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An Environmentally benign Synthesis and Characterization of Novel Sugar based Silver Nanocomposite Hydrogel for Antibacterial Applications

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Abstract : The incorporation of nanometals into hydrogels have been extensively studied and found to have immense potential for medical, therapeutic and diagnostic applications. In the present investigation, we report an environmentally benign synthesis of sugar based semi interpenetrating hydrogel (SIH) networks of cross-linked poly(acrylamide) utilizing carboxymethylcellulose (CMC) – starch (SR) biocomposites as a grafting backbone and N,N’–methylenebisacrylamide (MBA) as the cross-linker. Invariably sized silver nanoparticles were generated in situ in the swollen hydrogel by the reduction of silver nitrate (AgNO₃) using azadirachta indica (neem) leaf extract at room temperature. UV-Visible spectroscopy, FTIR spectroscopy, scanning electron microscopy (SEM) and thermogravimetric analysis (TGA) were used to characterize the formation of silver nanoparticles in the hydrogel. The antibacterial activity of the semi interpenetrating silver nanocomposite hydrogel was also investigated.

Keywords: Silver nanoparticles, environmentally benign synthesis, acrylamide, carboxymethyl cellulose, starch, hydrogel, nanocomposites.

Introduction

Hydrogels are generally synthesised from natural and synthetic polymers. They possess numerous advantages over inorganic materials such as lower material cost and easy fabrication, better biocompatibility and biodegradability and more versatile processability and functionalization. Recently silver nanoparticles with unique optical, electronic and antibacterial properties have attracted much attention and have found many applications in the field of medicine and pharmaceutics. The most attractive features of silver nanocomposites is due to extraordinary enhancement in properties of hydrogels such as mechanical toughness, large deformability, high swelling/deswelling rates, excellent electrical conductivity, high transparency and remarkably strong antibacterial activity in combination with a fairly low toxicity against human tissues. Hydrogels with large free space among the cross-linked networks can not only act as a reservoir for massive nanoparticles loading, but also function as a nanoreactor template for the nucleation and growth of nanoparticles. Free radical graft copolymerization of vinylic monomers onto sugar backbones followed by cross-linking of their chains is a well known method for the synthesis of sugar based hydrogels. The general methods used to incorporate nanoparticles into the hydrogels includes gelation of preformed nanoparticles in a solution of hydrogel forming monomers, embedding of nanoparticles in the hydrogel networks by swelling – shrinking process, repeated heating, centrifugation, redispersion and entrapment of nanoparticles in hydrogel matrix followed by reduction.
with common reducing agents. However, drawbacks such as aggregation of nanoparticles in monomer solution before and during the gelation process, leaching of nanoparticles out of the network\textsuperscript{12}, forcing conditions for synthesis and the use of toxic reducing agents limit the potential of hydrogel nanocomposites for biomedical applications. This paves the way for biological methods, using plant extracts, which are cost effective and environmentally friendly\textsuperscript{13}.

To the best of our knowledge, based on the wide literature survey on super absorbent hydrogels, it is concluded that there is no published report on the synthesis of hydrogels in which two sugars (CMC and SR) and a synthetic monomer (AM) are used. The above findings inspired us to synthesize poly(acrylamide) – carboxymethylcellulose – starch hydrogel with certain hydrophilic nature for embedding the silver ions and reducing it to silver nanoparticles insitu using neem plant extract\textsuperscript{14} to obtain sugar based hydrogel – silver nanocomposite (HSN). The structure of HSN, effect of pH, monomer concentration on the swelling behavior of HSN and its antibacterial activity were studied.

**Experimental**

**Materials**

Acrylamide (AM), carboxymethylcellulose (CMC), starch (SR), ammonium persulphate (APS), silver nitrate (AgNO\textsubscript{3}), N,N\textsuperscript{-}methylenebisacrylamide (MBA), N,N,N\textsuperscript{-},N\textsuperscript{-}tetramethyl ethylenediamine (TEMED) of reagent grade were kindly supplied by S.D. Fine Chemicals (Mumbai, India) and used without further purification. Double distilled water (DDW) was used for the preparation of hydrogel and other solutions required in this study.

**Synthesis of sugar based semi interpenetrating hydrogel**

An improved single step method for the synthesis of SIH was conducted\textsuperscript{15} as follows. CMC (0.05 g) and SR (0.05 g) were mixed in 5 ml of DDW to get a homogeneous mixture. AM (1.0 g) was dissolved in 6 ml of DDW followed by cross-linker MBA (0.01 g in 1 ml of DDW) and initiator APS (0.005 g in 1 ml of DDW). Both the solutions were mixed and TEMED (0.02 ml in 1 ml of DDW) was added which, together with APS, acts as redox-initiating pair and initiates free radical polymerization. The reaction mixture was stirred and heated at 60º C for 5 minutes. The polymerization reaction results in the formation of SIH within 10 minutes of reaction time. The formed hydrogel was equilibrated with water for 3 days to remove unreacted monomers and reagents. The hydrogel was dried in hot air oven to constant weight.

**Plant information**

Scientific Name – Azadirachta Indica  
Common Name – Neem

Plant part used – Leaves  
Family Name – Maliaceae

**Preparation of plant extract**

Fresh leaves of Azadirachta indica (neem) were collected from the local territory. The leaves were cleaned by washing with double distilled water and dried using water absorbent paper. About 40 g of leaves were cut into small pieces using a sterilized scissor and ground in mortar and pestle to obtain a paste. This paste was dispersed in 100 ml of double distilled water and heated for 15 minutes at 70 – 80º C. The extract was filtered using Whatman’s No.1 filter paper and the filtrate was collected in a presterilized conical flask and used on the same day.

**Preparation of semi interpenetrating hydrogel – silver nanocomposite**

Dried hydrogel (500 mg) was equilibrated with double distilled water for 3 days at room temperature. The swollen semi interpenetrating hydrogel was transferred to beaker containing 50 ml of 0.005 M silver nitrate solution and allowed to equilibrate for 24 hours. These silver salt loaded hydrogel was transferred into another beaker containing 50 ml of neem leaf extract and allowed to stand for 12 hours. During this period the silver ions were reduced to silver nanoparticles which were confessed by the development of brown color in the
hydrogel. The silver nanoparticles loaded semi interpenetrating hydrogel was dried at ambient temperature and often termed in the forthcoming sections as HSN.

