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Development of Functional Guar Gum-Based Highly Water Absorbent and Investigation of Reaction Parameters

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Abstract: The present article describes guar-gum (GG) polysaccharide-based eco-friendly highly water absorbable polymer network with itaconic acid (IA) and acrylamide (AAm) as monomers, prepared due to characteristic features such as natural abundance, biocompatibilities, and biodegradability as well as biological and physicochemical properties. The solution polymerization technique was used successfully to synthesize the GG-g-PIA-co-AAm co-polymer. In this method, IA and AAm monomer polymerize onto polysaccharides GG in an aqueous medium at 70 °C with potassium persulfate (KPS) acting as a chemical initiator and N, N′-methylene-bisacrylamide (N-MBA) acting as a cross-linker. The maximum allowed time to complete the reaction was 1 hour and 20 minutes. The consequence of the neutralization degree of itaconic acid, GG, AAm, N-MBA, and KPS was optimized. Surface morphology and binding behavior of prepared GG-g-PIA-co-AAm were characterized by FTIR and SEM microscopy. The grafting (between GG and IA and AAm and crosslinking reactions were produced by a solid chemical empathy between the NH_2 groups in the guar gum and the carboxylic groups in the poly itaconic acid, leading to the formation of amide bonds. Various parameters like free-absorbency capacity (FAC) as high as possible and (AUL) were also studied for efficient absorbent polymers. A hypothetical mechanism for polymeric reaction during polymerization has been proposed.

Keywords: Solution Polymerization, highly absorbent polymer, free-absorbency capacity (FAC), Absorbency under load, FTIR

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INTRODUCTION

Highly absorbent polymers are cross-linked polymers (hydrophilic) that can engage and recall enormous capacities of water and solute particles in a swollen state, because of the presence of -COOH,-NH_2 -OH, and other hydrophilic groups attached to the polymeric backbone (1-2). The highly absorbent polymer is usually composed of ionic monomers and is branded by a short cross-linking density, which results in large watery curiosity sizes (up to 1000 times their weight) (3-4). Remarkably, these competent absorbent nets can grip and recall aqueous solutions up to several hundred times their weight, even while under pressure. Highly absorbent polymers are used in a wide range of applications, including sanitation goods such as adult incontinence products, baby diapers (5) and feminine napkins (6), forestry, agriculture, horticulture, drug distribution, food storage, waste water management (11), tissue work, and biosensor (12).Because of their biocompatibility and biodegradability (15–16), natural polymers (13–14) and their derivatives (from wood or plant-based biomass) act as highly absorbents to store water and have an excellent ability to absorb water multiple times their weight.

Furthermore, GG is a naturally occurring polysaccharide derived from the guar plant that is water-soluble, and normally nonionic. The GG is unsolvable in carbon-based solvents such as esters, alcohols, ketones, and hydrocarbons with only the exclusion of formamide. For GG, water is the most suitable solvent, and its forms at high viscosity appear uniform at very low GG concentration and at high viscosity from colloidal solution. With the existence of several OH groups across the chain, GG forms H_2 bonding in an aqueous solution, which can be explained by the big deal of its performance. The main problem of GG hurts from some interruptions like biodegradability, which restricts its application but it possesses applications like stabilizing, thickening, gelling, emulsifying, binding, etc., which can be improved through the addition of vinyl monomers (=CH_2) (IA/AAm) hydrophilic nature and extremely sensitive for polymerization.

The GG has been added by various monomers (17-20) to modify the properties of GG and greatly expands into a highly absorbent, which involved water absorbency and swelling amount. Many initiators, such as $KMnO_4$ (21-22), Cu^{2+}(23), potassium bromate ($KBrO_3$) (24), KPS (25), ammonium peroxodisulfate ($(NH_4)_2S_2O_8$) (26-27), ceric ions (Ce IV), adopted in the different type of polymerization (28-29). In case of polysaccharides, more research was done in the case of chitosan based superabsorbent polymers (30-34)

Feasible alternatives to acrylic acid and methacrylic acid-based polymers are IA and their derivatives. The main feature of IA today is that its manufacture does not depend on sources such as petrochemicals. IA deliberate its polymerization in an aqueous phase and water as a solvent for polymerization (redox pair/ initiators) received more attention by detailed investigations of methacrylic acid, (35) acrylic acid, (36) and acrylamide (37). In natural polysaccharides, AAm is widely used as grafting material and is the most significant vinyl monomer for large polymer-add-on due to its hydrophilic nature. Antimicrobial properties are also developed by grafting of water-loving polymers such as PAAm.

