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RESEARCH PAPER

Preparation and Superabsorbent of a Novel Copolymer Nanocomposite: Optimization of Swelling Behavior

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ABSTRACT

Biosorbents are environmentally friendly, readily available, have high absorption efficiency, and are desirable for the treatment of contaminated water. Herein, a covalently cross-linked green macro particle hydrogel nanocomposite CMC(IA-coAm-Ca) as a bio-adsorbent was prepared through a polysaccharide carboxymethyl cellulose reaction with acrylamide (Am)-Itaconic acid (IA) modified carboxymethyl cellulose (CMC) and further Ca(II) crosslinking polymerization. To limit the structure and characteristics of the nanocomposite composite several techniques were used such as (HRTEM), (FESFM/EDX), (XRD), and (FTIR),. The practical experiments included the study of improving the preparation conditions produced by a nanocomposite with the maximum SR%: the effect concentration of CMC, the effect of Am, the effect of IA, the effect of Ca(II) ion crosslinking effect of pH, and effect of temperature. The results show that employing (1 g) CMC, (2 g) Am, 2 ml IA, and (2 g) Ca(II) ion crosslinking. The hydrogel nanocomposites highest swelling ratio SR% in DW was 2100%. It was found that the mechanical water retention properties are strongly affected by monomer to CMC ratio and concentration of Ca(II) ion crosslinking. thus, the hydrogel displayed swelling behaviors that were monomers-dependent.

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INTRODUCTION

Hydrogels are described as having a three-dimensional network. They are classified as polymers that can swell significantly and also maintain their shape and structure in water over long periods of time. Hydrogels maintain their structure and cross-linked nature of each polymer chain, which enables them to retain a large amount of water. [1] Hydrogels are prepared by cross-linking polymer chains chemically or physically to form a network three-dimensional. Hydrogels are characterized by their high-water absorption capacity due to their crystalline network and high swelling capacity; however, they are not able to dissolve in water. Therefore, bio-based hydrogels have gained biocompatibility, environmental friendliness, and biodegradability. It is not enough for hydrogels to consume large

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amounts of water or biological fluids due to their high swelling capacity and remain insoluble in water. Biopolymer-based hydrogels have gained popularity due to their biocompatibility, swelling capacity, low-cost, high-water absorption, and biodegradability.[2]. Differences in the structure and composition of the polymer affect the crosslinking of the biopolymer and the different methods of crosslinking. For example, the great ability of biopolymers to swell and absorb water leads to Various applications such as in medicine and agriculture such as using them as wound dressings, personal care products, sanitary pads and diapers. [1, 3]. Poly-acrylamide is the primary network in several tough hydrogels. Poly acrylamide hydrogels containing salts are utilized as stretchable, transparent, ionic conductors in ionotropic. Polyacrylamide hydrogels have been utilized as a model material to study the growth of cracks in hydrogels under static cyclic and dynamic loads [4, 5]. Carboxy methyl cellulose (CMC) is a soluble in-water cellulose derivative that is bio-compatible and can be further chemically modified to form gel through physical interactions [6-8]. CMC is a soluble in water, anionic polysaccharide and is one of the most commonly used industrial cellulose ether. CMC is applied in systems where hydrophilic colloids are involved and has some applications in some industrial areas, including cosmetics, detergents, pharmaceuticals, textiles, food, etc. CMC displays the capability to suspend solids in an aqueous solution, stabilize emulsions, absorb moisture from the atmosphere, and is mostly utilized as a thickener, suspending aid, binder, gelling agent, stabilizer, and water retention agent, etc [9-12].

MATERIALS AND METHODS

Synthesis of CMC(IA-co-Am-Ca) gel

A CMC(IA-co-Am-Ca) gel was synthesized by applying polymerization. different quantities of Am (0.1–2 g/ 10 ml. After that, several quantities of CMC (0.15–4 g/ 20 ml) to 40 °C and IA (0.15–3 g/ 10 ml) the reaction mixture was stirred at 150 rpm for 90 min at 25 °C. The CMC(IA-co-Am-Ca) gel solution was dropped into a 100 mL $CaCl_2.2H_2O$ solution of 10–40 g/L. After the reaction was completed, the gel spheres continued to be cured in solution for 3 h, then they were removed, washed in distilled water, and dried. CMC(IA-co-Am-Ca) gel spheres were then prepared. Fig. 1 shows the image of gels.

