

SYNTHESIS, CHARACTERIZATION AND APPLICATION OF CARBON-BASED NANOMATERIALS FOR REMOVAL OF WATER POLLUTANTS**Seecharan Banjare and Sandhyarani Panda***

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ABSTRACT

Water and wastewater treatment, including industrial water, is crucial for protecting the environment and public health and carbon-based nanomaterials shows the adsorption of water pollutants. The paper focused on the green synthesis and characterization of Carbon-based bio adsorbents and their potential application in water pollution remediation. The Green synthesized Carbon-based bio adsorbents was synthesized using a modified Hummers method, resulting in uniform spherical structures. The synthesized Green synthesized Carbon-based bio adsorbents was characterized using various techniques including UV-Vis spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray (EDX) mapping, and EDX imaging. UV-Vis spectroscopy analysis revealed the presence of characteristic absorption peaks, confirming the successful synthesis of Green synthesized Carbon-based bio adsorbents. XRD analysis showed a distinct peak at 2θ , indicating the presence of a well-ordered layered structure. SEM imaging displayed the spherical morphology of the Green synthesized Carbon-based bio adsorbents with a relatively uniform size distribution. EDX mapping and imaging provided elemental composition information, confirming the presence of carbon and oxygen in the Green synthesized Carbon-based bio adsorbents. Furthermore, the absorption capacity of the synthesized Green synthesized Carbon-based bio adsorbents for water pollution was investigated. The Green synthesized Carbon-based bio adsorbents exhibited a high adsorption capacity for various water pollutants, including organic dyes and heavy metal ions. This suggests their potential as an effective adsorbent for water treatment applications.

Keywords: Green synthesized Carbon-based bio adsorbents; UV-Vis spectroscopy; X-ray diffraction (XRD); scanning electron microscopy (SEM); energy-dispersive X-ray (EDX) mapping and EDX imaging.

1. INTRODUCTION

Water scarcity is a major concern due to limited water resources and a growing population. Traditional methods of wastewater treatment involve various technologies such as ion-exchange, reverse osmosis, and oxidation. Newer approaches, like the use of nanomaterials, have also been explored for removing contaminants from water (Anjum et al., 2019). Nanomaterials can be used for processes like adsorption, photocatalysis, and filtration to remove pollutants from water. Emerging contaminants, including pesticides, dyes, plasticizers, and pharmaceuticals, pose health risks and can be effectively removed by carbonaceous nanomaterials (Nasrollahzadeh et al., 2021a). These nanomaterials have high surface areas and reactivity, making them efficient in removing hazardous substances.

The development of environmentally sustainable techniques, such as green chemistry and nanotechnology, is crucial in addressing emerging challenges (Pokrajac et al., 2021). Using safer solvents and reducing the use of chemicals in production can improve the sustainability of water treatment processes (Welton, 2015). Green nanotechnology has the potential to reduce costs and improve efficiency in wastewater treatment. The demand for clean drinking water is increasing, and micropollutants are contaminating water sources (Magalhães-Ghiotto et al., 2021). Conventional decontamination methods often require excessive chemical use and can generate toxic by-products. Nanomaterials offer a promising solution with their high surface area, reactivity, and cost-effectiveness (Ahmed et al., 2022a). Advanced nanomaterials are being used to develop water treatment systems that can continuously monitor water quality. These materials can effectively remove contaminants and ensure the long-term accessibility and feasibility of water supply (Nasrollahzadeh et al., 2021a). Nanomaterials like graphene-based nanomaterials and carbon-based nanotubes are specifically used for wastewater treatment and the removal of pharmaceutical contaminants.

In conclusion, nanomaterials show great potential in addressing water scarcity and removing pollutants from water. By utilizing advanced nanomaterials and adopting green and sustainable techniques, we can improve water treatment processes and ensure a clean and accessible water supply for all (Nishu & Kumar, 2023).

Nanomaterials offer promising alternatives for wastewater treatment due to their unique advantages. However, challenges such as potential health risks, high production costs, selectivity, sustainability, and recyclability still exist. Further studies are needed to standardize analytical methodologies, understand removal kinetics, develop simulation models, evaluate environmental behaviors of contaminants, address toxicity issues, and assess the environmental effects of nanomaterials (Ahmed et al., 2022b). Researchers are focusing on producing efficient and sustainable nanomaterials at an affordable cost for eco-friendly treatment of water and wastewater. This includes targeting pharmaceuticals, endocrine disrupting chemicals, industrial wastes, pesticides, organic dyes, personal care products, detergents, and other emerging pollutants (Nasrollahzadeh et al., 2021b).

