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RESEARCH PAPER

Green Synthesis and Characterization of Guar Gum/ Polyacrylamide/Activated Carbon Hydrogel for Efficient Methylene Blue Removal

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ABSTRACT

An innovative approach was developed for removing highly potent dyes, such as methylene blue (MB), from simulated solutions under various application conditions. The sorbent material was green hydrogels based on guar gum/polyacrylamide (GG/PAm) fabricated with activated carbon (GG/PAAm/AC). The crosslinker N, N'-methylene bisacrylamide (MBA) and the initiator potassium persulfate (KPS) were used in minimal amounts to adhere to green chemistry principles. The synthesis was performed under mild conditions, and the polymerisation was carried out at 60 °C, indicating a lower energy requirement. Different combinations were tried, varying the concentrations of GG (0.12–0.2 g), PAm (0.3–3 g), AC (0.01-0.1g) , KPS (0.01–0.1 g), and MBA (0.02–0.12 g). Of the ratios investigated, the best configuration of hydrogels was 0.15 g GG, 2.0 g , PAM, 0.08 g AC , 0.05 g KPS, and 0.08 g MBA, which generated a hydrogel with increased water-holding and hardness, and a higher adsorptive rate. The effect of AC on the crystallographic and thermal properties of the hydrogel composites was verified via the XRD and TGA analyses, respectively. The reactivation efficiency of the prepared composite surface initially reached 98% after the first regeneration cycle and decreased to 81% by the fifth cycle. This gradual decline indicates partial loss of active sites over successive uses. However, the presence of active carbon (AC) played a critical and effective role in maintaining regeneration performance by providing a stable, high-surface-area network that supports dye desorption and functional group recovery. The reusability investigation demonstrated that the sorbents were highly effective in removing significant amounts of pollutants after four adsorption–desorption cycles.

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INTRODUCTION

Freshwater scarcity, a pressing global issue, is addressed through green solutions in green chemistry. Several options for recovering and reusing existing resources have been proposed. Pollution of water by various contaminants appears to be one of the significant threats to the globe, as water serves as a vital source of life for all living organisms on Earth. These contaminants may be inorganic, organic, or biological in nature. Some, such as biological pollutants and certain dyes, can be broken down by bacteria, while others are resistant to this process. Heavy metals are among the most hazardous types of contaminants, as they persist up the food chain, leading to severe health problems and a significant mutagenic impact. Additionally, the decomposition of dye derivatives kills microorganisms and algae in aquatic organisms by blocking sunlight and altering the genetic information of various cellular systems. Thus, the removal of these pollutants is one of the most promising solutions and has aroused considerable attention [1, 2]. Dye molecules consist of two key components: the chromophores, which are primarily responsible for producing the color, and the auxochromes, which not only supplement the chromophore but also render the molecule soluble in water and enhance its affinity (to attach) toward the fibers [3]. Due to their toxic effects, dyes have generated much concern regarding their use. It has been known to cause mutagenesis, chromosomal fractures, carcinogenesis, and respiratory toxicity. Therefore, focusing on specific methods and technologies to remove dyes from various types of wastewater streams is desirable [4-9]. In fact, multiple materials derived from agricultural waste and natural resources have been utilised in this field. Among the various methodologies applied to water purification, adsorption has proven to be highly effective due to its high efficiency, low

