Photocatalytic Degradation of Organic Pollutants Using Graphene Quantum Dot-Loaded Sodium Alginate Hydrogels: Synthesis and Characterization

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Abstract

Rapid industrialization and population growth are placing increasing pressure on global clean water resources, resulting in a decline in water quality and associated environmental and health risks. Nanotechnology offers promising and cost-effective solutions for water remediation. Among nanomaterials, graphene quantum dots (GQDs) exhibit strong potential due to their chemical stability, high surface area, and photo luminescent properties. Embedding GQDs within hydrogels enhances their stability, dispersion, and photocatalytic performance. This study aimed to synthesize GQDs from organic waste—specifically waste tea leaves (WTL)—using an environmentally friendly microwave-assisted method. The synthesized GQDs were characterized using UV–Vis spectroscopy, dynamic light scattering (DLS), zeta potential, and scanning electron microscopy (SEM), revealing an absorption peak at 272 nm, particle sizes below 100 nm, high colloidal stability, and spherical morphology.

GQDs were embedded in sodium alginate (SA) hydrogels through ionic crosslinking. The resulting GQDs-loaded hydrogels were characterized by UV–Vis spectroscopy, swelling behavior tests, and visual inspection under UV light. Photocatalytic degradation tests against methylene blue (MB) demonstrated effective dye removal under both UV light (25.1%) and dark conditions (17.85%), confirming both photoreactive and passive adsorption capabilities. These results highlight the synergistic effect of GQDs and SA hydrogels and offer a sustainable, dual-function platform for wastewater treatment applications.

Keywords: Graphene quantum dots, microwave-assisted synthesis method, hydrogel, methylene blue, photocatalytic degradation

Introduction

Currently, global water resources are under serious pollution threats due to rapid industrialization and the increasing world population. Water pollution has become a critical problem due to toxic substances continuously discharged into natural water sources such as rivers, lakes, and oceans (Mohammad Sharifi et al., 2024; Safardoust-Hojaghan & Salavati-Niasari, 2017). This situation causes serious adverse effects both on ecosystems and human health. Among these pollution sources, organic dyes released from industries (textile, leather, and plastic) are of particular concern (Lanjwani et al., 2024). Organic dyes are pollutants that are commonly found in industrial wastewater and are resistant to biodegradation. These dyes are classified according to their ionic structures; MB, rhodamine 6G (Rh6G), malachite green (MG), and crystal violet (CV) are among the cationic dyes, while acid orange 7 (AO7), alizarin red S (ARS), methyl orange (MO), and eriochrome black T (EBT) are classified as anionic dyes. In order to reduce the effect of such organic pollutants, photocatalysis is a sustainable and effective water treatment method that completely mineralizes compounds such as MB through reactive oxygen species (ROS) and eliminates their toxicity (Mohammad Sharifi et al., 2024; Sajjadi et al., 2017). Organic dyes are toxic, carcinogenic, and persist in the environment due to their stable aromatic structures. Additionally, these persistent pollutants pose serious risks to the marine ecosystem even when present in trace amounts (Lanjwani et al., 2024). There are traditional methods such as chemical precipitation, coagulation, and biological treatment available for the removal of these persistent compounds from water. These methods generally suffer from significant shortcomings in terms of high cost, risk of secondary pollution, and low efficiency (Wang et al., 2025). Herein, advanced oxidation processes (AOPs) like photocatalysis stand out as a promising alternative that offers the potential to completely mineralize organic pollutants into harmless components (carbon dioxide and water using light energy) (Yang et al., 2025). The effectiveness of this process is based on the production of highly ROS, hydroxyl radicals (•OH) that attack dye molecules (Umrao et al., 2015). The application of

nanotechnology presents an exciting potential to tackle these problems, offering novel and affordable water remediation methods. This advancement has demonstrated that quantum dots such as zinc selenide, silver-tin oxide, titanium dioxide, and quantum dots (carbon, graphene) have great potential for water purification (Gozali Balkanloo et al., 2023; Mahmoud et al., 2023; Sajjadi et al., 2017; Wang et al., 2025; Yang et al., 2025). In recent years, GQDs have attracted attention as a leading material in this field. As shown in figure 1, They have many features sought for modern photocatalysts, such as adjustable band gap, large surface area, high photostability, excellent electron mobility, and most importantly environmentally friendly and metal-free structure (Gozali Balkanloo et al., 2023; Ibarbia et al., 2020; Jeon et al., 2016; Sun et al., 2023).

It is emphasized in the literature that the precursor material and method used in the synthesis of GQDs are decisive on the properties of the obtained product. Accordingly, the use of WTL biomass waste is gaining more and more importance because it offers a solution to the problem of recycling wastes and provides a low-cost and renewable carbon source. Similarly, the use of MW as a synthesis method provides significant advantages for being faster, a single-step process, and less energy-consuming compared to conventional hydrothermal methods.

