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(RESEARCH ARTICLE)

Treatment of lead-contaminated water using a PVDF-expanded graphite membrane composite synthesized from carbon-based waste bottles

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Abstract

Lead contamination in water supplies poses significant risks to both environmental and human health. Simultaneously, the accumulation of waste Polyethylene Terephthalate (PET) bottles presents a persistent environmental challenge due to their slow decomposition rates. These dual concerns necessitate innovative solutions that address both water treatment and waste management. This study investigates the synthesis and application of composite membranes fabricated from expanded graphite and polyvinylidene fluoride (PVDF) derived from waste PET bottles for lead removal from contaminated water. Atomic Absorption Spectroscopy (AAS) confirmed lead presence in water samples collected from the Wewe River, while Fourier Transform Infrared (FTIR) spectroscopy characterized each adsorbent material. The lead removal efficiency was evaluated across varying concentrations of expanded graphite filler. Results demonstrated that membranes containing the highest expanded graphite concentration (10 wt.%) achieved optimal lead removal performance. This research demonstrates the environmental potential of PVDF-expanded graphite composite membranes, offering dual benefits through effective lead remediation and PET waste reduction.

Keywords: PVDF; Expanded graphite; PET; Composite membrane; Adsorption

1. Introduction

Water is essential for all living organisms, making its quality critical for survival [1]. Among heavy metal contaminants found in water, lead is of particular concern due to its inherent toxicity and disproportionate adverse health effects on children. Lead is typically found in drinking water due to the corrosion of plumbing materials, especially those containing lead [2]. In Ghana, lead in various river bodies has been attributed to illegal artisanal and small-scale mining, locally referred to as *galamsey*. These unregulated operations often involve heavy metals and chemicals, which contaminate nearby water sources [3]. Exposure to lead can lead to neurological damage, developmental delays, and other health problems [4,5]. Beyond its human health impacts, lead contamination severely damages aquatic ecosystems and biodiversity [6], and creates additional health risks through bioaccumulation in aquatic organisms [7].

The Environmental Protection Agency (EPA) has established a maximum contaminant level (MCL) for lead in drinking water at 15 parts per billion (ppb). This threshold represents the action level, beyond which water systems must implement corrective measures to reduce lead concentrations and safeguard public health [8]. The World Health Organization (WHO) recommends that lead concentrations in drinking water should not exceed 10 µg/l [9]. Therefore, treating lead-contaminated water is a crucial step in ensuring safe drinking water for communities.

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Various traditional methods are available for treating different pollutants in the environment, including lead, but these conventional methods have limitations [10]. Due to this, alternative methods for removing heavy metals such as lead from water have been developed, offering more effective and less toxic solutions with better outcomes. Key processes include coagulation, flocculation, adsorption, and membrane filtration. Among these, composite membranes have emerged as a promising technology due to their high selectivity, low fouling tendency, and easy scalability for larger applications [11]. PVDF-expanded graphite composite membranes have received significant attention due to their excellent mechanical, thermal, and chemical properties [12]. It also has promising properties for adsorption [13].

Waste PET bottles are a major contributor to global plastic pollution and pose a significant local challenge in Ghana [14]. Recycling these materials is crucial for reducing landfill and ocean waste [15]. PET waste bottles can be converted into carbon through pyrolysis, a process that involves heating a material in the absence of oxygen. The carbon produced from this process can be used as a precursor for the synthesis of expanded graphite. Using waste materials to synthesize expanded graphite has been explored in recent years as a cheap way of producing graphite. Waste PET bottles are a significant environmental problem, as they take hundreds of years to decompose and can release toxic chemicals into the environment [16]. Therefore, developing a PVDF-expanded graphite composite membrane synthesized from carbon obtained from waste PET bottles offers a promising, environmentally friendly, and economically viable solution for water treatment.

Objective

The primary objective of this research is to synthesize PVDF-expanded graphite composite membranes from waste PET bottle-derived carbon for the removal of lead from contaminated water. This approach simultaneously tackles water treatment challenges and plastic waste reduction, offering both environmental and cost-effective advantages.

2. Methodology

2.1. Research Design

The methodology adopted in this project comprised four (4) steps: synthesizing carbon from waste PET bottles, converting the carbon to expanded graphite, producing PVDF-expanded graphite membrane composites, and treating lead-contaminated water with the composite membranes.

The synthesis of carbon from waste PET bottles was carried out through a controlled pyrolysis process. Waste PET bottles were initially collected, thoroughly washed, and cleaned to eliminate impurities and unwanted residues. The cleaned PET bottles were then mechanically shredded using an industrial shredder.