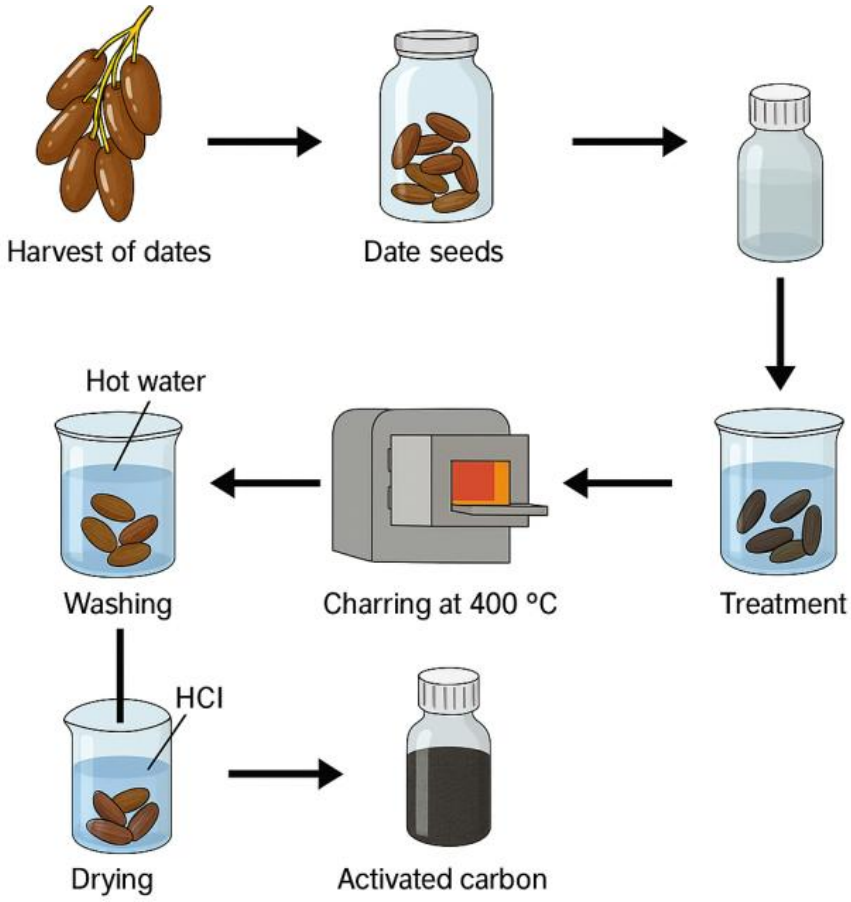

Fig. 1. Preparation of Date Pits as activated carbon.

waste generation, versatility, and reusability. Over the past two decades, hydrogels have emerged as promising candidates for water treatment. The most important of these are those prepared from or based on natural materials, such as guar gum, chitosan, pectin, cellulose, and starch [10, 11].

Hydrogels have several advantages, including high hydrophilicity, biocompatibility, and a three-dimensional porous structure that closely resembles the extracellular matrix, making them one of the most competitive materials for wound dressings. Compared to traditional wound dressings (such as gauze, bandages, and cotton), hydrogel dressings can be easily modified to meet the diverse demands during various stages of wound repair. Gellan gum, an exopolysaccharide derived from non-pathogenic bacteria of the Sphingomonas group, can be easily converted into hydrogels through gentle heating and cooling. Recently, gellan gum has received extensive attention in the biomedical field, particularly in soft tissue engineering, due to its structural similarity to the extracellular matrix of soft tissues. These excellent properties make gellan gum an ideal scaffold for loading PDA particles, and its porous structure can maintain good dispersion and controlled release of heat energy [12-14].

Hydrogels have numerous applications due to their structural adaptability, high water content, and biocompatibility. These have been widely applied in biomedical applications such as drug delivery systems, wound dressings, tissue engineering scaffolds, and contact lenses. Regarding environmental engineering, hydrogels have been proved as efficient adsorbents for the removal of heavy metals, dyes and other pollutants from wastewater. Additionally, they are employed in agriculture as soil conditioners to maintain water content and can be combined for their stimulus-responsive properties in actuators and sensors. Advances in hydrogel technology, such as nanocomposite hydrogels and stimuli-responsive ("smart") hydrogels, have enhanced their applicability and created new opportunities

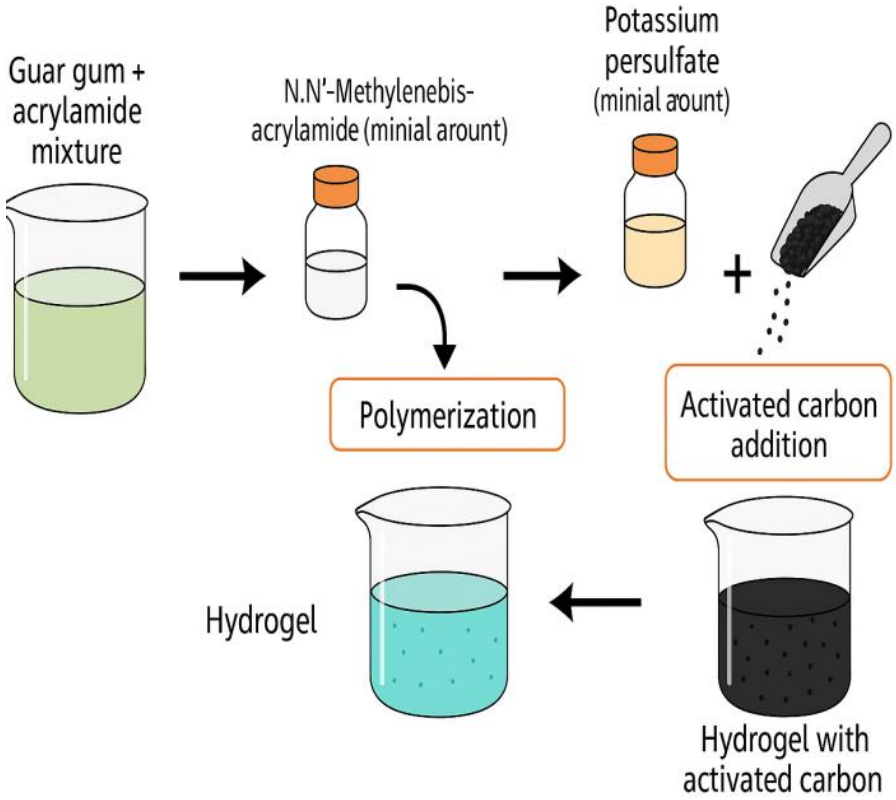

Fig. 2. Preparation of GG/PAAm/AC Hydrogel Composite.

in modern applications [15-18].

