

Synthesis of Superabsorbent Polymers *via* Itaconic Acid Graft Copolymerization onto Acacia Gum Polysaccharide for Improved Material Performance

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In this study, novel superabsorbent polymer composites based on natural acacia gum (AG) were synthesized *via* graft copolymerization of itaconic acid (IA) in the presence of inorganic kaolin powder. The polymerization was initiated by ammonium persulfate (APS) and crosslinked with *N,N'*-methylenebisacrylamide (N-MBA). Key variables influencing the polymerization process, including the concentrations of KPS, IA, N-MBA and the AG/kaolin weight ratio, were systematically studied to optimize water swelling capacity. The structural characterization of the synthesized composites was performed using FTIR spectroscopy and SEM analysis. The FTIR spectrum revealed a new absorption band at 1671 cm⁻¹, confirming the successful integration of kaolin powder and the formation of inorganic-organic polymer linkages. Additionally, the degree of neutralization was examined, along with the evaluation of critical parameters such as free absorbency capacity, absorbency under load of 16.9 g/g and centrifuge retention capacity of 55.1 g/g. The results demonstrate the potential of these superabsorbent polymer composites for various applications due to their enhanced water absorption properties.

Keywords: Superabsorbent, Free-absorbency capacity, Absorbency under load, Centrifugal retention capacity, Inorganic clay.

INTRODUCTION

Superabsorbent polymers are specified as loosely cross-linked; 3D structure, hydrophilic polymeric set-up can swell, absorb and retain huge amounts [100 to 1000 times its own mass] of water or other biotic liquid due to availability of hydrophilic clusters in the structure of superabsorbent polymers while preserving the physical dimension structure [1-5]. Superabsorbent polymers currently may have found many areas of application including sensors, hygienic products, drug delivery [2], gel actuators, agriculture [6] and removal of heavy metals [7] and organic pollutants [7-11], water blocking tapes and coal dewatering [12-19]. In recent years, a wide investigation towards optimization and development of superabsorbent polymers was performed [20]. The commercial superabsorbent materials are used in above-mentioned applications that include neutralization (partial) of crosslinked polyacrylic acid (pAAc), hydrolysis (partial) of acrylonitrile-starch copolymers; acrylic

acid-starch graft copolymers and so on well-known reference available in the literature. Nowadays, an important research work focused on biodegradability of the materials has received much attention towards issues based on protection of the environment [6,21,22]. In recent, years these special classes of biodegradable polymers attained much focus and were renamed as 'globe-compatible materials.

Arabic gum, which is commonly known as acacia gum, natural occurring polysaccharides consists of arabinogalactan, which is made up of galactose and arabinose monosaccharides with liner core chain of β -1,3-linked galactose units with wide splitting at the C6 position [23,24]. The galactose and arabinose branches involve termination with glucuronic acid [25]. The arabic gum possess many common rewards such as biocompatibility, biodegradability, non-toxicity and renewability make it involvement in the various applications which use a marketable polymer in wide zones such as pharmaceutical [6], suspending agents, food and industries for cosmetic production [26,27].

The existence of functional groups in the raw gum limits its performances and applicability. Graft copolymerization [28] (vinyl) is a well-known effective technique to synthesize natural superabsorbent polymers with the help of natural polysaccharides [28-37]. The presented groups available in the parent natural polysaccharides may alter molecular weight, flexibility, mobility, viscosity and thermal stability will help in new applications towards grafted gum in various industries [35,38-40]. The natural gums have the tendency to behave as good reinforcement as fibers in polymer hydrophilicity, matrices [6] and swelling in cold water for viscous dispersion in colloidal form, which is easily biodegradable [41]. This study uniquely explores the synergy between kaolin and natural polysaccharides, setting it apart from reported superabsorbent polymers (SAPs) utilizing fillers like bentonite, montmorillonite or silica. While bentonite-based SAPs often face limitations from higher cross-linking density reducing absorbency, the acacia gum-itaconic acid-ammonium persulfate (AG-IA-kaolin) composites demonstrate superior water retention due to lower crosslinking density and optimized hydrogen bonding as confirmed by FTIR analysis. Additionally, incorporating kaolin enhances swelling, thermal stability, mechanical strength and water barrier properties, making these composites more versatile and robust. Further emphasis on their biodegradability and real-world applications would enhance the novelty and relevance of the findings.

