Chapter 5

Green fabrication of novel Au-core Ag-shell nanocomposite hydrogels for various biomedical applications

A = CP980 or NPC hydrogel. B = Au-core Ag-shell nanocomposite hydrogel. C = core shell stabilized by hydrogel network.

Development of Au-core Ag-shell nanocomposite hydrogels
5.1 Introduction

The rapid growth of nanotechnology has provided many types of functional materials for biomedical applications. One such type of functional material is metal nanocomposite hydrogels, obtained by the impregnation of metal nanoparticles within the hydrogel network [1,2]. These nanocomposite hydrogels have showed their influence in diverse areas, particularly in medical science for various biomedical applications [3-6]. Of late, nano particles of either monometallic or bimetallic (as alloy) had been administered to develop metal nanocomposite hydrogels. However to enhance further their applicability, bimetallic core-shell nano particles are introduced into the hydrogel matrix in the present investigation. Bimetallic core-shell nanocomposite hydrogels form a special type of nanocomposite functional materials, where the impregnated bimetallic core-shell comprises a metallic core covered with a shell of another metal [7]. The addition of a second metallic component enhances the activity, selectivity and stability of the pure monometal [7], that form the primary basis for their utilization in the present investigation. Furthermore, the core-shell nano particles were employed to a larger extent to facilitate their efficiency in the areas of biomedical, magnetic, catalytic and optical applications [8,9].

Hydrogels are three-dimensional (3D), hydrophilic polymeric networks capable of imbibing large amounts of water or biological fluids [10,11] and resemble like natural living biological tissue more than any other class of synthetic biomaterials due to their high water contents and soft consistency similar to natural tissue [12]. Because of their high water content, swollen hydrogels can provide a better feeling for the skin in comparison to conventional ointments and patches [13]. These well defined characteristic properties offered researchers to utilize hydrogels for various biomedical applications which include, production of wound dressing materials, trans-
dermal systems, drug delivery carriers, sanitary pads, disposable diapers, dental materials, implants, injectable polymeric systems, ophthalmic applications and hybrid-type organs (encapsulated living cells) [14-18]. Owing to their wide applications, and the increasing demand to curb microorganisms made researchers to develop different hydrogel nanocomposite systems from time to time through metal nano particles impregnation [1,2].

In our earlier investigations, nano particles of gold (Au) or silver (Ag) or both (as alloy) were impregnated successfully to develop nanocomposite hydrogels as potential anti-bacterial wound dressing materials [6,19-22]. In the present investigation, core-shell nano particles (Au-core Ag-shell) are impregnated to develop novel nanocomposite hydrogels for antimicrobial applications in the fields of surgical and sanitary zones. Gold and silver are specifically chosen because of their gaining renewed attention for combating the threat of bacterial infection. The additional advantage of these materials being that, unlike antibiotics, metal nano particles (either Ag or Au) do not act via cell receptors to kill the micro organisms [23]. So, the chance of immune response in microbes to develop resistance against nano particles is not possible. Hence, the problem of disease transmission/contamination through various micro organisms that occur usually after the disposal of wound dressing materials or sanitary articles could be greatly eradicated without bringing any resistance in micro organisms [24].

In general, bimetallic core shell nano particles were synthesized through seed-mediated growth, template synthesis, chemical reduction and laser ablation. However, these methods utilize not only chemical reductants but also auxiliary stabilizers, and often require specialized expensive equipment [25] and even hazardous to environment. Due to, increasing environmental concerns, it is necessary to develop
new and eco-friendly techniques for the synthesis of nano particles. Some of these methods include green process utilization of leaves’ extracts to develop core shell nano particles [26-28]. The present research work is one such green process that do not involve any toxic chemicals in the core-shells synthesis protocol. During this process, the extracts of naturally available mint leaves are utilized for synthesizing core-shell metal nano particles. This is the most appropriate and cost-effective method which utilizes ambient conditions for nucleation.

