

Nanocomposite-Engineered Polyurethane Foam for Accurate and Fast Oil-Water Separation

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ABSTRACT: The creation of effective and affordable separation materials is crucial because oil-water contamination poses serious environmental risks. In this study, reduced graphene oxide (rGO), magnetite (Fe₃O₄) nanoparticles, and recycled polystyrene (PS) from thermocol waste were combined to create a nanocomposite-modified polyurethane foam (PUF). FTIR and XRD analyses were used to confirm the formation of Fe₃O₄ and rGO/ Fe₃O₄ nanocomposites synthesized by the co-precipitation method. A dip-coating method was used to create the modified PUF, which guaranteed consistent nanomaterial deposition and improved surface hydrophobicity. The PUF structure was successfully coated with rGO/ Fe₃O₄ and PS, demonstrating successful surface modification, according to SEM-EDS analysis. Strong superhydrophobic and oleophilic properties of the composite allowed for the quick and selective adsorption of oils and organic solvents, including waste oil, engine oil, toluene, and chloroform. Chloroform showed the best separation efficiency among these.

All things considered, the rGO/ Fe₃O₄/PUF/PS composite shows great promise as a recyclable and highly effective material for oil-water separation, industrial wastewater treatment, and oil spill remediation.

KEY WORDS: Reduced graphene oxide (rGO), polyurethane foam (PUF), Fe₃O₄ nanoparticles, oil-water separation, magnetic adsorbent.

I. INTRODUCTION

Pollution levels in water bodies are rising as a result of the quick development needed to meet today's high standards of living. The marine environment is being devastated by large-scale oil spills, including those caused by tanker accidents, offshore oil drilling, and operational discharges. By poisoning, suffocating, and destroying habitat, they kill seabirds, marine mammals, and other marine life. The most prevalent pollutants in large aquatic systems, like oceans and seas, are oil spills and other contaminants [1]. Urbanization and the expansion of modern, high-energy industrial businesses are the primary causes of the massive discharge of oily waste into clean water sources. Rivers, lakes, and groundwater in residential areas are tainted by oil and grease from industrial discharges, urban runoff, and illicit used oil disposal. Because it can settle in sediments, contaminate drinking water supplies, and harm freshwater habitats, this contamination presents long-term risks to human and environmental health. Mining, transportation, refining, and oil exploration are the most frequent sources of water pollution [2-3]. Ecosystems and habitats can be destroyed by oil spills, which can have long-term negative effects on the terrestrial and marine environments. Contaminated water exposure is linked to skin irritation, neurological effects, and an increased risk of cancer. Fishing and tourism can be negatively impacted by water pollution, which can result in job losses and financial losses. The World Wildlife Fund for Nature claims that 12% of marine pollution is caused by oil spills [4]. The three main types of oil separation technologies are physical, chemical, and biological [5]. Mechanical oil skimming, chemical dispersion, bioremediation, material adsorption, and in situ burning were the main techniques used in the past to clean up oil spills. Likewise, a variety of physical and chemical processes, including ion exchange, flocculation, coagulation, ultrafiltration, and adsorption membrane filtration, are involved in the treatment of water [6]. In order to produce low-cost, effective, high-removal capacity, stable, and long-lasting materials, researchers concentrate on the physical treatment of oil-water [7]. Because oil is denser than alcohol and less dense than water, its molecules cannot pack as tightly as those of water. They need more space per unit area and are less dense. Recently, it has been discovered that oleophilic porous metals, polymers, and fibers are efficient substitutes for the selective adsorption of dyes, heavy metals, and oil. The widely accessible polyurethane (PU) sponge can be an excellent substrate because of its 3D porous structure, high surface area, high adsorption capacity, high mechanical strength, excellent chemical resistance, ease of fabrication, low density, and high stability [8]. Commercial sponges can adsorb both water and oil at the same time because they are inherently hydrophilic and oleophilic [9]. Polyurethane foam (PUF) is made oleophilic and hydrophobic by loading onto it, which reduces fouling and enhances its performance to develop desired adsorption characteristics [10]. The excellent water oil separation performance of PUF has been enhanced by the use of Fe₃O₄, PMMA, SiO₂, MoS₂, ZnO, Al₂O₃, and GO as effective separation materials [11-13]. Fe₃O₄ NPs

enhance polyurethane foam's hydrophobicity, magnetism, and foam stabilization. These nanoparticles give the system magnetic characteristics that enable recovery and recycling in response to an external magnetic field.