MATERIALS AND METHODS

Activation of Date Pits

Activated carbon was produced from date pits collected from Iraqi farms. The pits were initially hand-separated from the date palm fruits and thoroughly washed with hot distilled water to eliminate any residual sugars and adhering impurities, followed by drying in the open air at room temperature for 24 h. The dried pits were carbonized by thermal. They were carbonised in a furnace at 400°C for two hours under a reduced oxygen environment to produce biochar. The carbonised product was ground and sieved to a uniform particle size after cooling. The biochar obtained was subsequently chemically activated. It was immersed in a 0.1 M hydrochloric acid (HCl) solution and mixed at room temperature for 2 h to remove inorganic impurities and increase the porosity. The mixture was filtered and washed a few times with hot distilled water until a neutral solution was obtained. The acid-treated carbon was eventually oven-dried at 105°C for 12 h, cooled in a desiccator and packed in clean, air-tight glass containers for subsequent use in adsorption studies. As shown in Fig. 1.

Preparation of GG/PAAm/AC Hydrogel Composite

The GG/PAAm/AC hydrogel was prepared using a free radical polymerization process at ambient condition. In the beginning, 0.1 g of guar gum (GG) was dissolved in deionized water of 20 mL with continuous magnetic agitation at 60°C until a homogeneous viscous solution (Solution A) was achieved. PAm monomer (1.2.0 g) was dissolved in 10 mL of de-ionized water in another beaker. 0.05 g of a radical initiator, potassium persulfate (KPS), was then added, and 0.08 g N, N'-methylene bisacrylamide (MBA) was used as a crosslinking agent (Solution B). The solution was then mixed at room temperature for 30 minutes to obtain a homogeneous dispersion. Then a certain quantity of activated carbon (AC) was added to Solution B and homogenised with mechanical agitation and ultrasonication (where necessary) for a good distribution. Solution A was followed by Solution B with continuous stirring, and the mixture was then cast into clean Petri dishes or moulds for polymerisation. The polymerisation was conducted in a hot-air oven for 1.5 h at 60°C. Hydrogel product was collected, rinsed with abundant deionized water to wash away unreacted monomers and soluble impurities and then placed in hydrated or dehydrated condition before the intended analysis· As shown in Fig. 2

RESULTS AND DISCUSSION

Scanning electron microscopy SEM

The SEM micrograph (Fig. 3a) displays the surface morphology of the activated carbon-loaded hydrogel before the adsorption of MB dye. At a magnification of 50,000×, the surface appears rough and highly porous, characterized by an interconnected network of irregularly shaped cavities and ridges. These features are indicative

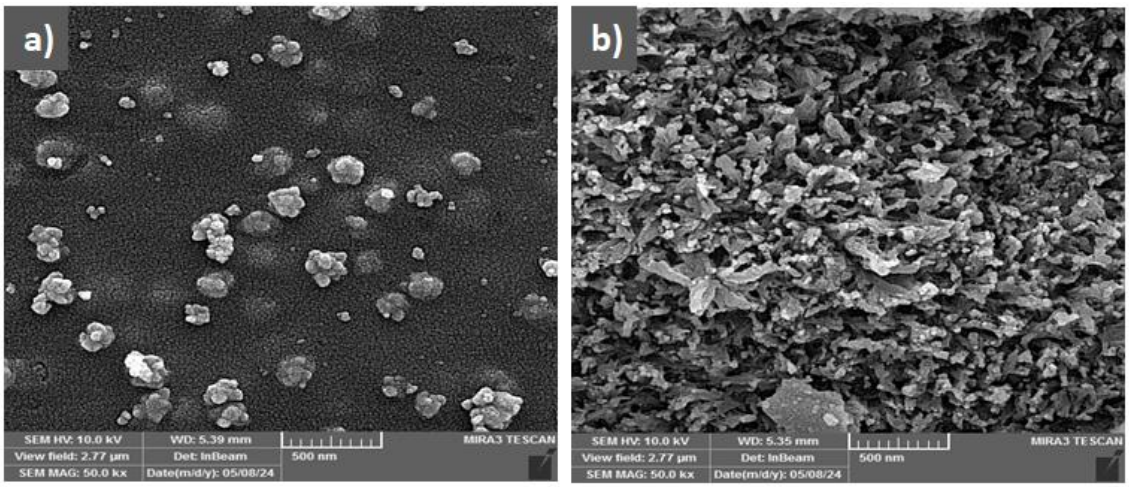

Fig. 3. FESEM image a) Hydrogel, b) hydrogel after adsorption of MB dye.

