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Synthesis of Superabsorbent from Mixed Polymer (Guar Gum and Starch) Containing Silver Nanoparticles

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ABSTRACT: The graft copolymerization of acrylamide (AM) on to mix polymers (guar gum and starch) using ammonium per sulfate (APS) as an initiator, N,N,N',N' Tetramethylethylene diamine (TEMED) as an activator and N, N'-Methylene bis-acrylamide (MBS) as a crosslinker in aqueous media has been carried out to obtain a super absorbent. This was followed by saponification of the grafted polymer and insertion of silver ions in to the hydrogel, which were further reduced with sodium borohydride (NaBH₄) for silver nanoparticle formation. The effects of different parameters were studied and reaction was optimized. The product so formed was dried and finely powdered sample was characterized using FT-IR, TGA, and SEM, TEM and XRD. Product showed maximum water absorbency of 550 g/g, and salt solution absorbency (NaCl) 59 g/g.

KEYWORDS: Mixed Polymers; Graft co-polymerization; Superabsorbent polymer; Nanoparticles.

I. INTRODUCTION

Superabsorbents hydrogel are loosely cross-linked network of hydrophilic polymers that can absorb and retain aqueous fluids up to thousands times of their own weight [1]. They are widely used in many fields such as medical, agriculture, horticulture, etc [2-6]. In the last decade, the synthesis and stabilization of metal nanosystems have been very interesting subjects of nanoscience and nanotechnology [7] and these nanosystems have potential applications in photonic, electronic, catalytic, chemical, and biosensor areas because of their size effects compared to those of bulk metal and molecular compounds [8-10]. Due to the lack of sufficient chemical and physical stability of metal nanoparticles, their development to real world applications has been restricted to some extent. To receive a better stabilization or dispersion of metal nanoparticles in aqueous media, various protective agents had been used and these agents also play a wonderful role in controlling the nanostructures of the particles [7, 11-14]. For this purpose, various polymeric stabilization agents including natural and synthetic polymers, dendrimers, latex particles, microgels, and hydrogels have been studied exclusively [11, 13]. Because hydrogels can afford to have free-space between the networks in the swollen stage, they can serve for nucleation and growth of nanoparticles and act as nanoreactors or nanopots. This approach was established by Wang et al [15]. Although a number of procedures were developed to prepare mono dispersed silver nanoparticles (Ag nanoparticles), most cases involve the stabilization of the generated particles using either surfactants or polymers [16]. In addition, synthetically varied methods were developed for Ag nanoparticles and Ag nanoparticle composites by incorporating metal ions into thin films of titania, sol-gels, porous polymers, Ag nanoparticles doped natural polymers such as carbohydrate polymers (starches and gums, etc) and viscous resins loaded Ag nanoparticles, which are highly suitable for biomedical applications [17]. Particularly, nanosilver based hydrogels have received approval for clinical applications but dermal toxicity is reported [18-20]. Ag nanoparticles are considered as nontoxic and environmentally friendly antibacterial materials, but due to their poor binding characteristic with surfaces, their utility has been restricted. Therefore, polymer-stabilized nanoparticles and nanoparticles embedded in hydrogel networks are outstanding approaches for antibacterial applications [21-23]. The present paper discusses work on application of mixed polymers (guar gum and starch) as a base material for synthesis of super absorbent polymer containing Ag nanoparticles, as they are very often tailored by modifications to develop functional properties such as solubility and enhanced absorbency. Polysaccharide based superabsorbent hydrogels (SHs) have emerged as promising substitutes because they are nontoxic biopolymers, abundant in nature, usually available at low cost, and intrinsically biodegradable and biocompatible [17, 24].

II. EXPERIMENTAL

II. A. MATERIALS:

Guar gum was supplied by Satguru Industries, Mumbai and all other chemicals used were of laboratory grade. Starch, acrylamide (AM), cross-linker (MBA) potassium persulfate, N, N, N',N'-tetramethylethylenediamine (TMEDA) and other chemicals were supplied by S.D.FineChemical Pvt. Ltd.