Graphene nanomaterials have significant physicochemical characteristics such as high surface area, simple functionalization, and hydrophobicity, making them suitable for treating wastewater. They can be used as nano sorbents to eliminate hazardous and toxic pollutants and can undergo catalytic degradation through various processes (Yang et al., 2018). However, their commercial production and use present challenges. Uniform nanoporous graphene sheets can filter and desalinate water by adjusting pore size and pressure, but their mechanical stability is a drawback. N-doped sandwich-type graphene composites have been developed as capacitive deionization electrodes, offering a large surface area and low resistivity along with efficient salt adsorption (Aghigh et al., 2015). Graphene oxide (GO) nanosheets can serve as selective barriers for water permeability, and super-hydrophobic and super oleophilic porous graphene can be utilized as highly selective and recyclable absorbents (Avornyo & Chrysikopoulos, 2024).

Carbon-based bio adsorbents nanofiltration membranes with porous polyacrylonitrile nanofibrous mats have been created for water treatment, effectively regulating thickness and generating a barrier to contaminants (Wang et al., 2016). Unlike carbon nanotubes (CNTs), graphene-based nano adsorbents have advantages in removing toxic dyes due to the availability of basal planes for adsorption. Additionally, graphene and its analogues can be easily fabricated without metallic catalysts or complex apparatus (Assad et al., 2024).

Carbon-based bio adsorbents nanomaterials do not require additional purification steps as they are free of catalytic residues. Carbon-based bio adsorbents nano sorbents have abundant oxygen-bearing functionalities that enhance their reactivity and hydrophilicity, allowing them to interact with various contaminants through electrostatic and hydrophobic interactions (Nasrollahzadeh et al., 2021a). Consequently, Carbon-based bio adsorbents has gained attention as an effective nano sorbent for contaminated water and wastewater.

In summary, graphene nanomaterials possess unique physicochemical properties that make them suitable for wastewater treatment. They can be used as nano sorbents, filtration membranes, and deionization electrodes, offering superior adsorption capabilities and recyclability (Rana et al., 2024). Their ease of fabrication and absence of catalytic residues are additional advantages in their application. Carbon-based bio adsorbents, in particular, has shown promise as an effective nano sorbent for contaminated water (Punia et al., 2020).

2. EXPERIMENTAL

2.1 Required Materials

Sigma-Aldrich in India for pristine graphite powder (99% graphene, 325 mesh) and Methylene Blue dye. All of the analytical-grade compounds were utilized in the experimental investigation without any sort of purification or treatment.

2.2 Method

2.2.1 Extract Preparation of the T. Cordifolia Plant Stem:

Stems of T. cordifolia plants that were disease-free were gathered, repeatedly cleaned with distilled water, sliced, and then crushed. In addition, 100 mL of distilled water and 15 g of cleaned T. cordifolia stems were put to a 250 mL flask, which was then heated to 80–90 °C for three hours while stirring continuously.^{34, 35} After the solution was cooled to room temperature, its color changed from colorless to brown. Filter paper (Whatman No. 1) was used to filter the prepared solution. After being collected in a glass vial, the sample was kept for a week at 0–5 °C in the refrigerator.

2.2.2 Preparation of Carbon-based Bio Adsorbents:

With a few minor modifications to Hummer's method, Carbon-based bio adsorbents were produced from natural graphite powder. In a 1000 mL flask kept in an ice bath (0–5 °C), 120 mL of concentrated sulfuric acid (0.1 M H₂SO₄) was mixed with 5.0 g of dry graphite powder and 0.015 g of boric acid (H₃BO₃). For around three hours, the solution was continuously swirled.³⁶ After putting the mixture in an ice bath (below 15 °C to prevent explosion), 15 g of KMnO₄ was gradually added. The solution needed to be taken out of the ice bath and heated to 35 °C for two hours while being continuously stirred.

After 450 mL of deionized water was added to the agitated mixture, it was heated to 95 °C for two hours. After then, the combination was given a full hour to come down to room temperature. To further remove the KMnO₄ residue, 35 mL of hydrogen peroxide (H₂O₂, 30%) was added. After the solution had been vacuum-filtered using Whatman No. 1 filter paper, the precipitate was thoroughly washed with 500 mL of deionized water and dried in a hot air oven at 85 °C for 15 hours.

