurities. Then theAcrylonitrile was washed with double distilled water and dried over andhydrous CaCl2. The acrylonitrile was then distilled inan atmosphere of Nitrogen at reduced pressure. It was thencollected in a clean dry amber colour bottle and kept in therefrigerator at 5°C(1)

Preparation of sodium acrylate (AcNa)

Sodium acrylate was prepared by neutralizing the acrylic acidusing Sodium hydroxide (1).

Poly(N-tert-amylacrylamide - co - Acrylamide / Sodiumacrylate) $CaCO_3$ Nanocomposite Hydrogels

The hydrogel were prepared by free radical copolymerizationof NTA, AM and Ac Na in the presence of MBA as crosslinkerand APS for initiating the polymerisation system. Aqueoussolution containing a weighed amount of NTA, AM, MBA,APS and certain amounts of AcNa (0.00, 0.10, 0.30, 0.50g)were dissolved in methanol – water (3:1) mixture and final volumewas made 10mLin a polymerization tube. A solution containing10mg of Calcium Carbonatenanoparticle was added with constantstirring. After bubbling nitrogen for 15 min, the contents wereplaced in thermostatic water bath at 60C and the polymerization[1].

FT-IR spectralCharacterization

FT-IR spectra were recorded in KBr matrix in 400-4000 cm-1by FT-IR spectrometer.

Collection of wastewaters

The water samples were collected fromCooum river at Mylapore and sea water from Merina Sea as shown in Figure 1.

Figure 1. Wastewater from Cooum and sea water

Swelling behavior of $CaCo_3$ Nanocomposite Hydrogel

The swelling behavior was measured by immersing weighed samples of dry hydrogels in double distilled water. The excess surface water in the swollen gel was removed by blotting and then weighed. The degree of swelling (DS%) commonly described as swelling ratio is expressed as increases in weight/g of dried hydrogels after keeping in contact with water forselected period of time. The swelling behavior of the hydrogels was carried out in water at room temperature. 0.1 g of hydrogel was immersed in 100 ml of salt water, normal water, cooum water, After the time interval the weight of the swelling gel noticed.

$$SR\% = \frac{W_D - W_S}{W_D}$$

W_D weight of the dry gel, Wsis the weight of the swollen gel at a given time.

3. RESULTS AND DISCUSSION

The required amount of monomers were particles were dispersed in the monomer solution. The solution were purged with nitrogen gas for 30 minutes. The free radical polymerization was carried out 60 C for 20 hours. The synthesized hydrogels were dried at room 50°C.

FT-IR spectral Characterization

The synthesized hydrogel was characterized by FI-IR spectroscopy. The FT-IR Spectrum of poly (NTA-CO-AM/AcNa) $CaCO_3$ nanocomposite hydrogel is shown in Figure 2.. The peak at 780, conform the presence of $CaCO_3$ Nanoparticle in the polymer matrix. The other peaks at 3300, 1770 and 1085 for NH, C=O and CH stretching respectively.

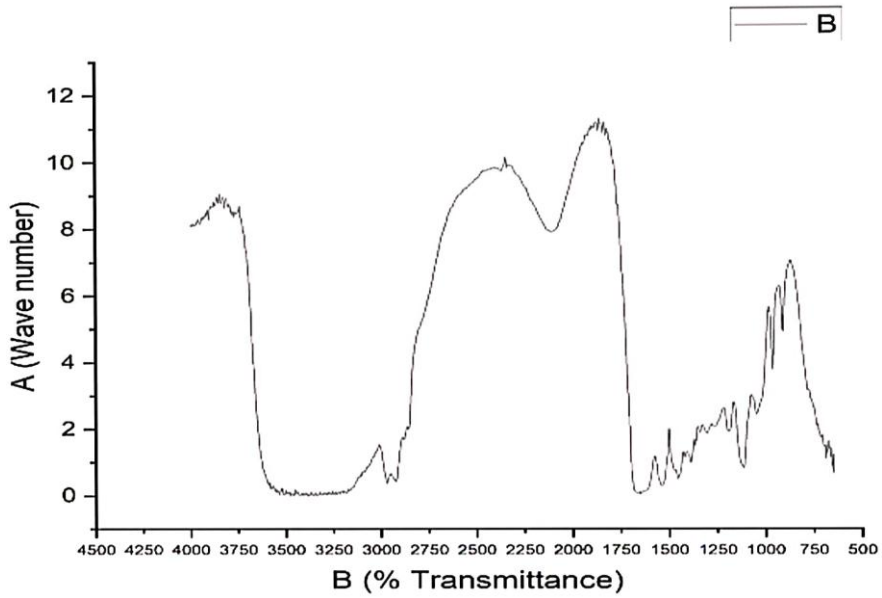

Figure 2. FT-IR spectrum of Poly (N-tert-amylacrylamide - co - Acrylamide / Sodiumacrylate) $CaCO_3$ Nanocomposite Hydrogels

Swelling behavior of $CaCO_3$ Nanocomposite Hydrogel

The swelling ratio is defined as the difference between the initial weight of dry hydrogel and the weight of fully swollen gel divided by the initial weight of dry gel. The swelling rate increases of sizes in salt solution (Ref: Table 1 & Figure 3). The presence of NaCl (electrolyte) suppress the swelling rate. The slowing SR rata may due to the reaction of NaCl with $CaCO_3$.cooum water after treatment having move swelling rate than the other type of water, because of free from metal ions and electrolyte.

Table 1.Swelling behavior of $CaCO_3$ Nanocomposite Hydrogel

	S.R%		
TIME	Diss.wat	sew.wa	salt water
15	95.45	113.8	77.8
30	108.18	188.8	81.74
45	118.18	240	103.2
60	140	245	116.5
90	204	263	145.4
120	272.72	304	168
180	338.18	406	173.7
240	352.7	412	186.3

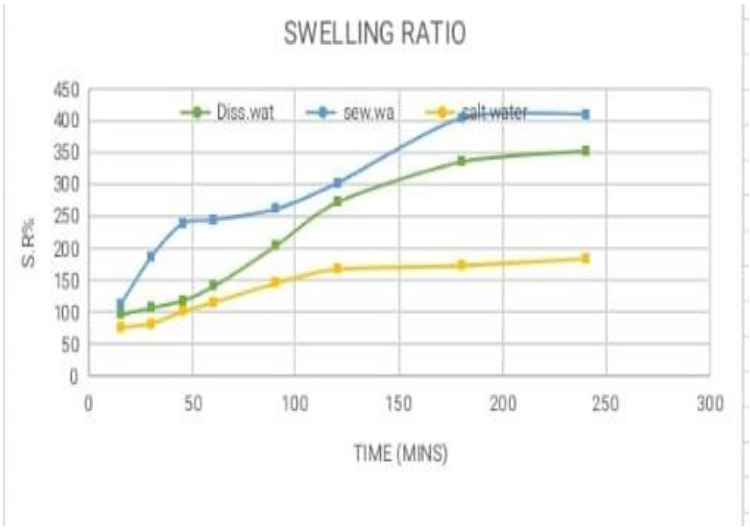

Figure 3. Plots of swelling behavior of $CaCO_3$ Nanocomposite Hydrogel

4.CONCLUSION

The Calcium carbonate Nanocomposite containing hydrogel were prepared by free radical polymerization at 60°C. The synthesized hydrogel was characterized by IR spectroscopy. The swelling rate of the hydrogel increases with respect to the types of water containingions.

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