II. B. METHOD

II. B.1. PHYSICAL CHARACTERIZATION OF MIXED POLYMERS

II. B.1.1. SWELLING POWER

The estimation of swelling power was carried out using the method reported by Subramanian and coworkers (25). Mixed polymers (0.6 g) was dissolved in 30 ml of distilled water. The mixture was then centrifuged (using CRU- 5000 centrifuge) at 5000 rpm for 15 min. The supernatant liquid was carefully removed and the swollen mixed polymers sediment was weighed. Swelling power (g/g) was calculated as the ratio of the weight of the wet sediment to the initial weight of the dry mixed polymers.

II. B.2.1. PREPARATION OF ACRYLAMIDE GRAFTED MIXED POLYMERS (AM-G-GG-ST)

Acrylamide grafted mixed polymers (AM-g-GG-ST) was prepared by using solution polymerization reaction by dissolving mixed polymers (Guar gum and starch) and acrylamide 10 gm (1:1.5) in approximately 100 ml of water in presence of a cross-linker (MBA) 0.5% of the reaction mixture and redox initiating system ammonium persulfate (APS) 1% on weight of mixed polymers and N,N,N',N'- tetramethylethylenediamine (TMEDA) 0.25 ml. Polymerization reaction was carried out at 30°C for 60 min. Product so formed was washed with distilled water to remove the unreacted monomer, oligomers, cross-linking agent, initiator, the soluble and extractable polymer and other impurities and dried in oven at 60°C to reach a constant weight. A series of experiments were carried out to optimize the reaction in terms of swelling capacity by varying different parameter such as time, mixed polymers: acrylamide ratio, concentration of initiator and concentration of crosslinker.

II. B.2.2. SAPONIFICATION

The graft copolymer product was saponified by adding 0.50 g of AM-g-GG-ST to 50 ml of NaOH solution in 100 ml flask which was immersed in thermostated water bath and was fitted with magnetic stirrer and reflux condenser. The varied NaOH concentrations of 0.05, 0.1, and 0.15 N, saponification temperatures (60°C to 100°C) and saponification time (60 to 120 min) were studied. After completion of reaction, the mixture was allowed to cool to room temperature. It was then washed with distilled water to be free from excess of NaOH and dried in oven at 60°C to reach a constant weight.

II. B.2.3. PREPARATION OF AM-G-GG-ST/AG NANOPARTICLES

The dry superabsorbent powder (AM-g-GG-ST) was accurately weighed (0.5 gm) and put in 500 ml of distilled water and equilibrated for 2 days. Aqueous solution of silver nitrate [50 ml of 5 mM (0.845 gm/100 ml)] was then added. It was again allowed to equilibrate for 24 hr. This silver salt loaded IPN (inter penetrating network) hydrogel was finally transferred to a beaker containing 50 ml of 10 mM (0.31 gm/100 ml) NaBH₄ aqueous solution and allowed for 2 hr to reduce Ag⁺ ions to nanosilver Ag⁰ particles at room temperature.

II. B.3. EVALUATION OF ANTIBACTERIAL PROPERTY

II. B.3.1. WELL DIFFUSION ASSAYS TEST

Antibacterial activity for these nanocomposites was carried out by using well diffusion assay method. All required solutions were prepared in distilled water. The agar medium was prepared in a conical flask and sterilized at a pressure of 15 psi for 20 min. Two test tubes of 20 ml saline solution were sterilized by autoclaving at 15 psi for 20 min. 1 ml of a loopful of *Staphylococcus aureus* was mixed in 20 ml of saline solution. One ml of saline containing the culture was added to a sterilized petri dish and was mixed thoroughly with 20 ml appropriately cooled nutrient agar (containing 1% agar-agar). This solution was allowed to solidify and then a well of 8 mm diameter was made in the agar by using a cork-borer. To this inoculated petri dish, one drop of sterilized nanocomposites gel particle solution (20 mg/10 ml distilled water) was added in each well using 50-microlitre tip and incubated for 24 hr at 37°C in the incubation chamber and then zone of inhibition was measured.