Green Production of carbon-based bio adsorbents Applying the Stem Extract of T. cordifolia. In a 100 mL Erlenmeyer flask, a 50 mL solution of distilled water containing 25 mg of Carbon-based bio adsorbents was sonicated for one hour. Additionally, 50 mL of T. cordifolia extract and 50 mL of carbon-based bio adsorbents suspension was combined in a 1:1 (v/v) ratio using a 250 mL Erlenmeyer flask. For three hours, the well-combined mixture was refluxed at 85 °C.³⁷ The resultant solution was then rinsed several times, filtered using Whatman filter paper (No. 1), and dried for fifteen hours at 85 °C in an oven. For use in other investigations, the dried carbon-based bio adsorbents were ground into a fine powder.

2.3 Characterization Studies

UV-visible absorption spectra were obtained by Shimadzu 1900i with 200–800 nm. The resolution was set to 4 cm⁻¹. XRD spectra were recorded on a Bruker AXS D8 Advanced (Cochin, India) using Cuα radiation with 1.5406 Å wavelength, and a Si (Li) position sensitive detector (PSD) detector was used. An Anton Paar TTK 450 attachment was added (Cochin, India), and the temperature range was 170°C to 450°C. Structural features were obtained by using a SEM and EDX JEOL Model JSM-6390LV (Cochin, India). The resolution was around point: 0.23 nm, lattice: 0.14 nm, magnification 2000× to 1,500,000×.

2.4 Dye Degradation

The MB adsorption experiments were conducted in batch conditions using the Green synthesized Carbon-based bio adsorbents nanocomposites. A standard MB stock solution was used and diluted with deionized water in order to obtain different concentrations. The obtained MB solutions were kept in a flask with fixed volume (10 mL of 5 ppm) and Green synthesized Carbon-based bio adsorbents nanocomposites were added. The flask was placed in a sonicator for 120 min, at pH 6, at room temperatures. The upper layer liquid was analyzed by UV-Vis (UV-Visible 1900i, Shimadzu, Japan), at a wavelength of 589 nm, after a certain time. The Green synthesized Carbon-based bio adsorbent nanocomposites were removed with the help of centrifugation, when the experiment was over. The removal (R, %) was calculated from Equation (1) as follows:

$$R(\%) = \frac{C_o - C_e}{C_o} \times 100 \quad \dots\dots\dots (1)$$

Where, C_o and C_e are the initial and equilibrium concentrations of MB (mg L^{-1}), respectively.

3. RESULTS AND DISCUSSION

3.1 Characterization Studies

3.1.1 UV-Visible Absorption Study

The UV-visible analysis was conducted to confirm the synthesis of Green synthesized Carbon-based bio adsorbents nanocomposites by absorption spectra along with the determination of optical properties. Typical UV-visible absorption spectra of Green synthesized Carbon-based bio adsorbents nanocomposites shown in Fig.1 show absorption peaks at 369 nm respectively. This shifting to longer wavelength decreases the band gap and enhances the photocatalytic reaction. The linear region of the plot ah versus h was used to calculate the optical band gap of Green synthesized Carbon-based bio adsorbents nanocomposites (Fig. 2). The obtained band gap of Green synthesized Carbon-based bio adsorbents nanocomposites is. 4 eV.