of a well-developed three-dimensional hydrogel structure. The dispersion of activated carbon particles is evident throughout the polymer matrix, contributing to the heterogeneous texture and enhancing the overall surface area available for adsorption. The porous architecture and the presence of micro- and mesopores are critical for facilitating rapid dye diffusion and interaction with active adsorption sites. The morphology suggests that the hydrogel composite has a high potential for efficient adsorption due to its increased surface roughness, pore density, and the synergistic effect of the incorporated activated carbon [19-21].

The SEM photomicrograph (Fig. 3b) presents a surface morphological view of the activated carbon laden hydrogel after the adsorption of methylene blue (MB) dye. The surface is significantly altered, as an abundance of spherical compounds and aggregates have formed within the hydrogel matrix compared to the pre-adsorption image. These surface deposits may be attributed to the adsorbed MB dye molecules in clusters, representing the available active site with which the successful interaction is maintained. The coarse and rough structure of the hydrogel surface, observed before treatment, is now compacted and partly blocked (resulting from the occupation of inner pores and surface functional groups by the dye). The reduction in apparent porosity was recorded and the enhancement of apparent particles deposed firmly on the surface was observed, which also reflect efficient adsorption. These morphological transformations also demonstrate the strong interaction of the hydrogel with the cationic dye molecules and thus may have potential applications in wastewater treatment technologies.

This TEM image in Fig. 4 of the hydrogel reveals its internal nanostructure, showcasing a well-defined, nearly spherical or slightly cuboidal nanoparticle embedded within the matrix. The particle size is approximately 100, and 200 nm, indicating nanoscale features consistent with a nanocomposite system. The relatively uniform contrast suggests a homogeneous distribution and incorporation of nanoparticles or dense regions within the hydrogel network. This morphology supports the presence of a compact internal structure, potentially contributing to enhanced mechanical stability and efficient interaction with target molecules during adsorption processes [22].

The TGA curve of the GG/PAAm/AC hydrogel exhibits a three-step weight loss trend. The first slight weight loss under 150°C is assigned to the evaporation of adsorbed moisture. A second, more stable plateau, valid up to approximately 300°C, is evidence of the stability of the polymer backbone. A significant weight loss in the range of 300–500°C is due to the thermal degradation of polymer chains, GG and polyacrylamide. Above 500°C, little

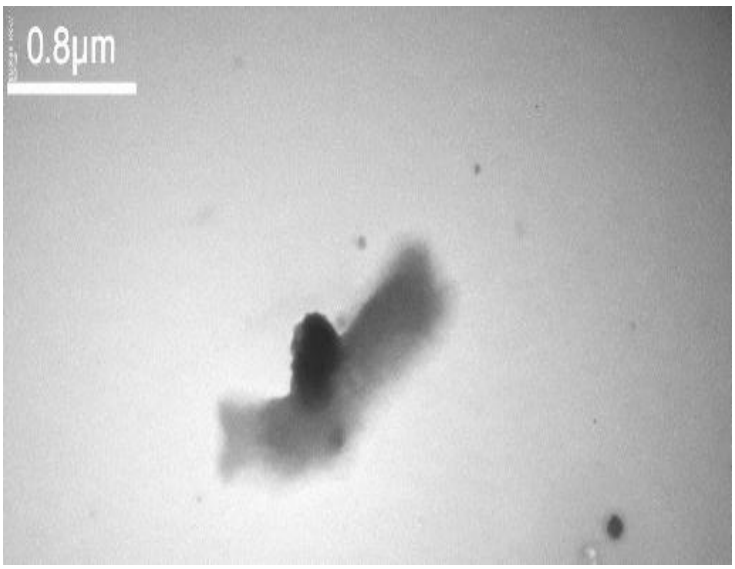

Fig. 4. TEM image of hydrogel composite.

residue is left, indicating that the organic material is sufficiently decomposed. These results suggest that the hydrogel exhibits good thermal stability, which is suitable for moderate-temperature work, including wastewater treatment. As shown in Fig. 5 a [5, 6, 23].