Studies of Swelling of CMC(IA-co-Am-Ca) gel

The pre-weighted CMC(IA-co-Am-Ca) gel was placed in DW and kept at 25 °C for 120 min without any disturbance. The swollen CMC(ITA/AM-Ca) gel was blotted with filter paper to eliminate the additional DW, and after that weighed. The extreme RS% was calculated by Eq. 1:

$$\%\,\mathrm{SR} = \frac{\mathrm{M1} - \mathrm{M2}}{\mathrm{M2}} \times 100 \qquad (1)$$

W1 denotes the mass of the swelled CMC(IA-co-Am-Ca) gel, and W_2 mass of the dried CMC(ITA/AM-Ca) gel.

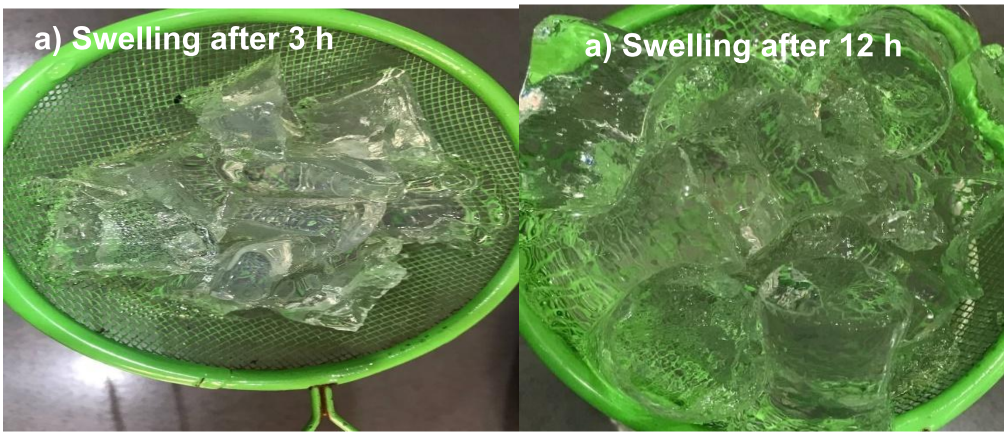

Fig. 1. Real image of preparation of CMC(IA-co-Am-Ca) gel.

RESULTS AND DISCUSSION

In the FTIR spectrum of the CMC(IA-co-Am-Ca) gel Fig. 2, The bands at 3400–3500 cm^{-1} arising from stretching group −OH converted to broader and fewer shifted at 3200–3400 cm^{-1}, The broadband from 3100 to 3300 cm^{-1} indicates stretching −OH in the polysaccharide. Peaks at 980, and 1000 cm^{-1} were assigned to C–O–C, and C–O–H stretching, of glucose stretching, respectively [13-15]. This can be interpreted as follows: A broad band from 3500−2750 cm^{-1} indicates −NH, −OH, and over-lapping bands from asymmetric cyclic CH, $-CH_2$, and $-CH_3$. Moreover, shifted peaks from 1500 – 1330 cm^{-1} represent aromatic rings C=C group, indicative of interactions [16, 17].

X-ray Diffraction (XRD)

Fig. 3 shows the XRD patterns of CMC(IA-co-Am-Ca) gel nanocomposite, that appeared broad peak in CMC(ITA/AM-Ca) gel nanocomposite, non-crystalline structure in the nanocomposite diffraction pattern, A peak looked at 2θ = 21.122°, and 2θ = 32.141° suggesting conversion from amorphous to semi-crystalline nature. The amorphous nature was also reported for the polymer of CMC with either IA or Am in the literature [18, 19]. This confirms the contribution of the grafting CMC. The broadness of XRD patterns for hydrogel shows the dispersal of CMC in gel [20, 21].

The FESEM morphology technique was used to determine the surface morphology of CMC(IA-co-Am-Ca) gel nanocomposite at different weights of carboxymethyl cellulose. The FESEM microscopic image revealed in the form Fig. 4a, the gel with weight 2 g of (CMC). The surface has structures resembling thin threads with random, unordered aggregates as a result of the lack of overlap of (CMC) within the gel matrix due to the lack of cohesion of the gel and its collapse at high temperatures. As for the Fig. 4b weight (3 g) the CMC of the gel has a rough surface without any crosslinking between the CMC molecules. Some small white balls in the form of small aggregates were also observed. The CMC(IA-co-Am-Ca) gel nanocomposite form also showed a porous, sheet-like surface due to gel formation and cross-linking by Ca+2 between the polymeric chains. At the same time, CMC(IA-co-Am-Ca) gel nanocomposite face resulting from the introduction of CMC contains rough particles within the polymeric chains. The prepared surface is characterized as a heterogeneous spongy porous surface containing voids and numerous active sites. Hence it can swelling [22, 23]. EDX analysis of the samples (Fig. 4c), that the Ca(II) ions in solution underwent a cross-linking polymerization reaction with CMC to form a CMC(IA-co-Am-Ca) gel nanocomposite with a three-dimensional network structure. The surface scan analysis of CMC(IA-co-Am-Ca) gel nanocomposite Ca, elements are

