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ZnO-Nanoparticle-Embedded Hydrogel Nanocomposite: A Sustainable Adsorbent for Malachite Green Dye Removal

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ABSTRACT

Hydrogels are hydrophilic, cross-linked, three-dimensional polymeric networks that are insoluble and feature high porosity and excellent water retention capabilities. In this work, a hydrogel (sodium alginate/acrylic acid (SA/AC)) and its nanocomposite (SA/AC/ZnO-NPs) were prepared successfully using a co-polymerisation method upon embedment of zinc oxide nanoparticles (ZnO-NPs) with an acrylic acid matrix SEM, and TEM thoroughly characterised the prepared hydrogel/nanocomposite. The adsorption capacity of these sorbents was evaluated in a batch system for the removal of malachite green (MG) dye under various operational conditions, including pH, contact time, and adsorbent dose. The removal efficiency of SA/AC hydrogel increased with the increase in the adsorbent dose, from 76.04% to 93.81%, along with the decrease in q value (777-25.94 mg/g), whereas the SA/AC/ZnO-NPs hydrogel nanocomposite exhibited the removal efficiency of 94.36-98.31%, and the qe value declined from 988.00 to 99.03 mg/g .The adsorption capacity of SA/AC hydrogel increased to 67.05% after 5 minutes of adsorption and to 90% after 40 minutes of adsorption. For the SA/AC/ZnO-NPs nanocomposite, the content increased from 90% to 98% within the same time duration .The higher adsorption efficiency can be attributed to the presence of –COOH groups, which provide strong hydrogen bonding and electrostatic interactions with the cationic dye molecules of MG. The results indicate that the SA/AC hydrogel and SA/AC/ZnO-NPs nanocomposite could be suitable, environmentally friendly adsorbents for removing dyes from wastewater. Nevertheless, the apparent pH-sensitive nature of sodium alginate and ZnONPs can complicate the performance of such materials, especially in the practical world, where precise pH control may not be feasible.

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INTRODUCTION

Hydrogels are three-dimensional, hydrophilic, and cross-linked polymeric networks characterized by their high porosity, structural integrity, and insolubility in water. These materials possess a large surface area and are rich in functional groups, enabling them to absorb and retain significant quantities of water or biological fluids without

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undergoing structural degradation [1, 2]. Due to their tunable chemical composition and sensitivity to external stimuli such as pH, temperature, light, and electric fields, hydrogels have gained widespread attention for diverse applications, including agriculture, enzyme immobilization, drug and cell delivery systems, and environmental remediation as effective adsorbents [3, 4].

The rapid expansion of industries such as textile, paper, printing, and leather processing has led to the excessive use of synthetic dyes and the consequent release of untreated dye-containing effluents into aquatic ecosystems . Among these dyes, malachite green (MG), a cationic triphenylmethane dye with the molecular formula $C_{52}H_{54}N_4O_{12}$, is widely used for coloring materials like paper, wool, nylon, and leather [5]. MG appears as a bright green crystalline solid and has been under scrutiny due to its high toxicity, carcinogenicity, mutagenicity, and non-biodegradability.Despite being strictly regulated and not approved for human consumption, there is no universally established permissible limit for MG in drinking water. However, under directive 2002/657/EC, a minimum required performance limit (MRPL) of 2 µg/kg was set for its concentration in aquaculture products. MG exposure is known to cause severe health complications, affecting the liver, kidneys, heart, spleen, skin, and eyes [6-9] . These factors underscore the urgent need for efficient and sustainable methods to eliminate MG from aqueous environments.A wide range of physical, chemical, and biological processes have been employed for the extraction of pollutants from aqueous systems, including but not limited to UV radiation treatment, phytoremediation, membrane filtration, chemical oxidation, reverse osmosis, and adsorption .Of these methods, adsorption has become the most commonly used one because of its simplicity of use, low cost, ease of design, and wide range of applications[10-13].