The XRD pattern of the hydrogel (Fig. 5 b) displays a broad and low-intensity diffraction peak located between 20° and 25°, which is assigned to an amorphous or semi-crystalline structure. Such a broad hump indicates the existence of randomly oriented polymer chains with short-range order, which is typical in natural polymer-derived or synthetic hydrogels. No sharp and

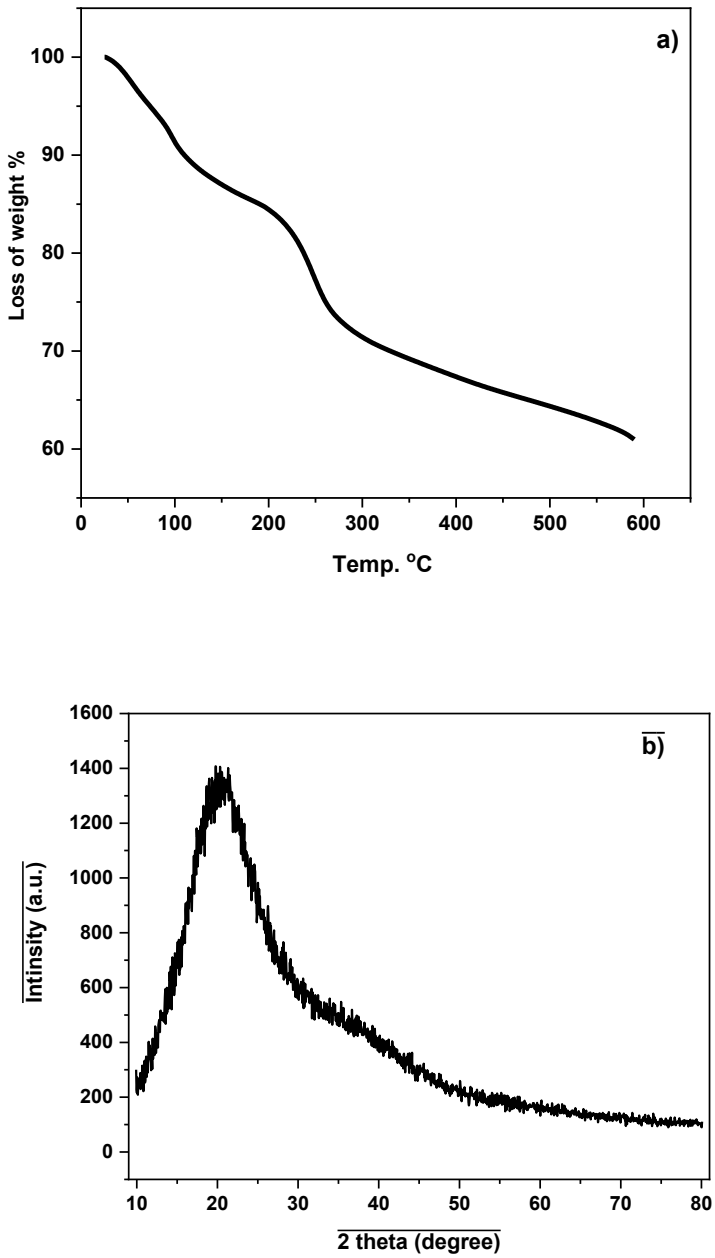

Fig. 5. a) Thermogravimetric Analysis TGA curve of the GG/PAAm/AC hydrogel, b) XRD of GG/PAAm/AC hydrogel nanocomposite.

well-defined diffraction peaks were observed, indicating that the hydrogel lacks a crystalline phase and is predominantly amorphous. Such structural disorder is common in the case of free-radical polymerisation-derived gels, particularly when involving natural biopolymers (e.g., guar gum). Minor and sharp peaks can be observed for composite hydrogels with AC . But if there are no peaks seen here, it must be that there is low filler present or that it is distributed well within the hydrogel. The amorphous characteristic is favorable for the swelling ability as well as dye adsorption due to its flexibility, pore accessibility, and functional group exposure.

Optimization of the swelling behavior for the synthesis of hydrogel

Effect of Acrylamide Monomer Content on Swelling Ratio

The monomer plays a dual role: it interacts with the cross-linker and initiator to form the hydrogel's 3D network and introduces hydrophilic groups that enhance water absorption. Varying the acrylamide (AM) content from 0.3 g to 2.0 g revealed its significant impact on swelling behavior. As the AM concentration increased, the swelling ratio peaked at 1500% with 1.2 g, indicating enhanced chain propagation and water uptake (Fig. 6). However, further increases led to a decline, with a swelling ratio of 1100% at 1.5 g.