In this paper, polysaccharides are a novel GG-based highly absorbent polymer offering enhanced performance. Polymers were synthesized by N-MBA working as a crosslinker, and KPS played the role of the initiator in polymerization for IA:AAm with water sponginess near 450 times its weight was investigated. The effect of the NaOH, N-MBA, and KPS, as well as the reaction on the resultant absorbent things, have conversed. Also, we studied the AUL of highly absorbent polymers.

EXPERIMENTAL

Materials

IA (Sigma-Aldrich), acrylamide (Merck), GG (viscosity average molecular weight: k300 Da) was purchased from Hindustan Gums Ltd. KPS, N-MBA, potassium bromate ($KBrO_3$), ammonium peroxodisulfate ($(NH_4)_2S_2O_8$), ceric ammonium nitrate ($(NH_4)_2Ce(NO_3)_6$), and potassium mono sulfate(PMS) acquired from Sigma-Aldrich were used exactly as received. NaOH and NaCl were bought from E-Merck. The remaining materials were AR grade and solution prepared with DI water.

Initiator Induced Polymerization of Comonomers (IA/AAm) onto GG

GG powder 0.4 g dm-3 and 75 mL DI water were added into the 3-necked reactor with stirring at 400 RPM until the uniform solution was formed. The predefined quantity of itaconic acid monomer was partly defused by the drop-wise addition of 8N NaOH solution below cooling in an ice bath to remove exothermic neutralization reaction temperature. Prepared mol ratios of itaconic acid monomerto 8N NaOH solution were 1:0.65, 1:0.70, 1:0.75, 1:0.80, and 1:0.85.The neutralization ratio prop is well-defined as follows:

$$\mathrm{prop} = \frac{n_a}{n_b} \qquad \text{(Eq. 1)}$$

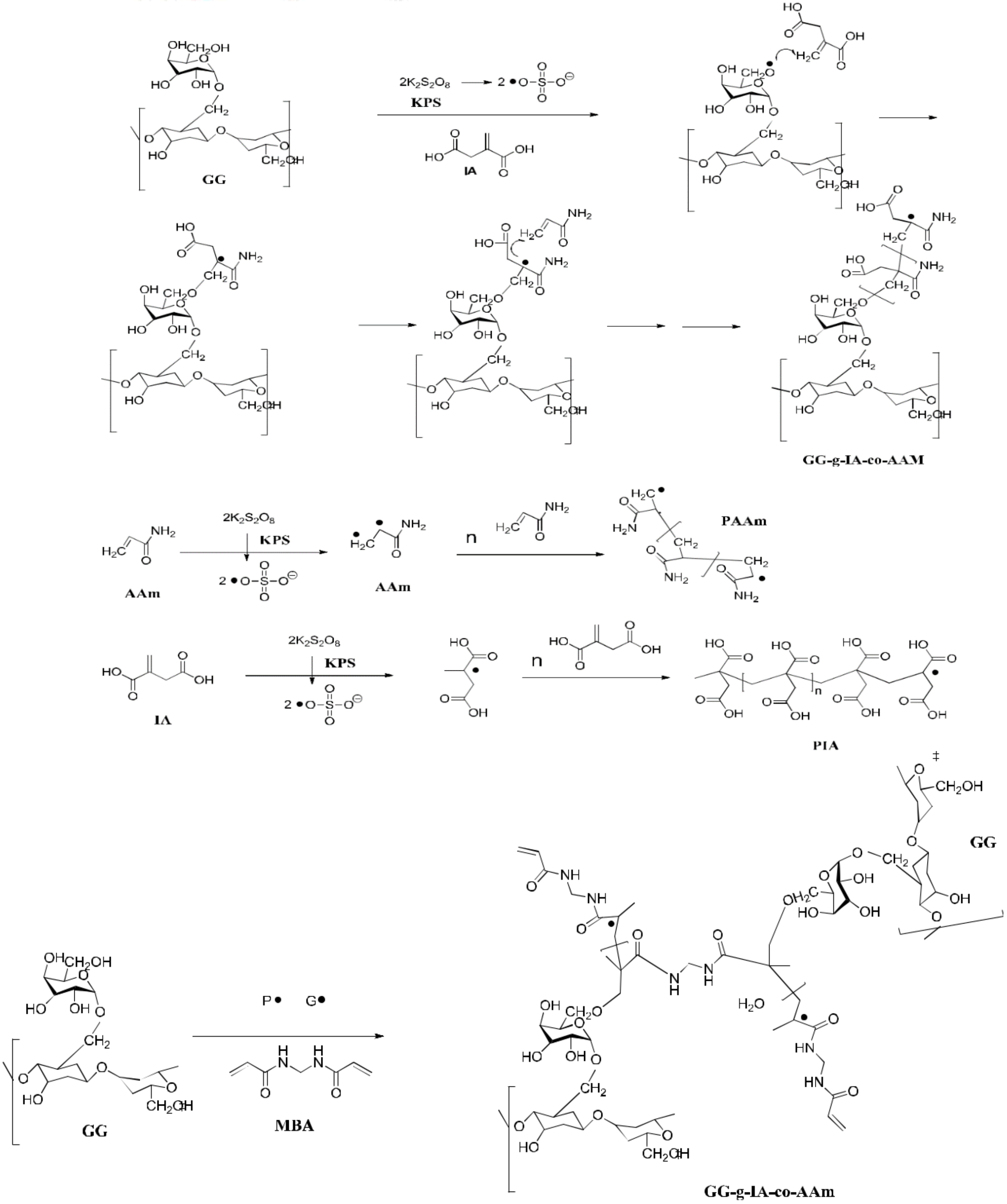
Where n_a and n_b are the mol figures of itaconic acid monomer and NaOH, respectively. Then a 3-necked reactor was positioned in an oil immersion predetermined at the preferred temperature (70ºC) filled with Nitrogen. The neutralized IA was drop-wise added to the 3 neck reactor with the help of dropping funnel containing GG, pre-determined amounts (1 g) of AAm, and (0.5–0.2 g) N-MBA, with stirring and degassed by blowing nitrogen for 30 minutes. After a desirable interval, 0.05 mol/L KPS (radical initiator) added. The induced polymerization of co-monomers was allowed to proceed at 70ºC in nitrogen, (inert medium) 2h with magnificent at 600 RPM. The blend was permissible to cool at ambient temperature. GG-g-PIA-co-AAm was washed away by ethanol and dehydrated in a 60ºC oven for 48h to persistent weight. After grinding, the powdered highly absorbent polymer (GG-g-PIA-co-AAm) was stored away from light heat and moisture. The yield of the polymerization was planned as the weight ratio of addition after the graft co-polymerization to IA/AAm monomer by the following equation-2:

$$Yield = \frac{W_{\mathrm{GG\text{-}PIA\text{-}co\text{-}AAm}} - W_{\mathrm{GG}}}{W_{IA+AAm}} \qquad \text{(Eq. 2)}$$

Where WGG-g-PIA-co-AAmis the weight of the graft copolymer (GG-g-PIA-co-AAm), WGG is the weight of the unique GG, and W_{IA+AA_m} is the weight of the comonomer.

The mechanism of polymerization and crosslinking reaction: (Scheme 1) planned as first the KPS as described in the previous study and then various reactions given as follows:

$$K_2S_2O_8 \rightarrow KSO_4^{*} + KSO_4^{-}$$



Scheme 1: Mechanism of polymerization and reaction involved.

CHARACTERIZATION

Infrared spectra (IR) were taken on a Bruker FT-IR spectrometer (Vector 33 Germany). SEM, (Hitachi S-5200 Japan) was used to detect the morphology of the highly absorbent polymer.

Free water absorbency measurement

Free water absorbency for prepared polymers was determined by the well-known tea bag method (38). In this method, acrylic gauze with fine meshes usually known as a teabag, in which precisely weighted highly absorbent polymer (+0.3-0.03 g) per unit size (average) between 255-395 micrometers, was absorbed totally in 300 mL of NaCl solution for 1h to spread equilibrium swelling. At that time the extra solution is decanted by dangling the tea bag until no water fell off. The allowed water absorbency calculated in relation to equilibrated swelling (Qeq) was specified by the equation:

$$Q_{eq} = \frac{W_{TB,f} - W_{TB,i}}{W_{¿-g-PIA-co-AAm}} \qquad \text{(Eq. 3)}$$

Where WGG-g-PIA-co-AAm is the weight of the dehydrated GG-g-PIA-co-AAmtriturate, WTB,f,and WTB, Iam the weight of the teabag afterward and beforehand water uptake, correspondingly.

Absorbency Under Load (AUL) measurement

AUL technique was adopted for measurement in which a cylindrical piston permitting pressure solicitation on the highly absorbent polymer placed in the medium, i.e., enlargement of particles. Water absorbance of polymeric material underneath deed for changing pressure was detected. The experiment established an easily varying cylindrical piston preserved exclusively on a glass tube fitted by pricked bottommost diskette. The calculated amount of highly absorbent polymer was retained in the cylindrical piston, and weight was put on the cylindrical piston to alter the pressure. All the assembly was secure and kept in a trench surrounding 0.8 wt. percent NaCl. In this method that solution height outside the width of a highly absorbent polymer. The swelling amount underfed was measured over a period of period for an assumed weight at ambient temperature.

AUL calculated with the use of Equation 4.

$$AUL\left(\frac{g}{g}\right) = \frac{W_1 - W_0}{W_0} \qquad \text{(Eq. 4)}$$

Where W_1 swollen gel weight (under the given pressure) and W_0 is polymer weight (dry).