Various adsorbents such as silica, activated alumina, ion exchange resins, zeolites, biochar, charcoal and polysaccharide-based materials have been used for wastewater treatment. Sodium alginate (SA) is one of these types of polysaccharides, a biodegradable, nontoxic, pH-responsive anionic biopolymer obtained from the cell wall of brown algae. Due to its natural source and environmental soundness, SA has received considerable attention in adsorption applications [14, 15]. Nonetheless, its adsorption performance can be greatly improved by doping with functional additives, which alter its structure and physicochemical properties. The frequently used additives for improving the properties of SA-based materials are: activated carbo, graphite, MXenes, TiO_2, montmorillonite, halloysite, zeolites, rice husk, metal-organic frameworks (MOFS), natural and sintetic polymers,and most importantly, silica. Silica is an amorphous inorganic polymer with thermal, chemical and mechanical stability and its relatively low cost, abundance and environmental friendliness make it a good candidate to be used as reinforcing agent. Dispersion of silica into SA hydrogels results in increase of porosity and introduction of new active binding sites, which is beneficial for the enhancement of overall adsorption efficiency [16-19].

From the series of metal oxide NPs, ZnO-NPs are known for their excellent adsorption efficiency for synthetic dyes in aqueous media, owing to their large surface area, high reactivity, and photocatalytic characteristics. Despite the promising results of ZnO-NP-based hydrogels, the literature reports in this field are few and scattered, when compared with other nano-composites. More recently, hydrogels in the form of agar/graphene oxide/ZnO nanocomposites have been found to remove 88% and 91% of methylene blue and methyl orange from industrial effluents, respectively, in reported studies. These results emphasise the potential of ZnO-NP incorporated hydrogels in dye uptake, and the need for further studies to develop ZnO-NP-integrated hydrogels as an efficient, sustainable, and environmentally friendly adsorbent for wastewater treatment applications[20, 21].

MATERIALS AND METHODS

Synthesis of SA/AC/ZnO-NPs Hydrogel Nanocomposite via Copolymerization Method

The SA/AC/ZnO-NPs hybrid hydrogel nanocomposite was synthesized using a copolymerization technique, as detailed below: Step 1: Preparation of Sodium Alginate Solution (Solution A) .A 10% (w/v) sodium alginate (SA) solution was prepared by dissolving 2.0 g of sodium alginate in 20 mL of deionized water. The solution was continuously stirred at 40 °C using a magnetic stirrer until a uniform and homogenous mixture was obtained. This solution was designated as Solution A.Step 2: Preparation of Acrylic Acid Precursor Solution (Solution B).In a separate beaker, 10 mL of acrylic acid was taken, followed by the addition of 3 mL of deionised water (to initiate pre-hydrolysis) and 3 mL of 0.1 N nitric acid (HNO_3), which served as a catalyst. This mixture, referred to as Solution B, was stirred for 1 hour using a magnetic stirrer to ensure complete pre-polymerization and formation of a transparent solution. Step 3: Synthesis of SA/AC Hydrogel For the formulation of the SA/AC hydrogel, Solutions A and B were gradually combined under continuous magnetic stirring, allowing uniform mixing and copolymer formation .Step 4: Incorporation of ZnO Nanoparticles. To prepare the SA/AC/ZnO-NPs nanocomposite hydrogel, both Solutions A and B were first mixed as described above, followed by the addition of 0.25 g of ZnO nanoparticles (ZnO-NPs). The resulting suspension was stirred continuously for 1 hour to ensure uniform dispersion of ZnO-NPs within the polymeric matrix.Step 5: Gelation, Aging, and Drying. The resulting mixture was transferred to a clean container and placed in a laboratory oven at 60 °C for 1 hour to allow for gelation, cross-linking, aging, and initial drying of the hydrogel structure.The resulting SA/AC/ZnO-NPs hydrogel nanocomposite was collected and stored for subsequent characterization and adsorption experiments. As shown in Fig. 1.