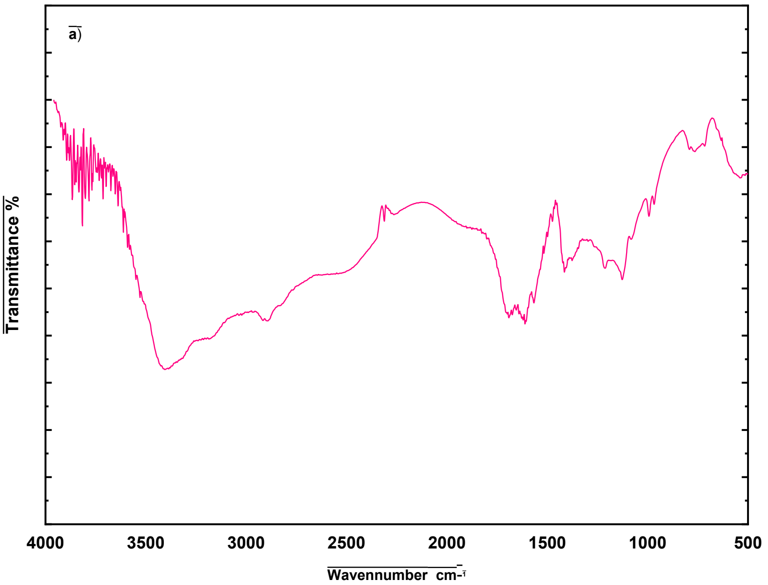

Fig. 2. FTIR spectrum of the a) CMC(IA-co-Am-Ca) gel.

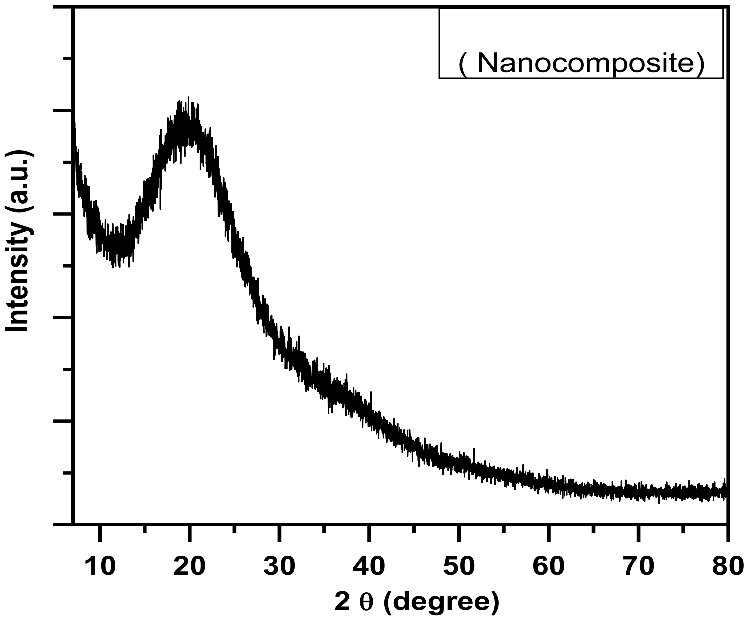

Fig. 3. XRD patterns of CMC(IA-co-Am-Ca) gel nanocomposite.