This reduction is attributed to diminished electrostatic repulsion and increased solution viscosity, which restrict free radical mobility. Additionally, homopolymer formation and self-crosslinking at higher AM concentrations limit network flexibility. Hydrogen bonding among hydroxyl groups may also contribute to network contraction, further reducing swelling capacity [24, 25]

Effect of Guar Gum on Swelling Ratio

Fig. 7 illustrates the effect of varying guar gum (GG) concentrations (0.2–1.5 g) on the swelling ratio of the hydrogel. The maximum swelling ratio (1500%) occurred at 1.0 g GG, likely due to electrostatic repulsion among negatively charged (COO^-) groups and lone electron pairs on oxygen atoms. Beyond this concentration, increased GG leads to more free radicals and active sites, promoting crosslinking and polymer chain entanglement, which limits water absorption and reduces the swelling ratio. Excessive GG also increases solution viscosity, hindering macromolecular mobility and reducing recombination efficiency. This results in a more heterogeneous structure and diminished swelling

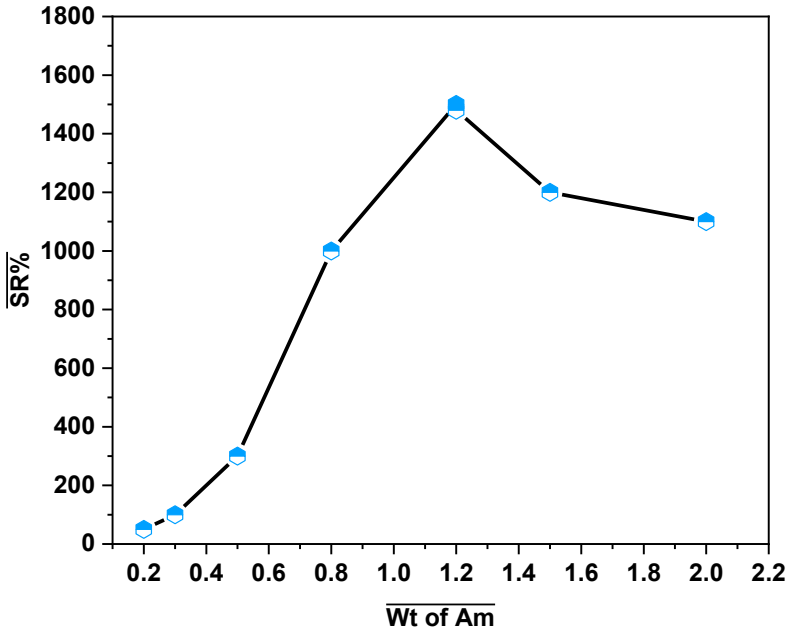

Fig. 6. Effect of acrylamide on the Swelling Ratio.

and adsorbate loading capacity [26, 27].

Effect of Activated Carbon Content on Swelling Ratio

The incorporation of activated carbon (AC) into the hydrogel significantly influences its swelling properties. At low AC content, the swelling ratio was relatively low, likely due to insufficient porosity and limited interaction between the hydrogel matrix and water molecules. As the AC content increased, the swelling ratio improved, reaching a maximum at 0.08 g as shown in Fig. 8. This enhancement can be attributed to the porous nature of AC, which increases water uptake and creates additional pathways for fluid diffusion within the hydrogel network [28, 29].

However, when the AC content exceeded 0.08 g, the swelling ratio began to decline. This reduction

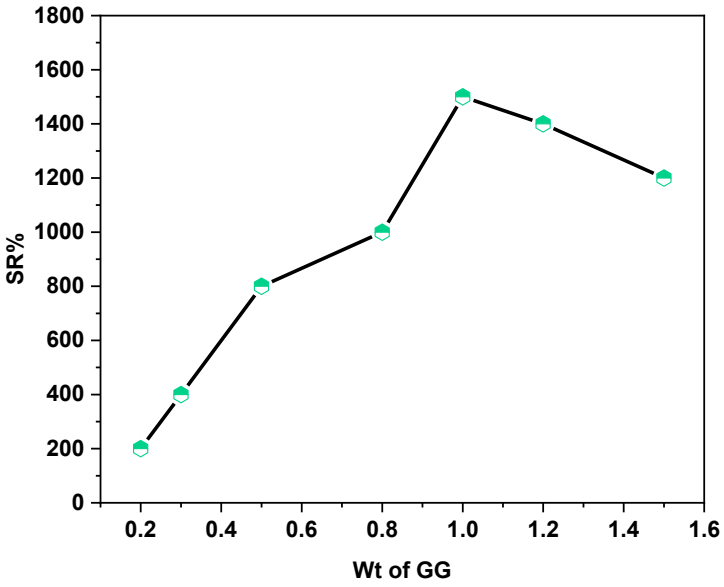

Fig. 7. Effect of the GG on the Swelling Ratio.