RESULTS AND DISCUSSION

The surface morphology of as-prepared hydrogel (SA/AC/ZnO-NPs) before adsorption at 50,000× (FESEM) Fig. 2a. The image depicts the general homogeneous presence of small, round or granular particles within the hydrogel matrix. These characteristics exhibit evidence of highly separated ZnO nanoparticles (ZnO-NPs) embedded in the polymeric chain of sodium alginate (SA) and acrylic acid (AA). The closed packed, compact conformation and fine surface texture indicate rough and porous nature of the surface, one of the reasons behind enhanced dye adsorption, since larger available area and active binding sites are accessible to dye molecules. Furthermore, the negligible agglomeration of particles reflects that the ZnO-NPs are well dispersed in the hydrogel matrix, and this good compatibility is beneficial for their mechanical stability and reproducible adsorption activities. The nanoscale structure of the hydrogel (presence of elliptical features and densely packed domains) is also important for an effective interaction of dye molecules interaction (malachite green) in the following adsorption assay [22, 23].

The provided FESEM image shows the surface morphology of the SA/AC/ZnO-NPs hydrogel nanocomposite after the adsorption process, likely following interaction with malachite green (MG) dye, at a magnification of 50,000× Fig. 2b .Compared to the pre-adsorption image, the surface has become significantly more compact and aggregated, indicating the accumulation of dye molecules on and within the hydrogel matrix. The porous and nanoparticulate texture

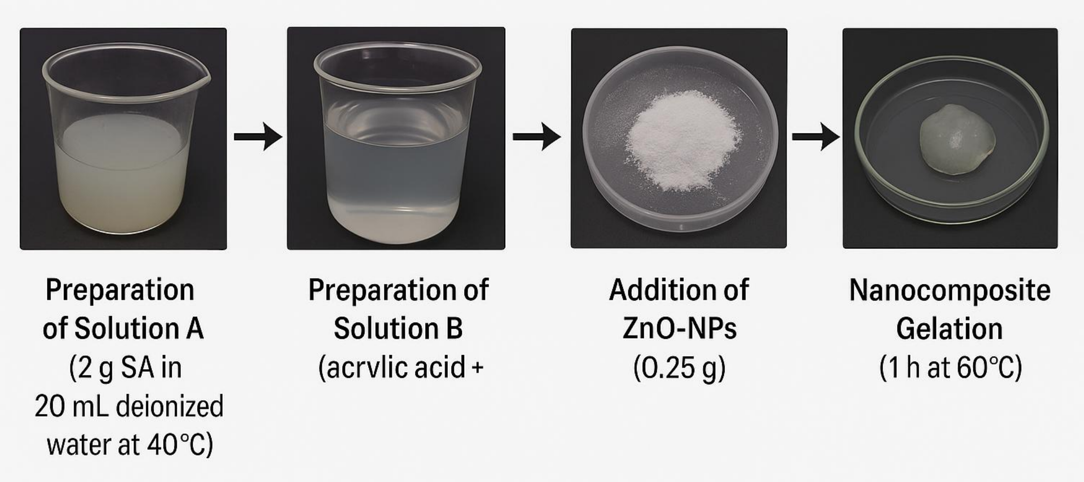

Fig. 1. Synthesis of SA/AC/ZnO-NPs Hydrogel Nanocomposite.

visible before adsorption appears now partially or fully occupied, which confirms successful dye uptake. The adsorbate (MG dye) likely filled available micro- and mesopores, reducing surface roughness and free volume. The post-adsorption morphology reveals larger, irregular flake-like structures, possibly resulting from intermolecular interactions and aggregation caused by dye binding. These structures may reflect the formation of dye–polymer complexes or hydrogen bonding/electrostatic interactions at the surface. The denser texture and reduced visible inter-particle space support the conclusion that the material has undergone structural densification, which is typical of successful adsorption behavior in hydrogel systems [17, 24, 25].