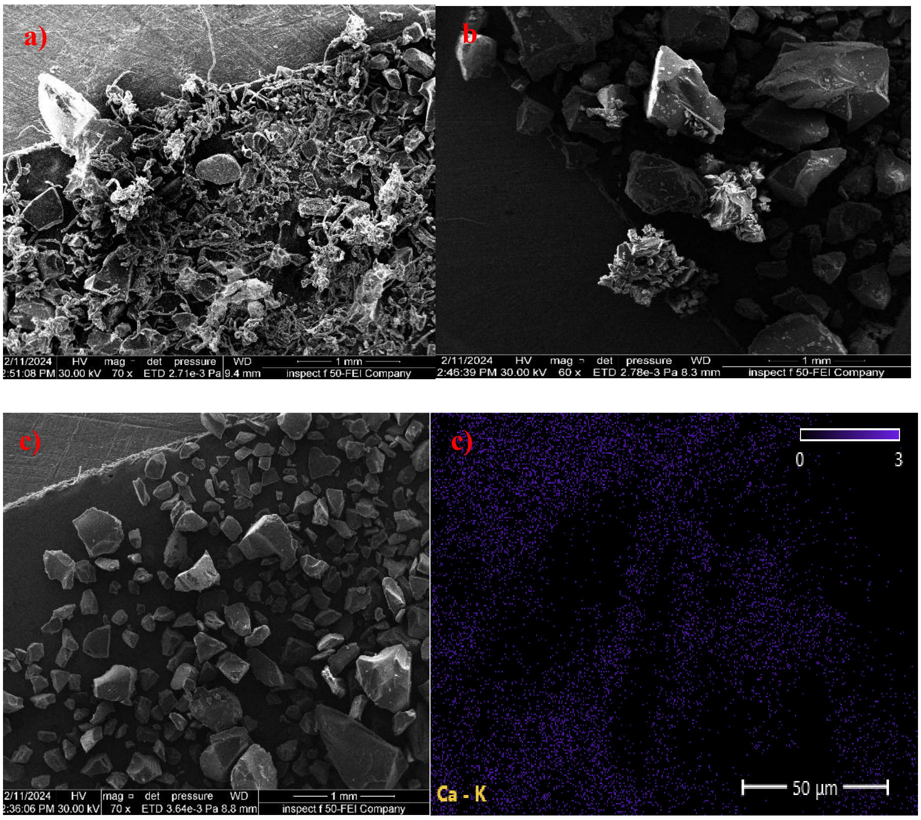

Fig. 4. FESEM a) 2 g of CMC in nanocomposite, b3 g CMC in nanocomposite c) 4 g CMC in nanocomposite, e) EDX of CMC(IA-co-Am-Ca) gel nanocomposite.

evenly distributed in the composite, indicating the successful preparation of the nanocomposite [24-26].

A higher resolution transmission electron microscope (HRTEM) image is shown in Fig. 5. This figure shows a carboxymethyl cellulose CMC(IA-co-Am-Ca) gel nanocomposite) embedded within a polymer matrix. It can be seen that the CMC(ITA/AM-Ca) gel nanocomposite appears in the form of small irregular random balls spread on the polymer surface with some patchy black aggregates and tends to form aggregates at 200 nm and 500 nm. Moreover, the surface of the CMC(IA-co-Am-Ca) gel nanocomposite is covered with a thin layer, in which carboxymethyl cellulose was observed incorporated within the CMC(IA-co-Am-Ca) gel and plays a pivotal part in enhancement stability and raising the surface area as an essential component. To synthesize ecologically friendly hydrogel[27-30].

Optimization Swelling of the CMC(IA-co-Am-Ca) gel nanocomposite)

The swelling behavior of the polymer is shown in Fig. 6a as a function of the IA. Different weights of IA from (0.5-2.5 ml), which shows the effect of several concentrations of IA. The magnitude of the effect of IA on swelling is similar to the Ca(II) crosslinking ratio because the incorporation of IA also increases the mechanical strength of the polymer and reduces the swelling behavior in the water of the resulting polymer, which occurs through the ability of IA to cross-link [31].