The aim of present study is to employ the graft copolymerization for the synthesis of eco-friendly superabsorbent polymers composites based on acacia gum with sodium salt of itaconic acid and the study of their water-holding properties and water absorbing capacity compared to other natural polysaccharides based superabsorbent polymers (SAPs). The novelty of this investigation led to the development of superabsorbent polymer composites with a higher enlargement rate (water) likened to those described earlier in the literature.

EXPERIMENTAL

Itaconic acid (IA), N,N-methylene-bisacrylamide (N-MBA), ammonium persulfate (APS) and kaolin powder were purchased from Sigma and used without further purification. Sodium hydroxide and sodium chloride were bought from E-Merck. The remaining materials used AR grade and solution prepared in deionized water (DI) and used throughout the experimental work.

Synthesis of superabsorbent polymer composites: In three-necked reactor, acacia gum 0.5 g dm^{-3} dissolved properly in 75 mL DI water. Simultaneously, itaconic acid (IA, 10 g) was neutralized with NaOH (6.1 g) in 5 mL DI water. Then crosslinker methylenebisacrylamide (N-MBA) solution (0.012 g MBA in 3.0 g water) was added to the monomer solution and 0.01 g kaolin powder in the same solution. Then placed three-necked reactor equipped with a thermometer and magnetic stirrer with stirring. The reactor was degassed by N_2 gas blowing for 45 min. Then, initiator APS (0.1 g) was introduced into the reaction reactor. The graft copolymerization reaction was allowed to proceed in an inert atmosphere at 70°C for 120 min with constant stirring at 600 rpm. After the propagation step, the oxygen gas passed to terminate the reaction and then

the reaction mixture was permissible to cool at room temperature. The mixture of superabsorbent polymer composites AG-g-pIA was precipitated in pure ethanol and filtered off on Buchner funnel dried out in a hot air oven at 60°C for 48 h to constant weight. After grinding, the obtained powdered superabsorbent polymer composites (AG-g-pIA) were stored in dark to avoid moisture, light and heat. The yield of the polymerization, grafting percentage (%G) and percent swelling were calculated by the following equations:

$$\text{Yield} = \frac{W_{\text{AG-g-pIA}} - W_{\text{AG}}}{W_{\text{IA}}} \quad (1)$$

where $W_{\text{AG-g-pIA}}$ is the weight of graft copolymer ($W_{\text{AG-g-pIA}}$), W_{AG} is the weight of AG and W_{IA} is the weight of monomer.

$$G (\%) = \frac{W_f - W_b}{W_b} \quad (2)$$

where W_f is the weight of synthesized superabsorbent composites and W_b is the weight of natural polysaccharides acacia gum.

Characterization: The superabsorbent polymer composites (AG-g-pIA) were analyzed for the available functional groups. The absorption spectrum of the sample were taken in KBr pellets using Agilent technologies Carry 630 FTIR spectrophotometer at the resolution of 2 cm^{-1} . To study the morphology behaviour of superabsorbent composites polymers (AG-g-pIA), the cross-section and surface area were examined electron microscopy (SEM) using JEOL JSM-6610LV model.

Swelling measurements using tea bag method: The superabsorbent polymer composites (AG-g-pIA) were evaluated for their water absorbency. To calculate such parameters, different swelling mediums such as water and saline solution (0.9% NaCl) were used. The average size of the per unit sample ($0.3 \pm 0.03 \text{ g}$) between $250\text{-}390 \mu\text{m}$ was placed into tea bag engrossed in large amount of the consistent swelling media such as deionized water, 0.9% saline solution and permitted to swell at room temperature to reach swelling equilibrium. The equilibrium absorbency was calculated using eqn. 3 [42-44]:

$$\text{Swelling ratio (g/g)} = \frac{W_s - W_d}{W_d} \quad (3)$$

where W_s and W_d are the weight of swollen and dried AG-g-pIA, respectively.

