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## Synthesis and characterization of *Gum ghatti* based biopolymer superabsorbent hydrogel

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### ABSTRACT

A novel biopolymer-based superabsorbent hydrogel (SAP) was synthesized through chemical cross-linking by graft copolymerization of acrylamide (AM) and itaconic acid (IA) onto Gum ghatti (Gg) via a redox initiator system of ammonium persulfate (APS) and *N, N, N', N'*-tetramethylethylenediamine (TMED), in the presence of *N, N'*-methylenebisacrylamide (MBA) crosslinking agent, sodium bicarbonate foaming agent, a triblock copolymer of polyoxyethylene/ polyoxypropylene/ polyoxyethylene as a foam stabilizer. Characterization of SAP was done by FT-IR, TGA, SEM, HPLC and GCMS. The effects of pH and salinity on the swelling aptitude of the SAP were investigated alongwith its degradability in *Streptococcus bovis* medium.

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### INTRODUCTION

Superabsorbent polymer (SAP) hydrogels are lightly crosslinked hydrophilic polymers that can absorb, swell and retain aqueous solutions up to hundreds of times their own weight. They are mostly used in hygienic (disposable diapers, sanitary napkins, surgical pads, etc.) and, to a less extent, agricultural applications. This ability to absorb water is mainly due to the presence of ionic functional groups<sup>[1]</sup>. Hydrogels which swell and contract in response to external conditions like salt, pH, temperature and electric stimulus have been studied<sup>[2-4]</sup>. In case of ionic hydrogels many structural factors like charge, crosslinking density, hydrophilicity and degree of ionization, pKa value of the ionisable groups affect the degree of swelling. Also, properties of swelling medium like pH, ionic strength of counter ion and its valency influences the swelling properties<sup>[5-8]</sup>. Workers from all over the world are working on the

synthesis of superabsorbents based on natural polysaccharides as backbone because of their exceptional properties like biodegradability, biocompatibility, renewability and non-toxicity<sup>[9,10]</sup>.

Several reports were available on the synthesis of porous hydrogels made from AM and IA monomers<sup>[11-13]</sup>, but no substantial work has been reported related to grafting of AM and IA onto Gg. The present article is based on the synthesis and characterization of a novel biodegradable superabsorbent hydrogel through chemical crosslinking by graft copolymerization of and IA onto Gg.

### MATERIALS AND METHODS

#### Materials

*Gum ghatti* (Gg) was purchased from Sd-Fine Chemicals Pvt. Ltd. Ammonium persulfate (APS), *N, N, N'N'*-tetramethylethylenediamine (TMED), *N,*

*N*-methylenebisacrylamide (MBA), Sodium bicarbonate ( $\text{NaHCO}_3$ ) were purchased from Fluka (Buchs, Switzerland). Lutrol F<sup>®</sup>127 was obtained from BASF (Ludwigshafen, Germany). Acrylic acid (Merck, Darmstadt, Germany) was vacuum distilled at 63 °C/12 mm Hg and 50 °C/50 mm Hg, respectively, prior to use in order to remove the inhibitor. Milli-Q grade deionized water was used for preparing the solutions.

Synthesis of graft copolymer, Gg-g-poly [AM-co-IA]

A pre-weighed amount of Gg (1.0g) was added to 30 ml deionized water in a 500 ml reactor equipped with a mechanical stirrer (RZR 2021, a three-blade propeller type, Heidolph, Schwabach, Germany) and stirred (250 rpm) for 10 min. The reactor was placed in a thermostated water bath to control the reaction temperature at 80 °C. After dissolving Gg and homogenizing the mixture, the monomers AM, IA and the crosslinker, MBA, Lutrol F<sup>®</sup>127 (foam stabilizer) were simultaneously added and the reaction mixture was stirred for 15 min. Then the initiator APS (oxidant) and TMED (reductant),  $\text{NaHCO}_3$  (forming agent) was added (TABLE 1). The solution was stirred at 400-500 rpm while maintaining the temperature and inert atmosphere. The temperature was maintained at 80 °C and the reaction mixture was stirred continuously for 4 h. The low molecular weight substances remaining in the samples after polymerization were extracted with boiling ethanol for 24 h. The product was collected by centrifugation and dried in the oven under vacuum at 60 °C for 24 h. The dried graft polymer was added to 300 ml deionized water. It was allowed to swell during agitation in a water bath at the constant temperature of 60 °C for 24 h. Then it was extracted with ethanol in a soxhlet for 6 h followed by water at 100 °C for 72 h. The precipitate was filtered and dried under vacuum at 60 °C.

### FT-IR spectra

FTIR spectra of individual and crosslinked polymers were recorded in the range 400-4000  $\text{cm}^{-1}$  on a Perkin Elmer Paragon 500 FTIR spectrophotometer using KBr pellets.

### Thermogravimetric analysis

The thermo gravimetric analysis data were recorded

with a shimadzu DTG-50 thermal analyzer. The samples were heated from room temperature to 600 °C at a

TABLE 1 : Composition of the feed mixture

Polymer code	MBA (g)	KPS (g)	TMED (ml)	AM (ml) (1%w/v)	IA (ml) 1% (w/v)	ES (g/g)
P <sub>1</sub>	0.1	0.1	0.1	2.5	2.5	41.2
P <sub>2</sub>	0.2	0.1	0.1	2.5	2.5	37.5
P <sub>3</sub>	0.3	0.1	0.1	2.5	2.5	27.2
P <sub>4</sub>	0.1	0.2	0.1	2.5	2.5	48.8
P <sub>5</sub>	0.1	0.3	0.1	2.5	2.5	39.5
P <sub>6</sub>	0.1	0.2	0.2	2.5	2.5	54.0
P <sub>7</sub>	0.1	0.2	0.3	2.5	2.5	41.5
P <sub>8</sub>	0.1	0.2	0.2	5.0	2.5	37.2
P <sub>9</sub>	0.1	0.2	0.2	7.5	2.5	33.2
P <sub>10</sub>	0.1	0.2	0.2	2.5	5.0	60.1
P <sub>11</sub>	0.1	0.2	0.2	2.5	7.5	54.7

Reaction conditions: Gum ghatti: 1g, Lutrol F<sup>®</sup>127: 100mg,  $\text{NaHCO}_3$ :0.5g,  $\text{H}_2\text{O}$ :30ml, Temperature: 80 °Cs

heating rate of 10 °C per min.