The present investigation was majorly aimed to fabricate novel microbial resistant inorganic Au-core Ag-shell nanocomposite hydrogels with high swelling properties that can find applications in surgical and sanitary zones as well. For this purpose, hydrogels were prepared separately from commercially available Carbopol® 980 NF (acrylic acid polymers cross-linked with allyl ethers of pentaerythritol) and Noveon® AA-1 Polycarbophil (acrylic acid polymer crosslinked with divinyl glycol) [29,30]. These materials are particularly selected because of their more relevance in pharmaceutical and biomedical applications, in formulation of buccal, vaginal, nasal, ophthalmic and rectal bioadhesive products [29,31,32]. For the developed hydrogels the swelling studies in phosphate buffered saline (PBS), pH 7.4 were performed, which showed that these hydrogels can absorb blood and secretion exudates from injured wounds or other bodily parts to an extent of 12-22 times (1200-2200%) more than its weight. Further, the antibacterial studies confirmed their excellent antibacterial efficiency. Overall, the results indicated that the novel nanocomposite hydrogels developed by environmental friendly green approach is a promising tool in improving the medical standards of both surgical and sanitary zones. The experimental findings pertaining to this novel inorganic Au-core Ag-shell nanocomposite hydrogels are presented in this chapter.
5.2 Experimental

5.2.1 Materials

Carbopol® 980 NF (CP980) and Noveon® AA-1 Polycarbophil (NPC) are obtained as gift samples from Lubrizol Advanced Materials, Europe. Acrylamide (AAm), N,N'-methylenebisacrylamide (MBA), Ammonium Persulphate (APS), gold chloride (HAuCl$_4$·xH$_2$O) and silver nitrate (AgNO$_3$) are purchased from S.D. Fine-Chem Ltd., Mumbai, India. All chemicals were used without further purification. Double distilled water was used throughout the experimentation. The required mint leaves were collected at locally available agricultural forms.

5.2.2 Preparation of the Mint leaves’ extract

Mint leaves’ extract was prepared following the standard procedure, similar to the methods described in the earlier study [33]. In brief, fresh mint leaves were collected and thoroughly washed with double distilled water. Mint leaves broth was prepared by taking 2.5 g of thoroughly washed and finely cut mint leaves in a 500 mL Erlenmeyer flask with 100 mL of sterile double distilled water. The contents of the leaves were extracted by heating the solution at 100 °C for 2 min, cooled to room temperature and filtered through 0.45 µm PVDF Millex Filter using a 50 mL syringe. The extracted solution was preserved at 4 °C and utilized for nucleation of Au$^{3+}$ and Ag$^+$ ions.

5.2.3 Fabrication of hydrogels

A set of Carbopol® 980 NF hydrogels (P(CP980-AAm)$_x$, $x = 1$-3) and Noveon® AA-1 Polycarbophil Hydrogels (P(NPC-AAm)$_x$, $x = 1$-3) were separately fabricated by dissolving AAm (14.06 mM) and various ratios (0.05-0.15g) of acrylic acid polymers (CP980/NPC) in 3 mL of distilled water, stirring at 300 rpm for 2 h at
25 °C. To this aqueous solution, MBA (0.648 mM) and APS (2.191 mM) were added, and the temperature was raised to 50 °C for 15 min to initiate the free-radical polymerization reaction. The reaction was maintained at ambient conditions for 4 h. During the reaction period, gelation occurs leading to the formation of hydrogels. The obtained hydrogels were immersed in distilled water at room temperature for 24 h to remove unreacted materials present in the hydrogel network. Finally, various formulations of CP980 and NPC hydrogels were dried out at ambient room temperature for 48 h. The feed compositions of the various formulated CP980 and NPC hydrogels are presented in Table 1.