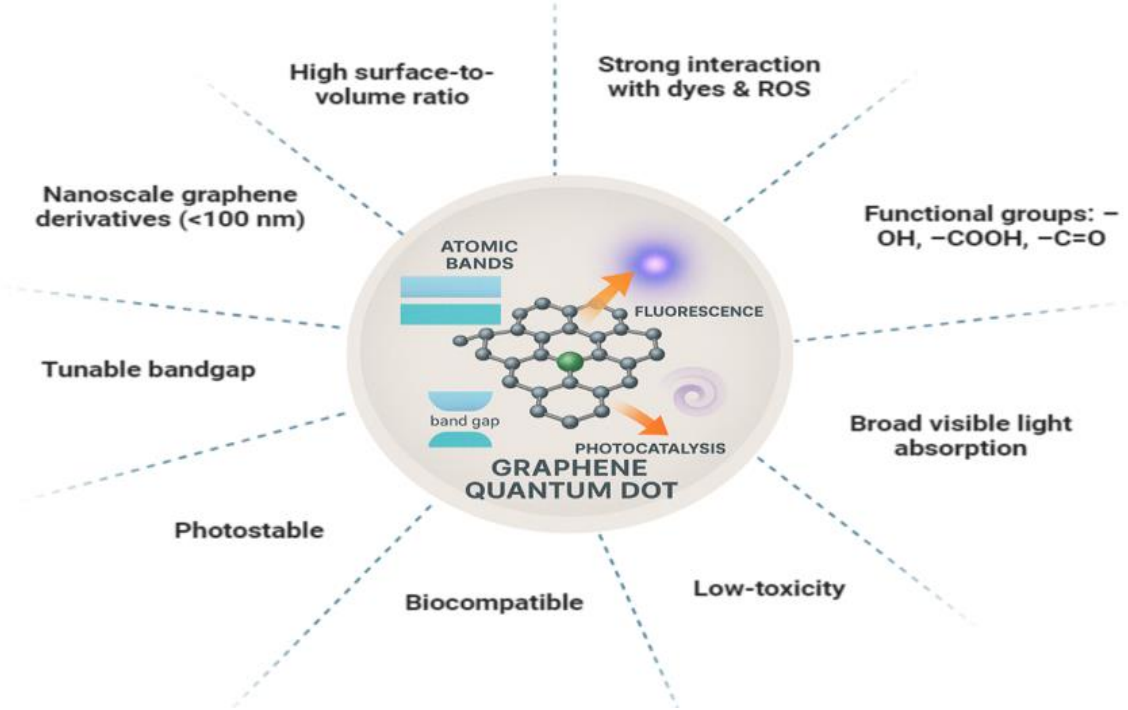


Figure 1. The key properties and applications of GQDs

This efficient method significantly increases the sustainability potential of GQDs (Abbas et al., 2020, 2023; Gozali Balkanloo et al., 2023; Sun et al., 2023). However, the use of nanocatalysts such as GQDs in powder form makes it difficult to recover them from water after the reaction and reduces their catalytic efficiency due to their tendency to agglomerate (Wang et al., 2025). To overcome these practical problems, it is critical to immobilize nanocatalysts in a stable matrix (Ibarbia et al., 2020). For this purpose, SA, a natural polysaccharide, stands out as an ideal support material (Sun et al., 2023). SA is used for biocompatibility, porous structure, and ability to easily form a three-dimensional hydrogel network with Ca^{+2} ions, providing both homogeneous distribution of GQDs and creating a synergistic effect by offering an adsorption surface for pollutant molecules (Mohammad Sharifi et al., 2024; Sun et al., 2023; Wang et al., 2025; Yang et al., 2025). In this research, a new composite photocatalyst was developed by integrating GQDs synthesized from WTL via MW method into SA hydrogel matrix. Also, the main objective of this study was to characterize GQDs-loaded SA hydrogel and evaluate its photocatalytic degradation activity against MB to establish a sustainable and efficient platform for water treatment (Abbas et al., 2020; Lu et al., 2015; Umrao et al., 2015).

Therefore, this study addresses a key challenge in the field: the recovery and reusability of nanocatalysts like GQDs in water treatment. By immobilizing GQDs within sodium alginate (SA) hydrogels, we aim to enhance photocatalytic performance while enabling stable dispersion and ease of recovery. This composite system was synthesized using an environmentally friendly, microwave-

assisted method and characterized through comprehensive optical and structural techniques. The study evaluates both the photoreactive and passive dye removal efficiency of the GQDs-loaded SA hydrogels against methylene blue (MB), aiming to establish a sustainable platform for advanced wastewater treatment.

Materials and Methods

Materials

Sodium alginate was purchased from Sigma-Aldrich and calcium chloride was purchased, methylene blue and ethanol absolute for analysis were purchased from Merck and used without any further purifications unless stated. Waste tea leaves were obtained after tea leaves in a domestic filter tea machines.