### Scanning electron microscopy

The SEM of gold-coated samples were obtained using JSM - 6390LV scanning electron microscope (Jeol Ltd, Japan) at a magnification of x 5 to 300,000 (Resolution-HV 3.0 nm).

### Determination of residual IA by high performance liquid chromatography (HPLC)

Residual IA was detected and quantified by HPLC (Prominence, Shimadzu Corporation, Japan). The chromatographic system consisted of a computer-controlled pump (model LC 20AT), autosampler (model SIL-10AF) equipped with a 200 $\mu$ l sample loop, photodiode array (PDA) detector (model SPD-M20A). Shimadzu LC Solution software was used for the system and data management. The separation was performed in isocratic mode at a flow rate of 1.0 ml/min and a temperature of 40°C on an analytical column Gemini 5 $\mu$  C18, 150 x 4.6 mm (Phenomenex, USA). An RPC18 Security guard (4 x 3mm, Phenomenex) was employed to protect the analytical column. The mobile phase was aqueous 0.05% orthophosphoric acid and the injection volume was 50 $\mu$ l. The observer backpressure values were in the range from 1400 to 1450 psi. Data was acquired and processed by LC solution software (Shimadzu, Japan).

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### Determination of residual AM by liquid chromatography mass spectrometry (LCMS/MS).

Residual AM was detected and quantified by Liquid Chromatography Mass Spectrometry (LCMS/MS). Analysis were performed on a Perkin-Elmer 200 Micro pump series system (perkin-Elmer, Uberlingen, Germany) coupled to an Applied Biosystem API 2000 triple quadrupole mass spectrometer equipped with a Turboionspary ionization source (Applied Biosystem, Foster City, CA). MS detection was performed in the positive ion mode using multiple reaction monitoring (MRM). Data was acquired and processed by Analyst software (version 1.4.1).

### Swelling measurements of superabsorbent hydrogel

Swelling behavior of the prepared SAP was performed by tea bag<sup>[17]</sup> method. About 0.100 g of sample was added to a small bag made of nylon (50 mm x 90 mm; 200 mesh). Then the bag was completely immersed in the swelling medium (200 ml) at room temperature for 24 h to reach the swelling equilibrium. It was removed from the swelling medium and hung up for 15 min to remove the excess fluid and weighed.

The equilibrium swelling (ES) was defined as follows:

$$ES (g/g) = \frac{(W_s - W_d)}{W_d} \quad (1)$$

Where  $W_s$  and  $W_d$  are the weights of the swollen sample and the weight of dried gel, respectively.

### Degradation study by *Streptococcus bovis*

A culture medium was prepared by taking MRS broth (de Man, Rogosa and Sharpe broth). The broth was sterilized by autoclaving for 15 min at 121 °C at a pressure of 15 lbs (pH= 5.7±2 at 25 °C). *Streptococcus bovis* was inoculated in this medium and the pure culture was maintained separately in the incubator. To 10 ml of the sterilized broth, 0.100 g each of the samples i.e., pAM, poly [AM-co-IA] and Gg-g-poly [AM-co-IA] were added aseptically in separate test tubes, and each tube of samples were supplemented with inoculome of the bacterial stains separately. The test tubes were kept at 37±1 °C in an incubator.

The degradation of samples by *Streptococcus bovis*

was monitored in time intervals of 1,8,15 and 30 days. After the required time period, the samples were washed repeatedly with deionized water, oven dried at 40±1 °C for 24 h. Then the samples were weighed to determine the weight loss.

### Quantitative estimation of free CO<sub>2</sub>

Chemicals requirement: Na<sub>2</sub>CO<sub>3</sub>, phenolphthalein indicator.

Procedure: The cultured sample ('X' ml) and blank tube was titrated against Na<sub>2</sub>CO<sub>3</sub> (N/50) ('Y' ml) using phenolphthalein indicator until the pink color persists for at least 30 s. This was continued till getting a concordant reading.

### Calculation

$$\begin{aligned} N_1 V_1 &= N_2 V_2 \\ (CO_2) (Na_2CO_3) \\ \Rightarrow N_1 \times X &= (1/50) \times Y \\ \Rightarrow \text{Strength} &= (Y \times 22) / (50 \times X) \\ \Rightarrow \text{Free CO}_2 &= [(Y \times 22 \times 1000) / (50 \times X)] \text{ mg/l} \\ \Rightarrow \text{Free CO}_2 &= [(440 \times Y) / X] \text{ ppm} \end{aligned}$$

## RESULTS AND DISCUSSION

In the present study, we have attempted to graft AM/IA comonomers on the Gg backbones. The reactive vicinal group where the grafting is initiated on Gg backbone is CH<sub>2</sub>OH. APS and TMED were used as the redox initiating system. The reaction between APS and TMED produces the trimethyl ethylene methylene diamine radical and hydrogen sulfate free radical. These radicals abstracts hydrogen from one of the existing functional groups in protein backbone (i.e. COOH, SH, OH, and NH<sub>2</sub>) to form corresponding macroinitiator.