<table>
<thead>
<tr>
<th>Hydrogel code</th>
<th>AAm (mM)</th>
<th>CP980 (g)</th>
<th>NPC (g)</th>
<th>MBA (mM)</th>
<th>APS (mM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P(AAm)</td>
<td>14.06</td>
<td>0.0</td>
<td>0.0</td>
<td>0.648</td>
<td>2.191</td>
</tr>
<tr>
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<td>0.0</td>
<td>0.05</td>
<td>0.648</td>
<td>2.191</td>
</tr>
<tr>
<td>P(NPC-AAm)_2</td>
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<td>0.0</td>
<td>0.10</td>
<td>0.648</td>
<td>2.191</td>
</tr>
<tr>
<td>P(NPC-AAm)_3</td>
<td>14.06</td>
<td>0.0</td>
<td>0.15</td>
<td>0.648</td>
<td>2.191</td>
</tr>
<tr>
<td>P(CP980-AAm)_1</td>
<td>14.06</td>
<td>0.05</td>
<td>0.0</td>
<td>0.648</td>
<td>2.191</td>
</tr>
<tr>
<td>P(CP980-AAm)_2</td>
<td>14.06</td>
<td>0.10</td>
<td>0.0</td>
<td>0.648</td>
<td>2.191</td>
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<td>P(CP980-AAm)_3</td>
<td>14.06</td>
<td>0.15</td>
<td>0.0</td>
<td>0.648</td>
<td>2.191</td>
</tr>
</tbody>
</table>

Table 1: Feed compositions of the various formulated CP980 and NPC hydrogels.

5.2.4 Fabrication of Au-core Ag-shell nanocomposite hydrogels

Approximately, 500 mg of dry hydrogels were allowed to swell in distilled water for 48 h to reach equilibrium swelling. The swollen hydrogels were transferred to a 50 mL glass beaker containing 20 mL of aqueous silver nitrate (5 mM) and 10 mL of aqueous gold (III) chloride (5 mM) solutions for 24 h time in order to permit equilibration. During this equilibrium stage, Ag⁺ and Au³⁺ ions were being exchanged from aqueous solution to the hydrogel networks. Finally, the ions loaded hydrogels
were immersed into mint leaves’ extract up to 6 h time at room temperature in order to nucleate Au$^{3+}$ and Ag$^{+}$ ions to Au-core Ag-shell nano particles. Subsequently, hydrogels with nucleated Au-core Ag-shell nano particles (P(CP980-AAm)$_x$ + Ag$^0$ + Au$^0$, x=1-3 and P(NPC-AAm)$_x$ + Ag$^0$ + Au$^0$, x=1-3) were dried out at ambient room temperature for 48 h and crushed for characterizations.

5.2.5 Characterizations

The core-shell nanocomposites were studied for morphological studies (TEM, SEM), elemental analysis (EDS), spectral analysis (FTIR), thermal analysis, swelling behavior and antibacterial test.

Transmission electron microscopy (TEM) was conducted on JEM-1200EX, JEOL (Tokyo, Japan). The TEM sample was prepared by dispersing two to three drops (1 mg/1 mL) of finely grinded core-shell nanocomposite hydrogel solution on 3 mm copper grid and dried at ambient temperature. Scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS) analysis was performed using JEOL JEM-7500F (Tokyo, Japan), operated at an accelerating voltage of 2 kV. All SEM-EDS samples were gold-coated, prior to examination. The required fourier transform infrared (FTIR) spectra were recorded on a Perkin Elmer FTIR spectrometer (Model Impact 410, Wisconsin, MI, USA). Samples were examined between 500 and 4000 cm$^{-1}$. Thermo gravimetric analysis (TGA) of the samples were carried out using SDT Q 600 DSC instrument (T.A. Instruments-water LLC, Newcastle, DE 19720, USA) at a heating rate of 20 °C/min under a constant nitrogen flow (100 mL/min).

5.2.6 Swelling ratio

The swelling ratio (Sg/g) of the hydrogels was determined by gravimetric method by allowing the hydrogels to swell in the Phosphate buffered saline (PBS), pH
7.4 for 24 h at 37 °C. The value of swelling ratio was calculated by using the Eq. (1):
[20,21].

\[
\text{Swelling ratio (Sg/g)} = \frac{W_s - W_d}{W_d}
\]

(1)

Where, \(W_s\) and \(W_d\) denote the weight of the swollen hydrogel at equilibrium and the weight of the dry hydrogel, respectively. The data provided was an average value of 3 individual readings of samples.