The Hettich Zentrifugen Universal 320 centrifuge and DLAB D2012plus Centrifuge for purifications, BIOBASE lyophilizator for freeze drying, SAMSUNG MS23F300E home type-microwave to synthesize GQDs, Thermo Scientific Evolution 201 UV-Visible Spectrophotometer, Malvern Instruments Zetasizer Nano ZS, ZEN3600, ZEISS GeminiSEM 560 Scanning Electron Microscopy for characterization have been used.

Methods

Preparation of Graphene Quantum Dots

GQDs were synthesized from WTL using an environmentally friendly and rapid household MW described in our previous study. One gram of WTL powder was weighed and placed in an erlenmeyer flask, then 100 mL of ethanol was added. This extract was stirred in a thermomagnetic stirrer at room temperature for 24 hours. Then, filter paper was used for filtration 3 times, and after filtration, the supernatant was heated in a microwave oven at 100 watts for 13 min, as shown in figure 2. For purification, centrifugation was performed twice; the first time, a centrifuge was used to remove large carbon particles by centrifuging the mixture at 8,000 rpm for 15 min and then at 12,000 rpm for 15 min. The obtained supernatant was filtered through a 0.22 μm membrane filter to remove the remaining sediment. The final supernatant containing GQDs was collected for characterization, and the stock solution was placed in the refrigerator and then lyophilized to obtain a powder of the synthesized GQDs. GQDs powder was stored in a refrigerator at approximately 4 °C to preserve its physicochemical stability and prevent potential degradation over time (Nassirzadehsaleh & Aykaç, 2024).

Preparation of SA Loaded GQDs

The SA solution was prepared by gradually adding 2 g of SA powder to 100 ml of distilled water and continuously stirring for four hours. Simultaneously, 5 mg of GQDs was dispersed in 250μl of distilled water. Subsequently, the GQDs solution was slowly added to the SA solution and stirred until a homogeneous mixture was obtained that is shown in figure1. After the loading process, the obtained SA-GQDs solution was added dropwise to a CaCl2 (1.11% w/v) solution and left at room temperature for 2 hours to crosslink. Finally, the hydrogels were washed with distilled water three times to eliminate unbound GQDs and lyophilized and stored for later use. Finally, the GQDs-loaded SA hydrogels were characterized as shown in Figure 4. using multiple analytical techniques (Ehtesabi et al., 2019; Monfared-Hajishirkiaee et al., 2023; Zhuang et al., 2016).

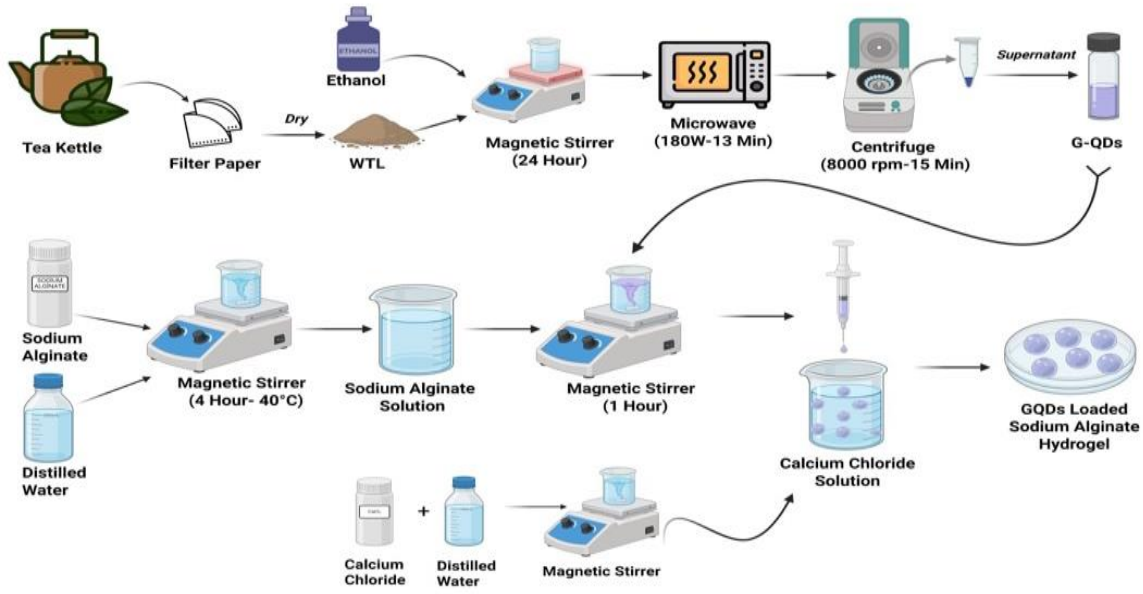


Figure 2. Synthesis method GQDs loaded SA hydrogels

Characterization

Ultraviolet Visible Spectrometry

UV–Vis spectrometry was used to evaluate the optical properties of the nanomaterials by measuring their absorbance in the ultraviolet and visible light regions. GQDs, which usually exhibit characteristic absorbance peaks due to π→π transitions around 280, were applied in this research to study their optical properties. UV-Vis spectroscopy is widely used for observing dye degradation processes photocatalytic degradation. For instance, MB shows a strong absorbance peak around 664 nm. By monitoring the decrease in absorbance over time, the efficiency of nanomaterials in degrading MB was evaluated in this study (Ahmet Aykaç et al., 2024; Lu et al., 2015; Nassirzadehsaleh & Aykaç, 2024).