The TEM (Transmission Electron Microscope) image shows the nanostructure of the prepared SA/AC/ZnO-NPs hydrogel with a submicron scale (0.8 µm). Fig. 3 shows clear, elongated, rod-like nanoscales, most likely attributed to the ZnO nanoparticles embedded in the hydrogel matrix. These are structures that indicate the growth of anisotropic ZnO crystals, which are widespread in ZnO nanorods/nanowires[26, 27]. The rods are strongly separated from one another and stand out well against the background, indicating that ZnO-NPs are homogenously distributed in the hydrogel matrix. This shape helps in better and faster access of dye molecules to the coated Ni particles as a higher surface area is available. The lack of strong clustering or aggregation indicates that the synthesis method yielded adequate particle stability and distribution, which is

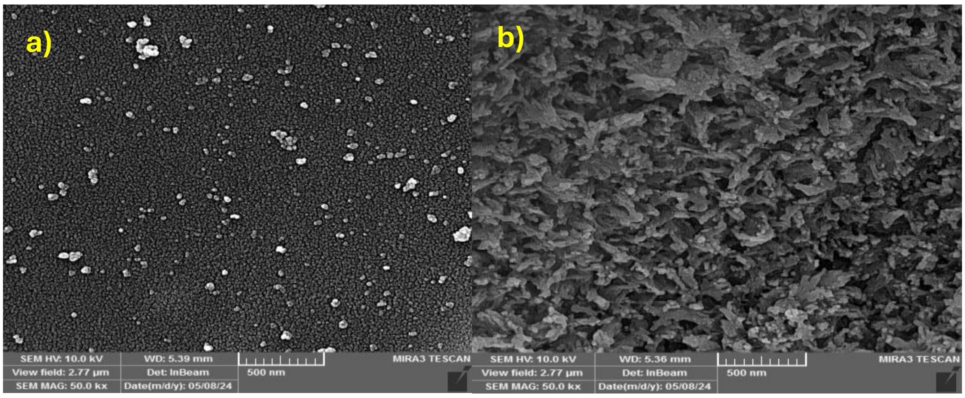

Fig. 2. FESEM image of a) hydrogel, b) hydrogel after adsorption.

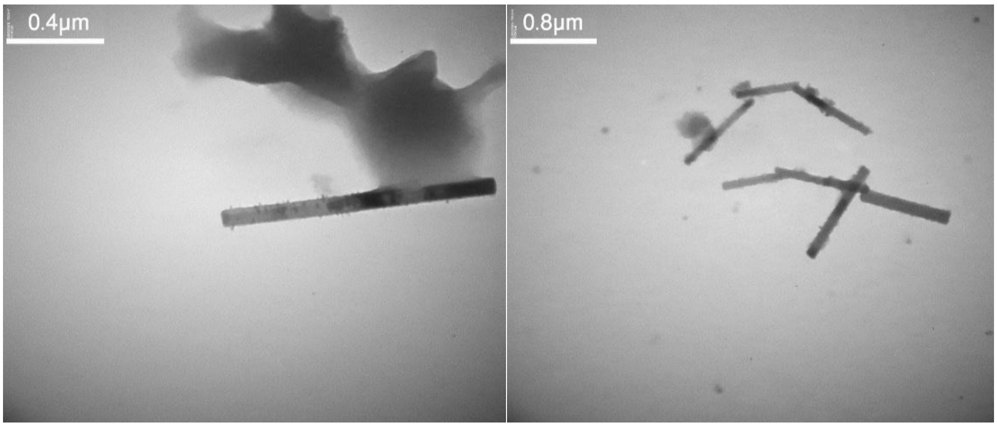

Fig. 3. Transmission Electron Microscope image of hydrogel.

essential for the even adsorption performance of the full hydrogel. The precise boundary between the ZnO rods and matrix (presumably which looks more arcade or more transparent) also discloses that the inorganic nanoparticles are successfully embedded in the framework of hydrogel without collapse or phase separation [28, 29].

