Synthesis and Swelling Characterization, of Eco-friendly Cellulose/poly(AAc-co-AAm)/Ag Nanocomposite

Fatmah A, Abdulsalam, [1] Moussa Khlifa[1] and Mahdi M. Almaky. [1]*

[1]Department of Chemistry, Faculty of Science, Sebha University, Sebha, Libya

** E-mail: Mou.khlifa@Sebhau.edu.ly*

***Abstract*—Eco-friendly cross-linked cellulose/poly(AAc-co-AAm)/ silver nanocomposite was successfully synthesized via simple free radical graft polymerization in presence of *N,N'*-methylene bisacrylamide (MBA) as crosslinking agent and potassium persulphate as an initiator.**

The grafted cellulose copolymers hydrogels AgNPs were characterized using several characterization techniques such as ultraviolet spectrometry (UV-Vis), scanning electron microscopy (SEM) and X-ray diffraction (XRD). Microscopic analytical techniques exhibited an average diameter of 39-55 nm for AgNPs, as well as give information about shape and surface topography of the nanocomposite. XRD pattern of all samples showed that the finger print of Ag, cellulose and poly(AAc-co-AAm) have been observed approving the preparation of superabsorbent Ag nanocomposites.

The influence of variables such as composition ratio and $AgNO_3$ concentration, on the swelling behavior of prepared hydrogels. The degree of swelling was substantially increased with decreasing AAc : AAm molar ratio from 1:3 to 1:2. Effect of silver nitrate concentration result show the water absorption increase with increase of $AgNO_3$ concentration. A maximum swelling of 669 g/g and 515 g/g respectively in distilled water and tap water were obtained when 15mmol of $AgNO_3$ was incorporated

***Keywords*— Acrylic acid, acrylamide, swelling, cellulose, Ag nanoparticles**

1.Introduction

Polymeric hydrogels (PHs) are considered as one of the most promising materials due to their unique properties and wide scale of applications. PHs are hydrophilic polymers that are cross-linked and have the ability to absorb significant amounts of water without disintegrating [1, 2].

Superabsorbent polymers (SAPs) are a special class of hydrogels with very high absorption capacities. Hydrogels have a capacity to swell or absorb up to 10 grams of water per gram of material. In comparison, Superabsorbent polymers have an astonishing ability to swell and absorb water ranging from 600 to 1000 grams per gram of material [3]. Their resistance to dissolving in water is due to their cross-linked 3-dimensional polymer structure. When superabsorbent polymers come into contact with water, they absorb the water and produce a gel-like substance. The water is contained within the polymer network, located between the cross-linked sites. The 3D polymer structure is formed by crosslinking polymer chains using physical and/or chemical methods. The hydrophilic functional groups, such as -OH, -COOH, -CONH-, -CHO, -COONa, -SO_3H, -NH_2, in the polymer chains play an active role in absorbing water between the crosslinks that join the chains [4, 5].

Superabsorbent polymers are hydrophilic polymers characterized by their remarkable ability to absorb and retain enormous amounts of water, salt solutions, and aqueous fluids. SAPs, first created in the 1960s for hygiene items such as diapers, have advanced to become essential in other industries [6]. These polymers consist of lengthy polymeric chains with a small degree of cross-linking. Their water absorption capability is due to hydrophilic functional groups connected to the polymer backbone, but their resistance to dissolution is due to the cross-links between network chains [7].

The SAP contains water that enables the unrestricted movement of certain solute molecules, with the polymer material acting as a structure to retain the water. These polymers are created by polymerizing water-soluble monomers by a free radical process in the presence of an appropriate cross-linking agent [8]. SAPs are widely used because to their distinctive physicochemical properties, which make them well-suited for applications that need water absorption and retention. superabsorbent polymers have transformed the agriculture industry by boosting soil water retention, which helps address water scarcity problems and boosts crop yields. SAPs are used in wound dressings and controlled medication release systems in the medical profession [9].