Dynamic light scattering

This method is a sensitive, non-invasive analytical characterization method used to measure the size and distribution profile of nanoparticles in solution. DLS is based on the scattered light intensity as a function of particle size. In this method, larger particles scatter more light than smaller particles, resulting in an intensity distribution that is skewed toward larger particles regardless of the amount. The DLS analysis results is discussed in the result and discussion section (Nassirzadehsaleh & Aykaç, 2024).

Zeta potential

Zeta potential occurs in the range of +30 and -30 mV to distinguish stable and unstable suspensions. Particles with zeta potential more positive than +30 mV or more negative than -30 mV are normally considered stable. In this study, the zeta potential of GQDs was investigated (Nassirzadehsaleh & Aykaç, 2024).

Scanning Electron Microscopy

SEM is an advanced imaging technique, produces high-resolution images by scanning the sample surface with a focused electron beam. It enables the investigation of nanoparticle distribution, size, shape, surface morphology and topographical features of a sample at high resolution. SEM is typically combined with other analytical techniques (UV-Vis, DLS, Zeta potential) to provide a more comprehensive characterization of GQDs, as illustrated in figure 3 (Ahmet Aykaç et al., 2024; Zhuang et al., 2016).

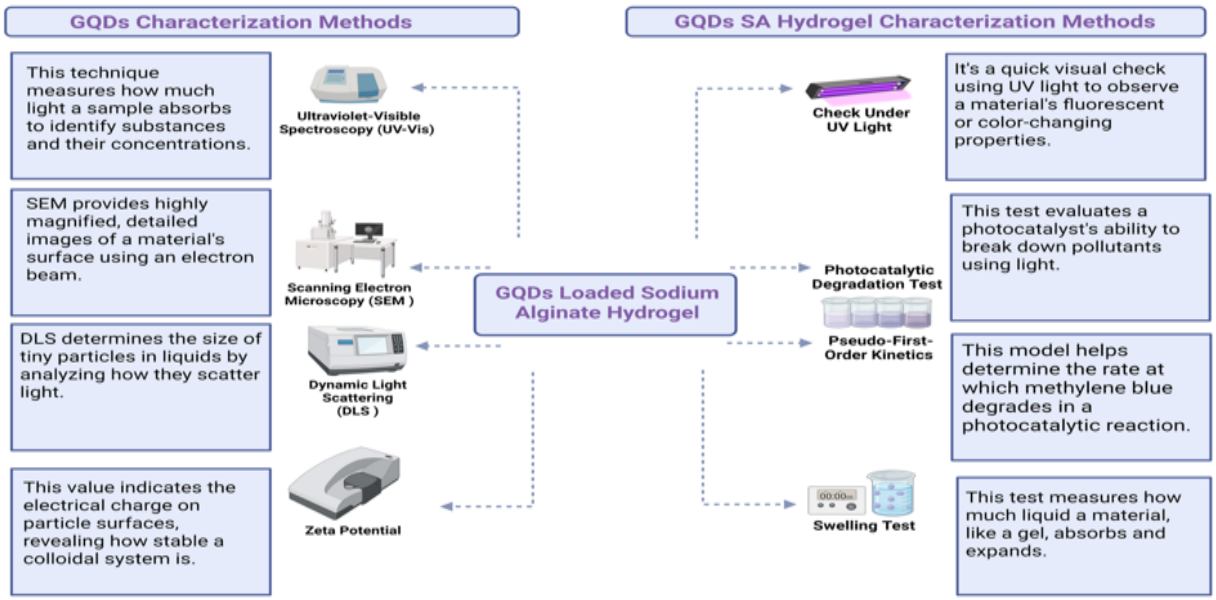


Figure 3. Characterization methods for GQDs and GQD-loaded SA hydrogels

Swelling test

Swelling behavior relates to the ability of hydrogels to absorb water and expand in volume. This method is an important parameter to evaluate the structural integrity, porosity and potential biomedical applications of hydrogel. In this study, the swelling behavior of SA hydrogel was evaluated in distilled water at pH:6 and room temperature to evaluate its water absorption capacity over time (Zhuang et al., 2016).

Check under UV light

The photoluminescence properties of GQDs-loaded hydrogels under UV light exposure were observed. This technique helps to verify the successful integration and optical activity of GQDs, which typically emit visible photoluminescence when excited by UV light (Ehtesabi et al., 2019).

Methylene blue study

The photocatalytic and passive degradation behavior of GQDs-loaded SA hydrogels was evaluated under both UV light (figure 4.) and dark conditions. The UV tests were conducted at multiple time intervals (15, 30, 60, 120, 180, and 240 minutes), and the dark condition study was performed after 24 hours of incubation. The results were analyzed using the Degradation Percentage (%) and Pseudo-First Order Kinetics model are shown in Eq. (1) and (2), respectively.