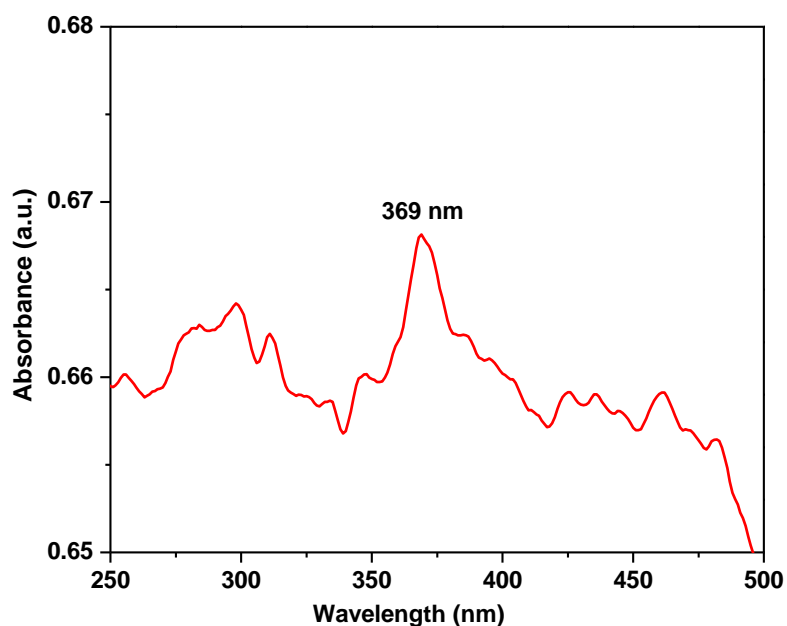


Figure:1 UV-vis Absorption Spectra of Carbon-based bio adsorbents

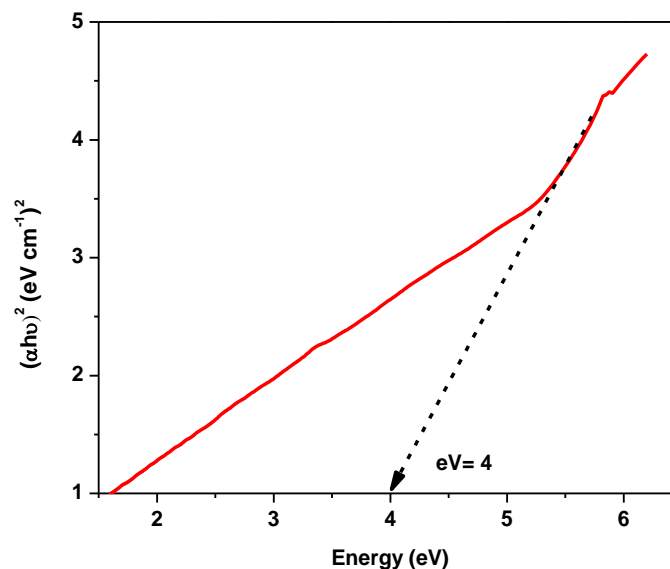


Figure:2 The optical band gap energy of the Carbon-based bio adsorbents

3.1.2 XRD Study

The XRD patterns of Green synthesized Carbon-based bio adsorbents nanocomposites are shown in Fig. 3. The characteristic diffraction (002) and (001) peak of Green synthesized Carbon-based bio adsorbents nanocomposites was observed at $2\theta = \sim 29^\circ$. The peaks indicate (002) the existence of oxygen-containing group and further confirmed that the graphite was fully oxidized into GO.

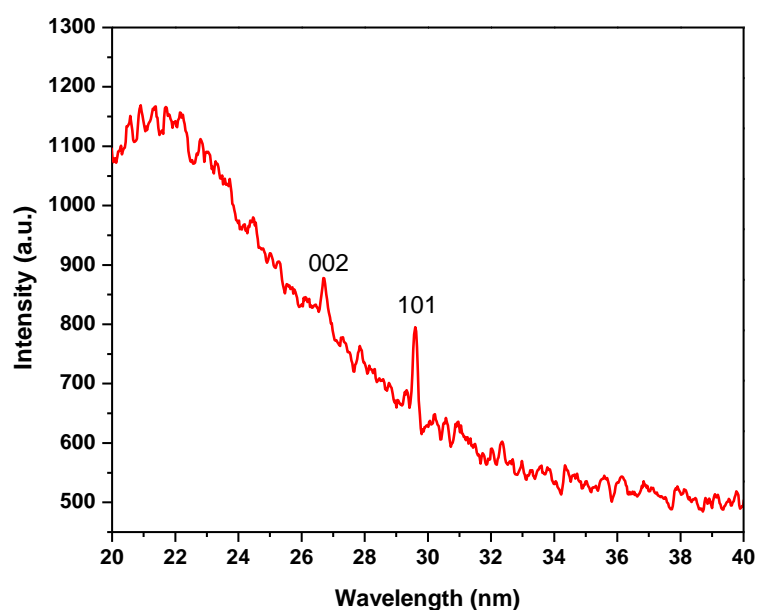


Figure: 3 XRD (X-ray diffraction) of Carbon-based bio adsorbents

3.1.3 SEM Study

The surface morphological properties of the synthesized Green synthesized Carbon-based bio adsorbents nanocomposites were investigated by Scanning electron microscopy (SEM) as shown in Figure 4. The nanocomposites were found to be spherical with some aggregates attributable to the surface energy and surface tension of the Green synthesized Carbon-based bio adsorbents nanocomposites.