These macroradicals initiate acrylamide/ itaconic acid acid grafting onto Gg backbone led to a graft copolymer. In addition, in the presence of a crosslinker, i.e., MBA, crosslinking reaction was occurred and finally a three dimensional network was obtained.

### FT-IR spectra

IR spectrum of Gg Figure1 (a) showed broad peaks at 3446.15 cm<sup>-1</sup> (O-H stretching of carbohydrates), 2900.00 cm<sup>-1</sup> (-CH<sub>2</sub> asymmetric stretching), 1430.77 cm<sup>-1</sup> (-CH and -CH<sub>2</sub> inplane bending in carbohy-

drates), 1023.08 $\text{cm}^{-1}$  (-CO stretching region as complex bands resulting from C-O and C-O-C stretching vibrations) and 630.77 $\text{cm}^{-1}$  (pyranose ring). For Gg-g-pAM Figure 1 (b) and Gg-g-Poly [AM-co-IA] (c), the peaks found at 3400.00, 1669.23, and 1609.31  $\text{cm}^{-1}$  indicate the N-H stretching, the C=O stretching and N-H bending of the amide bands, respectively,

which are characteristics of the  $\text{—CONH}_2$  group containing in the acrylamide. In addition, the peak at 1230.76  $\text{cm}^{-1}$  is for the  $\text{—C—N}$  stretching and 630.76  $\text{cm}^{-1}$  for the weak band N-H out of plane bending. These are the typical absorption bands of the amide. Moreover, band at 1753.76  $\text{cm}^{-1}$  Figure 1 (c) was assigned to carboxylic carbonyl group of itaconic acid.

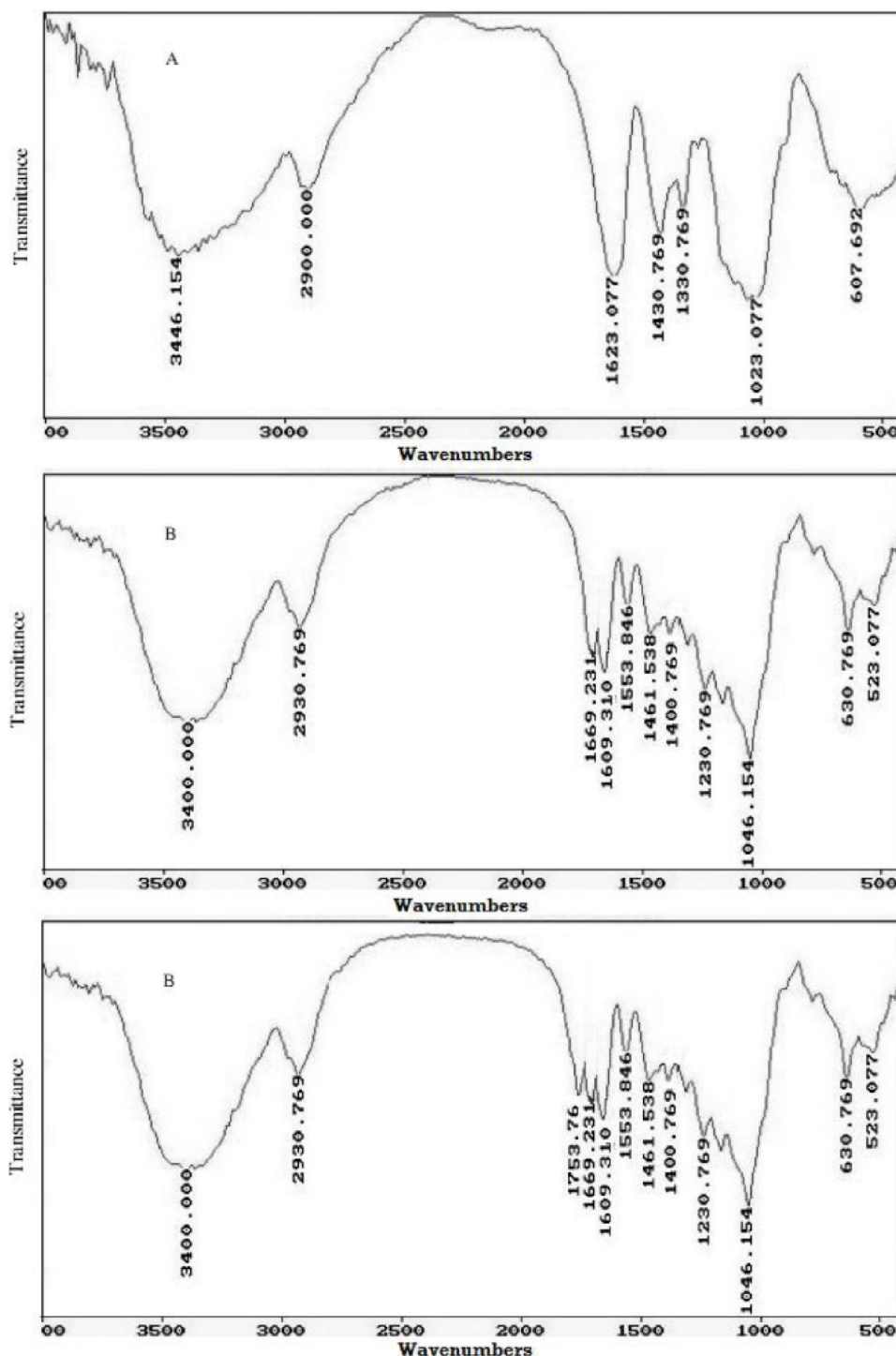


Figure 1 : FTIR spectra of (a) Gg, (b) Gg-g-pAM, (c) Gg-g-Poly [AM-co-IA]