The worldwide community is increasingly focused on developing (SAPs) that fulfill performance standards and adhere to sustainable practices in response to environmental issues [10]. The environmental consequences related to conventional SAPs, especially their poor biodegradability, have led to a search for environmentally friendly substitutes. Among these hydrogels polyacrylamide (PAAm) is one of the most popular[8]. In addition, partially neutralized polyacrylic acid (PAAc) has also been proven to give high performance SAP[11]. Cellulose, a natural biopolymer derived from plants, is a great base for creating nano-composites due to its biocompatibility and sustainability. Integrating synthetic polymers like poly(AAc-co-AAm) improves the mechanical characteristics and durability of the nano-composite, enabling specific functions [8]. Adding silver nanoparticles (AgNPs) to SAPs enhances their antibacterial capabilities. Silver nanoparticles are recognized for their strong antibacterial properties and show potential

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for use in situations where inhibiting microbial growth is crucial [9]. Researching SAPs using customized copolymers and silver nanoparticles is driven by the goal of achieving a balance between superior performance and environmental sustainability [11, 12].

The aim of this paper is concerned with the study of the effect of numerous synthesis parameters, such as, composition ratio, $AgNO_3$ concentration on the swelling behavior of prepared cellulose/poly(AAc-co-AAm)/Ag nanocomposite. The results were then compared with other reported hydrogels AgNPs.

2. Materials and methods

2.1 Materials and Chemicals

Acrylic acide (AAc), Acrylamide (AAm), N,N'-methylene bisacrylamide (MBA), and Potassium persulphate (KPS) are analytical grades and were purchased from; LOBA CHEMIE *Co.* India and used as received. Silver nitrate($AgNO_3$) from(SCARL) and Cellulose($C_6H_{10}O_5)_n$ from(Scharlau). All other used agents were analytical grade, and all solutions were prepared with distilled water.

2.2 Synthesis of Cellulose/Poly(AAc-co-AAm)Ag NPs

one-pot method was used to synthesize the nanosilver-containing biodegradable superabsorbent polymer/cellulose composite. An amount of 7.11 g acrylamide was dissolved in 50 ml distilled water and mixed in a three necked round bottom flask and heated to 40 °C under nitrogen protection in a water bath. The solution is then mixed for 20 minutes, then 2.5 ml acrylic acid was added to the solution. 0.05 g of cellulose was added into the flask while stirring, followed by 0.05 g of MBA as crosslinking agent. An amount of 0.1 g KPS as initiator dissolved in 10 ml distilled water, was slowly added into the flask to initiate the polymerization process. Silver nitrate solutions of different dosages (5, 10 and 15 mmol) and glucose (5,10 and 15ml) were added into the system and mixed at 65 °C for 1 h to reduce Ag+ into silver particles. The solution was then heated at 80 °C while mixing for 2 hours. The polymers thus formed are composites of biodegradable cross-linked cellulose poly(AAc-co-AAm)/silver nanoparticles. The obtained SAP composites were dewatered using ethanol and then poured into a petri dish and dried in the oven for 24 h at 60 °C. Finally, the dry samples were crushed into powder and sifted for subsequent experiments and tests.

2.3 Characterization Techniques:

The goal of nanocomposite characterization not only conform synthesis of nanocomposite in size less than 100 nm (at least one dimension) but also give some information about shape of particles, crystallinity, surface topography and zeta potential, etc.

A series of samples which are prepared using different concentrations of silver nitrate solution.

- Poly(AAc-co-AAm) (**SAP1**) and Cellulose/Poly(AAc-co-AAm) (**SAP2**)
- Superabsorbent copolymer/Ag nanocomposite when the concentration of $AgNO_3$ is 5 mmol (**SAP3**)
- Superabsorbent copolymer/Ag nanocomposite when the concentration of $AgNO_3$ is 10 mmol (**SAP4**)
- Superabsorbent copolymer/Ag nanocomposite when the concentration of $AgNO_3$ is 15 mmol (**SAP5**)

2.3.1. X-ray diffraction (XRD)

The as prepared nanoparticles were studied utilizing a X-beam diffractometer (XRD, D8-Find, Bruker, with CuKα radiation (1.5418 Å), Madison, WI, USA) working at a current of 40 MA, voltage of 40 kV and step filter 0.01º. in any case, the dried samples should be ready before estimation, tests processing utilizing basic planetary ball factory (LZQM0.4L, Shicheng Desert spring Mineral Gear Assembling Co., Ltd.) in which ball factory of treated steel of 0.1 cm breadth put in processing measure with tests for 1 hour at 1500 rpm.