III. CHEMICAL CHARACTERIZATION

III.A. FT IR

The IR spectra of original and AM-g-GG-ST samples were recorded using FTIR spectrophotometer (Shimadzu 8400s, Japan) using ATR sampling technique by recording 45 scans in % T mode in the range of 4000 to 600 cm⁻¹.

III.B. TGA

The thermograms of mixed polymers, grafted co-polymer of mixed polymers with Acrylamide and grafted mixed polymers containing Ag nanoparticles samples were recorded using Shimadzu 60H DTG using aluminium pan between temperature range 30-500 °C and under inert atmosphere of N₂ at a flow rate of 50ml/min.

III.C. SCANNING ELECTRON MICROSCOPY (SEM)

Analysis of the morphology of dried and grafted sample was carried out using scanning electron microscope (JEOL, Japan).

III.D. TRANSMISSION ELECTRON MICROSCOPY (TEM)

Transmission electron microscopic image for nanocomposite was recorded using a CM200 (PHILIPS) at an electronic voltage of 20 kV. For TEM measurement sample was prepared by 10-20 microliter of finely grinded hydrogel nanocomposite dispersion on a copper grid and dried in a sonicator.

III. E. XRD

X-ray diffraction measurements were recorded using Rigaku diffractometer (Cu radiation at 0.1546 nm) running at 40 KV and 40 mA (Rikagu, Japan) at an angular (2θ) range of 4° to 32°.20'. In order to have precise results XRD analysis was carried at four different places, from the radial scans of intensity versus 2θ.

III.F. WATER ABSORBENCY MEASUREMENT

The dry sample was weighed (0.2 g) and immersed in water for 24 h to reach absorption equilibrium. The fully swollen hydrogel was separated from the unabsorbed water with a 65-mesh screen and thereafter, the hydrogel was weighed. The relative water absorbency was calculated as follows [26]:

$$\text{Water absorbency (g/g)} = \frac{(M_2 - M_1)}{M_1}$$

III. G. SWELLING VARIATION WITH PH

The procedures for these experiments are same as those described in "Water absorption measurement" section. The pH dependence of hydrogel swelling was evaluated by considering certain amounts of the hydrogel samples (0.5 ± 0.001 g) in solutions (200 ml) with different pH. The various solutions were adjusted to the desired pH value by addition of dilute HCl or NaOH

IV. RESULTS AND DISCUSSION

Results of characterization of mixed polymers indicate that the swelling power of the mixed polymers was 12.58 g/g.

IV.A. EVIDENCE OF GRAFT CO-POLYMERIZATION ON MIXED POLYMERS (AM-G-GG-ST)

The FTIR spectrum of Acrylamide on to mixed polymers (AM-g-GG-ST) (Fig.1) clearly indicates the peak for N-H stretching vibration at 3375 cm⁻¹ which confirms the introduction of NH₂ group after grafting of mixed polymers with acrylamide. The other peak at 1659 cm⁻¹ confirmed the presence of CO group which otherwise was missing in ungrafted sample of mixed polymers.

Fig. 1: FTIR spectra of mixed polymers, AM-g-GG-ST and AM-g-GG-ST containing Ag nanoparticles