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### Thermal analysis

The results of thermogravimetric analysis (TGA) technique employed to characterize the thermal properties of the obtained graft copolymers as shown in Figure 2. Gg (a) shows a two-step characteristic thermogram, wherein the major weight loss (70%) takes place in the second step within the temperature range of 175–330 °C, the temperature for a maximum decomposition was 315 °C. The thermogram of Poly (AM-co-IA) (b) showed three decomposition stages. The first decomposition stage in the range 35–105 °C was attributed to the loss of bound water. The second one in the interval of 179.1–295 °C had been described to the decarboxylation of IA coupled with the chain scission. The Weight loss in the third or main stage of decomposition (290–390 °C) can be assigned to the degradation of acrylamide portion. In case of Gg grafted with Poly (AM-co-IA) (c), four stages of decomposition were observed. It is suggested that in an initial stage of the thermal diagram, when the temperature in a range from ambient temperature to about 110 °C, the weight loss is a result of the dehydration process of the water contained in such a hydrophilic polymer. At the second stage from 150 to 264 °C, there is a decomposition peak in the side groups and branches of the graft copolymer (carboxyl group in itaconic acid proportion). At the third stage from 265 to 340 °C, there is a degradation of Gg in the graft copolymer. However, at the fourth stage about 350 °C, the weight loss was found as a result of the degradation of the polymer chain and matrices (degradation of polysaccharide and acrylamide portion). From the TGA curves, it can be concluded that the thermal stability of the polysaccharide decreases with the grafting of Poly (AM-co-IA) chains onto the polysaccharide backbone. This may be attributed to the low thermal stability of poly (itaconic acid) as a result of decarboxylation reaction observed in the second degradation step. This phenomenon has also been reported by N. Isýklan<sup>[14]</sup>. He has indicated that thermal stability of the polymer reduced with the grafting of IA onto sodium alginate.

### Scanning electron microscopy

Scanning electron microscopy (Figure 3) allows much high magnification of the hydrogel structure in which we can see the surface. This picture verifies that

the graft copolymers prepared in this work have a porous structure. The surface of the hydrogel is rougher and the approximate diameter of the pores was found to be in between 40 to 60 µm. The uneven surface may be due to a quite high viscosity of the gel and to the solvent evaporation process. It is supposed that these pores are the regions of water permeation and interaction sites of external stimuli with the incorporated drug or hydrophilic groups of the graft copolymers. Therefore, the porous structure is the predominant reason for the higher swelling rate.

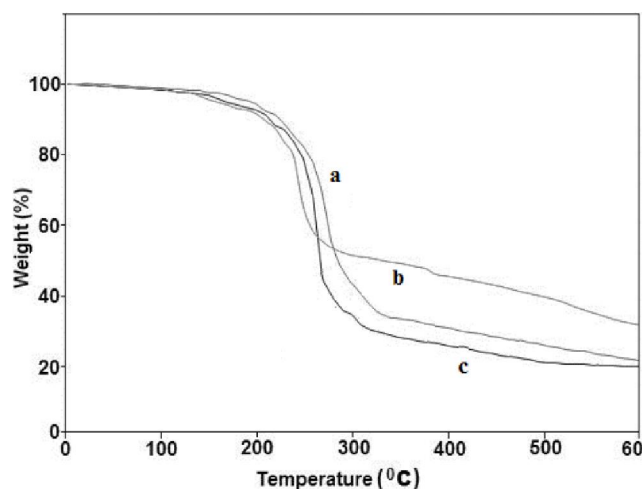


Figure 2 : TGA of (a) Gg, (b) Gg-g-pAM, (c) Gg-g-Poly [AM-co-IA]

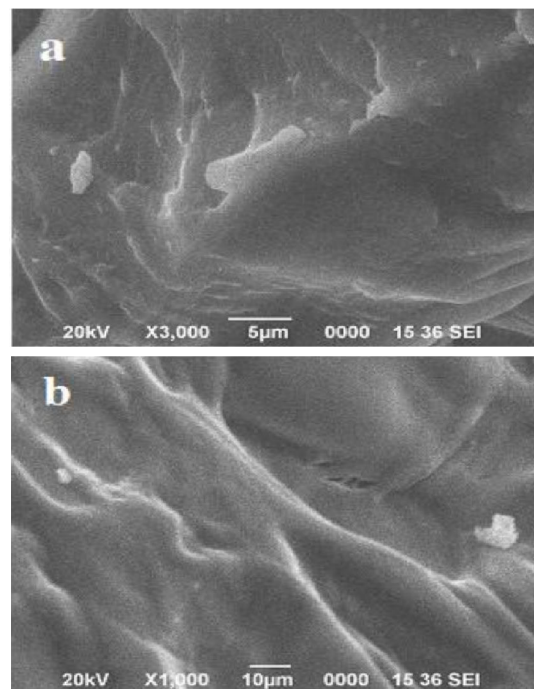
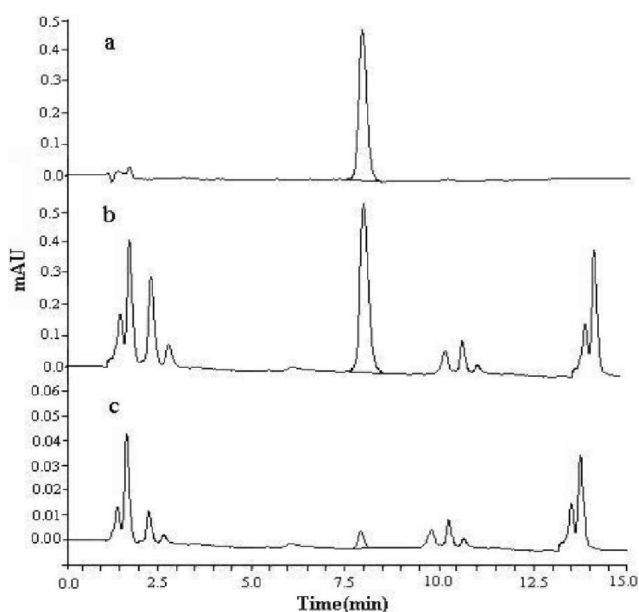