5.2.7 Antibacterial test

The antibacterial activity was tested against two bacteria E. coli (G -) and Bacillus (G +). The method followed was ‘Disc diffusion method’, as described in the literature. The required nutrient agar medium was prepared by mixing peptone (5.0 g), beef extract (3.0 g), sodium chloride (5.0 g) and agar (15.0 g) in 1000 mL of distilled water and the pH was adjusted to 7.0. The agar medium was sterilized in a conical flask in an autoclave (MAC, MACRO scientific works Pvt. LTD., Delhi) at a pressure of 6.8 kg (15 lbs) for 30 min and transferred into sterilized petri dishes in a laminar air flow chamber (Microfilt Laminar Flow Ultra Clean Air Unit, Mumbai, India) for solidification. Later, 50µL of microbial culture was uniformly streaked over the solid surface. To this inoculated Petri dish, pre-impregnated discs with a standard gel concentration (20 mg/10 mL distilled water) were placed and incubated at 37 °C for 48 h to obtain the inhibition zones. Finally, the formed inhibition zones were measured and photographed.

5.3 Results and discussions

In the present investigation, Carbopol® 980 NF (CP980) and Noveon® AA-1 Polycarbophil (NPC) were successfully utilized to fabricate the hydrogels. During this process gelation occurs through linking of CP980/NPC molecular chains with AAm which initially forms polydisperse soluble branched polymers called ‘sol’. The
process of linking leads the branched polymer to increase its size to form an insoluble infinite polymer called ‘gel’ or ‘network’. The transition of a system from finite branched polymers to infinite polymer is called ‘sol-gel transition’ (or ‘gelation’) and the critical point where gel first appears is called ‘gel point’ [34]. Once the hydrogels were formed, bimetallic Au-core Ag-shell nano particles were impregnated into the gel network by swelling method [2]. Core-shell metal nano particles that are normally produced by chemical reducing agents are usually associated with environmental toxicity or biological hazards. From this perceptive, extracts of naturally occurring mint leaves were chosen for the nucleation of core-shell nano particles.

**Mechanism of Au-core Ag-shell formation**

In a competitive process involving both silver and gold ions, the reduction of gold ions occurs first than silver ions [27]. The comparatively slower reduction rate of silver ions relative to that of gold ions is due to their differences in the reduction potentials of the two metal ions, the redox potential being considerably lower for \( \text{Ag}^+ \) to \( \text{Ag}^0 \) (0.80 V) than \( \text{Au}^{3+} \) to \( \text{Au}^0 \) (1.50 V) [27]. Once, the gold ions are reduced, the surrounded silver ions eventually get reduced and deposit over nucleated \( \text{Au}^0 \) in the form of layer, giving Au-core and Ag-shell nano particles [27]. Further, the size reduction of Au and Ag to nano dimension increases not only the surface energy but also the number of binding sites between Au and Ag atoms, which stabilizes the total energy [35]. Hence, the formation of bimetallic Au-core and Ag-shell nano particles might be explained by the combination of these factors involving balance between the binding energy and the surface energy. The successfully fabricated inorganic Au-core Ag-shell nanocomposite hydrogels via green process was pictorially presented in Scheme 1.
The mint leaves used for the synthesis of core-shell nano particles are rich in many vitamins and minerals such as Niacin, Carotene, Folic Acid, Thiamine, Riboflavin, Magnesium, Calcium, Phosphorus, Iron, Magnesium, Copper, Manganese, Zinc, Chromium. Apart from these chemical constituents mint leaves majorly contains Menthol [36]. The plant possesses carminative, antibacterial, diaphoretic and antispasmodic properties that enhance the medicinal value of mint to a large extent [37].