RESULT AND DISCUSSION

Proof of Grafting

The Fourier transfer spectra of polysaccharide guar gum explained in Figure-1 and peak at 3346 and 2909 cm^{-1} due to –OH vibration (widening) and -C-H vibration (stretching) respectively.Peak 1420 and 1300 cm^{1} assigned due to scissoring (-CH$_2$) and vibration bending (-OH) respectively. CH-O-CH$_2$ stretching (39) is assigned by the peak at 1080 cm^{-1}. Figure 2 explains the Fourier transfer spectra of highly absorbent polymer GG-g-PIA-co-AAm obtained by the copolymerization process. The shifting of peak 3344 cm^{-1} to 3610 cm^{-1} when related with spectra of guar gum due to–OH vibration (splayed) guar gum which shows dipping asset elaborate in polymeric reactions. The peak at 2955 cm^{-1} assigned for stretching vibration (C-H) and angular distortion (widening) CΞC stretching at 1610 cm^{-1} further confirmed the polymerization reaction. -CN bond peak allocated at 1411 cm^{-1} and 719~668 cm^{-1} accredited to NH flapping vibration.

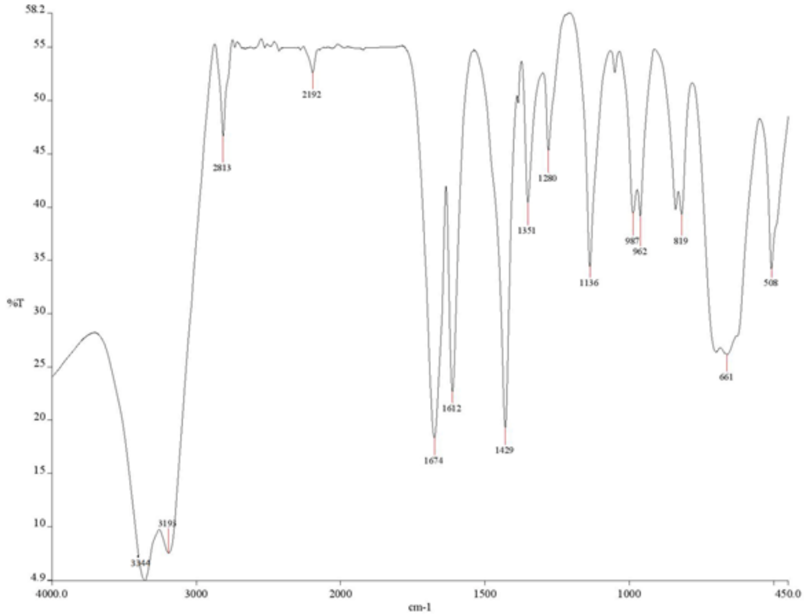


Figure 1: FT-IR spectrum of GG.

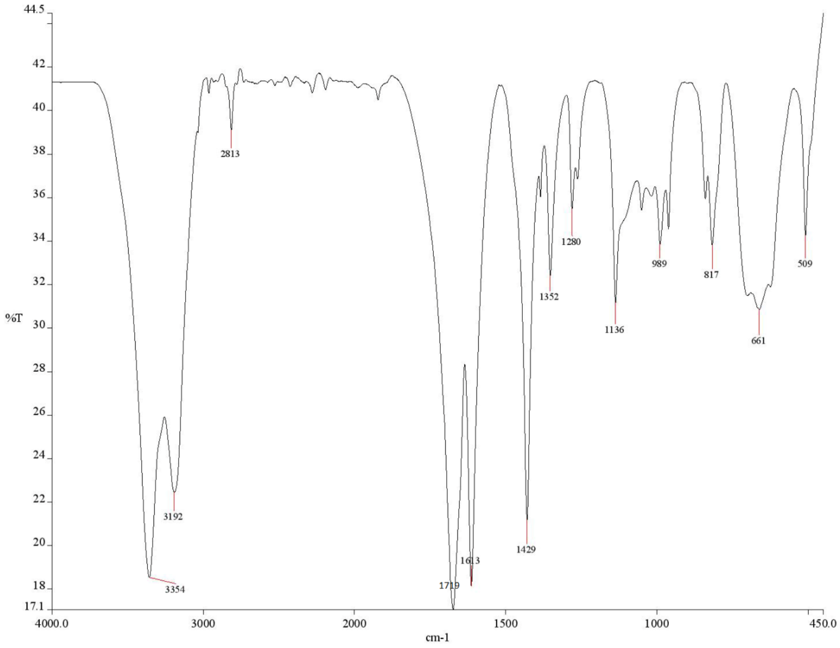


Figure 2: FT-IR spectrum of GG-*g-PIA-co-*Aam.