Degradation Percentage (%): (1)

$$\text{Degradation (\%)} = [(A_0 - A_t) / A_0] \times 100$$

Where A_0 is the initial absorbance and A_t is the absorbance at time t.

Pseudo-First Order Kinetics model: (2)

$$\ln (C_t / C_0) = -kt$$

Where C_0 is the initial concentration, C_t is the concentration at time t and k is the rate constant (min^{-1}) (Safardoust-Hojaghan & Salavati-Niasari, 2017; Umrao et al., 2015)

Figure 4. Photocatalytic degradation of MB by GQDs-loaded SA hydrogel under simulated sunlight using a UV light lamp

Results and Discussion

UV-Vis spectroscopy

The optical properties of the synthesized GQDs were analyzed by UV–Vis spectroscopy. An absorption peak at 272 nm wavelength was observed in the GQDs, as shown in Figure 5. This peak corresponds to the π→π* electronic transitions of the C=C bonds in the sp^2 hybridized carbon structure. These results indicate that GQDs with conjugated π-electron system were successfully synthesized and indicate that these quantum dots are suitable for photocatalytic degradation.

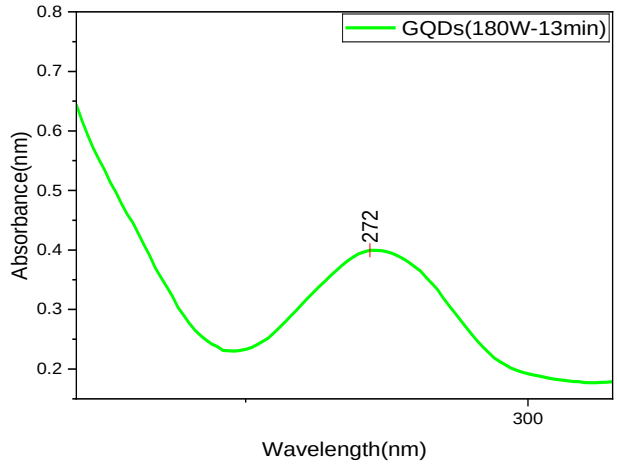


Figure 5. UV-VIS spectrometry characterization technique for three different GQDs

Dynamic light scattering

As a result of this analysis, the Z-average (average hydrodynamic diameter) of the synthesized GQDs was determined as 73.05 nm, and the polydispersity index (PdI) was found to be 0.878. The main peak was observed at 11.87 nm in the distribution by number, while this value was determined as 22.97 nm in the distribution by volume that are shown in figure 6. These results show that GQDs are nano-sized and dispersed in the aqueous medium and they are suitable for integration into the hydrogel system.

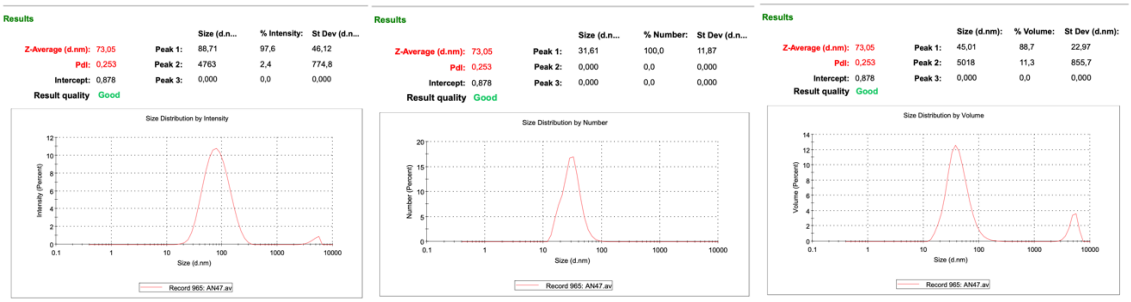


Figure 6. DLS analysis report by (a) intensity, (b) number, (c)volume of GQDs

Zeta potential

The average surface charge of GQDs was determined to be -36.9 mV as shown in figure 7. This results illustrates their robust colloidal stability for consistenting dispersion in aqueous environments without particle agglomeration. Moreover, negative zeta potential in this analysis reflects excellent colloidal stability for facilitating homogeneous dispersion within hydrogel networks and enhances photocatalytic efficiency.