Fig. 6b displays the swelling capability of

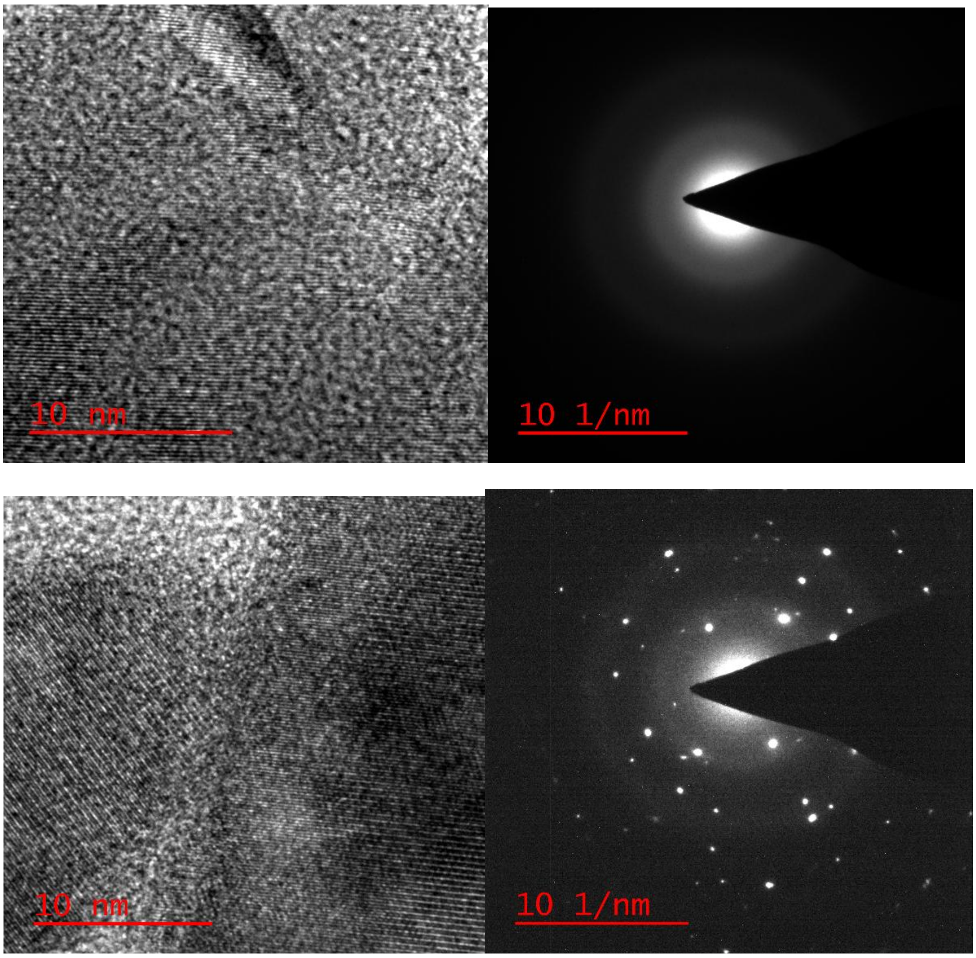

Fig. 5. Higher resolution transmission electron microscopy (HRTEM) image of CMC(IA-co-Am-Ca) gel nanocomposite)

nanocomposite with several concentrations of CMC (0.1–2g/20 ml). The RS% is firstly very low due to the repulsion between the negative charge caused via pairs of electrons on O2 and N2 charge on –OH in CMC. However, the rising concentration of CMC, increases cross-linking and difficulty absorbing water As a result, the RS% rises with increasing concentration of CMC. thus, greater quantities of CMC created extra active sites, leading in cross-linking optimal to best adsorbate loading on the adsorbent [32, 33].

The effect of acrylamide (Am) concentration on the bio-composite was examined by measuring the swelling ratio. Different amounts of Am (0.1-2.5 g) were dissolved in 5 ml of DW and added while stirring Fig. 6b, a maximum RS% of 1200% was achieved with 0.5 g. Similar to how insufficient monomer caused decreased swelling when the AM dose was less than 2.0 g, a decrease in the amount of monomer present resulted in a decrease in swelling by more than 2.0 g. Concentrations of Am above the recommended level. Absorbance may decrease with Am concentration above 1.0 g[34, 35]

To prevent the polymer chains from dissolving in water, Ca(II) was used as a cross-linker during polymer preparation. Use different amount of Ca(II) (0.1-2 g/L) via dissolving it in 100 ml of H_2O to increase the amount of cross-linker dimer. To reduce the water absorption of water absorbed