Figure 3 : SEM image of the SAP with (AM/IA) (wt/wt) ratio 0.5

### Residual IA determination

Powdered hydrogel sample (0.300g) was accurately weighed and added to 10 ml methanolic orthophosphoric acid (10:90, pH=2.3) in a polypropylene tube. After vortex (Cyclo mixer, CM 101, Remi Instruments, India) for 10 minutes the sample hydrogels were placed in an ultrasonicator bath (Toshcon, SW-7, India) for half an hour followed by placing in an orbital shaker (Labline instruments, India) at 37 °C with constant agitation (200 rpm) for 12 h. Then it was centrifuged (Eppendorf, 510R, Germany) for 10 minutes at 3500 rpm at 4 °C. The supernatant was taken by means of a syringe, then filtered through a 0.45µm syringe filter (Millipore millex-HV, Hydrophilic PVDF) and finally put in a sample vial (Waters, USA).

Methanolic orthophosphoric acid (10:90) was used as extraction solvent. The coiled and packed chains of hydrogel matrix unfold and make rooms or voids for solvent molecules as it was allowed to swell and the total residual monomer in form of either acid or its salt diffuses from gel network to the extracting solution. Representative HPLC chromatograms of itaconic acid in different hydrogel matrix are shown in Figure 4. In this case also residual IA graft co-polymer decreased considerably after soxhlet extraction i.e. from 24.06 µg/g to 9.73 µg/g.



**Figure 4 :** Representative HPLC chromatograms of standard itaconic acid (0.1µg/ml) (a), residual itaconic acid detected in Gg-g-Poly [AM-co-IA] (before soxhlet extraction) (b), after soxhlet extraction (c).

### Residual AM determination

Dried xerogel (0.100 g) was accurately weighed and added to 4.5 ml water in an amber vial (Supleco, USA). 500 µl of the internal standard (100 ng/ml) solution, d3-acrylamide, is added. The samples are respectively shaken during 1 min on a Vortex and 10 min by orbital rotation. The extract is centrifuged at 5 °C with a speed of 4000 rpm; the supernatant was collected filtered on a nylon membrane. The supernatant was passed through Oasis® HLB SPE cartridges previously conditioned with 5ml of methanol and 5ml of water. Elution occurs with 5ml of water. A second SPE purification involves the Bond Elut-Accucat® cartridges conditioned with 5ml of methanol and 5ml of water before loading with the totality of the extract. Eluent is directly collected and filtered. 20 µl was injected into LCMS/MS.

For the detection by LC-MS/MS, identification occurs with the relative retention time (RRT) and diagnostic ions consisting mainly of the precursor ion at  $m/z$  72.04 and one daughter ion (quantifier) resulting from a loss of HCN at  $m/z$  55.10. For confirmatory purpose, a comparison with quality control samples is made using acceptable deviations of  $\pm 2.5\%$  for relative retention time and of  $\pm 20\%$  for the ionic relative abundance as described in the European Commission Decision 2002/657/EC. It can be seen from the Figure 5 that the amount of the residual AM was decreased markedly after soxhlet extraction (the amount was decreased from 24.01 µg/g to 0.908 µg/g). The decrease in residual AM content was due to its diffusion from gel network to the water, as it was allowed to swell in the excess of water, but a little amount was still detected as few monomers were trapped in the polymer chain.

### Swelling studies

#### Effect of pH on swelling

Equilibrium swelling studies indicated that the hydrogels were sensitive to environmental pH. So, the swelling behavior of the SAP was studied at various pH values between 1.0 and 13.0, at room temperature (Figure 6). To prepare the pH media, standard aqueous HCl (pH 1.0) and NaOH (pH 13.0) solutions were diluted with distilled water to reach the desired acidic and basic pH, respectively. Under acidic pH values,