Measurement of absorbency under load (AUL): The absorbency under load was calculated using long piston association permitting the accumulation of sample weights on top of the superabsorbent polymer composite materials [45]. A microporous fritted glass filter plate ($h = 8 \text{ mm}$, $d = 82 \text{ mm}$) were located into petri-dish ($h = 14$, $d = 120 \text{ mm}$) and the weighed dried AG-g-pIA sample ($0.5 \pm 0.01 \text{ g}$) was homogeneously spread on the top superficial of a polyester transparent fabric (tea bag) located on the fritted glass. A tubular shape solid load which was made up by Teflon, $d = 65 \text{ mm}$, adjustable tallness) was placed and put some calculated amount of dried AG-g-pIA samples during this procedure the sample could skipped in cylinder made by glass ($h = 52 \text{ mm}$ $d = 62 \text{ mm}$). During this the wanted load or functional compression 0.4,

0.7 and 0.10 psi were located on the superabsorbent polymer composites. Then the prepared 0.9% saline solution was added to obtain the height available liquids with a sintered glass filter. The overall setup was to avoid superficial loss and likely alteration in the saline concentration. After 65 min, the swollen superabsorbent composite polymers were again weighed and AUL was calculated using eqn. 3.

Centrifuge retention capacity (CRC): To study the centrifuge retention capacity (CRC) of sample of superabsorbent composite polymer (AG-g-pIA) (0.1 g) placed into polyester transparent fabric (tea bag) and deep into 0.9 wt.% saline solution for 45 min. After the desired interval of time, the tea bag was then removed from the saline solution and centrifuged at 300 g for a maximum time of 3 min. The preserved sample was detached from the tea bag and weighed [46]. The CRC was then calculated using eqn. 4:

$$\text{CRC} = \frac{S_1 - S_0}{S_0} \quad (4)$$

where S_1 and S_0 are the weight of dry and swollen AG-g-pIA.

RESULTS AND DISCUSSION

FTIR studies: The synthesized superabsorbent polymer composite (AG-g-pIA) was characterized using FTIR spectroscopy to confirm successful grafting and analyze structural features influencing performance. The FTIR spectrum of AG-g-pIA exhibited additional peaks, such as a C-H stretching band at 2931 cm^{-1} and an O-H bending band at 1424 cm^{-1} . A peak at 1618 cm^{-1} was attributed to the COO^- group, indicating carboxyl functionalization (Fig. 1). The spectrum also displayed N-H stretching bands at 3348 cm^{-1} and 3201 cm^{-1} , attributed to 2° amides, with another peak at 1686 cm^{-1} corresponding to their presence. Furthermore, a band at 1419 cm^{-1} suggested symmetrical COO^- group vibrations, indicative of polyacrylamide hydrolysis.

The FTIR band at 1671 cm^{-1} , a key feature in this study, signified the interaction between the carboxylic groups of itaconic acid and kaolin particles, confirming the formation

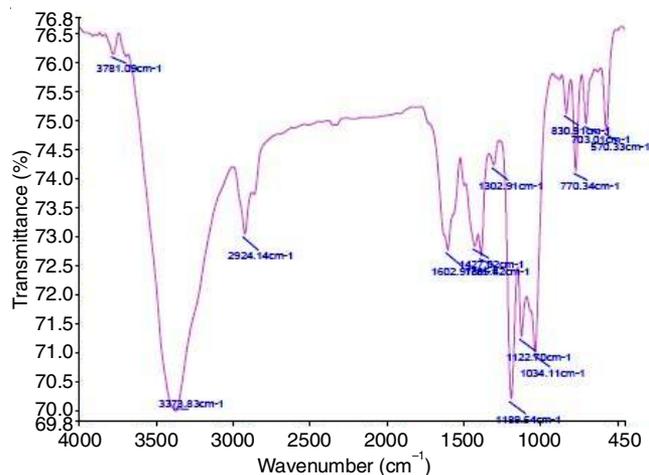


Fig. 1. FT-IR spectrum of superabsorbent composite polymer (AG-g-pIA)

of hydrogen bonds and crosslinked networks within the polymer matrix. These inorganic-organic linkages play a crucial role in the enhanced swelling behaviour and performance of the superabsorbent composites. Hydrogen bonding between kaolin and the polymer matrix facilitates the formation of a hydrophilic network, which significantly improves the water retention capacity. Additionally, the uniform dispersion of kaolin, supported by these linkages, reduces the crosslinking density in the matrix. This structural optimization leads to higher swelling capacity and overall improved performance of the synthesized composites.