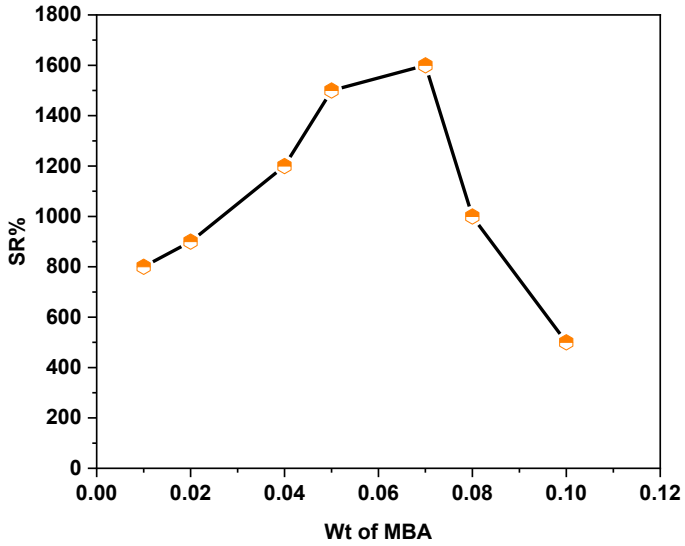

Fig. 8. Effect of Activated Carbon Content on Swelling ratio.

is likely due to excessive AC loading, which may lead to aggregation, reduced flexibility of the polymer chains, and obstruction of the hydrogel network. Such effects limit the hydrogel's ability to expand and retain water, resulting in decreased swelling performance at higher AC concentrations [28, 30].

Effect of Crosslinker Content on Swelling Ratio

The crosslinker is essential for forming a stable, insoluble hydrogel network by reinforcing structural integrity and controlling crosslink density, which directly influences fluid absorption. In this study, N, N'-methylenebisacrylamide (MBA) was used in varying amounts (0.02–0.09)

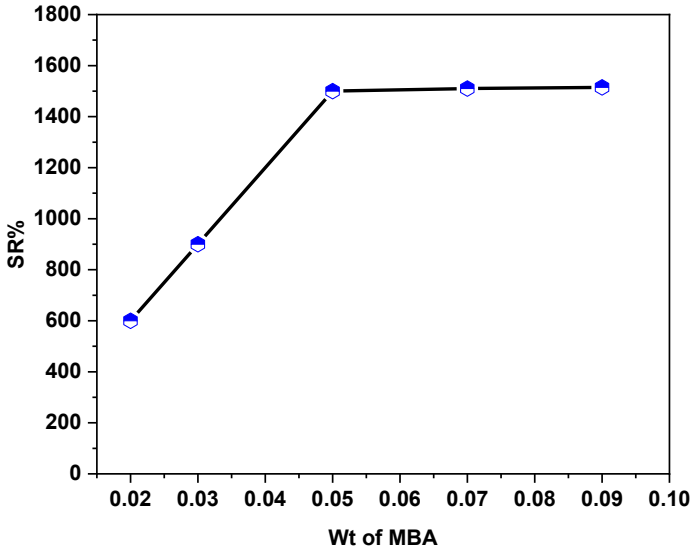

Fig. 9. Effect of cross-linker on the swelling behavior.

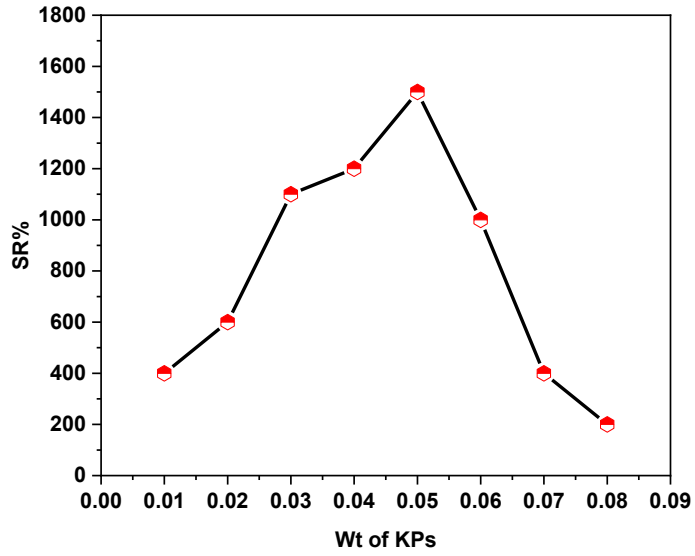

Fig. 10. Effect of the initiator on the swelling ratio.