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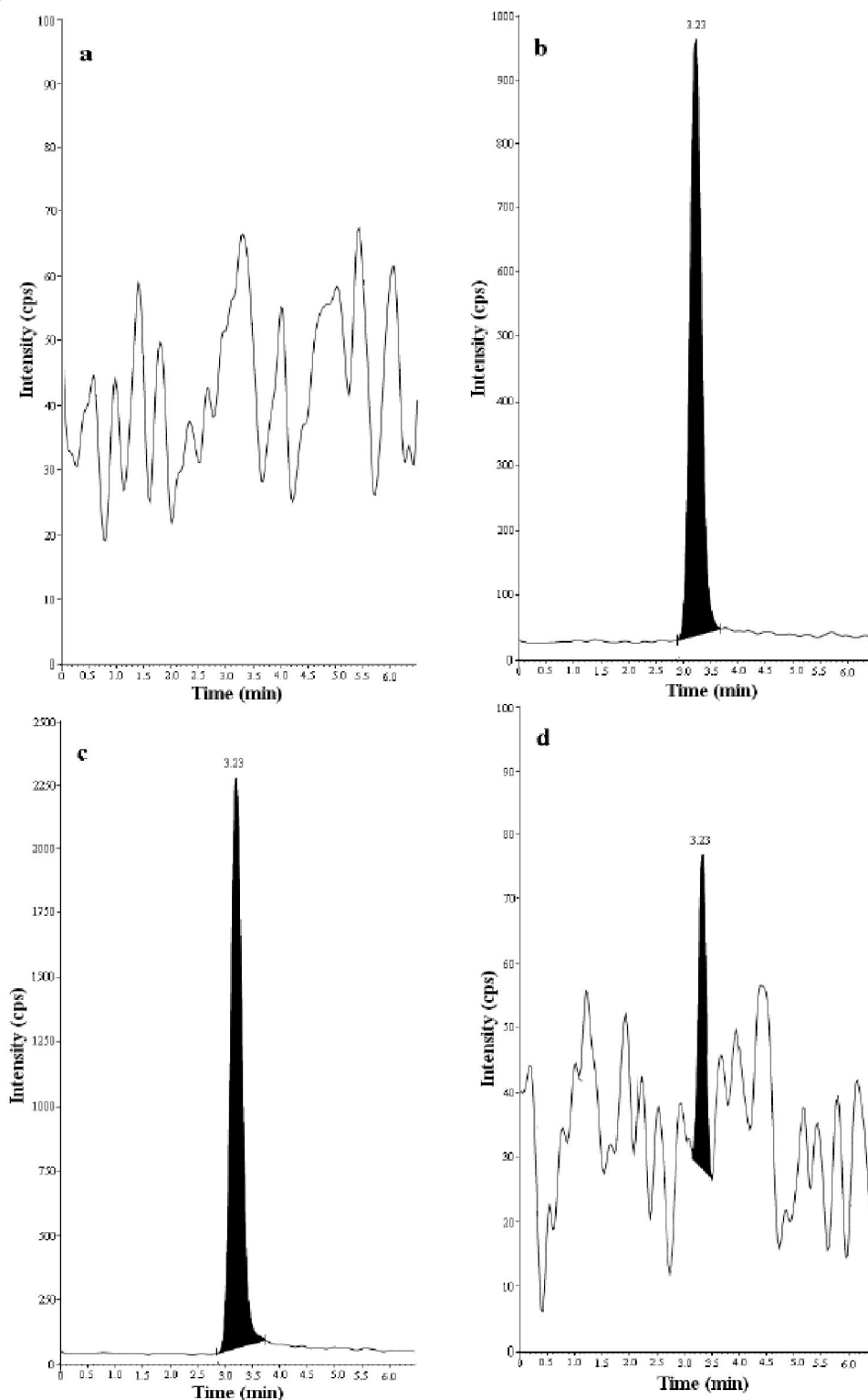
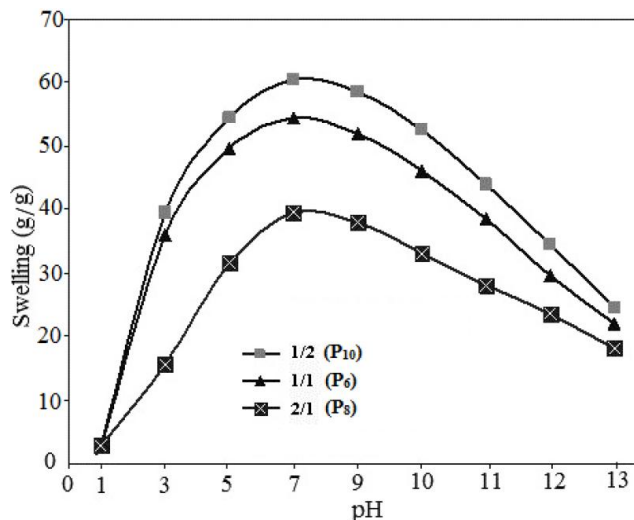


Figure 5 : LCMS/MS chromatogram of residual AM in H10 (a) Blank (Methanol), (b) AM standard 0.250  $\mu\text{g/ml}$ , (c) AM in sample ( $P_{10}$ ) 24.01  $\mu\text{g/g}$  (before extraction), (d) AM in sample (H10) 0.908  $\mu\text{g/g}$  (after soxhlet extraction).

most of the carboxylate anions are protonated, so the main anion–anion repulsive forces are eliminated and consequently swelling values are decreased. At higher pH values (5–7.4), some of the carboxylate groups are

ionized and the electrostatic repulsion between  $\text{COO}^-$  groups causes an enhancement of the swelling capacity. Again, a charge screening effects of the counterions (cations) limits the swelling at higher basic pH values

(>pH.7.4). Figure 6 also shows the effect of the AM/IA (v/v) ratio on the swelling behavior of hydrogels at various pH. The highest swelling capacity was obtained at 1/2 (v/v) ratio of AM/IA. It is known that a high concentration of charged ionic groups in the hydrogel increases the swelling due to osmosis and charge repulsion. In other words, the presence of more ionic groups in the polymer chains results in increased swelling, because the ionic groups are more strongly solvated than non-ionic groups in the aqueous medium.



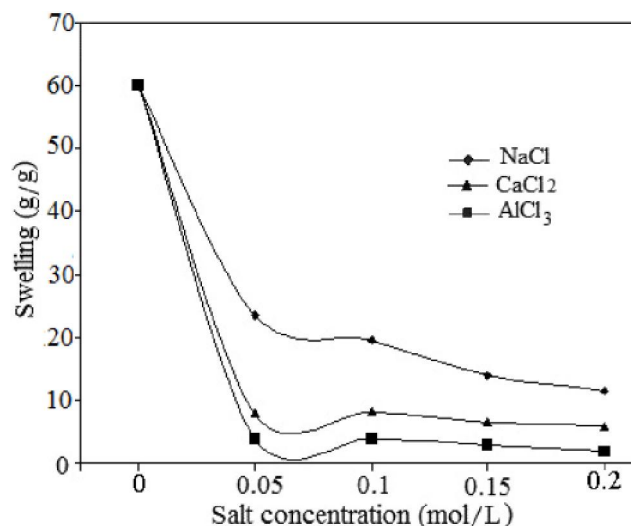
**Figure 6 :** Influence of pH values on equilibrium swelling of SAP with various (AM/IA) (wt/wt) ratios at 25 °C.