II. METHODS AND MATERIAL

A. Materials

Ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), ferrous chloride tetrahydrate ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$), ammonium hydroxide (NH_4OH , 25–30%), graphite powder, hydrazine hydrate, polystyrene (thermocool waste), chloroform, commercial polyurethane (PU) sponge, toluene, engine oil, motorcycle waste oil, methyl red, methyl orange, and distilled water were used exactly as received. After being dissolved in chloroform, thermocol (polystyrene) scrap was utilized as the polymer binder.

B. General Procedure for PUF Synthesis

a. Fe_3O_4 Nanoparticle Synthesis

Chemical co-precipitation was used to prepare Fe_3O_4 nanoparticles. Solutions of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (10.4 g in 20 mL DI water) and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ (4 g in 30 mL DI water) were prepared separately and combined at 90°C under nitrogen. A black Fe_3O_4 precipitate was formed when ammonium hydroxide was added dropwise until the pH reached approximately 8–9. After separation using magnetic decantation, the product was repeatedly washed with distilled water and dried at 60°C .

b. Preparation of $\text{rGO}/\text{Fe}_3\text{O}_4$ nanocomposite

Graphene oxide (GO) was prepared using a modified Hummers method. An ultrasonic bath was used to dissolve GO (150 mg) in 50 ml distilled water. After adding FeCl_3 and FeCl_2 solutions to dissolve GO while stirring magnetically, NH_4OH was added dropwise until the $\text{rGO}/\text{Fe}_3\text{O}_4$ nanocomposite was completely formed. After cleaning and magnetic separation, the product was dried in an oven.

c. Production of Modified PUF ($\text{rGO}/\text{Fe}_3\text{O}_4/\text{PUF}/\text{PS}$)

Cut into $2 \times 2 \times 1$ cm pieces, commercial polyurethane sponge was ultrasonically cleaned with acetone and distilled water before being dried at 50°C . To create a homogenous PS solution, thermocol waste (polystyrene) was dissolved in chloroform. Ultrasonication was used to disperse Fe_3O_4 or $\text{rGO}/\text{Fe}_3\text{O}_4$ nanoparticles into the PS solution. The samples were dried after the cleaned PU foams were dip-coated in the PS-nanoparticle dispersion for five minutes and any excess solution was removed. To guarantee a consistent coating of the PUF framework, the dipping-drying cycle was repeated three to four times.

d. Oil–Water Separation Testing

Oil absorption and separation efficiency were evaluated using water dyed with methyl red or methyl orange and various oils (engine oil, motorcycle waste oil, toluene, chloroform). A modified PUF piece was placed on the oil–water mixture, and absorption capacity (C_t) and separation efficiency ($\text{Eff}\%$) were calculated using:

Adsorption capacity

$$C_t = W_t - W_0 / W_0$$

Separation efficiency

$$\text{Eff} = M_t / M_0 \times 100$$

where W_0 and W_t are initial and final sponge weights, and M_0 and M_t are the initial and final masses of oil [21].



Fig.1. Representation through experimentation.
The creation of nanoparticles.



Fig.2. PUF modification ($\text{rGO}/\text{Fe}_3\text{O}_4/\text{PUF}/\text{PS}$).