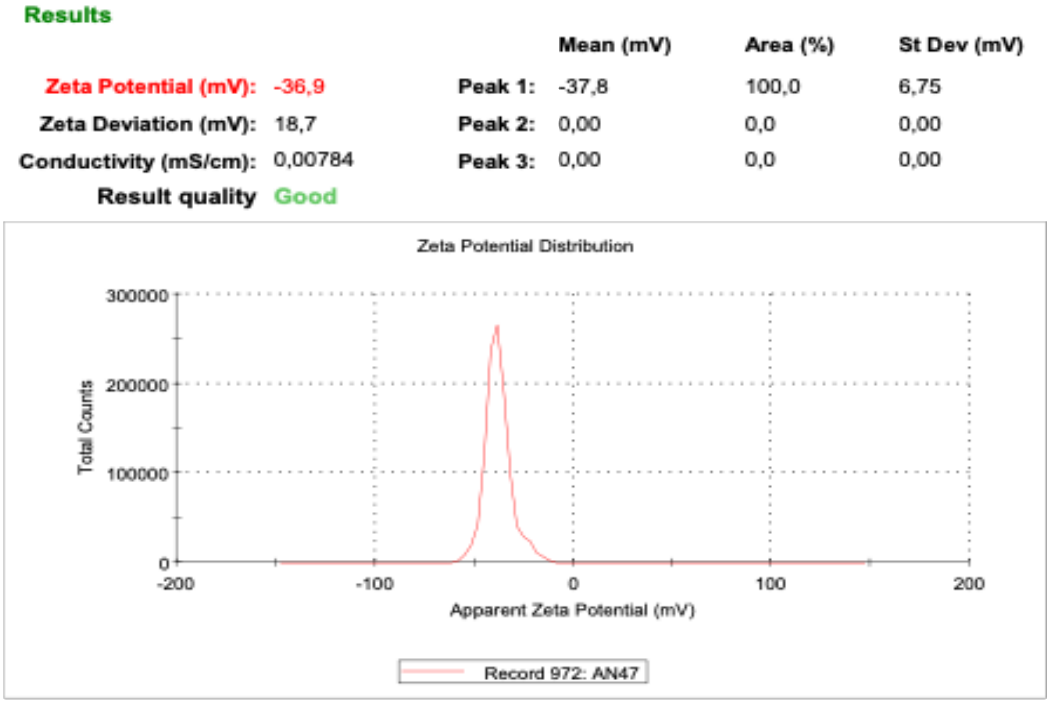


Figure 7. Zeta potential analysis record of GQDs

Scanning Electron Microscopy

The surface morphology and particle size of GQDs were measured and as it shown in Figure 8, the GQDs were uniformly distributed with a predominantly spherical shape. The observed particle sizes are approximately 14.56 nm, 16.78 nm, and 17.01 nm, confirming the nanometer-scale dimensions of the material. These SEM results confirm that GQDs were made successfully with a controlled shape, backing up the findings from UV-Vis and DLS tests.

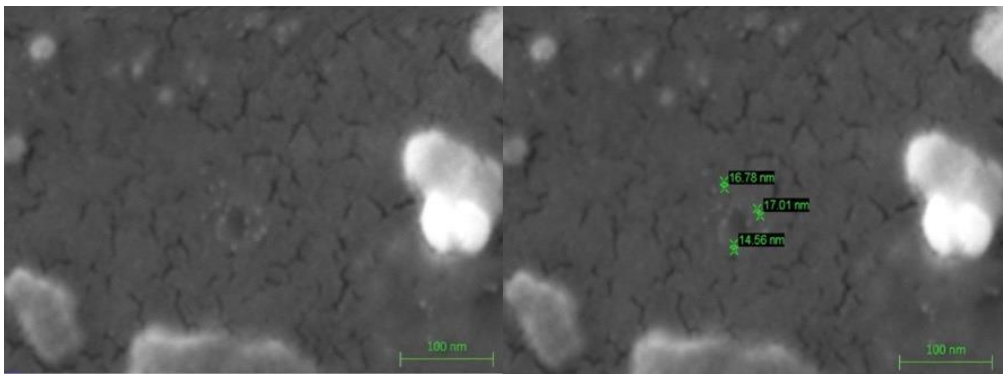


Figure 8. SEM analysis result of GQDs

UV-light

The optical activity of GQDs-loaded SA hydrogels was visually confirmed under UV light exposure. As seen in Figure 9(a), the hydrogel beads exhibited obvious blue fluorescence, indicating the successful incorporation of GQDs into the polymer matrix and their photoluminescence behavior. In contrast, the same samples shown in Figure 9(b) did not show any visible fluorescence under natural light. This visual confirmation supported other analyses in terms of confirming the formation of optically active GQDs and their stable integration into the SA hydrogel network.

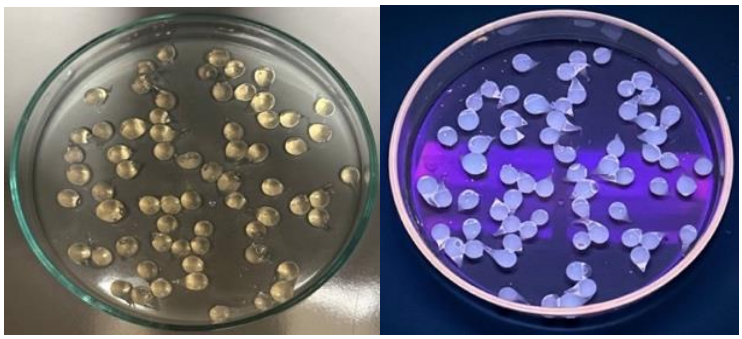


Figure 9. (a) GQDs-loaded SA hydrogels under natural sunlight showing no visible fluorescence, (b) GQDs-loaded SA hydrogels under UV light exhibiting characteristic fluorescence