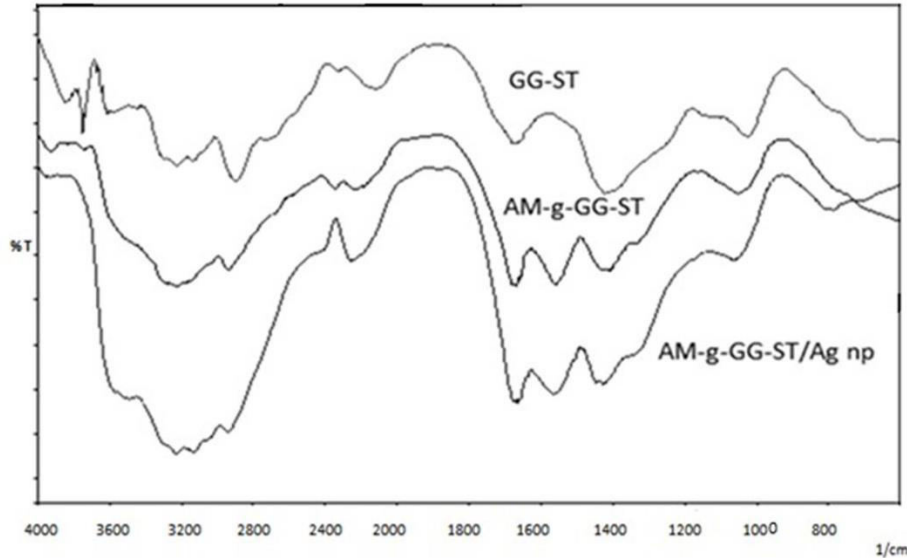
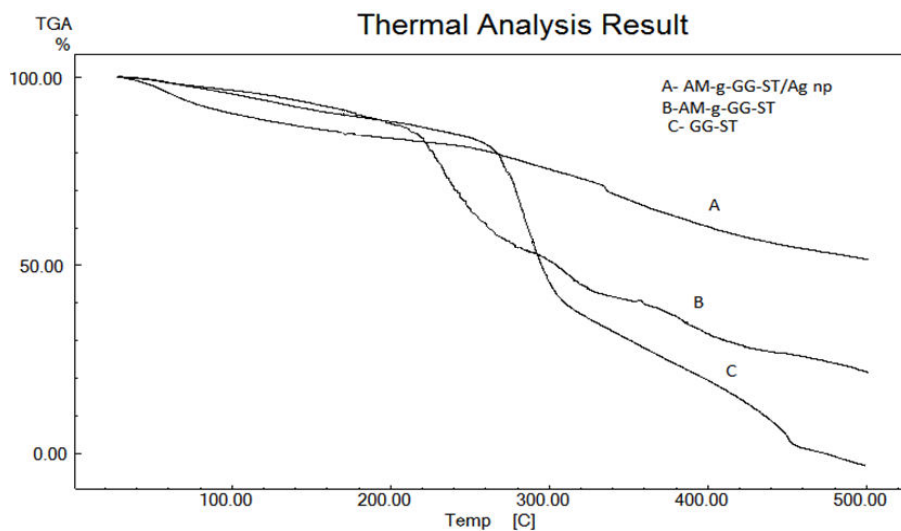


Fig. 2 shows the thermogram of mixed polymers, grafted mixed polymers and grafted mixed polymers containing Ag nanoparticle. In the initial stages weight loss values of all the samples were 16.21%, 8.30 and 7.40 % at 150oC, respectively. Beyond this temperature, the drastic decomposition of the sample resulted in to significant weight loss which was 33.18% for mixed polymers, 28.46% for AM-g-GG-ST copolymer and for AM-g-GG-ST containing Ag nanocomposites 15.67% at 300oC. However, beyond 300oC, the loss in weight was slowed down and finally at 500 °C, weight loss values observed were 98.75 % for mixed polymers, 71.86% for grafted mixed polymers and 48.53 % for polymer containing Ag nanoparticles, respectively. This clearly indicates that the grafted mixed polymers with AM and grafted mixed polymers containing Ag nanoparticles showed relatively higher thermal stability as compared to that of ungrafted mixed polymers. This could be attributed to the formation of crosslinked network as a result of graft copolymerization of AM monomer and also due to incorporation of metal particle on to a mixed polymer back bone.