REACTION



III. RESULTS AND DISCUSSION

Fe_3O_4 formation was validated by FTIR spectra that showed Fe–O stretching vibrations below 700 cm^{-1} . Remaining surface hydroxyl groups were indicated by broad O–H stretching around 3470 cm^{-1} . Characteristic Fe_3O_4 diffraction peaks were seen in XRD patterns at 30° , 35.5° , 43° , 53° , 57° , and 63° , which corresponded to the (220), (311), (400), (422), (511), and (440) planes. An additional weak peak corresponding to rGO was observed in the rGO/ Fe_3O_4 sample. Successful nanocomposite deposition along the PUF walls was shown by SEM-EDS analysis, with PS forming a thin, continuous layer over some pores. Absorption tests confirmed the composite's hydrophobic and oleophilic properties as well as its recyclability by showing quick and selective oil uptake with high separation efficiencies, especially for chloroform.

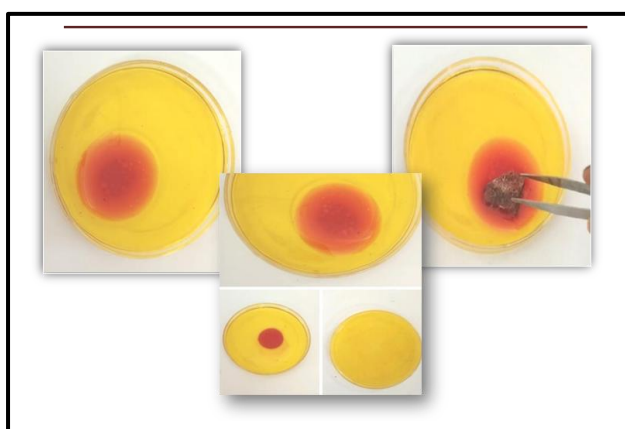


Fig.3. Absorption of engine oil from water with rGO/ Fe_3O_4 /PUF/PS.



Fig.4. Absorption of chloroform from water with rGO/ Fe_3O_4 /PUF/PS.

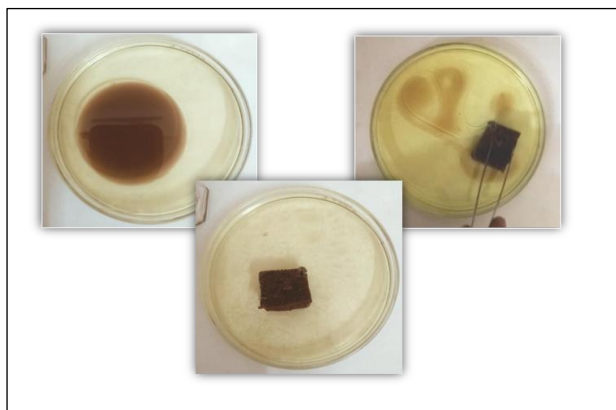


Fig.5. Absorption of waste engine oil from water using rGO/ Fe_3O_4 /PUF/PS.

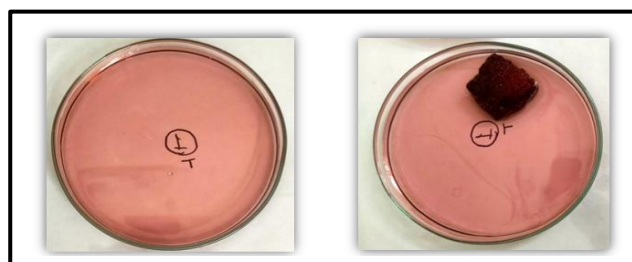


Fig.6. Absorption of Toluene from water with rGO/ Fe_3O_4 /PUF/PS.

- The absorption capacity and separation efficiency of various types of oils were calculated, and the results showed that chloroform has the highest separation efficiency.