**Spectral methods**

FTIR spectra of completely dried SIH and HSN were recorded with a Perkin Elmer FTIR spectrometer – Spectrum.RX1 (USA). UV – visible spectra of HSN (10 mg in 1 ml of methanol) was carried out on a Shimadzu 160A UV – visible spectrophotometer (Japan). For this, the HSNin methanol was grinded and stored for 10 days to leach out silver nanoparticles and then filtered using Whatman’s No. 1 filter paper. This filtrate was used for recording the spectra.

**Scanning Electron Microscopy (SEM)**

Scanning Electron Microscopic (SEM) analysis was performed using Tescan Vega3 SBU variable pressure scanning electron microscope with 0.2 ml of finely grinded SIH and HSN dispersions on a copper grid dried at room temperature after removing excess solution using filter paper.

**Thermogravimetric analysis**

The thermal stability of HSN was evaluated using Mettler Toledo 851e thermal system (Switzerland) at a heating rate of 10°C per minute and a flow rate of 10 ml per minute under nitrogen atmosphere.

**Swelling studies**

The pre-weighed HSN of different CMC – SR concentrations were immersed in DDW at 37°C. After specific time intervals, the gels were taken out and weighed after removing the surface adhered solution. Similarly, the swelling ability of HSN in various buffer solutions was studied. The swelling ratio (Q) of the gels was calculated from the equation

\[ Q = \frac{W_e}{W_d}, \]

where \( W_e \) is the weight of swollen hydrogel and \( W_d \) is the weight of dry hydrogel.

**Antibacterial studies**

The antibacterial activity of HSN was carried out on Mueller Hinton Agar (MHA) medium (Hi-Media Pvt. Ltd. Mumbai) using Kirby – Bauer disk diffusion method16. About 5 ml of the Mueller Hinton agar medium was poured into the sterile test plates and allowed to solidify. The plates were inoculated with test pathogens using sterile swabs. Sterile wells were dug inside the culture plates with the aid of a sterile cork borer at aseptic conditions. Samples (1 mg/ml) were then added to the wells at aseptic conditions. Stock solutions of the samples were prepared using DMSO. The test plates were incubated for 24hours. The zone of inhibition (in mm diameter) was measured and taken as the activity of the prepared HSN against the test organisms.

**Results and discussion**

In the present work, we have designed a novel pathway for the synthesis of HSN exploiting sugars (CMC and SR) which usually couple there biodegradability with stimuli sensitive response. In this method, the reduction potential/anchoring ability of the hydrogel is increased along with the stabilization of embedded nanoparticles17-20. In any conventional hydrogel networks, the functional groups present and its cross-linking density decides the stability of nanoparticles. Hence, we have synthesized nanometer sized homogeneous dispersion of silver nanoparticles in SIH networks employing poly(acrylamide) with CMC and SR matrices. A prominent feature of this methodology is that the nanoparticles were simply obtained at room temperature in presence of environmental friendly stabilizers. In general, the PAM – CMC – SR cross-linked networks acts as reservoir for metal ions uptake and anchoring it through carboxylic, amide and hydroxyl groups. The polymeric network also facilitates the reduction of silver ions into nanosilver and stabilizes it by preventing the aggregation of silver nanoparticles. It is quite interesting to indicate that the silver nanoparticles were formed solely inside the hydrogel networks and not in the surrounding medium. This confirms the binding of silver nanoparticles to the hydrogel networks as well as its storage without releasing into the media. The scheme of formation of silver nanoparticles inside the SIH networks is given below:
SIH + AgNO₃  $\rightarrow$ SIH – Ag⁺ Nee leaf extract $\rightarrow$ SIH – Silver nanocomposite.

UV – Visible spectra

In this work, there is a development of opaque brown color after the addition of neem leaf extract to the swollen hydrogel. This observation can be explained based on the in situ formation of silver nanoparticles (Ag⁺) by the reduction of silver ions (Ag⁺) in the entire hydrogel. This also represents that the nanoparticles were entangled inside the swollen hydrogel networks through strong localization and stabilization established from the macromolecules of sugars. The perseverance of silver nanoparticles in the hydrogel networks was confirmed by UV – visible spectral analysis. Silver nanoparticles loaded hydrogel solution have shown a distinct peak around 400 – 414 nm in the UV – visible spectra due to the surface plasmon resonance (SPR) effect caused by the quantum size of the silver nanoparticles.

FTIR spectra

IR spectra of the poly(acrylamide) based hydrogel (Fig.1) shows the presence of absorption bands characteristic to cross-linking bridges. Absorption band attribution was made in agreement with the values given in literature. The absorption bands at 3450 and 1663 cm⁻¹ corresponds to –NH stretching vibrations from the cross-linking bridges and –C=O extension vibrations of amide groups respectively. Two bands at 3336 and 3194 cm⁻¹ corresponds to –OH stretching as well as intra and intermolecular hydrogen bonding in the glycosidic ring. The symmetric valence vibration at 2932 cm⁻¹ is assigned to the –CH₃ groups between the macromolecular chains and cross-linking bridges. Another band at 2862 cm⁻¹ is attributed to the –N–CH₂ vibrations from the PAM – SR cross-linking bridges. The stretching vibration of ester groups of CMC is observed at 1610 cm⁻¹. Absorption bands assigned to the deformation vibrations of the –CN, –OH and –CH₂ groups in the 1400 to 1200 cm⁻¹ range were also identified. Cluster of peaks from 800 to 1130 cm⁻¹ range are assigned to stretching of C – O in C – O – C and C – O – H of glycosidic bonds. In the IR spectra of HSN, the peaks were shifted to lower wave numbers (3310, 3182, 2919, 1655, 1601 cm⁻¹) due to the interaction of silver nanoparticles with hydrogel networks. Therefore, we can confirm the presence of silver nanoparticles in the hydrogel networks.

Figure 1. FTIR Spectra of (a) SIH and (b) HSN

Scanning Electron Microscopy (SEM)

The surface morphology of placebo SIH and the HSN were investigated with SEM. The placebo hydrogel showed a smooth surface feature (Fig. 2a), whereas HSN showed a shrunken surface throughout the gel (Fig. 2b). The particle size analysis confirms the presence of small, spherical and highly dispersed silver nanoparticles with an average size of about 50 – 100 nm. It is noticed that no individual silver particles were observed outside the HSN, suggesting a strong interaction between the polymer matrix and the silver particles.
Thermal stability

The thermogram of HSN displayed distinctive thermal stability as shown in (Fig.3). The hydrogel has followed two decomposition steps and only 72.31% degradation of the hydrogel occurred below 508°C. The high thermal stability of the HSN can be attributed to the presence of large quantity of silver nanoparticles inside the hydrogel network.