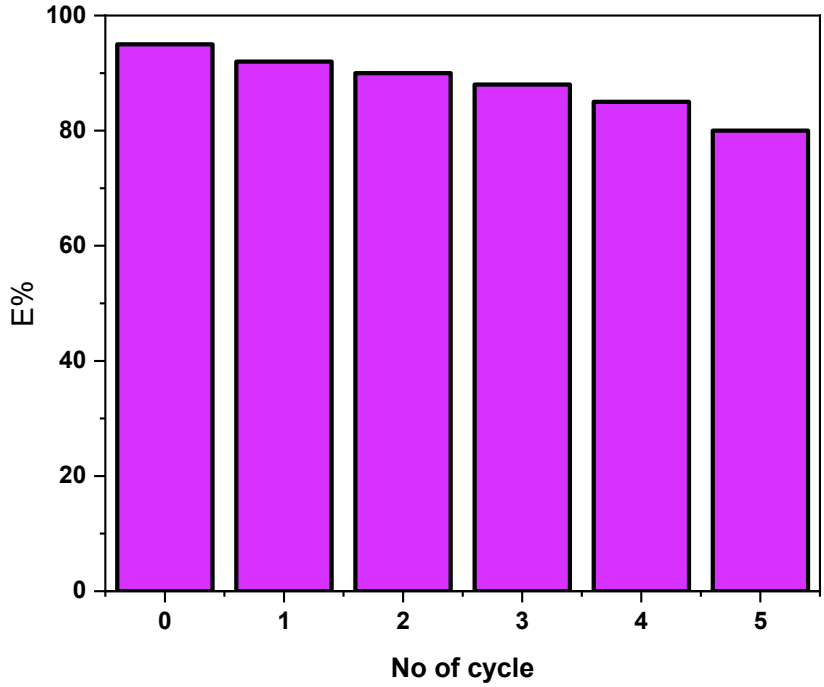

Fig. 11. Reusability Performance of the GG/PAAm/AC Hydrogel. experimental conditions (0.05 gm of the surface with 100 mg/L of the dye under condition 25 °C, pH 7 and equilibrium time 60 mins.).

g, dissolved in 5 mL distilled water. to evaluate its effect on the hydrogel's swelling behavior (Fig. 9). The highest swelling ratio (1500%) was observed at a dosage of 0.05 g MBA. At lower concentrations, insufficient crosslinking limited the network's ability to trap dye molecules, while higher concentrations led to excessive crosslinking, reducing pore size and water uptake capacity, Fig. 9 [31, 32].

Effect of Initiator Concentration on Swelling Ratio

To investigate the impact of initiator concentration, varying amounts of potassium persulfate (KPS) were tested, ranging from 0.01 to 0.08 g (Fig. 10). The swelling ratio peaked at 1500% with 0.05 g of KPS, attributed to optimal generation of free radicals that initiate effective grafting onto the κ-carrageenan backbone and enhance network expansion via interaction with sulfate radicals. However, at concentrations above 0.08 g, the swelling ratio declined. This reduction is likely due to excessive radical generation, leading to increased bimolecular termination, shorter polymer chains, and structural irregularities, as explained by Flory's theory. Higher initiator levels may also favour homopolymerization over grafting and decrease polymer molecular weight, resulting in reduced water absorbency [33, 34].

Regeneration Efficiency of the Hydrogel

Fig. 11 presents the regeneration efficiency (E%) of the GG/PAAm/AC hydrogel over five adsorption–desorption cycles for methylene blue (MB) dye, experimental conditions (0.05 gm of the surface with 100 mg/L of the dye under condition 25 °C, pH 7 and equilibrium time 60 mins.). The initial adsorption efficiency exceeded 85%, indicating a strong affinity toward MB. However, a gradual decline was observed with each cycle, reaching approximately 50% by the fifth cycle. This decrease is likely due to partial saturation of active sites, structural degradation, or irreversible dye adsorption during repeated use. Nevertheless, the hydrogel maintained a substantial portion of its adsorption capacity, demonstrating good reusability and structural stability, which supports its potential for practical wastewater treatment applications [35, 36].

CONCLUSION

In this study, a novel and environmentally friendly hydrogel based on gaum Gum (GG) grafted with polyacrylamide (PAM) was successfully synthesized via free radical polymerization.The synthesis process utilized biodegradable and low-toxicity components, demonstrating a green chemistry approach. By optimizing the monomer and cross-linker ratios, a hydrogel with excellent mechanical integrity, high porosity, and adequate swelling capacity was obtained. Hydrogels blended with activated carbon were employed in the removal of MB dye pollutant from simulated solutions under various application conditions. The reusability investigation proved that the active carbon supported hydrogels can be repeatedly applied four times with comparable quantities of pollutant removed. The obtained findings depict that the prepared materials can be used as effective candidates in wastewater remediation with repeated applicability. Its eco-friendly nature, ease of preparation, and effective dye removal performance support its potential application in sustainable wastewater treatment technologies.

CONFLICT OF INTEREST

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

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