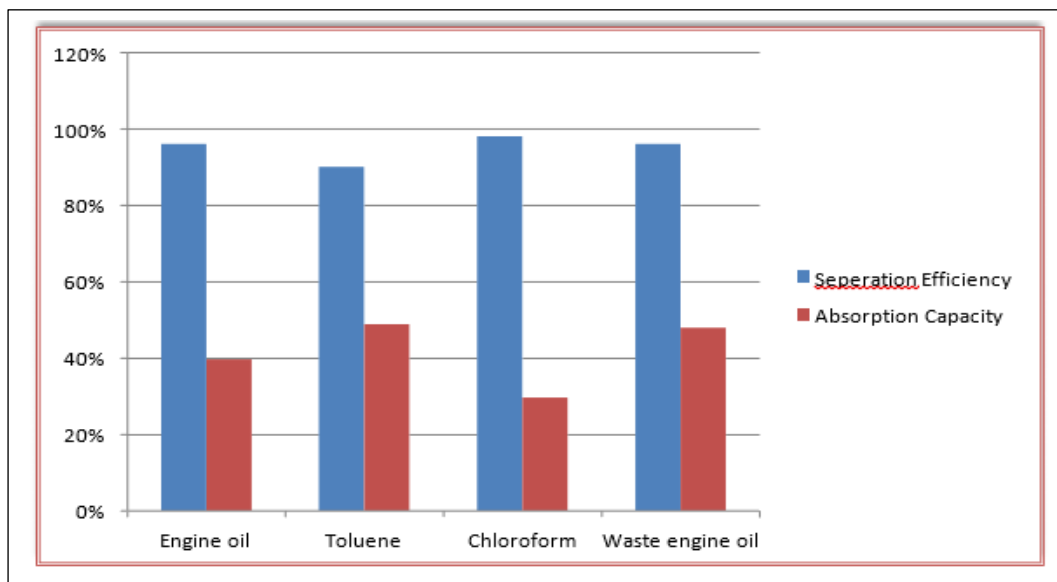
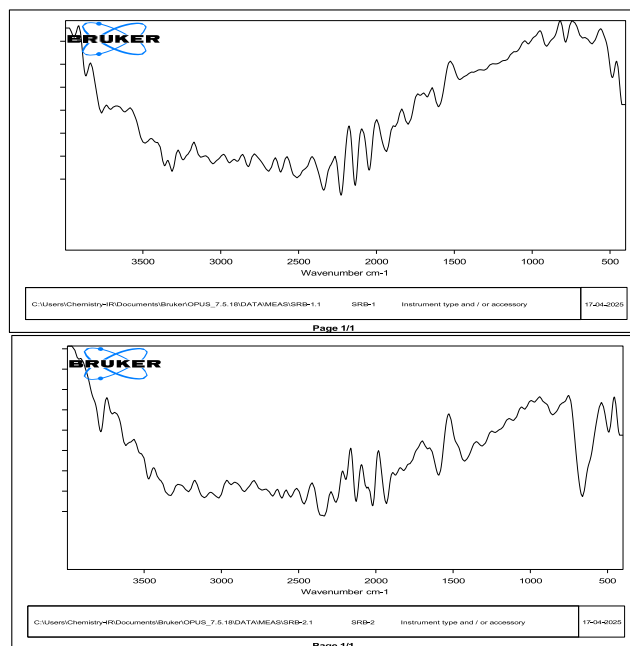


Fig.7. Separation efficiency and absorption capacity are used to separate different types of oil.