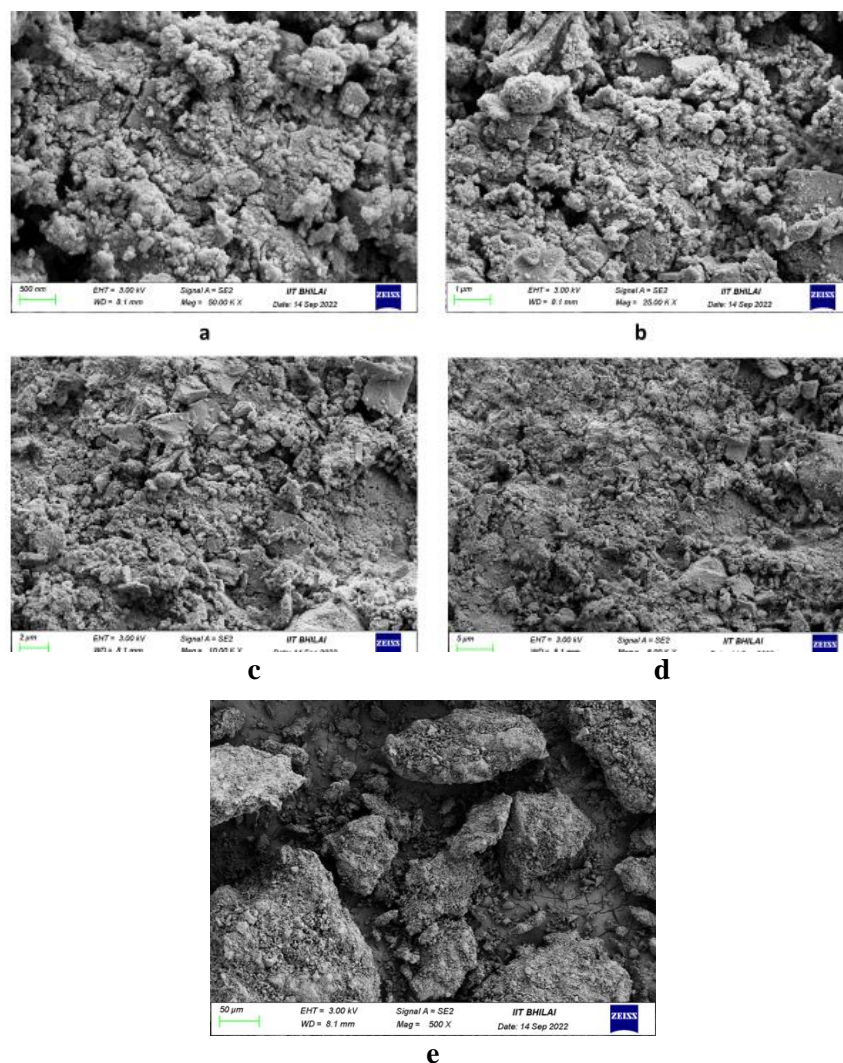
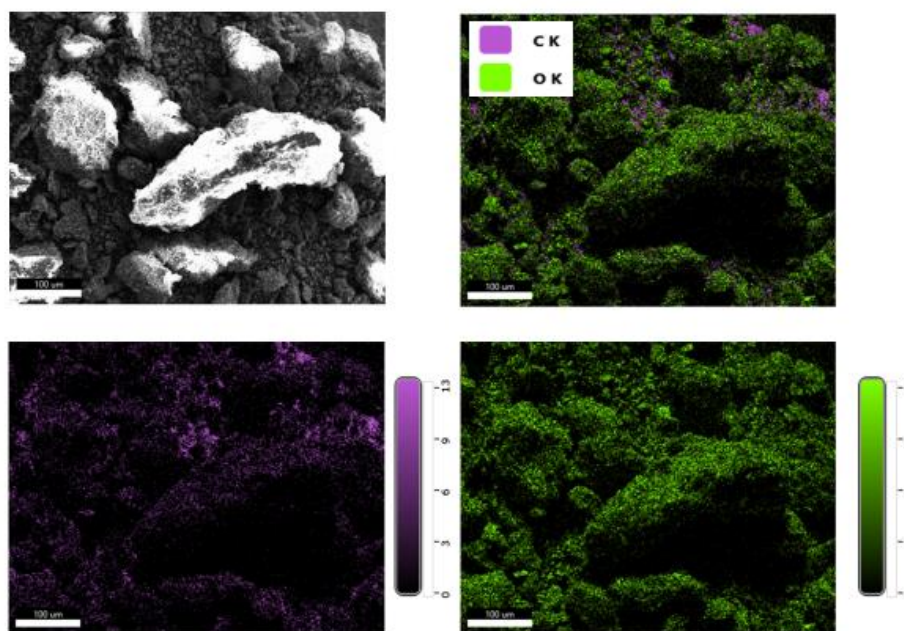