Swelling test

The swelling behavior of SA hydrogel was investigated in distilled water at pH:6 and room temperature. As shown in Figure 10, the initial dry weight of the hydrogel was 505 mg and a significant weight gain was observed within the first few hours. The hydrogel reached approximately 800 mg at 12 h and gradually approached the swelling equilibrium at 24 h and maintained a constant weight up to 48 h. This behavior indicated the strong water absorption capacity and structural integrity of the GQDs hydrogel matrix, which are required for sustained release and environmental applications.

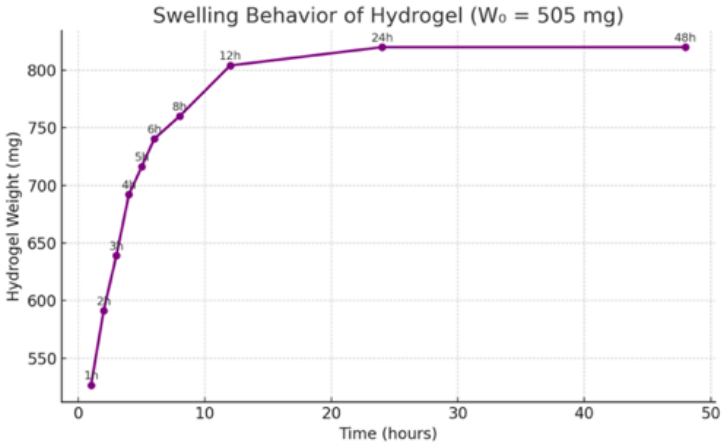


Figure 10. Swelling behavior of SA hydrogel in distilled water at pH:6 and room temperature

Photocatalytic Degradation

Calibration curve was constructed using MB solutions with ten different concentrations ranging from 0.5 to 5 ppm. A strong linear relationship was observed between concentration and absorbance, and the high coefficient of determination ($R^2 = 0.9985$) confirmed the accuracy and reliability of the UV–Vis spectrophotometric method for quantitative analysis in Figure 11.

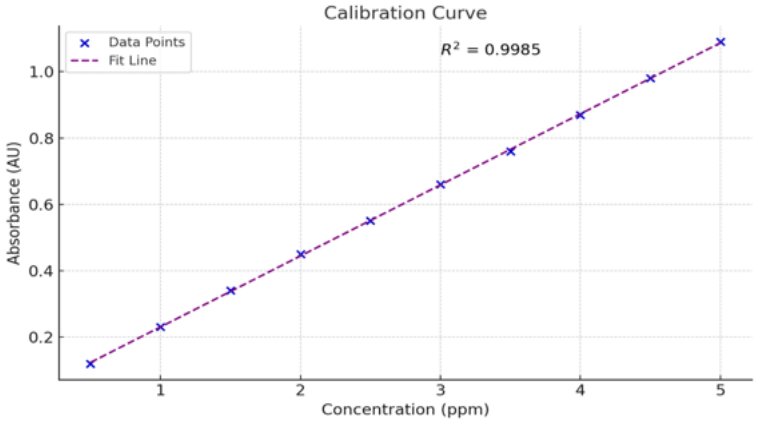


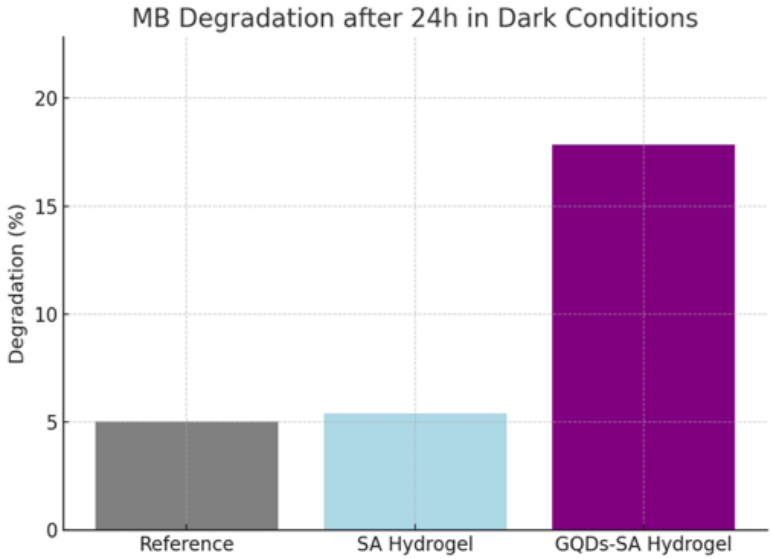
Figure 11. Calibration curve of MB solutions with different concentrations (0.5–5 ppm)

Pseudo-First-Order Kinetics:

MB photocatalytic degradation was evaluated under light and dark conditions using reference samples, plain SA hydrogels, and GQDs-loaded SA hydrogels. Under UV light, after 240 min, GQDs-loaded hydrogels reached the highest degradation percentage (25.1%), followed by SA hydrogels (25.4%) and the reference group (21.0%). Although these results indicate the enhanced surface interaction and photoreactive potential of GQDs, the degradation rate appeared similar to SA due to the complex kinetic behavior. In contrast, under dark conditions (after 24 h), GQDs-loaded SA hydrogels showed 17.85% degradation, which was significantly higher than both plain SA hydrogels (5.40%) and the reference (5.02%). This confirms that GQDs not only contribute to photoreactive degradation but also passive dye adsorption, most likely due to the high surface area and strong π-π interactions with MB molecules.

Degradation Percentage (%):

In this research, the photocatalytic and passive degradation behaviors of MB were evaluated under both UV light and dark conditions using reference samples, plain SA hydrogels, and GQDs-loaded SA hydrogels. The results were analyzed using Degradation Percentage (%) and pseudo-first-order kinetic modeling. As shown in Figure 12, under dark conditions, both the reference and plain SA hydrogels achieved only about 5% degradation after 24 h, while the GQDs-loaded hydrogel reached 17.85%.



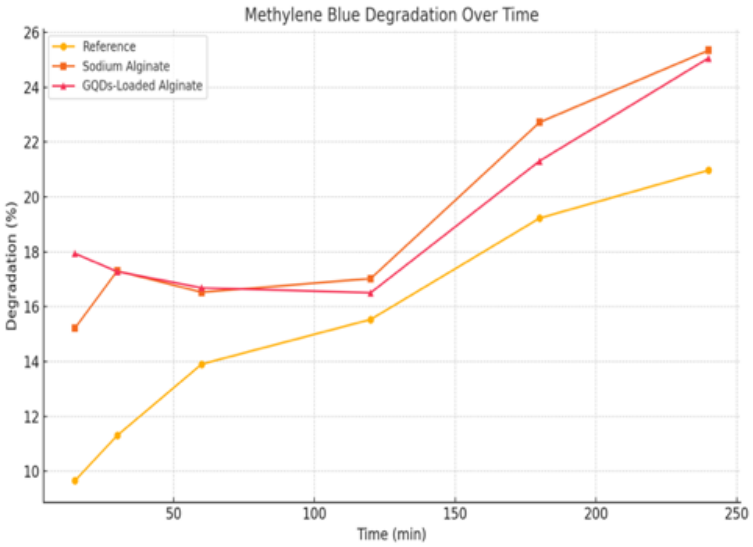


Figure12. a) The reference, plain SA hydrogel, and GQDs-SA hydrogel samples degradation. b) This graph illustrates the degradation efficiency of MB over 240 minutes under light for three different systems (reference, plain SA hydrogels, GQDs loaded SA hydrogels)

This significant increase highlights the strong adsorption capacity of GQDs and their interaction with MB molecules even in the absence of light, confirming their dual roles in passive dye removal. Furthermore, the time-dependent degradation under UV light is presented in Figure 12. Over an exposure time of 240 min, the reference sample achieved ~21.0% degradation and the plain SA hydrogel reached ~25.3%. This may be attributed to enhanced dye adsorption by the SA network. In particular, GQDs-loaded SA hydrogels showed faster and more efficient degradation, reaching 25.1% at the end of the test. This performance is attributed to the enhanced surface reactivity and photocatalytic activity of GQDs, which facilitate MB degradation through the formation of ROS and improved light absorption. As a result, it clearly shows that the integration of GQDs into hydrogel matrices provides a dual-functional system capable of both adsorptive and photocatalytic MB degradation under various environmental conditions.

Conclusions

In this research, GQDs were successfully synthesized by WTL using MW method and effectively incorporated into the SA hydrogel matrix. Characterization using UV-Vis spectroscopy, DLS, zeta potential, SEM, and fluorescence imaging under UV light confirmed the successful formation, stability, and optical activity of GQDs and their homogeneous distribution in the hydrogel. The swelling behavior showed high water sorption capacity and structural integrity, which made the GQDs-loaded SA hydrogel suitable for aqueous media. Most importantly, photocatalytic degradation experiments against MB under UV light and dark conditions revealed the bifunctional nature of the composite. The GQDs-loaded hydrogel achieved 25.1% degradation efficiency under UV light and 17.85% in the dark, while the plain SA hydrogels and reference samples showed significantly lower performance. Although a lower pseudo-first-order rate constant, the GQDs composite achieved higher overall degradation, suggesting a fast initial adsorption and surface-limited kinetic behavior. This study demonstrates the effect of GQDs and SA hydrogel on dye degradation and further provides a promising, low-cost, and environmentally friendly platform for wastewater treatment (waste from textile industries containing various dyes, soaps and salt). Future studies can investigate the sustainability and degradation performance of these hydrogels under natural sunlight (UV) in real wastewater environments.

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