IV.CHARACTERIZATION

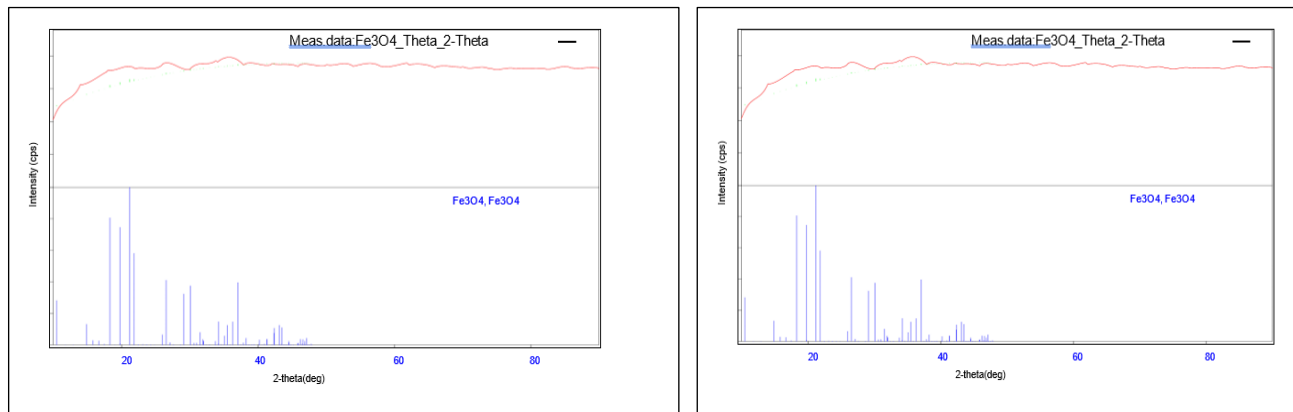
FTIR :



The FTIR spectra were recorded from 3500 to 400 cm⁻¹.

The FTIR 3470 cm⁻¹ is associated with the stretching vibration of the OH groups. The peak below 700 cm⁻¹ corresponds to the stretching vibration of Fe-O, which confirms the presence of Fe₃O₄.

- XRD patterns for Fe_3O_4 and $\text{rGO}/\text{Fe}_3\text{O}_4$.

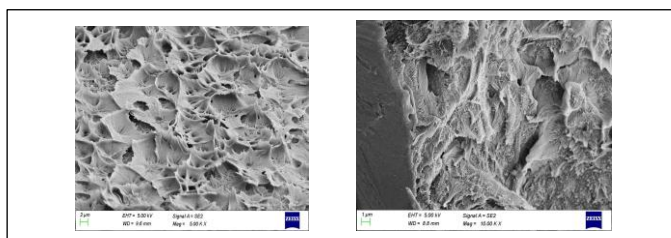


1) Fe_3O_4 (top graph) has standard peaks with strong reflection at ~ 300 , ~ 35.50 , ~ 430 , ~ 530 , ~ 570 , and ~ 630 , corresponding to the diffraction planes 220, 311, 400, 422, 511, and 440.

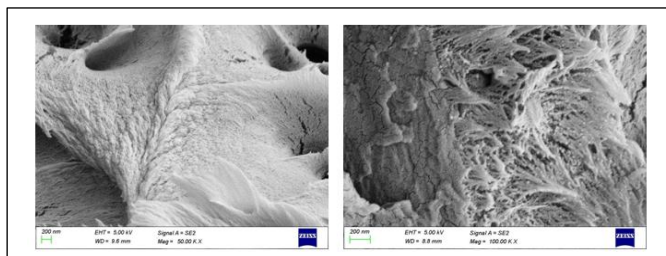
2) The estimated peak position for $\text{rGO}/\text{Fe}_3\text{O}_4$ (Bottom Graph) has standard peaks with strong reflection at ~ 180 , ~ 300 , ~ 35.50 , ~ 430 , ~ 530 , ~ 570 , ~ 630 , and ~ 10 - 120 with possible phase of Fe_3O_4 , 220, 311, 400, 422, 511, 440, and weak intensity peak of GO/rGO .

MODIFIED PUF CHARACTERIZATION

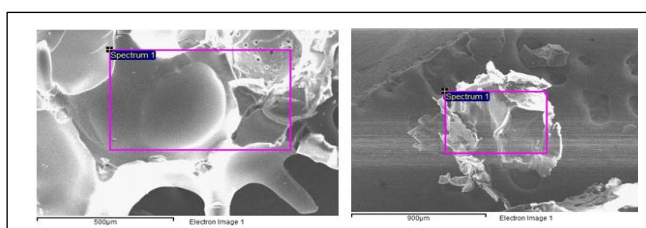
- EDS-SEM of modified PUF containing magnetic Fe_3O_4 nanoparticles and $\text{rGO}/\text{Fe}_3\text{O}_4$ nanocomposite.