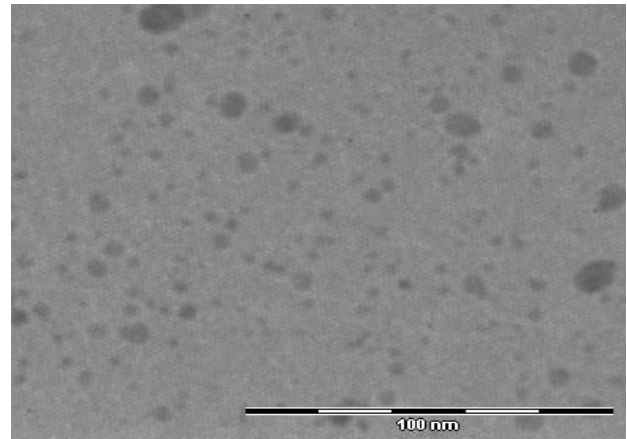
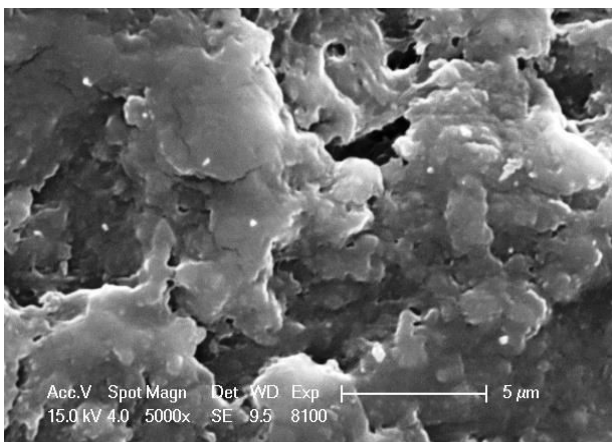
Fig. 2: Thermogravimetric analysis of mixed polymers, AM-g-GG-ST and AM-g-GG-ST containing Ag nanoparticles



SEM micrograph (Fig. 3) of mixed polymers system containing Ag nanoparticles i.e AM-g-GG-ST clearly shows surface modification such as presence of cavity and formation of Ag nanoparticles, further confirming the crosslinking between acrylamide and formation of Ag nanoparticles on to mixed polymers.

Fig 4 shows typical TEM image of Ag nanocomposites which reflects an excellent stability to the nanoparticles throughout the network structure, and the smallest particle dimension seems to be about 10 nm. This image demonstrates a highly uniform distribution of Ag nanoparticles it is possible that the Ag nanoparticles formed in the cross-linked network are spherical, highly dispersed and small in size.

Fig. 3: SEM of AM-g-GG-ST containing Ag nanoparticles **Fig. 4:**TEM of AM-g-GG-ST containing Ag nanoparticles



Figs 5 and 6 show the XRD pattern of hydrogel and its silver nanocomposites, respectively. A common broad diffraction peak 2θ can be seen at 20. The diffractogram of Ag nanocomposites, are assigned to diffraction at 2θ value of about approximately 38, 44, 65, and 78, corresponding to the bragg reflection of (111), (200), (220), and (311), plane of face centred cubic (fcc) structure Ag nanocomposites. The sharp peak in case of hydrogel Ag nanocomposite with intense peak represents its highly crystalline nature.

Fig. 5: XRD of AM-g-GG-ST

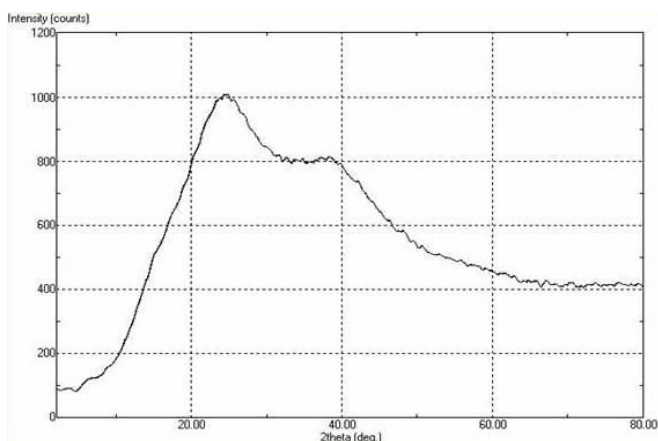
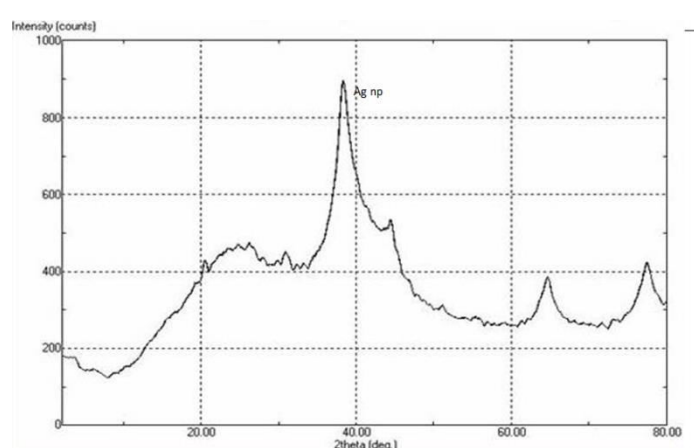