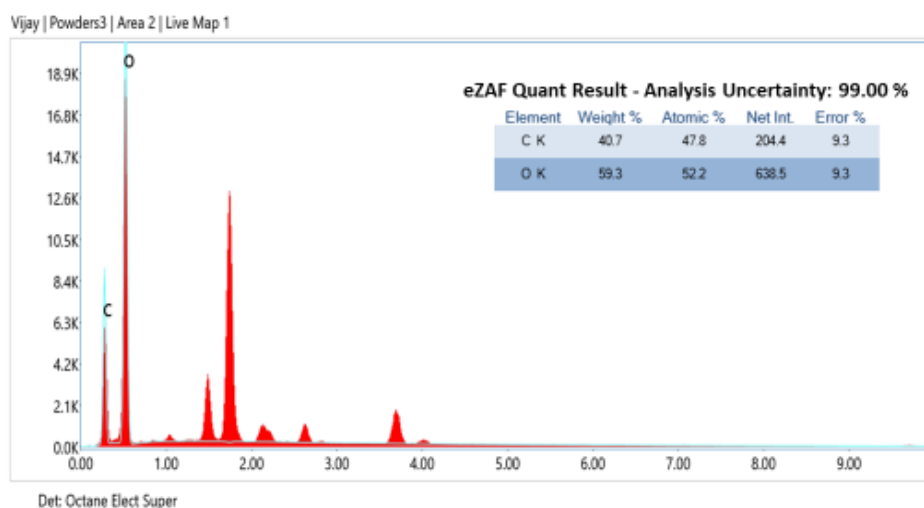
Figure:4. SEM (Scanning Electron Microscope) analysis of Carbon-based bio adsorbents

3.1.4 EDX and Mapping

Energy dispersive X-ray analysis (EDX) was used to confirm the composition and purity of Green synthesized Carbon-based bio adsorbents nanocomposites. EDX analyzed the precise G and O content and found only G and O peaks without other peaks due to contamination as seen in Figure 5a, and 5b.



(a)



(b)

Figure:5 EDX (Energy Dispersive X-ray) analysis of Carbon-based bio adsorbents (a) EDX image of Carbon-based bio adsorbents (b) EDX mapping of Carbon-based bio adsorbents

3.1.5 Dye Remediation

The scanning range between 200 and 800 nm was used for the solution of methylene blue from and λ_{max} obtained at 589 nm. The catalytic activity of Green synthesized Carbon-based bio adsorbents nanocomposites was studied with methylene blue under sonophotocatalytic condition. Further course of the reaction was monitored for methylene blue degradation at 589 nm through a decrease in absorbance (Fig. 6). Figure 6 also established that at this wavelength, other reactants present in reaction mixture do not interrupt.