![Mint leaves](image)

**Scheme 1:** Pictorial representation of green fabrication of inorganic Au-core Ag-shell nanocomposite hydrogels.

5.3.1 Transmission electron microscopic (TEM) analysis

Evidence for successful formation of core-shell nano particles was confirmed by transmission electron microscopy (TEM), as shown in Fig 1. Similar to the observation of many research groups, a boundary between Au and Ag elements can be distinguished by bright and dark contrast, where the dark portion was assumed to be core (Au) and the bright portion was assumed to be as shell (Ag) [38]. From the additional information provided by TEM data it is concluded that the nano particles
formed were within nano dimension of \( \sim 5 \pm 3 \text{ nm} \) and without any aggregation. It may be predicted that the core-shell nano particles were highly stabilized through the available hydrophilic functional groups of the hydrogel network [20]. Hence, core-shell nano particles without aggregation were formed.

**Fig 1:** images core-nano particles present in hydrogel nanocomposites of: A) P(CP980-AAm)\(_3\); B) P(NPC-AAm)\(_3\) with (a) lower magnification and (b) higher magnification.

5.3.2 Scanning electron microscopy-energy dispersive spectroscopic (SEM-EDS) analysis

The morphological studies done through SEM examination showed a clear surface pattern for pure P(CP980-AAm)\(_3\) (Fig 2A(a)) and P(NPC-AAm)\(_3\) (Fig 2B(a)) hydrogels and nano particles distribution pattern for core-shell nanocomposite hydrogels, P(CP980-AAm)\(_3\) + Ag\(^0\) + Au\(^0\) (Fig 2A(b)) and P(NPC-AAm)\(_3\) + Ag\(^0\) + Au\(^0\) (Fig 2B(b)). The formation of core-shell was supported by scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS), as shown in Fig 3, which
showed signals corresponding to Au and Ag in the spectra of both P(CP980-AAm)₃ + Ag⁰ + Au⁰ (Fig 3A) and P(NPC-AAm)₃ + Ag⁰ + Au⁰ (Fig 3B), indicating that the core-shell was composed of Au and Ag nano particles [9,28]. Further, this supports the presence of core-shell nano particles in the hydrogel network.

SEM images of A) (a) pure P(CP980-AAm)₃ hydrogel (b) P(CP980-AAm)₃ + Ag⁰ + Au⁰ hydrogel; B) (a) pure P(NPC-AAm)₃ hydrogel (b) P(NPC-AAm)₃ + Ag⁰ + Au⁰ hydrogel.

Fig 3: EDS images of A) P(CP980-AAm)₃ + Ag⁰ + Au⁰ hydrogel; B) P(NPC-AAm)₃ + Ag⁰ + Au⁰ hydrogel.

5.3.3 Fourier transform infrared (FTIR) analysis
The evidence for the successful preparation of Au-core Ag-shell nanocomposite hydrogel was analyzed through hydrogel–core-shell nanoparticles interaction from FTIR spectral data, as shown in Fig 4. The FTIR spectra of pure P(CP980-AAm)\textsubscript{3} and core-shell nanocomposite of P(CP980-AAm)\textsubscript{3} + Ag\textsuperscript{0} + Au\textsuperscript{0} were presented in Fig 4A. The spectrum of P(CP980-AAm)\textsubscript{3} hydrogel showed absorption bands at 3335 cm\textsuperscript{-1} and at 3190 cm\textsuperscript{-1} corresponding to –OH and –NH groups stretching vibrations respectively [3,4]. The other important bands at 2924 cm\textsuperscript{-1}, 1643 cm\textsuperscript{-1}, 1184 cm\textsuperscript{-1} and 1114 cm\textsuperscript{-1} were assigned to stretching vibrations of –C–H, carbonyl and –C–O groups respectively present in P(CP980-AAm)\textsubscript{3} hydrogel [21,39]. These peaks were shifted in the case of P(CP980-AAm)\textsubscript{3} + Ag\textsuperscript{0} + Au\textsuperscript{0} due to interactions of the core-shell nanoparticles with the functional groups of the hydrogel. The FTIR spectra of pure P(NPC-AAm)\textsubscript{3} and core-shell nanocomposite of P(NPC-AAm)\textsubscript{3} + Ag\textsuperscript{0} + Au\textsuperscript{0} were presented in Fig 4B. For pure P(NPC-AAm)\textsubscript{3}, absorption bands at 3330 cm\textsuperscript{-1} and at 3182 cm\textsuperscript{-1} corresponding to –OH and –NH groups stretching vibrations respectively [3,4]. The other important bands at 2932 cm\textsuperscript{-1}, 1644 cm\textsuperscript{-1}, 1181 cm\textsuperscript{-1} and 1119 cm\textsuperscript{-1} were assigned to stretching vibrations of –C–H, carbonyl and –C–O respectively groups present in P(NPC-AAm)\textsubscript{3} hydrogel [21,39]. These peaks were shifted in the case of P(NPC-AAm)\textsubscript{3} + Ag\textsuperscript{0} + Au\textsuperscript{0} as noticed in the earlier case. Over all, the shifting of peaks confirms the formation of core-shell nanoparticles with in the hydrogel network, providing significant stabilization for the formed core-shells nanoparticles [33].
5.3.4 Thermal Analysis