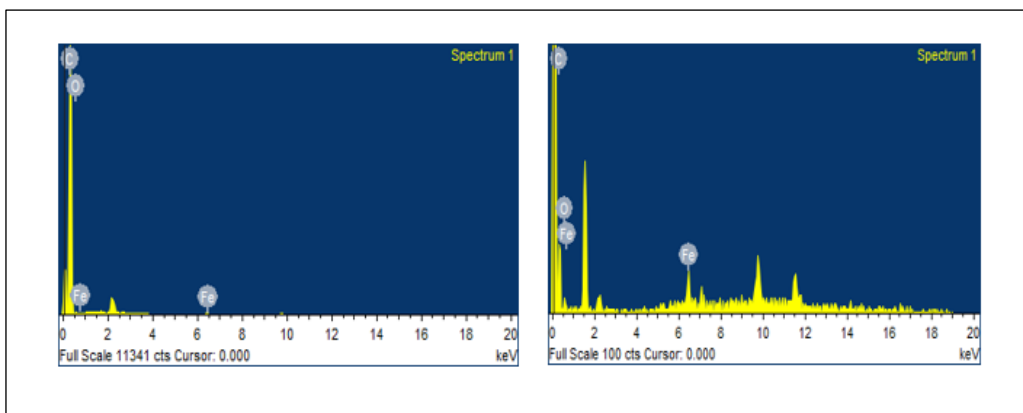


- EDS-SEM analysis of modified PUF after oil- water separation using magnetic Fe_3O_4 nanoparticles and $\text{rGO}/\text{Fe}_3\text{O}_4$ nanocomposite.



- EDS-SEM of $\text{rGO}/\text{Fe}_3\text{O}_4$ nanocomposite and modified PUF loaded with magnetic Fe_3O_4 nanoparticles.





The micrograph of rGO/Fe₃O₄/PUF/PS with oil after absorption is displayed in the EDS-SEM. The PS aims to produce a dark, continuous layer that blocks some of the pores in the PUF skeleton. The surface of the PU foam is covered by PS, but the inner pores of the rGO sheet are visible because it is transparent. The side wall and a few open PU foam pores were successfully coated with rGO/Fe₃O₄, according to the EDS-SEM. In order to achieve effective oil-water separation, the study successfully demonstrated the synthesis and characterization of a modified polyurethane foam (PUF) nanocomposite made of reduced graphene oxide (rGO), Fe₃O₄ nanoparticles, and thermocol (polystyrene, PS). Fe₃O₄ and rGO/Fe₃O₄ nanocomposites were successfully synthesized, according to FTIR and XRD analyses, with distinctive peaks confirming the existence of functional groups and crystalline structures related to these materials. EDS-SEM imaging showed that the addition of thermocol and the rGO/Fe₃O₄ nanocomposite to the PUF matrix produced a hierarchically porous structure. The uniform distribution of rGO and Fe₃O₄ on the foam surface was further verified by elemental mapping. The modified PUF composite shows promise for effective and reusable oil-water separation applications due to its combined superhydrophobicity, oleophilicity, and magnetic responsiveness.

V. CONCLUSION

This study's modified PUF shows great promise for oil-water separation. The rGO/Fe₃O₄/PUF/PS composite possessed strong hydrophobicity, oleophilicity, magnetic responsiveness, and structural stability. Successful nanoparticle synthesis, incorporation, and homogeneous surface coating were verified by characterization. High efficiency toward various oils and organic solvents was demonstrated by absorption and separation measurements, establishing this material as a viable, affordable, and eco-friendly option for wastewater treatment and oil spill cleanup.

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