Influence of Adsorbent Dose on Malachite Green Elimination

The effect of adsorbent dosage on the efficiency of malachite green (MG) removal was investigated in the range of 0.01 to 0.1 g of dry adsorbent, while all other parameters were held constant (Fig. 4). The removal efficiency of SA/AC hydrogel increased with the increase in the adsorbent dose, from 76.04% to 93.81%, along with the decrease in q value (777-25.94 mg/g), whereas the SA/AC/ZnO-NPs hydrogel nanocomposite exhibited the removal efficiency of 94.36-98.31%, and the qe value declined from 988.00 to 99.03 mg/g[30, 31]. The higher removal percentage for the higher adsorbent dose may be due to the higher available surface area and binding sites available, which can bind more dye molecules. But the decline of adsorption capability per unit mass may be attributed to the aggregation of the particles at high dosages, which would consequently decrease the effective surface area and block the active sites to the internal dye molecules. It is interesting to note that the introduction of ZnO nano-particles in the SA/AC matrix showed increased textural properties of the hydrogel, namely, the surface area and porosity. These improvements could lead to the better removal ability and adsorption capacity of the SA/AC/ZnO-NPs nanocomposite,

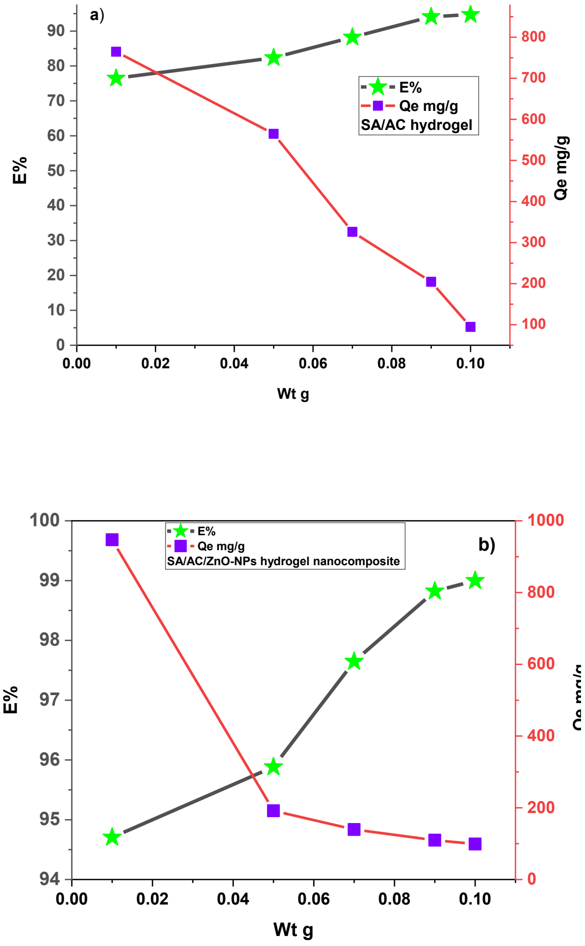

Fig. 4. Effect of a) SA/AC hydrogel, b) SA/AC/ZnO-NPs hydrogel nanocomposite on Malachite Green Elimination.