Swelling studies

The swelling behavior of any polymer network depends upon the nature of the polymer, polymer–solvent compatibility and degree of cross-linking. Hence, we have chosen sugar polymers (CMC and SR) to improve the swelling capacity of poly(acrylamide) hydrogel. The capability of hydrogels to swell in water is due to the hydrophilic groups present in the polymeric chains and its mechanical resistance is due in part to the physical or chemical cross-linking. It was known that polar head groups of polymeric chains such as hydroxyl, thiol, amine, and nitrile groups have high affinity for salts. The loading of silver ions throughout the gel networks causes repulsion among the networks, which ultimately leads to an improved swelling behavior of hydrogel systems. Further increase in swelling capacity was observed after the addition of reducing agent (neem extract) to silver ions loaded SIH. This is due to the formation of silver nanoparticles throughout the gel networks which increases the overall porosity of system and makes entry for more number of water molecules easier.
molecules inside the gel. The other reason can be that the formed particles have different sizes and surface charges which cause absolute expansion of the networks. The order of swelling was: HSN > Ag\(^+\) loaded SIH > SIH

The effect of CMC – SR concentration on water absorption of the HSN was investigated by varying the amount of CMC – SR from 0.1 g [0.05g + 0.05g] to 1.0 g [0.5g + 0.50g] whereas the concentration of AgNO\(_3\) was kept as 0.005M. As shown in (Fig. 4a), increasing CMC – SR amount up to 0.4g [0.2g + 0.2g] increases the swelling capacity. This was due to the introduction of more number of hydrophilic polymer chains inside the gel networks that assists in improving the swelling characteristics of gel systems. The decrease in swelling with further increase in CMC – SR amount [0.50g + 0.50g] can be attributed to the increase in viscosity of the medium which in turn hinders the movement of ions.

The swelling behavior of HSN in various pH was observed by placing 0.1 g of HSN in pH 2, 4, 6, 8 and 10 solutions respectively and examining them after an hour. It was noticed that the amount of swelling increases with increase in pH from 2 to 6, reaches maximum at pH 8 and then decreases as shown in (Fig. 4b). The repulsion between – COO\(^-\) groups in the hydrogel is the main reason for the maximum swelling at pH 8. Below pH 8, the H\(^+\) ions in the external medium effectively suppress the ionisation of the carboxyl and hydroxyl groups of CMC – SR. This decreases the number of mobile ions inside the HSN which causes decrease in osmotic pressure and hence the swelling capacity of HSN. Above pH 8, the –OH groups can ionise to – OR which reacts with – COO\(^-\) to form esters. This increases the number of networks inside the hydrogel and also decreases the hydrophilicity which is responsible for the decrease in swelling.

Antibacterial analysis

The main course of this study is to unfold a new antibacterial material. The antibacterial activity of different molar weight ratios of HSN were examined against Staphylococcus aureus, E. coli and Aeromonas spp. All HSN proved effective against the tested microorganisms and the growth inhibitory effects showed variation form one another. Fig. 5 exhibits the antibacterial property of HSN. The results indicate that the HSN exhibited greater reduction of bacterial growth which is due to the sustained release of silver nanoparticles form the hydrogel networks. The inhibition area of HSN for different organisms is given in Table. 1.
Figure 5. Antibacterial activity of HSN.

Table 1. Zone of Inhibition (mm)

<table>
<thead>
<tr>
<th>Organisms</th>
<th>Zone of Inhibition (mm)</th>
<th>Antibiotic (1mg/ml)</th>
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<tr>
<td></td>
<td>Concentration(µg/ml)</td>
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<tr>
<td></td>
<td>1000</td>
<td>750</td>
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<tr>
<td>Staphylococcus aureus</td>
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<td></td>
<td>24</td>
<td>20</td>
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<tr>
<td>E. coli</td>
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<td></td>
<td>21</td>
<td>17</td>
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<tr>
<td>Aeromonas spp.</td>
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Conclusion

CMC – SR / Poly(acrylamide) semi interpenetrating hydrogel – silver nanocomposite was successfully prepared via free radical polymerization followed by in situ reduction of silver ions to nanosilver using neem leaf extract as a clean and green reducing agent. The methodology developed for the synthesis is very simple and cost effective which makes it easy to implement in the industries. A number of HSN were formulated with high dispersion rates by varying the concentrations of CMC, SR and monomers. The formed HSN was characterized by different techniques. In the UV – visible spectra, nanosilver have shown good surface plasmon resonance behavior. In FTIR spectra, shifting of peaks to lower wave number occurred. SEM images confirm the presence of well defined silver nanoparticles and its stability was further confirmed by thermal analysis. The antimicrobial activities shown by silver nanoparticles make this method a potential route for metal nanoparticles preparation to be used for biomedical applications.

References


An eco-friendly synthesis, characterisation and antibacterial applications of novel almond gum – poly(acrylamide) based hydrogel silver nanocomposite

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ABSTRACT

Polysaccharide based semi interpenetrating hydrogel (SIH) networks of cross-linked poly(acrylamide) was synthesised through a redox initiating free radical polymerization utilizing almond gum as a grafting backbone, N,N′-methylenebisacrylamide (MBA) as the crosslinker and ammonium persulphate (APS) – N,N,N′,N′-tetramethyl ethylenediamine (TEMED) as the redox initiator pair. Silver ions were introduced into the hydrogel matrix and silver nanoparticles of invariable size were developed insitu of the swollen hydrogel by the reduction of silver ions (Ag⁺) using azadirachta indica (neem) leaf extract. The prepared hydrogel - silver nanocomposite (HSN) was characterized by UV-visible diffused reflectance spectroscopy (DRS), fourier transform infrared spectroscopy (FT-IR), high resolution scanning electron microscopy (HR-SEM), energy dispersive X-ray analysis (EDX) and thermogravimetric analysis (TGA). The influence of pH on the swelling behavior of HSN was studied and the antibacterial activity of the developed nanocomposite was evaluated.

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1. Introduction

Hydrogels are the most distinctive class of three dimensional polymeric networks which possess good hydrophilic character and can hold large quantity of aqueous solution without undergoing dissolution. The hydrophilicity of the hydrogel is due to the presence of functional groups such as –OH, –COOH, –NH₂, –CONH₂, –SO₃H within the polymer backbone or in lateral chains and its stability and insolubility are attributed to three dimensional networks developed by either physical or chemical cross-linking. These networks not only prevent the hydrogels from dissolution but also make them to swell in aqueous solution [1]. They can be derived from natural or synthetic sources and are being extensively explored for their potential in tissue engineering [2], wound dressing [3], biosensors and drug delivery devices [4–6]. Although synthetic polymers have well established structures suitable for such applications but natural polymers are preferred because of numerous advantages such as better biodegradability, easy fabrication, lower material cost, biocompatibility [7], hypotoxicity [8], more versatile processability and functionalization [9].