Fig. 6: XRD of AM-g-GG-ST containing Ag nanoparticles



Results in Table 1 show the effect of various parameters of reaction on water absorbency of the final product. The optimized conditions for highest absorbency were 60 min, initiator concentration 1% with respect to mixed polymers and 0.25 ml TEMED. Mixed polymers: monomer mixture concentration was 1:1.5 (w/w), and crosslinker was 0.5%. Either side of these optimized conditions, the product showed the decrease in water absorbency, which may be attributed to reduced extent of crosslinking due to not so favourable conditions for reactions.



Table 1: Effect of different parameters on graft co-polymerization yield and water absorbency of AM-g-GG-ST copolymer

Sr. No.	CG-ST: AM Ratio(w/w)	Conc. of Crosslinker (%)	Initiator Conc. (%)	Time (min)	Polymer Yield (%)	Water Absorbency of AM-g-GG-ST in (g/g)	
						Unsaponified	Saponified
1 Effect of Time							
A	1:01	0.5	1	45	80	50	270
B	1:01	0.5	1	60	98	95	510
C	1:01	0.5	1	75	98	92	510
2 Effect of Initiator Concentration							
A	1:01	0.5	0.05	60	65	60	255
B	1:01	0.5	1	60	98	95	510
C	1:01	0.5	1.5	60	85	70	380
3 Effect of Crosslinker							
A	1:01	0.25	1	60	70	50	240
B	1:01	0.5	1	60	98	95	510
C	1:01	1	1	60	98	58	360
4 Effect of GG:ST: AAM							
A	01:00.5	0.5	1	60	48	50	210
B	1:01	0.5	1	60	75	60	260
C	01:01.5	0.5	1	60	98	95	510
D	1:02	0.5	1	60	83	90	450

It is reported that the saponified copolymer showed increased water absorbency (27). In case of AM-g-GG-ST, the –CONH2 groups are converted in to –COOH and –COONa groups during saponification. The effect of various parameters of saponification and on conversion of –CONH2 to –COOH and –COONa is seen from the results in Table 2.

Table 2: Effect of various parameters on water absorbency of saponified AM-g-GG-ST Graft copolymer

Sr. No.	Effect of NaOH Conc. (N)	Time (min)	Temp (°C).	Water Absorbency of AM-g-GG-ST in (g/g)
				Saponified
1 Effect of Time				
A	0.1	60	80	280
B	0.1	80	80	510
C	0.1	120	80	350
2 Effect of Temp.				
A	0.1	80	60	310
B	0.1	80	80	510
C	0.1	80	100	260
3 Effect of NaOH Conc.				
A	0.05	80	80	380
B	0.10	80	80	510
C	0.15	80	80	250



The optimized parameters corresponding to maximum absorbency observed were temperature 80°C, time 90 min and NaOH concentration 0.1 N. With increasing temperature, time and NaOH concentration beyond optimized limits, the product shows decreased water absorbency. This may be due to degradation of polymer network because of over hydrolysis. The amount and variety of hydrophilic groups such as $-\text{CONH}_2$, $-\text{COONa}$ and $-\text{COOH}$ will govern the absorbency of superabsorbents. The enhanced absorbency behavior may be attributed to the collaborative absorbency effect of $-\text{CONH}_2$, $-\text{COONa}$ and $-\text{COOH}$ groups which is superior to that of individual category of $-\text{CONH}_2$, $-\text{COONa}$ and $-\text{COOH}$ groups [27].