### Swelling behavior in salt solutions

The swelling ability of “anionic” hydrogels in various salt solutions decreases appreciably compared to the swelling values in distilled water. This undesired swelling loss is often attributed to a “charge screening effect” of the additional cations causing a non-perfect anion–anion electrostatic repulsion. Also, in salt solution, the osmotic pressure resulting from the difference in the mobile ion concentration between gel and the aqueous phase is decreased and consequently the absorbency is also diminished.

Figure 10 indicates the swelling capacity of the SAP at various salt solutions. It is obvious that decrease in swelling is strongly dependent on the “type” and “concentration” of the salt added to the swelling medium. As shown in the Figure 7, multivalent cations decrease the swelling capacity considerably. This dramatic decrease of water absorbency in multivalent cationic solutions could be related to the complexing ability of the carboxylate groups inducing the formation of intramo-

lecular and intermolecular complexes, which resulted in an increased crosslinking density of the network.



**Figure 7 :** Swelling capacity variation of the SAP (P<sub>10</sub>) in saline solutions with various concentrations

### Re-swelling ability

To evaluate the re-swelling ability and the pH sensitivity of the hydrogels, the gel samples were put in pH 7.4 (PC 510, Eutech Instruments pvt. Ltd, Singapore) solution, then transferred to pH 1.0 solution. This operation was done in four cycles. It can be seen from Figure 8, that swelling ratio values almost remained unchanged in pH 7.4 and pH 1.0 solution. It is clear from the figure that the gel took almost 16.3 min to swell up to maximum (i.e., 60.0g”g), while it required almost 14.1min to de-swell completely (i.e., 10.0 g”g). The de-swollen gel was again allowed to undergo further swelling–de-swelling cycles. The initial deswelling rate is very fast i.e., in the first 5.0 min, the equilibrium mass swelling decreases from 60.0g “g to 16.5.0 g”g. In other words, the equilibrium swelling decreases by nearly 72.5 % in the first 5.0 min when the swollen gel is placed in the medium of pH 1.0. The explanation for these results goes like this. When the completely dried hydrogel sample is placed in the swelling medium of pH 7.4; the solvent diffuses into the outer surface of the gel through the macropores, resulting in the plasticization of macromolecular chains. At the same time, the carboxylic groups, attached along the polymer backbone, undergo ionization to yield –COO<sup>−</sup> groups (since the pH of the swelling medium is more than the pK<sub>a</sub> value of IA inside the gel matrix). This results in the formation



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of a charged hydrated layer through which the counter ions along with the solvent molecules embed into the interior dry core region and allow the gel to swell. In this way, the dry core slowly disappears and the gel matrix continues to swell. The swelling is further enhanced due to relaxation (or unfolding) of macromolecular chains owing to the repulsion among similarly charged  $\text{-COO}^-$  groups which also promotes the swelling process. When the fully hydrated gel is placed in the medium of pH 1.0,  $\text{H}^+$  ions present in the external solution, diffuse into the gel matrix through water filled macropores which have existed in the fully hydrated gel. These  $\text{H}^+$  ions protonate the  $\text{-COO}^-$  groups to yield uncharged  $\text{-COOH}$  groups which ultimately results in folding of the macromolecular chains as the repulsive forces no longer exist, thus letting the solvent molecules to come out of the polymer matrix. The above results show that the hydrogel has good ability and maintain its sharp response to pH variation. These sharp swelling and de-swelling behaviors of the hydrogels make them suitable candidate for controlled drug delivery systems. Such on-off switching behavior as a reversible swelling and de-swelling has also been reported for other ionic hydrogels.

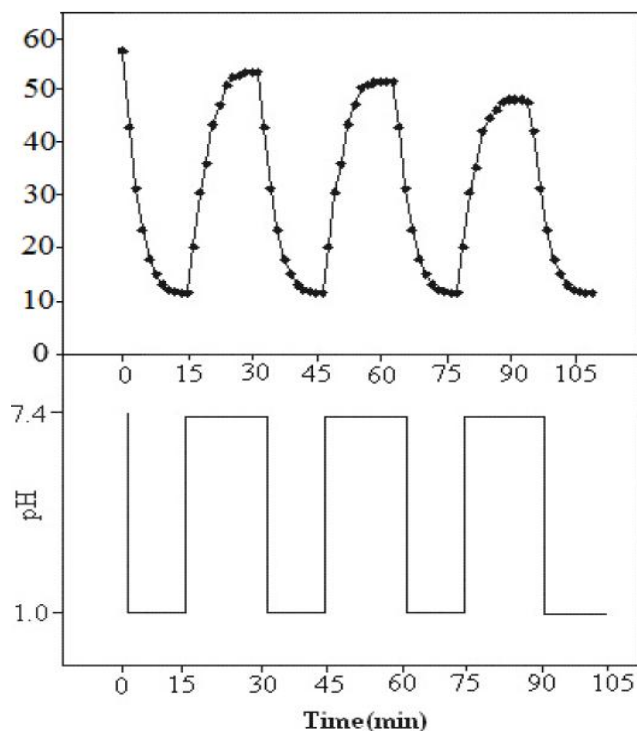


Figure 8 : Equilibrium reswelling behavior of the SAP ( $P_{10}$ ) transferred from a solution of pH 1.0 to 7.0 for four cycles at 37 °C

## Biodegradation by streptococcus bovis

From the comparative biodegradation study of pAM, pIA, Poly [AM-co-IA] and Gg-g-poly [AM-co-IA], it was found that pIA showed accelerated rate of degradation (by weight loss), after different periods of incubation (i.e. 1, 8, 15 and 30 days) in *S.bovis* medium. But the synthesized SAP, Gg-g-poly [AM-co-IA] showed more amount of weight loss than in case of pAM and Poly [AM-co-IA] as shown in Figure 9. This can be explained on the basis that, high water content in SPH facilitates the growth of *Streptococcus bovis* rapidly, thus enhancing the biodegradation, leading to weight loss. The order of biodegradation is as follows:

pIA > Gg-g-poly [AM-co-IA] > Poly [AM-co-IA] > pAM.