Metal nanoparticles such as silver, gold and copper are reported as highly toxic to microorganisms [10]. In recent years, silver nanoparticles have been extensively preferred for the production of medical products because of its strong cytotoxicity for various microorganisms and its wide use in controlling infections since ancient times [11,12]. However, the poor binding characteristic with surfaces limited its use as antibacterial agent. Therefore, silver nanoparticles are incorporated in the hydrogel networks for the preparation of hydrogel–silver nanocomposites [13]. Hydrogels with large free space among the cross-linked networks can not only act as a reservoir for massive nanoparticles loading, but also function as a nanoreactor template for the nucleation and growth of nanoparticles [14]. The incorporation of silver nanoparticles introduces, by synergetic effects between their organic and inorganic components, new properties such as enhancement in mechanical toughness, large deformability, high swelling/deswelling rates, excellent electrical conductivity, high transparency [15–17] and remarkably strong antibacterial activity in combination with a fairly low toxicity against human tissues [18].

The general methods used to incorporate nanoparticles into the hydrogels includes gelation of preformed nanoparticles in a solution of hydrogel forming monomers, embedding of nanoparticles in the hydrogel networks by swelling – shrinking process, repeated heating, centrifugation, dispersion and entrapment of nanoparticles in hydrogel matrix followed by reduction with common reducing agents. However, drawbacks such as aggregation of nanoparticles in monomer solution before and during the gelation process, leaching of nanoparticles out of the network, forcing conditions for synthesis and the use of toxic chemicals as reducing agents limits their use in the development of potential hydrogel nanocomposites. This pave the way for biological methods, using plant extracts, which are cost effective and environmentally friendly [19].

Among the natural polymers, polysaccharides in particular have some remarkable characteristics and considerable research has been directed towards them for the production of hydrogels [20]. Almond gum is one of the natural polymers exuded from the trunk of the almond tree (Punins dulcis). Carbohydrates of the almond gum are mainly constituted of arabinose, xylool, galactose and uronic acid with traces of rhamnose, mannose and glucose, thus suggesting an
arabinogalactan structure of the gum [21]. It is cheap and has many properties that have generated interest in its use such as water binding capacity, biodegradability, biocompatibility, non-toxicity, bioactivity, metal uptake capacity, fat binding capacity and antimicrobial activity [22–24].

Based on the literature survey, we report on the novel synthesis of poly(acrylamide)– almond gum hydrogel with certain hydrophilic nature for embedding the silver ions and reducing it to silver nanoparticles in situ using neem plant extract [25] to obtain polysaccharide based hydrogel – silver nanocomposite (HSN). The structure of HSN, effect of pH and its reversibility, almond gum and silver nitrate concentration on the swelling behavior of HSN were studied. Finally the antibacterial activity was evaluated against staphylococcus aureus, escherichia coli and pseudomonas aeroginosa.

2. Experimental

2.1. Materials

Acrylamide (AM), ammonium persulphate (APS), silver nitrate (AgNO₃), N,N'-methylenebisacrylamide (MBA), N,N,N',N'-tetramethyl ethylenediamine (TEMED) of reagent grade were kindly supplied by S.D. Fine Chemicals (Mumbai, India) and used without further purification. Almond gum (AG) was also of reagent grade and purified before use. Double distilled water (DDW) was used for the synthesis of hydrogel and for the preparation of solutions required in this study.

2.2. Purification of almond gum

Purification of almond gum was carried out as follows [26]. The gum was dried well and powdered using mortar and pestle and riddled through sieve No.100. It was solubilised in double distilled water and concentrated by heating. The concentrated gum was precipitated in an ice cold ethanol. The precipitated gum was separated and dried at 60 °C. The pure gum was finely grained and stored in air tight container.

2.3. Synthesis of polysaccharide based semi interpenetrating hydrogel (SIH)

SIH was prepared by an improved one pot synthesis [27] as follows. AG (0.1 g) was mixed in 5 ml of DDW to get a homogeneous solution. AM (1.0 g) was dissolved in 6 ml of DDW followed by initiator APS (0.005 g in 1 ml of DDW) and cross-linker MBA (0.01 g in 1 ml of DDW). Both the solutions were mixed and TEMED (0.02 ml in 1 ml of DDW) was added which, together with APS, acts as redox-initiating pair and initiates free radical polymerization. The reaction mixture was stirred and heated at 60 °C for 5 min. The polymerization reaction results in the formation of SIH within 10 min of reaction time. The formed hydrogel was equilibrated with water for 3 days to remove unreacted monomers and reagents. The hydrogel was dried in hot air oven to constant weight.

2.4. Preparation of plant extract

Azadirachta indica (neem) leaf extract was used for the green synthesis of silver nanoparticles because of its cost effectiveness, ease of availability and medicinal property [28]. Fresh leaves were collected from the local region. They were surface cleaned with running tap water to remove adhered impurities, followed by double distilled water and air dried at room temperature. About 20 g of leaves were cut into small pieces and grinded well to obtain a paste. This paste was dispersed in 100 ml of DDW and heated for 30 min at 70–80 °C. The extract was cooled down and filtered using Whatman’s No.1 filter paper and the filtrate was collected in a presterilized conical flask, stored at 4 °C and used on the same day.

2.5. Green synthesis of semi interpenetrating hydrogel – silver nanocomposite

Dried hydrogel (500 mg) was equilibrated with double distilled water for 3 days at room temperature. The swollen SIH was transferred to beaker containing 50 ml of 0.005 M silver nitrate solution and allowed to equilibrate for 24 h. These silver salt loaded hydrogel was transferred into another beaker containing 50 ml of neem leaf extract and allowed to stand for 12 h. During this period, the silver ions were reduced to silver nanoparticles which were confessed by the development of brown color in the hydrogel. The silver nanoparticles loaded SIH was dried at ambient temperature and often termed in the forthcoming sections as HSN.

2.6. Characterisation techniques

2.6.1. UV–visible diffused reflectance spectra (DRS)

UV–Visible DRS of SIH and HSN were recorded on the Shimadzu 2550 spectrophotometer using BaSO₄ as the reference material.