2.3.2. Ultraviolet Spectrometry (UV - Vis)

UV –vis spectra were recorder using an evolution 300 spectrophotometer with a double beam principal system with date recording using vision software version on windows xp/2000. (Biochrom,Cambridge,uk).

2.3.3. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) was used to visualize the composites nanoparticles and investigate their morphology. SEM images were taken using a Zeiss LEO Supra 55VP Field Emission and SEM Zeiss 1530. For sample preparation, composite nanoparticle suspensions were diluted 10 times with their dispersion medium, and then, a drop of the diluted nanoparticle suspension was directly deposited on a polished aluminum sample holder. Samples were dried under a vacuum. Samples were then coated in gold using EMITECH K450X sputter coater

2.3.4. Transmission Electron Microscope (TEM)

The shape and texture of nanoparticles were scanned via the high-resolution Transmission Electron Microscope (HRTEM, JEOL TEM-2100, Japan) with an accelerating voltage of 250 kV and magnification of 20 X. however, samples have been prepared before measurement by sonicated infusion extract-nanoparticles by sonication prop under condition of plus every 1 seconds at 85% amplitude power maximum temperature for 30 Minutes. Finally, 50 micron added to SEM grade with air dry for 5 hours.

2.3.5. Degree of swelling

The degree of swelling of SAPs represents the percentage increase of the volume of SAPs in water. Weighted quantities of the as prepared Poly (AAm-co-AAc) dry samples were immersed in excess distilled water and tap water at room temperature to reach an equilibrium of swelling. Swollen samples were then separated from unabsorbed water by

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filtered over a 0.25 mm screen. Degree of swelling (g/g) was determined by weighting the swollen samples and then calculated using the following equation:

$$\mathbf{Ds = W - W_o / W_o}$$

Where; W, Wo are the weights (g) of swollen and dry samples respectively.

3. Results and discussion

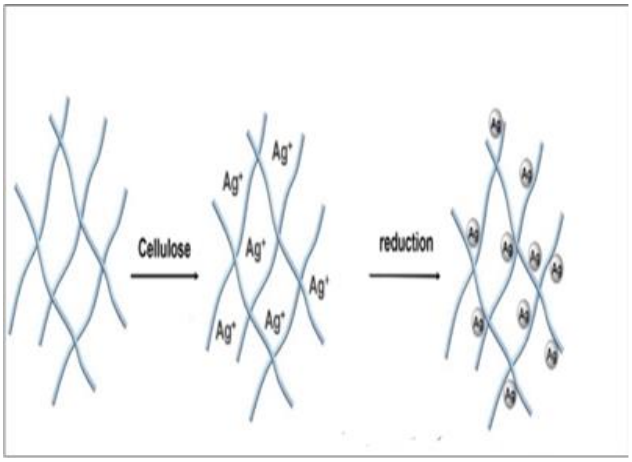
3.1 Synthesis of Cellulose/ Poly (AAc-co-AAm) Ag Nanocomposite

The superabsorbent hydrogel Cellulose/ Poly (AAc-co-AAm) AgNPs under investigation was prepared according to a procedure involved graft copolymerization both AAc and AAm on Cellulose under the free radical initiating effect of KPS and MBA as crosslinker. In order to initiate the grafting process, the initiator was first decomposed by heating to 60 °C to produce the free radicals which further initiated cellulose.