Scanning Electron Microscopy (SEM)–Guar gum and GG-g-PIA-co-AAm morphology was studied by SEM analysis characterized native guar gum (Figure 3a) precast with rotund structures but highly absorbent polymer showed that (GG-g-PIA-co-AAm), uneven shaped (junkyard) gatherings were invented due to IA/AAm combined and represented in Figure 3(b).

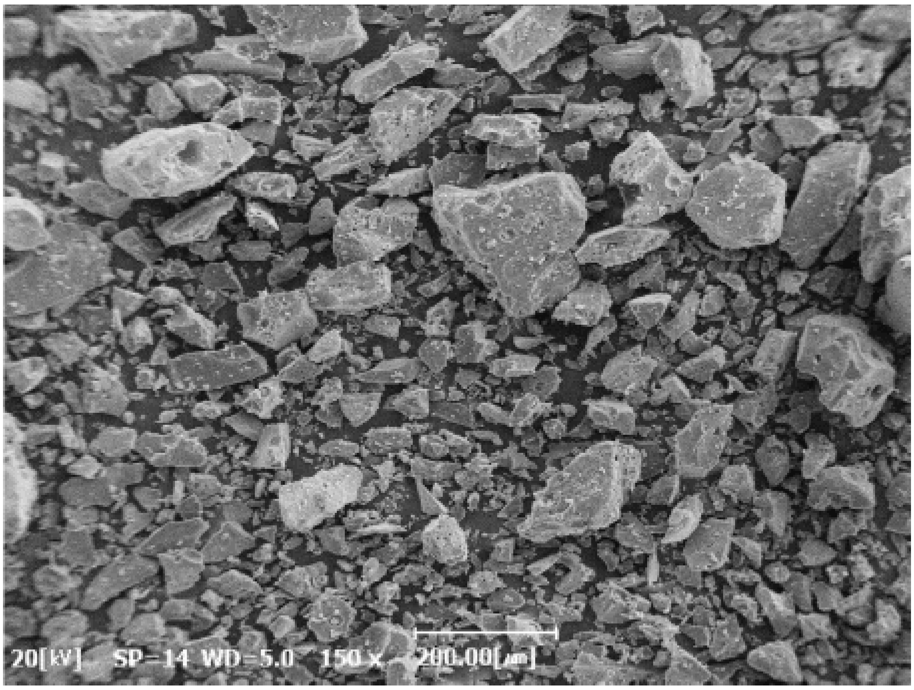


Figure 3(a) SEM micrograph of GG.

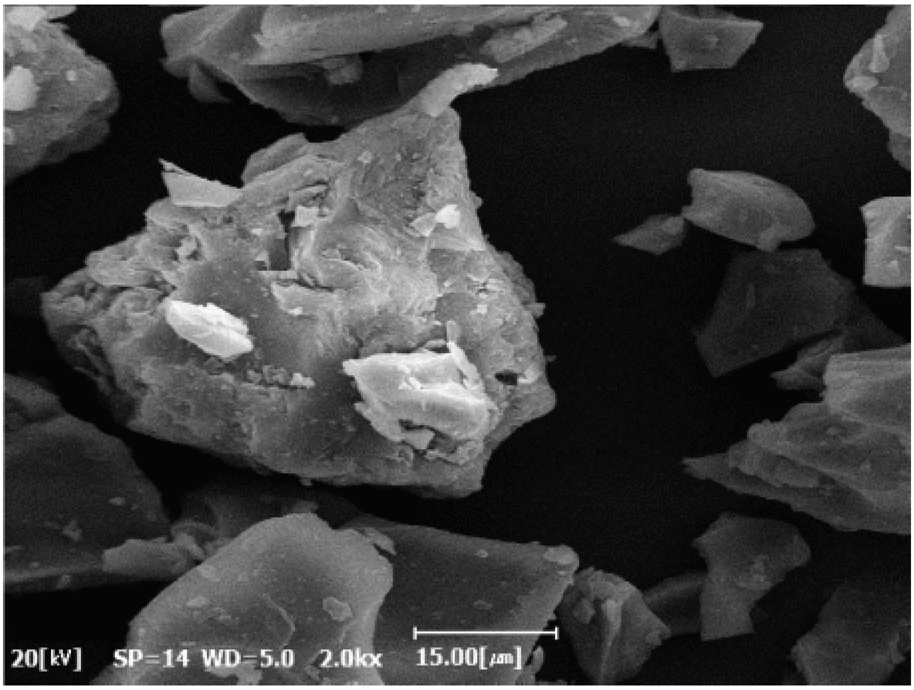


Figure 3(b): SEM micrograph of GG-*g*-*P*IA-*co*-Aam.