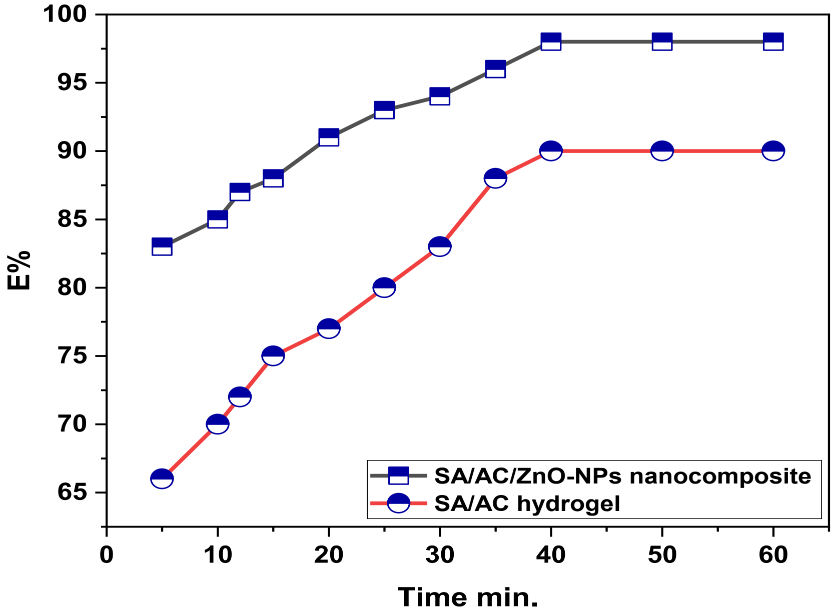

Fig. 5. Effect of contact time on the removal of MG dye.

which could be a potential adsorbent for adsorption of the dye when compared with the unmodified hydrogel [32, 33].

Influence of Contact Time on the Adsorption of Malachite Green

To achieve a better comprehension of the MG adsorption kinetics, the effect of contact time on the adsorption of MG on SA/AC hydrogel and SA/AC/ZnO-NPs hydrogel nanocomposite was also examined (Fig. 5). The adsorption capacity (qe) increased significantly with prolongation in contact time (5–60 min) and reached equilibrium after 40 min for both materials. The adsorption capacity of SA/AC hydrogel increased to 67.05 %after 5 minutes of adsorption and to 90% after 40 minutes of adsorption [3, 4, 34]. For the SA/AC/ZnO-NPs nanocomposite, the content increased from 90% to 98% within the same time duration. This extremely fast initial adsorption can be attributed to the abundance of active sites and large surface area, which lead to effective dye removal. The equilibrium required 35 minutes, and no significant increase in adsorption was noticed after that time, indicating that the active sites had been saturated with the dye molecules. The increased SA/AC/ZnO-NPs hydrogel nanocomposite removal capacity as compared to the pristine hydrogel is likely due to the interaction- and accessibility-enhancing nature of the ZnO nanoparticles, which results in increased surface area and porosity. According to these results, a contact time of 40 min was chosen as the optimal equilibrium time for all following adsorption experiments [35, 36].

CONCLUSION

This research relied on the development of a nanocomposite of SA/AC hydrogel and SA/AC/ZnO-NPs nanoparticles. Using a batch method, these gels were applied to evaluate their efficiency in the adsorption of MG dye from aqueous solution. An increase in MG removal efficiency ranging from 57.04 to 92.81 for SA/AC, and from 64.36 to 97.31 for SA/AC/ZnO-NPs was observed when using these adsorbents from 0.01 to 0.09 g. The data show that the addition of ZnO-NPs to the SA/AC hydrogel resulted in an increase in the surface area and porosity of the SA/AC/ZnO-NPs nanocomposite hydrogel compared to the SA/AC hydrogel. This FESEM image confirms the successful synthesis of a nanocomposite hydrogel with well-integrated ZnO nanoparticles. The observed surface morphology—marked by nanoscale uniformity, porosity, and structural integrity—provides a strong foundation for efficient dye adsorption, highlighting the material's suitability for environmental remediation applications. This TEM image confirms the successful incorporation of ZnO nanoparticles into the SA/AC hydrogel network, forming rod-shaped nanostructures with good dispersion. These morphological features are critical for the hydrogel's function as a high-performance adsorbent for dyes like malachite green, offering a large surface area, high stability, and reactive binding sites.

CONFLICT OF INTEREST

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

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