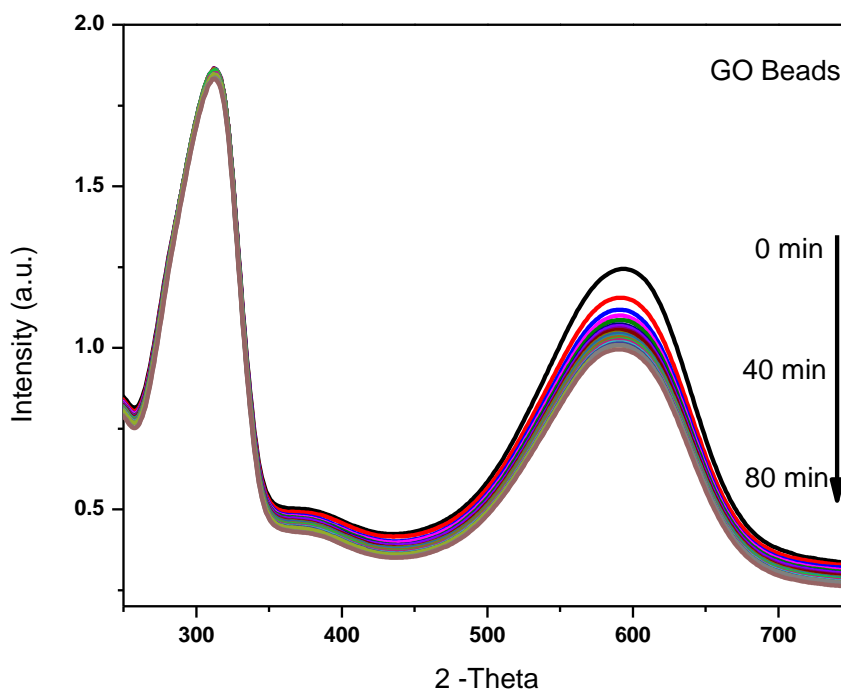


Fig.6: Absorption spectra of the MB aqueous solution during sonophotocatalysis in the presence of Green synthesized Carbon-based bio adsorbents nanocomposites at optimum condition (10 ml of 5 ppm, pH 6 time – 80 min, 10 mg Green synthesized Carbon-based bio adsorbents nanocomposites)

4. CONCLUSION AND FUTURE PROSPECTS

The treatment of water and wastewater, including industrial water, is crucial for protecting the environment and human health. Recent advancements in environmental pollution control have made significant progress. Greener nanotechnology and sustainable nanomaterials offer potential solutions for treating wastewater and addressing hazardous contaminants. However, obstacles such as inadequate infrastructure and resource utilization remain to be addressed. Concerns about the toxicity of nanomaterials also need to be examined, with carbon-based materials showing promise. Nanotechnology can enhance industrial water treatment by efficiently removing contaminants, but further research is needed. Carbon nanotubes, carbon and graphene quantum dots, and graphene-based nanomaterials may serve as effective, cost-efficient, and environmentally-friendly alternatives for water treatment. The modification of carbon nanotubes can improve their ability to remove contaminants, but comprehensive toxicity evaluations are necessary.

Green synthesized Carbon-based bio adsorbents nanocomposites are effective nano adsorbents for eliminating heavy metals and hazardous contaminants. However, their production cost is high compared to activated carbon, and additional equipment is necessary to prevent emission of nanocomposites into water or air. The commercialization of Green synthesized Carbon-based bio adsorbents nanocomposites for wastewater technology depends on their impact on the aquatic ecosystem. Conducting toxicity analyses, life cycle assessments, strategy evaluations, and dispersion studies in water systems helps assess the risks of Green synthesized Carbon-based bio adsorbents nanocomposites to human health.

Future Prospects

The efficiency of using nano-materials in wastewater treatment is undeniable; however, there are serious concerns regarding their impact on the environment. Nano-particles can be released during the preparation and treatment processes, posing long-term accumulation and risks. Future research should focus on developing catalysts with minimal environmental toxicity to reduce health risks. Re-evaluating ecotoxicity potential and conducting life cycle assessments of nano-materials is crucial to understand their overall benefits and risks. Mass adoption of nano-technology has been limited due to cost-competitiveness, so future applications will emphasize efficient processes requiring small quantities. Additionally, cost-effective methods and large-scale testing are necessary for successful field application.