Optimization of Solution Polymerization

Choice of Initiators

There are several initiation systems available for the polymerization, but for the selection of the best initiation system for the vinyl monomers' polymerization onto natural polysaccharides, we tested five altered arrangements which includes potassium bromate ($KBrO_3$), KPS, ammonium peroxodisulfate ($(NH_4)_2S_2O_8$), ceric ammonium nitrate ($H_8N_8CeO_{18}$), and potassium mono sulfate (Table-1). KPS gave the best result, which included the highest yield and conversion after 3-hour polymerization. But in the case of $KBrO_3$, ($H_8N_8CeO_{18}$), PMS ,and ($(NH_4)_2S_2O_8$) gave 85, 75, 64, and 60 of conversion, respectively. Absorbency of the highly absorbent polymer was the peak value (21.7 g/g) for the KPS scheme in saline solution (0.9 wt% NaCl). Firstly, potassium persulfate ions ($O_8S_2^{2-}$) are readily disconnected to sulfate-anion-radicals (thermally), which abstract H_2 from the -OH groups on C_2 atom in the GG chain to form consistent alkoxy radicals (40). These radicals started the grafting of IA/AAm onto GG polysaccharides, which lead to cross-linked polymers in the company of N-MBA cross-linker.

Table 1: Effect of Various Initiators on Free Radical Polymerization of GG-*g*-*P*IA-co-AAm [a.]

Initiator	Yield (%)	Absorbency (g/g)
Potassium persulfate (KPS)	92	28
Potassium Bromate ($KBrO_3$)	87	24
Ceric Ammonium Nitrate (CAN)	77	20
Potassium Monosulfate (PMS)	66	17
Ammonium Peroxodisulfate ($(NH_4)_2S_2O_8$)	62	15

Influence of initiator concentration

The influence of the initiator (KPS) concentration on the reaction (polymeric) and H2O absorbency for highly absorbent polymer was also investigated. The KPS concentration changed from 0.1 to 1.1 weight percent to monomers. The water absorbency of the highly absorbent polymer was improved primarily by growing the KPS concentration up to 0.5 wt. percent, but it reduced later, as shown in Figure-4. When KPS was used as in free radical polymerization it was clear that the molecular weight will fall with the increase of the KPS amount. A further reason for falls in the MW is that the comparative quantity of polymeric arrangement surfaces increases at levels of KPS concentration. A rise in the status of the dismissingstep reaction by bimolecular collisions, which in turn raises the cross-linker density (19).

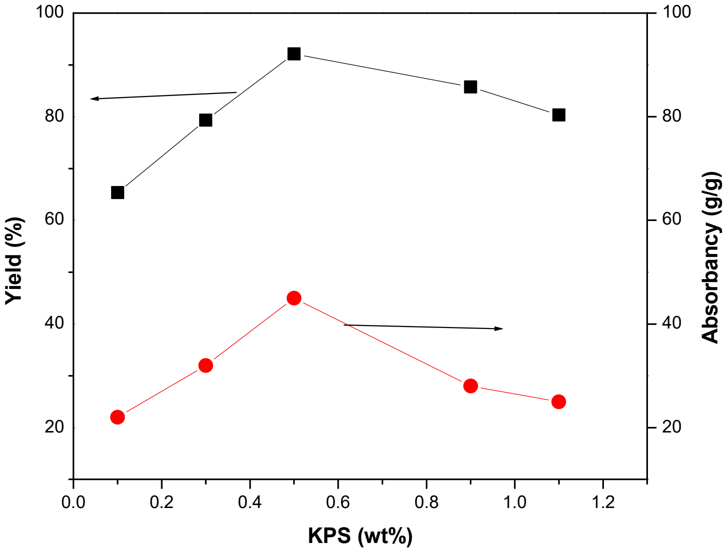
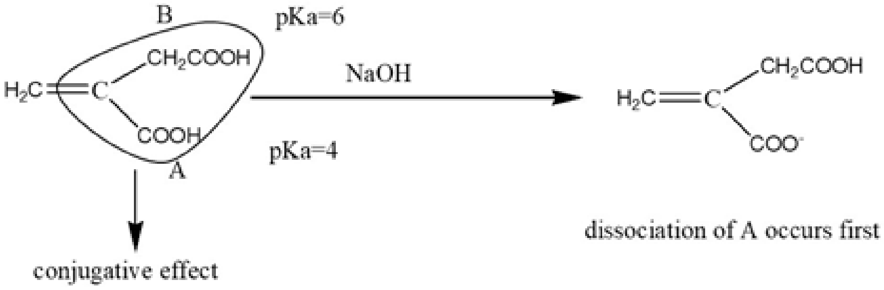


Figure 4: Effect of KPS concentration on (a) yield of graft polymerization and (b) absorbency of the resulting GG-*g*-*PIA*-*co*-AAm.