Thermo gravimetric analysis (TGA) is another piece of evidence, indicates simultaneously the existence of core-shell nano particles within the three dimensional (3D) networks of hydrogels and the thermal stability of the hydrogels. As shown in TGA curves (Fig. 5), at 700 °C, pure P(CP980-AAm)\textsubscript{3} (Fig. 5A) and pure P(NPC-
AAm)\textsubscript{3} (Fig. 5B) degrade almost completely but the core-shell nanocomposite hydrogels of P(CP980-AAm)\textsubscript{3} (Fig. 5A) and P(NPC-AAm)\textsubscript{3} (Fig. 5B) have with residue of 4.77 % and 6.58 % masses respectively. This is due to the existence of inorganic Au-core Ag-shell nano particles internally in the 3D networks of hydrogels. This phenomenon can be comparable with Ag or Au impregnated nanocomposites systems [1,2,40]. The data also concludes that the of core-shell nanocomposite hydrogels have higher thermal stability than pure P(CP980-AAm)\textsubscript{3} and P(NPC-AAm)\textsubscript{3} hydrogels.

![Thermo-gravimetric analysis of: A) pure P(CP980-AAm)\textsubscript{3} hydrogel, P(CP980-AAm)\textsubscript{3} + Ag\textsuperscript{0} + Au\textsuperscript{0} hydrogel; B) pure P(NPC-AAm)\textsubscript{3} hydrogel, P(NPC-AAm)\textsubscript{3} + Ag\textsuperscript{0} + Au\textsuperscript{0} hydrogel.](image)

5.3.5 Swelling studies

The present investigation was carried out with an aim to develop antibacterial hydrogels for various biomedical applications. This includes the design of transdermal wound/burn dressing materials, incontinence articles and sanitary napkins to absorb various biological fluids. The functioning of the hydrogel as a proper biomedical absorbent is defined through ‘swelling property’ or ‘swelling ratio’. Swelling property is a characteristic parameter which indicates the absorption capacity of a functional material for blood, bodily fluids and secretion exudates from the injured wounds.
In the present investigation, from the swelling ratio values of the hydrogels as shown in Fig 6, it is concluded that the value of swelling ratio of the developed nanocomposite hydrogels ranges from: 12 to 22 (g/g), indicating that they can absorb blood or any other bodily fluids to an extent of 1200-2200 % to its dry weight. This is a tremendous achievement. Further, the value of the swelling ratio was proportional to the concentration of CP980/NPC. From the swelling data, it is also evident that with increase of CP980/NPC concentration, the values of swelling ratio also increases. The observed phenomenon was due to the hydrophilic nature of CP980/NPC. Further, the core-shell nanocomposite hydrogels shows still higher swelling ratio than respective pure P(CP980-AAm) and pure P(NPC-AAm) hydrogels. The reason being that, when Au\(^{3+}\) and Ag\(^{+}\) ions loaded hydrogels were treated with mint leaves’ extract, nucleation of ions occurs resulting in the formation of core-shell nano particles. This allows the hydrogel to expand three dimensional (3D) networks and promotes higher water molecules uptake capacity [1,2]. One more reason for the expansion of 3D networks was the ‘size factor’. Nano particles formed with varying nanodimension results in the acquisition of different surface charges over their surfaces, causing absolute expansion of the networks [4].