The persulphate initiator decayed to generate anionic radical upon heating. This anionic radical can abstract hydrogen from the hydroxyl group of Cellulose to form macro-radicals which initiate the monomers polymerization, resulting to the formation of the grafted chain. It should be note that the polymerization and crosslinking of AAm were independent of the added Cellulose, despite the possibility that the small part of Cellulose could be grafted or copolymerized with the AAm polymer chains since free radical are generated in the structure of cellulose and AAm at the same time. Because of the presence of the crosslinking agent, MBA, the chain structure polymerized into a network structure.

On the other hand, the presence of the cellulose also influenced the swelling behavior sine it interacted through hydrogen bonds with AAm, causing a restriction in the polymer chains mobility [13].

Since silver is a transition metal with empty orbital, it can form carboxylate metal ion complexes [14] . Because of the strong localization of the silver ions within the network, the Ag NPs are immobilized throughout the network as a result of complexation of the silvers ions by the carboxyl groups and carboxylic acid groups of the polymers [15]. Cellulose contains carboxyl groups that can stabilize the silver ions. Then, the added glucose can reduce Ag ions to Ag NPs (Scheme 1). It should be monition that, the generation of silver nanoparticles in cellulose grafted poly (AAc-co-AAm) results in more pores, which corresponds to the increase in water absorbency of the SAP. In accordance with previous reports [16]. The free network space between hydrogel networks reserve and stabilize Ag NPS. Furthermore, the SAP loaded with silver nanoparticles exhibit great antibacterial activity [17].



Scheme1. shows the procedure of the preparation of Ag NPs loaded SAP.

3.1.1 UV-Vis of Cellulose/poly(AAc-co-AAm)/Ag Nanocomposite

Three representative $AgNO_3$ concentrations (5, 10, and 15mmol) were taken to study their influence on the formation of AgNPs. **Figure 3.1** displays UV-vis spectra of Cellulose/poly(AAc-co-AAm)/Ag-NPs sheets. The maximum absorbance occurs in the range of 463-467 nm regardless of $AgNO_3$ concentration, but the intensity increase as a function of molar concentration. This peak corresponds to the surface plasmon resonance absorption of silver NPs [18, 19], indicating the formation of AgNPs in the polymer composites.

It can be seen that the higher $AgNO_3$ concentrations, the more intense the SPR absorption peak. This could be due to the reduction of Ag^+ to Ag atoms increased which resulted in an increase the AgNPs in the reaction [20]. By increasing the $AgNO_3$ concentrations from 5 to 10 mmol the reduction of Ag^+ increases and large number of particles are formed. Furthermore, broad SPR band was observed for lower $AgNO_3$ concentrations (5mmol). As the amount of $AgNO_3$ concentrations increased, SPR became sharper along with an enhancement in its intensity. However, the intensity of SPR band decreases and broadens for a lower $AgNO_3$ concentrations. This results confirms formation of AgNPs in the polymer matrix which are in agreement with our SEM and XRD observations.

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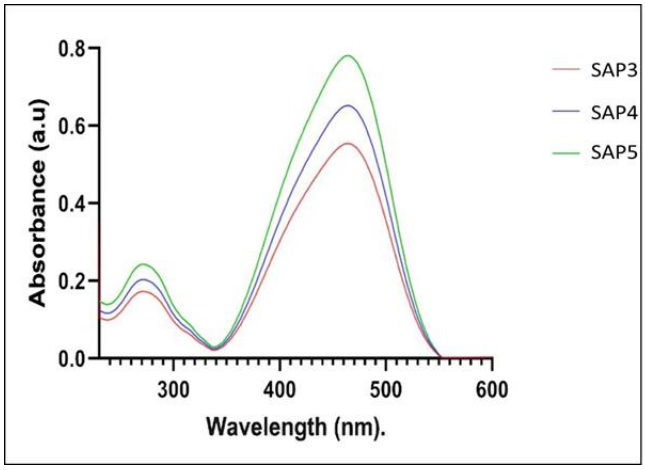