2.6.2. Fourier transform infrared spectra (FT-IR)

FT-IR spectra of completely dried SIH and HSN were recorded with a Perkin Elmer FT-IR spectrometer – Spectrum RX1 (USA) of scan range 500–4000 cm⁻¹. The instrument was purged with dry air before data collection and during measurements to eliminate error due to moisture absorption.

2.6.3. High resolution scanning electron microscopy (HR-SEM)

HR-SEM analysis was performed using Tescan Vega3 SBU variable pressure scanning electron microscope with 0.2 ml of finely grinded SIH and HSN dispersions on a copper grid dried at room temperature after removing excess solution using filter paper.

2.6.4. Thermogravimetric analysis

The thermal stability of SIH and HSN were evaluated using SII EXSTAR 6000 thermal system (Japan) at a heating rate of 10 °C per minute and a flow rate of 10 ml per minute under nitrogen atmosphere.

2.6.5. Swelling studies

The pre-weighed HSN of different AG concentrations were immersed in DDW at 37 °C. After specific time intervals, the gels were taken out and the surface adhered solution was removed and weighed. Similarly, the swelling ability of HSN in various buffer solutions was studied. The swelling ratio (Q) of the gels was calculated from the equation, \( Q = \frac{W_d}{W_g} \), where \( W_g \) is the weight of swollen hydrogel and \( W_d \) is the weight of the dry hydrogel.

2.6.6. Antibacterial studies

The antibacterial activity of SIH and HSN were carried out on Mueller Hinton agar (MHA) medium using agar disc diffusion method [29]. MHA medium was poured into the sterile petriplates. After the medium was solidified, the inoculums were spread on the solid plates with sterile swabs moistened with the bacterial suspension. The discs were placed in MHA plates and 20 μl of samples...
(Concentration: 1000 μg, 750 μg and 500 μg) were placed in the disc. The plates were incubated at 37 °C for 24 h. Then the antimicrobial activity of the prepared HSN against the test organisms was determined by measuring the diameter (mm) of zone of inhibition.

3. Results and discussion

3.1. Characterisation of SIH and HSN

3.1.1. UV–visible diffused reflectance spectra (DRS)
The development of opaque brown color after the addition of neem leaf extract to the swollen hydrogel indicates the in situ formation of silver nanoparticles (Ag+) by the reduction of silver ions (Ag+) in the entire hydrogel. This also represents that the nanoparticles were entangled inside the swollen hydrogel networks through strong localization and stabilization established from the macromolecules of polysaccharides. The perseverance of silver nanoparticles in the hydrogel networks was confirmed by UV–visible DRS spectral analysis. Silver nanoparticles loaded hydrogel have shown a distinct peak around 380–400 nm in the diffused reflectance spectra [30] (Fig. 1(a)) due to the surface plasmon resonance (SPR) effect caused by the quantum size of the silver nanoparticles [31] whereas the placebo hydrogel have shown no such peak in the diffused reflectance spectra (Fig. 1(b)).

3.1.2. Fourier transform infrared spectra (FT-IR)
IR spectra of the poly(acrylamide) based hydrogel (Fig. 2(a)) shows the presence of absorption bands characteristic to cross-linking bridges. Absorption band attribution was made in agreement with the values given in literature [32]. The absorption bands at 3335 and 1647 cm⁻¹ corresponds to –OH stretching vibrations from the cross-linking bridges and –C=O extension vibrations of amide groups respectively. A band at 3202 cm⁻¹ arising from the stretching vibration of the amide group, which is obvious in the spectrum of polyacrylamide proved the presence of acrylamide in the structure of synthesised hydrogel [33]. The symmetric valence vibration at 2916 cm⁻¹ is assigned to the –CH₂ groups between the macromolecular chains and cross-linking bridges. The asymmetric stretching vibration of ester groups of AG is observed at 1600 cm⁻¹. Absorption bands assigned to the deformation vibrations of the –CN, –OH and –CH₂ groups in the 1400 to 1350 cm⁻¹ range were also identified. Cluster of peaks from 500 to 1100 cm⁻¹ range are assigned to stretching of C – O in C – O – C and C – O – H of glycosidic bonds. In the IR spectra of HSN (Fig. 2(b)), the peaks were shifted to lower wave numbers (3326, 3192, 1642, 1591 cm⁻¹) due to the coordination between the nano-silver and electron rich groups (relating to –OH, –NH, –C=O, –COO⁻) [34,35] present in the hydrogel networks. Therefore, we can confirm the presence of silver nanoparticles in the hydrogel networks.

Fig. 1. Diffused Reflectance spectra of (a) HSN (b) SIH.

Fig. 2. FT-IR spectra of (a) SIH (b) HSN.
3.1.3. High resolution scanning electron microscopy (HR-SEM)

The surface morphology of SIH and the HSN were investigated with HR-SEM. The SIH showed a smooth surface feature (Fig. 3(a)), whereas HSN showed a shrunken surface throughout the gel (Fig. 3(b)). The particle size analysis confirms the presence of small, highly dispersed and rod shaped silver nanoparticles with an average size of about 75 nm. It is noticed that no individual silver particles were observed outside the HSN, suggesting a strong interaction between the polymer matrix and the silver particles.

3.1.4. Energy dispersive X-ray (EDX) analysis

Fig. 4(a) and 4(b) depicts the chemical composition of SIH and HSN as obtained by EDX. Fig. 4(a), which is the EDX spectrum of SIH, shows the presence of C and O signals only confirming the high purity of SIH. Fig. 4(b) shows the EDX spectrum corresponding to the presence of C, O and Ag elements only. The weight percentage of Ag as obtained from EDX spectrum is 6.10%. It is important to notice that the prepared HSN is pure and free from impurities.

3.1.5. Thermal stability

The thermogram of HSN displayed distinctive thermal stability as shown in Fig. 5(a). The HSN mainly showed two stage decomposition behavior [36]. An early weight loss (8%) in the range 80–120 °C was also observed and is due to the loss of absorbed moisture. The first major stage of decomposition is characterized by the initial decomposition temperature (IDT) in the range 230–260 °C and final decomposition temperature (FDT) in the range 310–380 °C. This stage resulted in 38% weight loss. For the second major decomposition stage, the IDT range was 420–450 °C and FDT range was 500–580 °C. The weight loss was around 31% which was attributed to the complete degradation of the hydrogel. However, the TGA thermogram of SIH (Fig. 5(b)) showed two decomposition curves [37] and degradation of the hydrogel starts around 230 °C and continues up to 500 °C. It is important to note that the thermal stability of HSN is higher than SIH. The high thermal stability of the HSN can be attributed to the presence of large quantity of silver nanoparticles inside the hydrogel network.