REFERENCE

- Aghigh, A., Alizadeh, V., Wong, H., Islam, M., Amin, N., & Zaman, M. (2015). Recent advances in utilization of graphene for filtration and desalination of water: A review. *Desalination*, 365, 389–397.
- Ahmed, S. F., Mofijur, M., Ahmed, B., Mehnaz, T., Mehejabin, F., Maliat, D., Hoang, A. T., & Shafiullah, G. M. (2022a). Nanomaterials as a sustainable choice for treating wastewater. *Environmental Research*, 214, 113807. <https://doi.org/10.1016/j.envres.2022.113807>
- Ahmed, S. F., Mofijur, M., Ahmed, B., Mehnaz, T., Mehejabin, F., Maliat, D., Hoang, A. T., & Shafiullah, G. M. (2022b). Nanomaterials as a sustainable choice for treating wastewater. *Environmental Research*, 214, 113807. <https://doi.org/10.1016/j.envres.2022.113807>
- Anjum, M., Miandad, R., Waqas, M., Gehany, F., & Barakat, M. A. (2019). Remediation of wastewater using various nano-materials. *Arabian Journal of Chemistry*, 12(8), 4897–4919. <https://doi.org/10.1016/j.arabjc.2016.10.004>
- Assad, H., Lone, I. A., Kumar, A., & Kumar, A. (2024). Unveiling the contemporary progress of graphene-based nanomaterials with a particular focus on the removal of contaminants from water: A comprehensive review. *Frontiers in Chemistry*, 12, 1347129. <https://doi.org/10.3389/fchem.2024.1347129>
- Avornyo, A., & Chrysikopoulos, C. V. (2024). Applications of graphene oxide (GO) in oily wastewater treatment: Recent developments, challenges, and opportunities. *Journal of Environmental Management*, 353, 120178. <https://doi.org/10.1016/j.jenvman.2024.120178>
- Magalhães-Ghiotto, G. A. V., Oliveira, A. M. de, Natal, J. P. S., Bergamasco, R., & Gomes, R. G. (2021). Green nanoparticles in water treatment: A review of research trends, applications, environmental aspects and large-scale production. *Environmental Nanotechnology, Monitoring & Management*, 16, 100526. <https://doi.org/10.1016/j.enmm.2021.100526>
- Nasrollahzadeh, M., Sajjadi, M., Iravani, S., & Varma, R. S. (2021a). Carbon-based sustainable nanomaterials for water treatment: State-of-art and future perspectives. *Chemosphere*, 263, 128005. <https://doi.org/10.1016/j.chemosphere.2020.128005>
- Nasrollahzadeh, M., Sajjadi, M., Iravani, S., & Varma, R. S. (2021b). Carbon-based sustainable nanomaterials for water treatment: State-of-art and future perspectives. *Chemosphere*, 263, 128005. <https://doi.org/10.1016/j.chemosphere.2020.128005>
- Nishu, & Kumar, S. (2023). Smart and innovative nanotechnology applications for water purification. *Hybrid Advances*, 3, 100044. <https://doi.org/10.1016/j.hybadv.2023.100044>
- Pokrajac, L., Abbas, A., Chrzanowski, W., Dias, G. M., Eggleton, B. J., Maguire, S., Maine, E., Malloy, T., Nathwani, J., Nazar, L., Sips, A., Sone, J., van den Berg, A., Weiss, P. S., & Mitra, S. (2021). Nanotechnology for a Sustainable Future: Addressing Global Challenges with the International Network4Sustainable Nanotechnology. *ACS Nano*, 15(12), 18608–18623. <https://doi.org/10.1021/acsnano.1c10919>

Punia, P., Naagar, M., Chalia, S., Dhar, R., Ravelo, B., Thakur, P., & Thakur, A. (2020). Recent advances in synthesis, characterization, and applications of nanoparticles for contaminated water treatment- A review. *Ceramics International*, 47. <https://doi.org/10.1016/j.ceramint.2020.09.050>

Rana, K., Kaur, H., Singh, N., Sithole, T., & Siwal, S. S. (2024). Graphene-based materials: Unravelling its impact in wastewater treatment for sustainable environments. *Next Materials*, 3, 100107. <https://doi.org/10.1016/j.nxmte.2024.100107>

Wang, J., Zhang, P., Liang, B., Liu, Y., Xu, T., Wang, L., Cao, B., & Pan, K. (2016). Graphene oxide as effective barrier on a porous nanofibrous membrane for water treatment. *ACS Applied Materials & Interfaces*, 8. <https://doi.org/10.1021/acsami.5b12723>

Welton, T. (2015). Solvents and sustainable chemistry. *Proceedings. Mathematical, Physical, and Engineering Sciences / The Royal Society*, 471(2183), 20150502. <https://doi.org/10.1098/rspa.2015.0502>

Yang, K., Wang, J., Chen, X., Zhao, Q., Ghaffar, A., & Chen, B. (2018). Application of Graphene-Based Materials in Water Purification: From Nanoscale to Specific Devices. *Environmental Science: Nano*, 5. <https://doi.org/10.1039/C8EN00194D>