Figure 3.1 UV-Vis spectra of Cellulose/poly(AAc co-AAm)/Ag NPs

3.1.2 XRD of Cellulose/poly(AAc-co-AAm)/Ag Nanocomposite

XRD pattern illustrate the success in preparation method to synthesisCellulose/poly(AAc-co-AAm)/Ag Nanocomposite. As shown in **Figure 3.2**, the finger print of XRD pattern of AgNPs, poly(AAc-co-AAm) and cellulose have been observed. However, characteristic peaks of cellulose was found at 2θ = 16.5 and 23°, which matched previously reported patterns in literature[21]. These results indicated that cellulose occurred of grafting with the poly(AAc-co-AAm. While the XRD characteristics peaks of poly(AAc-co-AAm) was not remarkable due to the higher intensity value of celluloses nanoparticle and silver nanoparticles.The XRD pattern of the composite silver nanoparticles exhibited four diffraction peaks at 2θ = 38.2, 44.5, 64.7, and 77.8°, which are indexed to the characteristic (111), (200), (220) and (311) diffractions of Ag crystals with face-centered cubic structure[110]. These results clearly suggest that the AgNPs were successfully embedded in the polymer matrix and modified the functional properties of SAP.

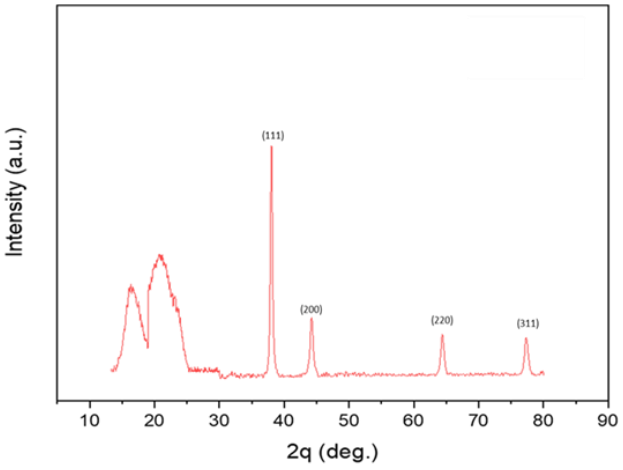

Figure 3.2 XRD of Cellulose/poly(AAc-co-AAm)/Ag Nanocomposite

3.1.3 SEM of Cellulose/poly(AAc-co-AAm)/Ag Nanocomposite

To investigate the formation and distribution of silver nanoparticles on cellulose fiber, the nanocomposites surfaces were examined and compered the results with blank sheet by SEM. **Figure 3.3** shows the SEM images of silver-cellulose/hydrogel nanocomposites. The images of plain hydrogel and silver-cellulose/hydrogel nanocomposites were observed throughout the surface of the samples: SAP1, SAP3, SAP4 and SAP5 respectively. The smoothness and cleanliness surface as seen from the pure polymer (SAP1). In contrast, SAP3, SAP4 and SAP5 show the in-situ reduction and immobilization method successfully produced AgNPs as a white dots dispersed across the cellulose surface. It can be seen that each particle is individually dispersed without any evidence of aggregate formation. Regardless of AgNPs amount, the morphology of the prepared Ag nanoparticles was clearly observed in cubic shape.