The overall swelling data significantly confirms that the developed hydrogels are good absorbents for blood, bodily fluids and secretion exudates from injured wounds or other body parts.
Devastation of bacteria is one of the properties that determine the utility of the developed hydrogels for various applications in bacterial prone areas. The efficiency of the developed nanocomposite hydrogels against bacteria was determined by conducting antibacterial activity against E. coli and Bacillus as shown in Fig. 7A and Fig 7B respectively. The results revealed that there is strong reduction in the number of bacterial colonies around core-shell nanocomposite samples. The mechanism of growth inhibitory effect of nano particles against the microorganisms is not clear so far [41]. However, among the various possible mechanisms proposed by many authors, ‘Inhibition by formation of pits,’ is considered to be suitable here. It is assumed that the interaction of nano particles with bacteria results in the formation of pits over the cell wall of the bacterial membrane. These pits cause the leakage of biologically important lipopolysaccharide molecules and membrane proteins, leading to microbial death [42-44]. According to Singh et al, the nano particles in range 1-10 nm attach to the surface of the cell membrane and drastically disturb its proper function by forming pits [45]. The core shell nano particles formed in the current approach exist in between ~5 ± 3 nm. This supports the assumed mechanism is appropriate.

The diameter of the inhibition zones exhibited by the nanocomposite samples is in the range 14.2–17 mm. The diameter of the inhibition zones for P(CP980-AAm)\(_3\) + Ag\(^0\) + Au\(^0\) and P(NPC-AAm)\(_3\) + Ag\(^0\) + Au\(^0\) against E. coli are 15.8 mm, 17.0 mm and against Bacillus are 14.2 mm and 15.1 mm respectively. According to the Standard Antibacterial test “SNV 195920–1992”, specimens showing more than 1 mm
microbial zone inhibition can be considered as good antibacterial agents [46]. Hence, the novel inorganic Au-core Ag-shell nanocomposite hydrogels developed from environmental friendly green process by mint leaves’ extracts can be considered as good antibacterial agents, effective in killing the bacteria. It should be noted from the literature survey of the various authors that the diameter of the inhibition zones obtained for individual monometallic Au or Ag nano particles were less than the inhibition zones’ values that obtained for Au-core Ag-shell nano particles in the present study. This supports the enhanced activity of the developed Au-core Ag-shell nanocomposite hydrogels over monometallic Au or Ag nanocomposite hydrogel systems. This characteristic property is particularly useful in designing antibacterial functional materials with improved activity.

Fig 7: Antibacterial activity of: (a) P(CP980-AAm)$_3$ + Ag$^0$ + Au$^0$ hydrogel, (b) P(NPC-AAm)$_3$ + Ag$^0$ + Au$^0$ hydrogel, (c) pure P(CP980-AAm)$_3$ + pure P(NPC-AAm)$_3$ against: A) E. coli; B) Bacillus.

5.4 Conclusion

Novel inorganic Au-core and Ag-shell nanocomposite hydrogels are fabricated from medicinally important gained Carbopol® 980 NF (CP980) and Noveon® AA-1 Polycarbophil (NPC). The process adopted is environmental friendly ‘green process’, where natural mint leaves’ extract, free from toxic chemicals are successfully utilized.
The developed hydrogels showed excellent antibacterial activity against *E. coli* (gram negative) and *Bacillus* (gram positive) and also exhibited pronounced swelling properties. Hence, from the view point of applications, the developed hydrogels can be utilized for various biomedical applications ranging from designing of wound dressing materials to sanitary appliances like: incontinence articles, tampons, nappy pants, nappy liners and sanitary napkins.

This work is an important contribution in the field of development of bimetallic Au-core and Ag-shell nanocomposite hydrogels using medically important commercial polymer samples via green process by utilizing the mint leaves extract for reduction process for various bio-medical applications.

The work presented in this chapter is communicated to International cited journal “Environmental Science: Nano”, Manuscript ID: N-ART-11-2013-000094. It is in the final process of Review for its acceptance.
5.5 References