Table 3: Effect of AgNO_3 concentration on absorbency of AM-g-GG-ST

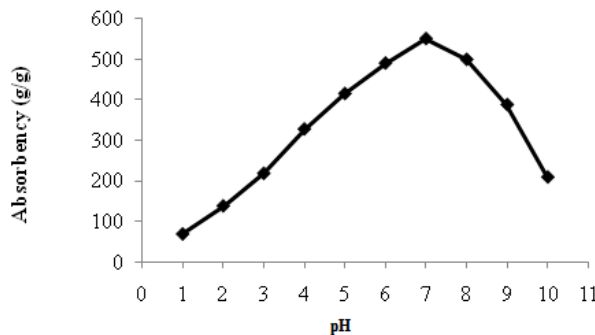
Sr. no.	Concentration of AgNO_3 used for preparation of Nanoparticles	Absorbency in g/g
1	2.5 mM (0.423gm/L)	530
2	5 mM (0.845gm/L)	550
3	7.5 mM (1.268gm/L)	490

Result in Table 3 show the effect of AgNO_3 concentration on absorbency. The sample equilibrated with 50 ml of 5mM solution of silver nitrate showed higher absorbency of 550 g/g. There after the absorbency decreased due to increase in concentration of metal in hydrogel network. Results in Table 4 show the effect of AM-g-GG-ST containing Ag nanoparticles on zone of inhibition when tested against *S.aureus* and *E.Coli* bacteria. Zone of Inhibition was 8.5 mm and 7mm, respectively and it is reported that zone of inhibition > 2 mm, [28, 29] qualified sample to be called as antibacterial. The results may simply be attributed to higher efficacy of Ag nanoparticles possessing greater surface area and thus inhibiting the growth of bacteria more effectively.

IV.B. VARIATION IN SWELLING IN SOLUTION OF DIFFERENT pH

It is recognized that the swelling behavior of superabsorbent is significantly influenced by factors such as salt solution and pH. To investigate the sensitivity of the hydrogel to pH, the hydrogel was studied at various pH ranging from 1.0 to 10.0 (Fig.7).

Fig. 7: Effect of pH on absorbency of AM-g-GG-ST copolymer containing Ag nanoparticles



No additional ions (through buffer solution) were added to the medium for setting the pH, in order to avoid possible effect of ionic strength on the absorption of a superabsorbent. Therefore, stock solution of NaOH (pH 13.0) and HCl (pH 1.0) were diluted with distilled water to reach desired basic and acidic pH, respectively. Fig. 5 indicates that the maximum water absorbency was observed in distilled water (at pH 7). Although it may also happen in very acidic or basic pH, but the ionic strength of the medium at extreme pH causes decrease in water absorption of entangled network. Furthermore, due to the moderate change of ionic strength of the medium at the pH close to 7, the water absorbency of hydrogel decreased more slowly than that at higher pH.



Absorbency in salt solution (0.1 N NaCl) was also studied and it was found to be 59 g/g. It is to be noted that maximum osmotic pressure is developed when hydrogels are placed in water and hence the maximum swelling is achieved giving very high degree of water absorbency. However, when the polymer is in salt solution (e.g., NaCl), extent of the development of osmotic pressure is much lower than that in case of water which is due to the external solution which contains Na⁺ and Cl⁻ ion. Therefore, extent of swelling is also drastically reduced as reported also in literature [30]. In general, the superabsorbents are expected to absorb water more than 100 times (or >1000%) that of their own weight [31, 32]. Our product showed maximum water absorbency of 550 g/g, and salt (NaCl) solution absorbency 59 g/g qualifying itself to be called as superabsorbent.

V. CONCLUSION

A new superabsorbent hydrogel containing Ag nanoparticles was successfully prepared by grafting AM on to a mixed polymers using APS/TEMEDA as initiating system followed by saponification. This convenient preparative method was conducted under normal atmospheric condition of cross linking and reduction of Ag⁺ ions to developed Ag nanoparticles in the hydrogel matrix. Also this promotes the new biomaterial having potential for use in biomedical/pharmaceutical technology or moisture maintenance material in agriculture/horticulture as superabsorbent. Water absorbency and saline water absorbency were found to be 550 g/g and 59 g/g, respectively. Antimicrobial activity of these hydrogels was also quite promising.

VI. ACKNOWLEDGEMENT

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