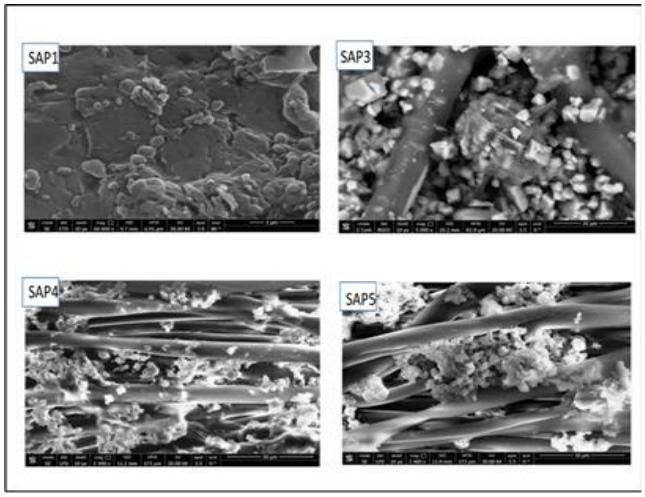

Figure 3.3 SEM images of Cellulose/poly(AAc-co-AAm)/Ag Nanocomposite

3.2 Swelling behavior

The main characteristic of hydrogels is their ability to retain a large amount of water or aqueous solutions, so it is important to characterize this property. Therefore, swelling property of SAPs is of interest for many applications.

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There are many factors influencing water absorbency of the cellulose Poly(AAc-co-AAm) sliver nanoparticles.

3.2.1 Effect of AAc:AAm molar ratio and water type on the degree of swelling

From the view point of practical applications, it is more important to study the swelling behaviors of SAP composites in various of monomers molar ratio. The swelling behaviors of superabsorbent composites and their effect of variation of AAc:AAm molar ratio on degree of swelling were investigated, as shown in Table 1. It is shown that degree of swelling of SAP increased by decreasing AAc:AAm molar ratio from 1:3 to 1:2.As the ratio of AAm monomer increased in the copolymerization feed, the active sites can react easily with AAc monomers, results in high viscosity of the medium and decrease in diffusion of monomers to active sites to produce cross-linked hydrogels, which result in decrease in the degree of swelling [22].

The swelling degree in deionized water increased from 58.0 g/g to 64.6 g/g, in distilled water Ds increased from 73.8 g/g to reach 103.8 g/g, while in tap water it increased from 32.6 g/g, to 53.6 g/g. Further decrease in AAc:AAm molar ratio to 1:1 causes decrease in degree of swelling up to 24.0 g/g in deionized water, 38.2 g/g in distilled water, and 14.2 g/g in tap water. Thus, the optimum degree of swilling is 103.8 g/g at molar ratio of 1:2 (AAc: AAm) in distilled water. Additionally, we can also understand from **Figure 3.4**. that the swelling capability of all samples (A, B, and C) in distilled water is higher than theirs in deionized and tap water. In the tap water the increase in ionic strength of the water leads to a decrease in swelling of SAP hydrogels. When the polymer is in tap water the osmotic pressure developed is much lower so the swelling is drastically reduced. An additional reason may be the increment of electrostatic attraction between anionic sites of chains and the cations in tap water resulting in an increase in the ionic crosslinking and subsequent loss of swelling [23]. Distilled water typically has a natural pH of 7. On the other hand, deionized water (pH<7) is produced by removing all ions but it may still contain dissolved gases and other impurities that can affect its pH. At higher pH, the carboxylic acid groups because ionized and the electrostatic repulsive force between the charged sites (COO^-)causes increasing in swelling[24]. This could be the reason for the distilled water has higher swelling compared to the ionized water (see **Table 3.1** and **Figure 3.4**).

Table 3.1 Degree of swelling (g/g) of the prepared SAP1 in different types of water

SAP1 Type	Degree of swelling, Ds (g/g)		
	Deionized water	Distilled water	Tap water
A	58.0	73.8	32.6
B	64.4	103.8	53.6
C	24.0	38.2	14.2

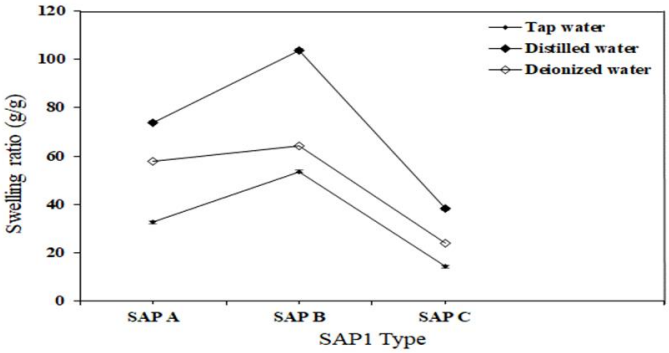

Figure 3.4 The effect of monomers molar ratio and water type on the degree of swelling of Poly (AAc-co-AAm).