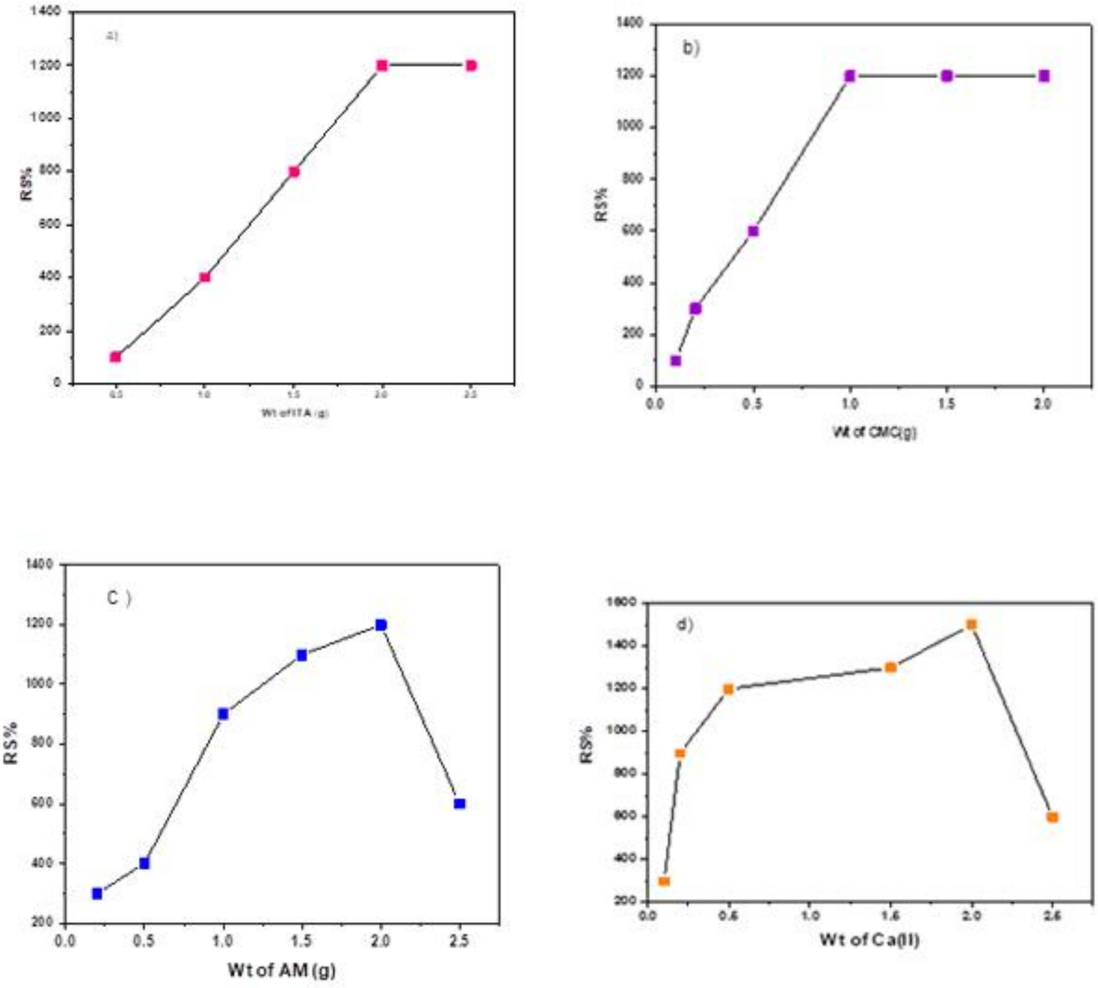

Fig. 6. Optimization Swelling of the CMC(IA-co-Am-Ca) gel nanocomposite, a) weight of IA, b) weight of CMC, c) weight of Am, and d) weight of Ca(II).

into the polymer network, which reduces the swelling ratio. Fig. 6d shows that less space among the copolymer chains resulted in more rigid 3D networks and even less surface area. The optimal mesh size for cross-linking affects the amount of contaminants trapped in the polymer due to the concentration of Ca(II) as a cross-linker. At little concentrations, the degree of crosslinking cannot adequately trap contaminant particles. Cross-linked Ca(II) was added during polymer preparation to form network crosslinks that prevent dissolution of the polymer chains in water. Increasing the concentration of calcium (II) led to a decrease in water absorption within the polymer network, which reduces the swelling behavior [33, 36].

CONCLUSION

A semi-natural, non-toxic CMC(IA-co-Am-Ca) gel nanocomposite was synthesized by polymerization of carboxymethyl cellulose (CMC). The optimization study revealed that the CMC(IA-co-Am-Ca) gel nanocomposite had a greater swelling ratio at the concentration of monomer (AM, 2 g), polysaccharide (CMC,1g), and crosslinking Ca(II) (2 g). It has been found that the mechanical properties of water retention are strongly influenced by the monomer-to-monomer ratio and the crosslinking concentration of calcium(II) ions. The best course of swelling was presented in deionized water by approximately 1200.3 %. the RS% rises with increasing concentration of CMC. thus, greater quantities of CMC created extra active sites, leading to cross-linking optimal to best adsorbate loading on the adsorbent, Cross-linked Ca(II) was added during polymer preparation to form network crosslinks that prevent dissolution of the polymer chains in water. Increasing the concentration of calcium (II) led to a decrease in water absorption within the polymer network, which reduced the swelling behavior.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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