SEM studies: The SEM micrographs of acacia gum (AG) and superabsorbent composites polymers (AG-g-pIA) are shown in Fig. 2. In order to study the morphology behaviour of the surface and the overall shape of the SAP particles, the SEM micrographs of high magnification (left) and low magnification (right) were confirmed, respectively. Acacia gum (AG) without monomers exhibits smooth and compact fracture surfaces, while the superabsorbent AG-g-pIA show a rough fracture surface. Furthermore, several large aggregates were also found which differed from the smooth polymer surface as observed in Fig. 2b.

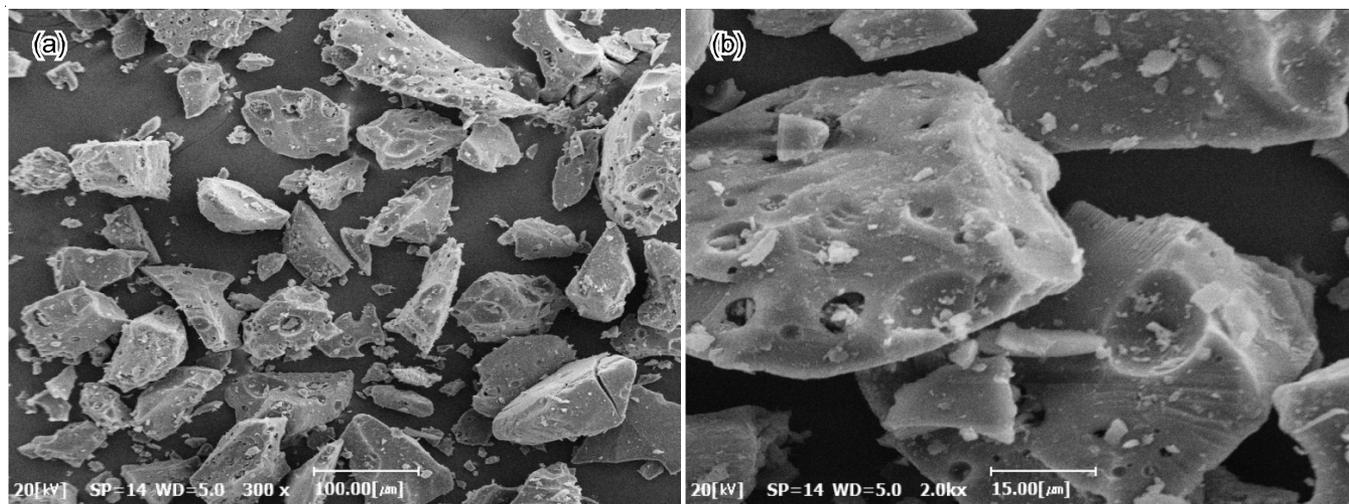


Fig. 2. SEM micrograph of acacia gum (a) and superabsorbent composites polymers (AG-g-pIA) (b)

Effect of initiator (APS) concentration: In Fig. 3, as the amount of APS increases, the water absorbency increases and then decreases. The highest peak of water absorbency in 0.9 wt.% NaCl solution was observed at g/g at 0.2 wt.% of APS concentration. It has been observed that there is a significant effect of APS concentration on water absorbency of the superabsorbent polymers (AG-g-pIA), which agreed with the relationship between the concentration of initiator in polymerization reaction and average chain length. APS initiator generated many free radical sites on the backbone, IA monomer could be grafted on these free radical sites available on the AG backbone.