Influence of degree of neutralization (Dn)

The effect neutralization degree ranges from 50 to 90 percent of IA on the water absorbency (Figure-5). As the neutralization degree of IA rised, water absorbency rised from 50 to 70%. The gripping effect of in-between carboxylate and carboxylic acid groups was explained by the fall in water absorbency and higher in each group, which could be explained the polymerization process naturalization mechanism (Scheme 2). In previously reported work, two carboxyl groups have dissimilar PKa (4 and 6) –COOH groups assigned by A and B in the scheme-3. When IA neutralize group A (represented by –COOH next to =bond in conjugative effect generated with A while B carboxyl group in which CH2 worked as an electron-donor. In this particular case, carboxyl group assigned by A dissociate first and easily explain the influence of the degree of neutralization during the polymerization process.



Scheme 2: Mechanism of partial neutralization of Ia monomer.

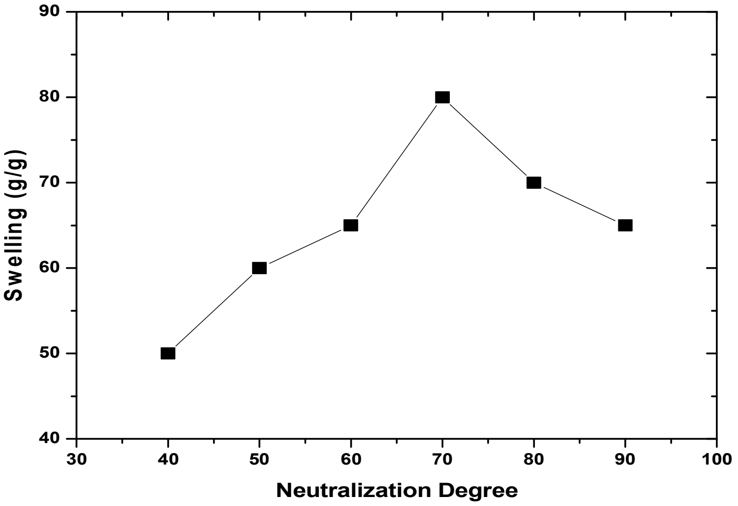


Figure 5: Effect of Neutralizing Agent on the swelling ratio.

The process of neutralization of 50 to 70% can be explained by the above discussion based on decarboxylation (41) and shifting of radicals to -CH2- group and associates extra radical –COOH group as represented in Scheme 2.

Estimation of AUL

The water absorbency of our synthesized highly absorbent polymer varied as a result of external factors such as swelling medium, applied pressure, and so on. Because absorbency under load AUL is proportional to mechanical strength to the gel, AUL can be calculated using the gelling power of adult diapers and superabsorbent polymers (42). 180 minutes was the appropriate time to reach supreme absorbency for several highly absorbent polymers. The AUL values are natural above this period. Based on the standard test technique approved for calculating AUL, the performance of the highly absorbent polymer swollen in 0.8 weight percent Na Clunder with a weight of 7200 Pa was unrushed. In highly absorbent polymers, the AUL values are 12-21g/g and are firm at many applied pressures also, to create the swelling pressure coefficient (SWC). The swelling pressure coefficient (SWC) was intended from the slope of the graph, plotted using applied load and swelling fraction, which was observed to be 0.7% Pa as opitomized in Figure-6.

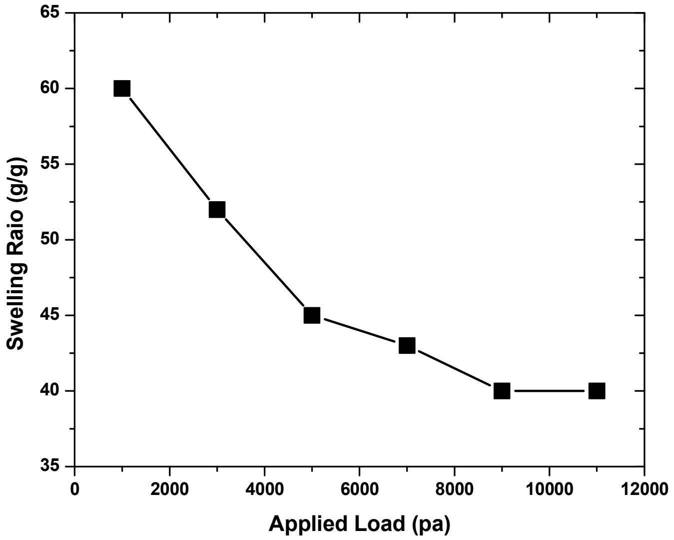


Figure 6: Influence of applied pressure on swelling ratio of GG-*g*-*P*IA-co-Aam.