3.2.2 Effect of AgNPs on the degree of swelling

The Cellulose/poly(AAc-co-AAm) swelling ratio can reach 185 g/g in distilled water and 118 g/g in tap water when silver nanoparticles are absent. However, when the silver nanoparticles are embedded in the SAP, the swelling ratio increased significantly[89]. **Figure 3.5 and Table 3.2** show the swelling behavior of the SAPs prepared in in different types of water. The SAP2 appear to show a slightly lower degree of swelling than other SAPs AgNPs (i.e. SAP3, SAP4 and SAP5). The water absorption capacity of the SAP is mainly due to its three-dimensional network structure and hydrophilic groups [23]. It also can be seen that with an increasing concentration of $AgNO_3$, the swelling ratio of the SAPs increases. The swelling ratio of Cellulose/poly(AAc-

co-AAm)/Ag nanocomposite's increased to 519 g/g and 469 g/g with 50 mmol of AgNO3 added to distilled water and tap water, respectively. By increasing the $AgNO_3$ concentrations applied to distilled water and tap water from 5 to 10 mmol the swelling ratio increased to599 g/g and 495 g/g. Furthmore, 15mmol of AgNO3 added in distilled water and tap water reached 669 g and 515 g / g respectively, which is much higher than some other SAPs[20, 24]. The reason could be that the formation of Ag NPs can increase the pores and free space within the networks structure and as a consequence, the SAP can adsorb more water than before.

Table 3.2 Effect of AgNPs on the degree of swelling

SAP Type	Degree of swelling, (g/g)	
	Distilled water	Tap water
SAP2	186	118
SAP3	519	469
SAP4	599	495
SAP5	669	515

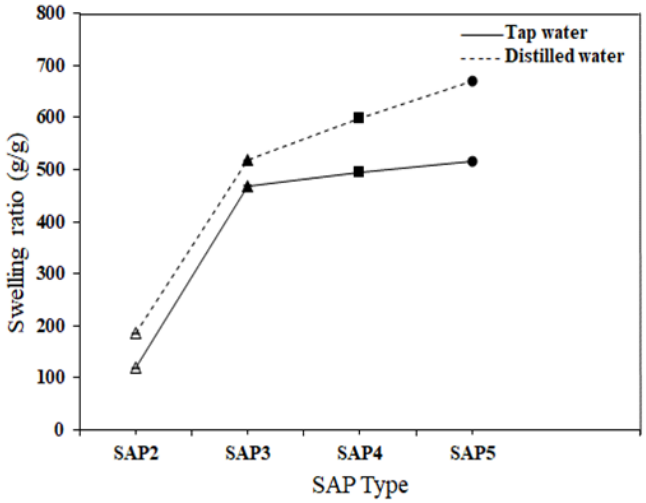

Figure 3.5 Effect of AgNPs on the degree of swelling

4. Conclusions

The overall conclusions are summarized in the following key points:

- Cellulose/poly(AAc-co-AAm) was synthesized by free radical polymerization at under nitrogen protection and used as a template for the fabrication of eco-friendly Cellulose/poly(AAc-co-AAm)/Ag nanocomposite.
- The formation of face centered cubic structured AgNPs imparted crystalline characteristics to the formed polymer/Ag nanocomposite.
- The AgNPs were formed in the diameters range of 39 to 55 nm and were evenly distributed throughout.
- The swelling behavior SAP hydrogels with different AAc:AAm molar ratios showed that the degree of swelling was substantially increased with decreasing AAc:AAm molar ratio from 1:3 to 1:2; then by decreasing AAc:AAm molar ratio to 1:1 the swelling ratio decreased.
- The effect of Ag NPs concentration on the swelling behavior of SAP copolymer was examined and showed that the increase in Ag NPs concentration resulted in a significant increase in the degree of swelling of such SAP in distilled water.

5. References

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