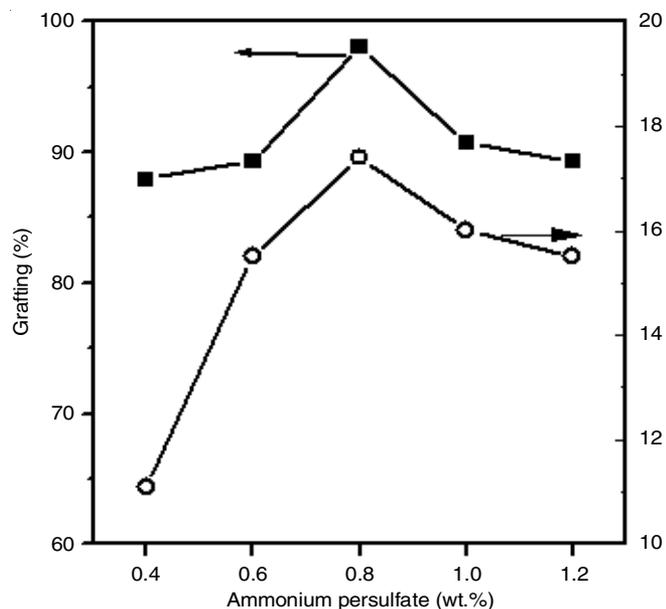


Fig. 3. Effect of ammonium persulfate (APS) concentration

Numerous free radical sites were developed by the APS initiator, IA monomers could be grafted onto the acacia gum (AG) backbone. A 3D AG-g-pIA sample was generated at the lowest concentration of APS initiator, resulting in greater water absorbency. When the concentration of APS initiator increases, the excess of primary radicals terminates the reaction of chain propagation mutually, but due to the reaction of homopolymerization started by primary radicals it is enhanced resulting in low water absorbency.

Influence of degree of neutralization on the absorbency properties: The influence of neutralization degree on the absorbency properties such as centrifugal retention capacity (CRC) and absorbency under load (AUL) of AG-g-pIA are shown in Fig. 4. As the degree of neutralization increases from 60% to 70%, a sharp increase in AUL and CRC value is observed. As the ionization of carboxyl groups of itaconic acid monomers results in the strong electrostatic repulsion and due to this size of the crosslinked network and water absorption capacity of AG-g-pIA increases. When increase the neutralization degree of 70%, absorbency properties such as CRC and AUL tended to continuously decrease due to the negative charges on carboxylic acid screening effect which decrease the electrostatic and osmotic pressure and reduce the overall absorption capacity of superabsorbent composites polymers (AG-g-pIA) [47].

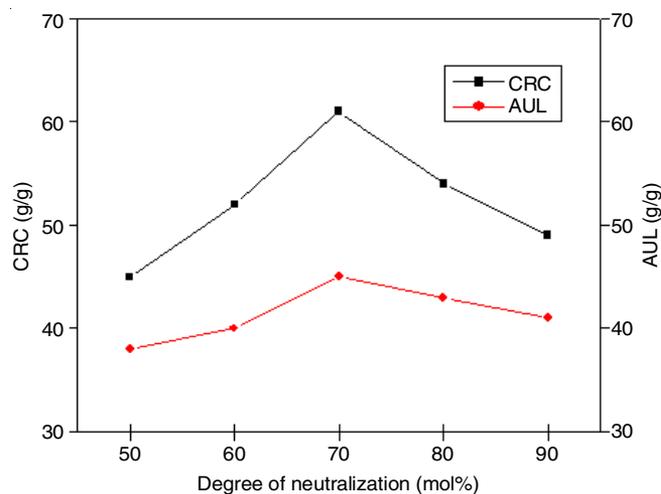


Fig. 4. Effect of neutralizing agent on the CRC and AUL

Effect of crosslinking agent content: In the formation of a 3D network the content of crosslinking agents plays an important role and has significant influence on the absorbency of polymer. After optimization the conditions such as amount of IA, AG and degree of neutralization to 60%, the effects of various amount of crosslinker N-MBA on the absorbency parameters such as CRC, AUL and gel fraction of AG-g-pIA are shown in Table-1. As the N-MBA concentration increased from 0.1 to 0.2 wt. %, the CRC also increased due to an increase in the gel fraction, as the initially insufficient and partially soluble network became more complete and resistant. But when the N-MBA amounts up to 0.4 wt. %, the crosslinking density and CRC decreases. On the other hand, the other absorbency parameter such as AUL was observed to increase uninterruptedly as the N-MBA amount increased from 0.1 to 0.4 wt. % due to the steady increase in the gel strength.