3.1.6. Swelling studies

The swelling behavior of any polymer network depends upon the nature of the polymer, polymer – solvent compatibility and degree of cross-linking. Hence, we have chosen carbohydrate polymer (AG) to improve the swelling capacity of poly(acrylamide) hydrogel. The capability of hydrogels to swell in water is due to the hydrophilic groups present in the polymeric chains and its mechanical resistance is due in part to the physical or chemical cross-linking. It was known that polar head groups of polymeric chains such as hydroxyl, thiol, amine, and nitrite have high affinity for salts [38]. The loading of silver ions throughout the gel networks causes repulsion among the networks, which ultimately leads to an improved swelling behavior of hydrogel systems. Further increase in swelling capacity was observed after the addition of reducing agent (neem extract) to silver ions loaded SIH. This is due to the formation of silver nanoparticles throughout the gel networks which increases the overall porosity of system and makes entry for more number of water molecules inside the gel. The other reason can be that the formed particles have different sizes and surface charges which cause absolute expansion of the networks. The order of swelling observed is: HSN > Ag⁺ loaded SIH > SIH.

3.1.7. Effect of AgNO₃ concentration on swelling capacity of SIH

The effect of AgNO₃ concentration on swelling characteristics of the SIH was studied by varying the concentration of AgNO₃ from 0.001 to 0.02M whereas the amount of AG was kept constant (0.1 g). Increasing the AgNO₃ concentration up to 0.005 M, the water absorbency of the hydrogel was increasing (Fig. 6). The reason was that the polar head groups of polymeric chains such as hydroxyl, thiol, amine, and nitrite groups have a high affinity for salts. A decrease in swelling when the AgNO₃ concentration was 0.02M may be attributed to the chelation of some hydroxyl and carboxylate groups of the hydrogel networks with silver nanoparticles which neutralizes the repulsions in the networks.

3.1.8. Effect of AG amount on swelling capacity of HSN

The effect of AG concentration on water absorption of the HSN was investigated by varying the amount of AG from 0.1 g to 0.5 g whereas the concentration of AgNO₃ was kept as 0.005 M. As shown in Fig.7, increasing AG amount up to 0.2 g increases the swelling capacity. This was due to the introduction of more number of hydrophilic polymer chains inside the gel networks that assists in improving the swelling characteristics of gel systems. The decrease in swelling with further increase in AG amount (up to 0.5 g) can be attributed to the increase in viscosity of the medium which in turn hinders the movement of ions.

Fig. 3. SEM image of (a) SIH (b) HSN.
3.1.9. Effect of pH on swelling capacity of HSN

The swelling behavior of HSN in various pH was observed by placing 0.1 g of HSN in pH of 2, 4, 6, 8 and 10 solutions respectively and examining them after one hour. It was noticed that the amount of swelling increases with increase in pH from 2 to 6, reaches maximum at pH 8 and then decreases (see Fig. 8). The repulsion between –COO– groups in the hydrogel is the main reason for the maximum swelling at pH 8 [39]. Below pH 8, the H+ ions in the external medium effectively suppress the ionisation of the carboxyl and hydroxyl groups of AG. This decreases the number of mobile ions inside the HSN which causes decrease in osmotic pressure and hence the swelling capacity of HSN. Above pH 8, the –OH groups can ionise to –OR which reacts with –COO– to form esters. This increases the number of networks inside the hydrogel and also decreases the hydrophlicity which is responsible for the decrease in swelling.

3.1.10. Effect of pH reversibility on swelling capacity of HSN

The reversible swelling–deswelling behavior of HSN in solutions with pH; 2 and pH; 8 was examined (Fig. 9) by placing the HSN in pH; 2 and pH; 8 solutions alternatively for every half an hour and swelling capacity was determined for every half an hour. At pH 8, the hydrogel swells due to anion–anion repulsive electrostatic forces, while at pH 2, it shrinks within a few minutes due to protonation of the carboxylate anions. This swelling–deswelling behavior of the hydrogel makes HSN a suitable material for drug delivery systems.

3.1.11. Antibacterial analysis

The main course of this study is to unfold a new antibacterial material. The antibacterial activity of different molar weight ratios of HSN and SIH were examined against *staphylococcus aureus*, *echerichia coli* and *pseudomonas aeruginosa*. All HSN proved effective against the tested microorganisms and the growth inhibitory effects showed variation form one another. Fig. 10 (a and b) exhibits the antibacterial property of SIH and HSN. The results indicate that the HSN exhibited greater reduction of bacterial growth as compared to SIH due to the sustained release of silver nanoparticles form the hydrogel networks. Another finding reveals that the positive charge on the silver ion is crucial for its antimicrobial activity through the electrostatic attraction between the negatively charged cell membrane of the microorganism and positively charged nanoparticles [40]. The inhibition area of SIH and HSN for different organisms is given in Table 1.

4. Conclusion

Novel almond gum/poly(acrylamide) semi interpenetrating hydrogel – silver nanocomposite was successfully prepared via free radical polymerization followed by *in situ* reduction of silver ions to nanosilver using neem leaf extract as a clean and green reducing agent. The methodology developed for the synthesis of SIH is very simple, rapid and cost effective which makes it easy to implement in the industries. A number of HSN were formulated with high dispersion rates by varying the concentrations of AG and monomer. The formed HSN was characterized by different techniques. In the UV–visible diffused reflectance spectra, nanosilver have shown good surface plasmon resonance behavior. In FT-IR spectra, shifting of peaks to lower wave number occurred. SEM images confirm the presence of well defined silver nanoparticles and its stability was further confirmed by thermal analysis. Variation in AG and silver ion concentration can greatly improve the swelling of corresponding HSN. The antimicrobial activities shown by silver nanoparticles make this method a potential route for...
Fig. 5. Thermogram of (a) HSN (b) SIH.

Fig. 6. Effect of concentration of $\text{AgNO}_3$ (M) on swelling capacity of SIH at $37^\circ$C.

Fig. 7. Effect of concentration of $\text{Ag}^{\text{II}}$ (g) on swelling capacity of HSN ($\text{AgNO}_3$ – 0.005 M) at $37^\circ$C.

Fig. 8. Effect of pH on swelling capacity of HSN at $37^\circ$C.