Effect of N,N′-methylenebisacrylamide concentration

Figure-6 represents the effect of N-MBA with a varying amount of 0.3 to 1.3 wt. percent monomer. In this case, detection yield was high at 0.3 to 0.7 wt. percent for monomer maximum swelling and high polymerization yield, with detection yield exceeding 85%. In the previous study (43) it was very low but in our case, it showed good permanence using squat N-MBA amount with high swelling water. The concentration of higher cross-linkers reduced space between polymer chains and, then, the highly cross-linked inflexible construction cannot be extended to hold a large quantity of water on the growing quantity of N-MBA. The smaller the space between the (polymer) units, the more the highly cross-linked rigid preparation cannot expand to capture a large amount of water (44).

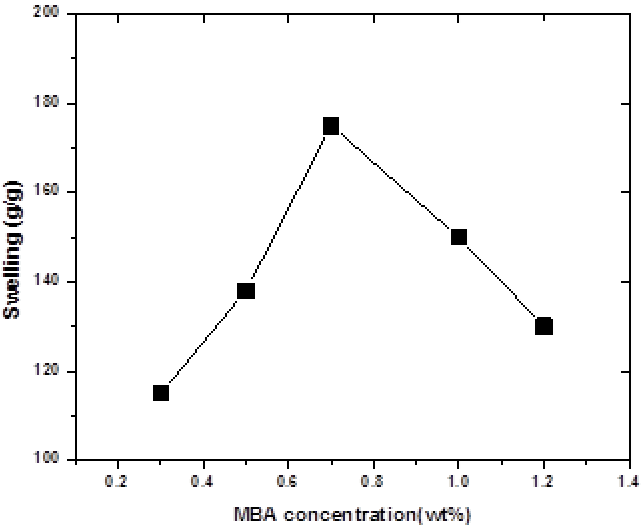


Figure 7: Effect of N-MBA concentration on the Swelling ratio.

Effect of GG concentration on Water Absorbency

Figure 8 represents the variation of guar gum between 0.7 to 1.7 g.dm^{-3} on water absorbency between 0.7 and 1.7 g.dm^{3} and falls with rise as the quantity of guar gum due to change in viscosity medium limits the monomer convenience and rise the polymer unit which falls in water absorbency.

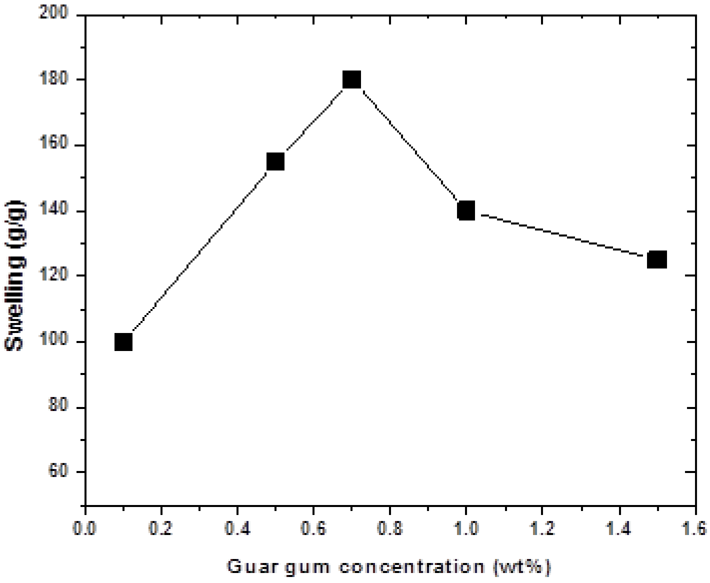


Figure 8 Effect of GG concentration on swelling.

CONCLUSION

In this investigation, the synthesis of highly absorbent polymer (GG-g-PIA-co-AAm) performed by polymerization (free radical) using KPS and N-MBA as initiator and cross-linker respectively, at 70ºC in an inert atmosphere, and their influence on polymerization, were studied. At 70% degree of neutralization, we obtained a high water absorption level, and Fourier transfer and SEM analysis confirmed the synthesis of the proposed highly absorbent polymer. In the presence of acrylamide, highly absorbent polymer materials expand the gel strength (swollen one) and minimize the price of the product. It was observed that highly absorbent polymers (GG-g-PIA-co-AAm) have comparatively decent potential to hold water and NaCl (saline solutions) both as values of the free absorbency (free swelling) and as values of AUL. The highly absorbent water-absorbing and water-holding polymer can be mainly applied to pads and diapers and unsanitary uses.

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