TABLE-1
CRC, AUL AND GEL FRACTION OF SAP ACCORDING TO THE CONTENT OF CROSSLINKING AGENT

N-MBA content (wt.%)	CRC (g/g)	AUL (g/g)	Gel fraction (%)
0.05	31.0	4.7	67.4
0.10	35.2	8.4	75.3
0.12	46.2	14.8	91.3
0.20	58.2	15.8	89.3
0.25	55.1	16.1	92.4
0.30	52.1	16.3	93.4
0.35	46.2	16.5	95.1
0.40	32.1	16.9	96.2

Comparative studies: Previous reports relied on the synthetic polymers, which are less biodegradable and environmentally sustainable. They often lacked optimization of filler content, resulting in the sub-optimal performance. Additionally, there was a weak correlation between polymer-filler interactions and functional properties, with minimal focus on integrating renewable, eco-friendly materials (Table-2). Thus, this study utilizes acacia gum, a natural polysaccharide, to improve biodegradability and sustainability, aligning with green chemistry principles. Optimized kaolin content enhances water absorption

TABLE-2
LIMITATIONS OF PREVIOUS WORK AND ADVANTAGES OF THE CURRENT STUDY

Feature/property	Previous studies on SAPs with inorganic fillers (<i>e.g.</i> , bentonite, montmorillonite, silica, kaolin) [Ref. 48]	Present study (Acacia gum-itaconic acid-kaolin SAPs)
Inorganic filler used	Kaolin, Bentonite, Montmorillonite, Silica	Kaolin
Polymer matrix	Synthetic polymers (<i>e.g.</i> , acrylic acid, polyvinyl alcohol), sometimes blended with natural polymers	Natural polysaccharide (Acacia gum) grafted with itaconic acid
Filler concentration range	Typically fixed or low concentration (<i>e.g.</i> , <5%)	Optimized range (0–10%), showing a significant improvement in water absorption and retention
Swelling behaviour	Limited improvement due to high crosslinking density or poor filler dispersion	Enhanced swelling due to reduced crosslinking density and optimized hydrogen bonding interactions
Mechanical strength	Variable, often dependent on polymer type and filler dispersion	Improved mechanical strength due to uniform distribution of kaolin in the polymer matrix
Thermal stability	Reported improvement, but less emphasis on biodegradable matrices	Enhanced thermal stability while maintaining biocompatibility through the natural Acacia gum-based matrix
Biodegradability	Limited biodegradability, especially for synthetic polymer-based matrices	High biodegradability due to the use of natural polysaccharide (Acacia gum)
FTIR analysis	Broad focus on polymer-filler linkages but limited emphasis on the effects of these linkages on swelling behaviour	Clear identification of carboxyl-kaolin interactions and their direct impact on swelling capacity
Environmental considerations	Focus on inorganic fillers but less integration of natural, renewable polymer matrices	Emphasis on green chemistry principles by combining a natural polymer with an inorganic filler
Applications	General SAP applications, <i>e.g.</i> , agriculture, hygiene products	Targeted applications with potential for high water retention in agriculture, waste management and soil conditioning
Comparative novelty	Often uses synthetic polymers or natural polymers without significant filler optimization	Innovative integration of kaolin and a natural polysaccharide (Acacia gum), highlighting filler-content optimization

and retention, while reduced crosslinking density further improves performance. The uniform dispersion of kaolin boosts thermal and mechanical properties. Moreover, the work emphasizes practical applications in agriculture and waste management with scalability potential for real-world use.

Conclusion

To prepare superabsorbent polymer composite (AG-*g*-pIA), itaconic acid (IA) was introduced *via* graft polymerization in the presence of inorganic kaolin powder in order to enhance the low absorption properties of natural polymer acacia gum (AG). The chemical composition of superabsorbent (AG-*g*-pIA) was characterized by FT-IR spectroscopy. The effect of various parameters which played a major role to increase the absorption properties and effect of ammonium persulfate (APS) and *N,N'*-methylenebisacrylamide (N-MBA) concentration were also examined. As the concentration of NaCl solution increased, all superabsorbent samples demonstrated a reduction in free absorption capacity due to the screening effect of sodium cations and a decrease in osmotic pressure. The superabsorbent polymer (AG-*g*-pIA) increased the value of centrifuge retention capacity (CRC) about 48%, compared to other superabsorbent polymer without presence of IA. Neutralization of monomer contributed to the expansion of superabsorbent network *via* the formation of anions. The highest centrifugal residual capacity and absorbency under load values of 55.1 and 16.9 g/g were obtained at 60% neutralization, respectively.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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