Fig. 9. pH reversibility of HSN with 30 min time interval at $37^\circ$C.

for metal nanoparticles preparation to be used for biomedical applications.
Table 1

<table>
<thead>
<tr>
<th>Organisms</th>
<th>Zone of Inhibition (mm)</th>
<th>Antibiotic (Ampicillin)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Concentration (µg/ml)</td>
<td>(1 mg/ml)</td>
</tr>
<tr>
<td></td>
<td>1000</td>
<td>750</td>
</tr>
<tr>
<td>SIH</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Staphylococcus aureus</td>
<td>04</td>
<td>03</td>
</tr>
<tr>
<td>Escherichia coli</td>
<td>06</td>
<td>04</td>
</tr>
<tr>
<td>Pseudomonas aeruginosa</td>
<td>04</td>
<td>[NA*]</td>
</tr>
<tr>
<td>HSN</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Staphylococcus aureus</td>
<td>08</td>
<td>07</td>
</tr>
<tr>
<td>Escherichia coli</td>
<td>11</td>
<td>08</td>
</tr>
<tr>
<td>Pseudomonas aeruginosa</td>
<td>08</td>
<td>07</td>
</tr>
</tbody>
</table>

[NA*] – not active.

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SYNTHESIS OF PH RESPONSIVE HYDROGEL–SILVER NANOCOMPOSITE FOR USE AS BIOMATERIALS

I. Syed Ahamed Hussain1, M. Gulsonbi2 and V. Jaisankar1

ABSTRACT
Hydrogels are the intelligent materials that can absorb and release biological fluids in response to specific external stimuli. Such unique properties of hydrogels prompted us to synthesize a pH responsive semi-interpenetrating polymer network based on cross-linked poly(acrylamide) prepared through an optimized redox initiating free radical polymerization utilizing almond gum as a grafting backbone, N,N'-methylenebisacrylamide (MBA) as the cross-linker and ammonium persulphate (APS) – N,N,N',N'-tetramethylethylenediamine (TEMED) as the redox initiation pair. Silver ions are introduced and silver nanoparticles are generated within the hydrogel networks via insitu reduction of silver ions using Azadirachta Indica (neem) plant extract under atmospheric conditions. The synthesized hydrogel–silver nanocomposite (HSN) was characterized by UV-Visible diffused reflectance spectroscopy (DRS), fourier transform infrared spectroscopy (FT-IR), high resolution scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and thermogravimetric analysis (TGA). Swelling properties are studied in solutions of different pH and found that the varying response of HSN in acidic and basic media make it suitable as body water retainers and stomach bulking agents.

Keywords: Hydrogel, nanosilver, green synthesis, swelling, pH responsive polymer.

INTRODUCTION
Hydrogels are the semi-interpenetrating polymeric networks which possess good hydrophilic character. They can swell and imbibe large amount of water without dissolution. The ability of hydrogels to absorb water arises from hydrophilic functional groups that are attached to the polymeric backbone while their resistance to dissolution is attributed to cross-linking that exists within the polymeric networks. These networks swell until the thermodynamic force of swelling is completely counterbalanced by the elastic, retroactive force exerted by the cross-links. The degree of swelling depends on both structural and environmental factors. Structural factors include degree of ionisation, density of cross-linking, extent of hydrophilicity, charge, concentration and pKa of the ionisable group.

Environmental factors like temperature, pH and ionic strength of the aqueous solution in contact with the polymer also affect the swelling characteristics. In addition to the swelling characteristics, the soft, flexible nature, alikeness to natural living tissue, hypotoxicity and biodegradability have made hydrogels suitable for the development of a variety of smart devices such as artificial heart valves, synthetic muscles, fabricated contact lenses and substrates for site specific drug delivery.

Novel hydrogels based on synthetic, natural or hybrid polymers with excellent swelling ability, biocompatibility, biodegradability, processability and functionalization have been synthesised. Innovative products are developed either as water absorbents for specific applications (e.g., personal hygiene products, underwater devices, water reservoirs for dry soils) or as biomedical devices, including lubricating research article.
almond gum as a grafting backbone, a reducing and capping agent was used. High swelling/deswelling rates, tunable electrical conductivity, extraordinary deformability, cytotoxicity and remarkably high electrical conductivity, large tough, non-toxic and non-cytotoxic silver nanoparticles have been incorporated in to the hydrogel structure of the gum which acts both as a binder and stabilizer and allows to stand for 12 h.

Recently, silver nanoparticles have been immobilized on three-dimensional cell culture scaffolds, thereby allowing their use as novel wound dressings. This novel approach is a cost-effective method of producing silver nanoparticles which can be applied directly onto the wound by a simple spraying method. In addition, these silver nanoparticles hold promise for cell culture applications. The highly biocompatible cellulose acetate based scaffold has been currently used for tissue engineering applications.

UV-Visible diffused reflectance spectra (DRS)

To evaluate the optical properties of pure SiH and HSN, UV-Visible diffuse reflectance spectroscopy was carried out. The samples were placed on a spectroscopic 1 cm path length cell for 10 minutes of reaction and the spectrum was recorded on the Shimadzu 2550 UV-Visible DRS spectrophotometer.

Fourier transform infrared spectra (FT-IR)

Fourier transform infrared (FT-IR) spectra were recorded on the Shimadzu FT-IR spectrophotometer using a KBr pellet technique. A total of 16 scans were collected at a resolution of 4 cm⁻¹. Spectra were recorded from 4000 cm⁻¹ to 400 cm⁻¹.

High resolution scanning electron microscopy (HR-SEM)

High resolution scanning electron microscopy (HR-SEM) was performed using a JEOL JSM 6401F HR-SEM operating at 15 kV accelerating voltage. The dried samples were coated with a thin layer of gold using a Hitachi E-1010 vacuum deposition chamber for further imaging.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) was performed on a Perkin-Elmer TGA 7 thermobalance to study the thermal stability of SiH and HSN. The samples were heated from 30°C to 800°C at a heating rate of 10°C min⁻¹ under nitrogen atmosphere.
RESULTS AND DISCUSSIONS

Swelling studies

Due to the presence of hydroxyl and amino groups, SIH and HSN possess high swelling properties. The swelling studies were performed in water, DDW, and DDW containing various concentrations of silver nitrate. The weight change of dry hydrogels was measured as a function of time for 24 h. The weight of dried hydrogels was measured before and after immersing in 100 mL of DDW, DDW containing various concentrations of silver nitrate, and water for 24 h. The swelling ratios were calculated using the following equation:

\[ \text{Swelling ratio} = \frac{W_a - W_0}{W_0} \]

where \( W_0 \) is the weight of the dry hydrogel and \( W_a \) is the weight of the swollen hydrogel.