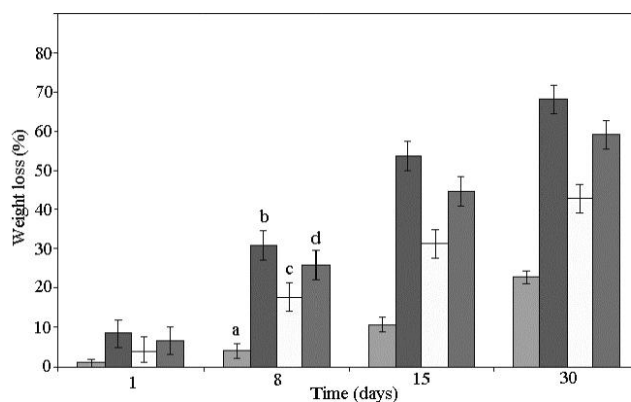


Figure 9. Biodegradation of (a) pAM (b) pIA (c) Poly [AM-co-IA] (d) Gg-g-poly [AM-co-IA] by *S. bovis* measured by weight loss.

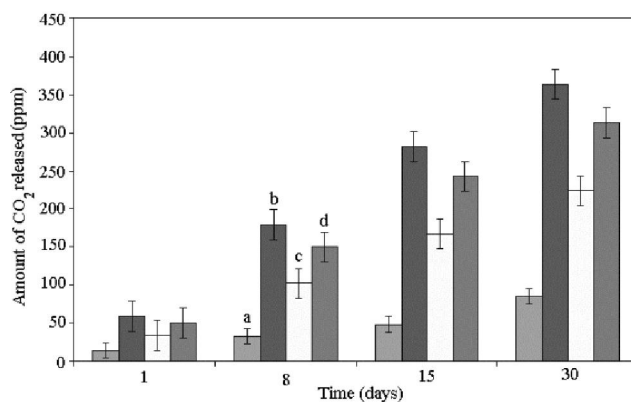


Figure 10 : Biodegradation of (a) pAM (b) pIA (c) Poly [AM-co-IA] (d) Gg-g-poly [AM-co-IA] by *S. bovis* measured by  $\text{CO}_2$  evolved.

Again, the rate of degradation was also measured by calculating the amount of  $\text{CO}_2$  evolved from the cultured medium at different times. The results in Figure 13 showed similar trend of degradation exhibiting more

biodegradability of the SAP than pAM and Poly [AM-co-IA]. By comparing both weight loss method and the CO<sub>2</sub> release method for the study of degradation of the SAP, it was inferred that the order of biodegradation was equivalent in both methods and it was further concluded that the SAP was biodegradable in nature.

### CONCLUSION

Gg-g-poly [AM-co-IA] SAP was synthesized by free radical graft copolymerization method. The surface morphology of the SAP was characterized by SEM, and found that the hydrogel was porous in nature. The SAP prepared through above method was suitable for various biomedical applications because of its high water absorption capability, good swelling and deswelling ability, biodegradability and lower content of residual monomers as evidenced from the HPLC and GCMS data. The SAP may potentially be used in agriculture, biomedicine, pharmaceuticals and controlled delivery of bioactive agents which is in progress in our laboratory.

### REFERENCES

- [1] K.Kabiri, H.Omidian, M.J.Zohuriaan-Mehr; J.Polym.Int., **52**, 1158 (2003).
- [2] S.W.Ali, R.S.A.Zaidi; J.Appl.Polym.Sci., **98**, 1927 (2005).
- [3] E.Karadag, O. B.Uzu"m, D.Saraydin; Eur.Polym.J., **38**, 2133 (2002).
- [4] F.L.Buchholz, T.Graham; Modern Superabsorbent Polymer Technology; Wiley-VCH: New York, (1998).
- [5] A.Sannino, A.Esposito, A.De Rosa, A.Cozzolino, L.Ambrosio, L.Nicolais; J.Biomed.Mater.Res., **3**, 1016 (2003).
- [6] M.J.Ramazani-Harandi, M.J.Zohuriaan-Mehr, A.A.Yousefi, A.Ershad-Langroudi, K.Kabiri; Polym.Test., **25**, 470 (2006).
- [7] A.Jamshidi, F.A.K.Beigi, K.Kabiri, M.J.Zohuriaan-Mehr; Polym.Test., **24**, 825 (2005).
- [8] A.Esposito, A.Sannino, A.Cozzolino, S.N.Quintilianod, M.Lambertia, L.Ambrosio, L.Nicolaise; Biomaterials, **26**, 4101 (2005).
- [9] R.Rodríguez, C.Alvarez-Lorenzo, A.Concheiro; J.Controlled.Release., **86**, 253 (2003).
- [10] J.Zhang, R.Liu, A.Li, A.Wang; Polym.Adv.Technol., **17**, 12 (2006).
- [11] K.Erdener, S.Dursun, G.Olgun; Polym.Adv.Technol., **8**, 574 (1996).
- [12] E.Vallés, D.Durando, I.Katime, E.Mendizábal, J.E.Puig; Polym.Bullet., **44**, 109 (2000).
- [13] A.Martínez-Ruvalcaba, J.C.Sánchez-Díaz, F.Becerra, L.E.Cruz-Barba, A.González-Álvarez; eXP.Polym.Lett., **3**, 25 (2009).