Fourier transform infrared spectra (FT-IR)

FT-IR spectra of PAM, SiH, and HSN were recorded in the 400-4000 cm\(^{-1}\) wavenumber range. The peaks at 1036 cm\(^{-1}\) are assigned to the C-O stretching vibration in the SiH hydrogel. The symmetric and asymmetric stretching vibrations of C-O were observed at 1213 and 1405 cm\(^{-1}\), respectively. A peak at 1642 cm\(^{-1}\) corresponds to the asymmetric stretching vibration of ester groups in the polymer. A peak at 3192 cm\(^{-1}\) corresponds to the C-H stretching vibration of the acrylamide unit. The peaks at 2074 and 2916 cm\(^{-1}\) correspond to the \( \text{C} = \text{O} \) and C-H stretching vibrations, respectively.

The IR spectra of HSN showed additional peaks at 1600 cm\(^{-1}\) due to the presence of nanosilver. The peaks at 3202 cm\(^{-1}\) correspond to the stretching vibration of the amide group, whereas the peaks at 1591 cm\(^{-1}\) correspond to the C=O stretching vibration of amide groups. The peaks at 1412 cm\(^{-1}\) correspond to the C-H bending vibration of the amide group.

Energy dispersive X-ray spectrum (EDX) analysis

The EDX spectrum of HSN showed the presence of C and O signals only, indicating the presence of nanosilver. The EDX spectrum of SIH showed the presence of C and O signals only, indicating the presence of silicon and nitrogen, respectively.

High resolution scanning electron microscopy (HR-SEM)

The HR-SEM images of SIH and HSN showed the presence of nanosilver. The images showed the formation of silver nanoparticles inside the hydrogel networks without releasing into the media. The scheme indicated that the silver nanoparticles were formed solely inside the hydrogel networks and not in the surrounding medium. This indicates that the silver nanoparticles were simply obtained at room temperature.

UV-Visible diffused reflectance spectra (DRS)

The UV-Visible DRS spectra of HSN showed a peak around 380 nm, which is due to the surface plasmon resonance (SPR) of the silver nanoparticles. The peak shifted to lower wave numbers (3326, 3192, 2916 cm\(^{-1}\)) due to the coordination of silver ions in the hydrogel matrices. A band at 3202 cm\(^{-1}\) corresponds to the stretching vibration of the amide group, whereas the bands at 1600 cm\(^{-1}\) correspond to the C=O stretching vibration of amide groups. The peaks at 1412 cm\(^{-1}\) correspond to the C-H bending vibration of the amide group.
Thermogravimetric analysis

Effect of pH reversibility on swelling capacity of HSN

Swelling capacity of Hydrogel, Hydrogel-Ag⁺, HSN

Applications

Body Water Retainers

Stomach Bulking Agents

Effect of pH on swelling capacity of HSN

Applications

Body Water Retainers

Stomach Bulking Agents

Effect of pH reversibility on swelling capacity of HSN

Swelling capacity of Hydrogel, Hydrogel-Ag⁺, HSN

Applications

Body Water Retainers

Stomach Bulking Agents
Supplements are claimed to act either by binding fats and so reducing fat absorption, as reported for cellulose- and chitosan-based products, or by directly reducing the appetite, as for different natural fibers and herbal products, that seem to absorb liquids and swell inside the stomach, thus giving a sense of fullness.

The basic idea of our work is that HSN-based pill can be administered orally before each meal and that the HSN powder swells once it reaches the stomach. In such a way, the space available for food intake is reduced giving a feeling of fullness. The swollen hydrogel is eliminated from the body by faecal way. In this perspective, the hydrogel is envisaged to pass through the gastrointestinal tract, thus it is supposed to encounter the different pH environments of the stomach and the intestine. Thus pH-responsive water retention capacity of HSN renders its use as stomach bulking agent.

CONCLUSION

Novel almond gum/poly(acrylamide) semi-interpenetrating hydrogel–silver nanocomposite was successfully prepared via free radical polymerization followed by in situ reduction of silver ions to nanosilver using neem leaf extract as a clean and green reducing agent. The methodology developed for the synthesis of SIH is very simple, rapid and cost effective which makes it easy to implement in the industries. A number of HSN were formulated with high dispersion rates by varying the concentrations of AG. The formed HSN was characterized by different techniques. In the UV-visible spectra, nanosilver have shown good surface plasmon resonance behavior. In FT-IR spectra, shifting of peaks to lower wave number occurred. SEM images confirm the presence of well defined silver nanoparticles and its stability was further confirmed by thermal analysis. Incorporation of silver nanoparticles increased the swelling capacity of hydrogel. The swelling response of the prepared HSN against pH made it suitable as body water retainers and stomach bulking agents for which specific tests will be performed subsequently.

TABLES AND FIGURES

Fig. 1: (a) SIH and (b) HSN

Fig. 2: Diffused Reflectance spectra of (a) HSN (b) SIH
Fig. 3: FT-IR Spectra of (a) SIH (b) HSN

Fig. 4: SEM image of (a) SIH (b) HSN
Fig. 5: EDX spectrum of (a) SIH (b) HSN and (c) Quantitative results of EDX spectrum of HSN

Fig. 6: Thermogram of (a) HSN and (b) SIH

Fig. 7: Effect of pH on swelling capacity of HSN at 37°C
Fig. 8: pH reversibility of HSN with 30 min time interval at 37 °C

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1. Green synthesis and characterization of pH responsive silver/almond gum – poly(acrylamide) hydrogel nanocomposite and its applications as body water retainers and stomach bulking agents

**I. Syed Ahamed Hussain, M. Gulsonbi, V. Jaisankar**

“National conference on emerging trends in chemistry (NCETC 17), 2-3 March 2017 at PG and Research Department of Chemistry, St. Joseph’s College of Arts and Science (Autonomous), Cuddalore, Tamil Nadu, India.

2. An environmental friendly synthesis, characterization and drug delivery applications of novel silver/almond gum – poly(acrylamide) hydrogel nanocomposite

**I. Syed Ahamed Hussain, V. Jaisankar**

“Second International Conference on Advanced Polymeric Materials (ICAPM 2017) 7-9 April 2017 at School of Chemical Sciences Auditorium, Mahatma Gandhi University, Kottayam, Kerala, India” and awarded best paper award (First Prize)