The shredded particles were loaded into a high-purity ceramic crucible and placed within a tubular furnace for thermal treatment. The furnace was operated under an inert atmosphere to ensure an oxygen-free environment, thereby preventing combustion. The heating protocol involved a controlled temperature ramp at a rate of 10 °C min^{-1} until the furnace reached a target temperature of 400 °C. This temperature was maintained isothermally for 2 hours to facilitate complete carbonization of the PET material.

Upon completion of the process, the furnace was allowed to cool to room temperature under the same inert conditions. The resulting carbonized material was then carefully collected from the crucible for further characterization and utilization. This method yielded a high-purity carbon product suitable for the subsequent synthesis of expanded graphite.

2.2. Synthesis Of Expanded Graphite from Carbon

50 g of carbonized PET was placed in a crucible and subjected to thermal treatment at 1000 °C for 2 hours in an inert atmosphere to produce synthetic graphite. The resulting graphite was then immersed in concentrated nitric acid (HNO_3) and stirred thoroughly to remove residual ions or impurities. After filtration and repeated washing with deionized water, the purified synthetic graphite was dried. Two grams of the purified graphite were then added to a reaction vessel containing 80 ml of concentrated sulfuric acid (H_2SO_4) and 24 ml of concentrated nitric acid under continuous stirring. While maintaining constant stirring, 20 g of potassium permanganate ($KMnO_4$) was gradually added in small portions to control the exothermic reaction and prevent overheating. The reaction mixture was then stirred for 6 hours at a controlled temperature below 50 °C to facilitate the oxidation and intercalation of the graphite layers. Afterward, the mixture was carefully diluted with cold deionized water, and the resultant material was washed and decanted repeatedly until the filtrate reached a neutral pH, removing residual manganese by-products such as

MnO_2. The washed material was then dried at 105 °C for 24 hours in a convection oven to remove moisture completely. Once cooled to room temperature, the dried oxidized graphite was obtained as expanded graphite, characterized by FTIR.

2.3. Production Of PVDF-Expanded Graphite Composite Membrane

To produce the composite membrane, 100 ml 20 wt.% PVDF solution was first prepared by dissolving 20 g of polyvinylidene fluoride (PVDF) in 80 ml of dimethylformamide (DMF). The solution was stirred on a magnetic stirrer for 12 hours at room temperature to ensure complete dissolution and homogeneity. For membrane fabrication, two concentrations of expanded graphite (EG) were incorporated into the PVDF solution to create composite membranes with 5 wt.% and 10 wt.% EG content. Specifically, 0.5 g of expanded graphite was added to 10 ml of the 20 wt.% PVDF-DMF solution for the 5 wt.% composite, and 1 g of expanded graphite was added to 10 ml of the 20 wt.% PVDF-DMF solution for the 10 wt.% composite. In both cases, the mixture was stirred for an additional 12 hours to achieve uniform dispersion of the graphite within the polymer matrix. Another composite membrane was also fabricated from pure PVDF, without expanded graphite, to serve as a control.

The composite solution was then cast onto a flat glass surface, with the aid of a rod to evenly distribute the material and form a uniform membrane. The cast membrane was immersed in deionized water and allowed to remain in the water bath for 48 hours to facilitate phase inversion and solvent removal. Afterward, the PVDF-Expanded Graphite composite membrane was retrieved from the water bath and air-dried at room temperature until fully dried, yielding a stable and homogeneous composite membrane.

2.4. Treatment Of Lead Contaminated Water

Water samples from the Wewe River near KNUST were collected and subjected to digestion and characterization to determine the initial concentration of lead. The treatment process employed adsorption, using PVDF-expanded graphite membrane composites as the adsorbent material. For the adsorption experiment, 100 ml of the water sample was transferred into two glass jars. A single membrane composite, 5 wt.% or 10 wt.% expanded graphite, was placed into each jar. The jars were agitated at controlled speeds for varying contact times of 5, 10, 15, 20, 30, and 60 minutes to assess the adsorption kinetics, which follow fundamental surface interaction principles [17]. At the end of each specified contact time, 10 ml of the water sample was extracted from the jars, transferred into labelled sample tubes, and stored for further analysis. The membranes were carefully removed after the experiment for post-adsorption characterization. Atomic Absorption Spectroscopy (AAS) was used to analyse the collected water samples and determine the remaining lead concentration. Fourier Transform Infrared Spectroscopy (FTIR) was also employed to characterize the membranes post-adsorption, evaluating changes in their surface functional groups. The lead removal efficiency for each membrane composite was calculated based on the reduction in lead concentration measured by AAS. This process provided insights into the performance of the PVDF-expanded graphite membrane composites in the adsorption of lead from contaminated water.

3. Results and discussion

3.1. Characterization Of Synthesized Carbon from Waste PET Bottles

Following the synthesis of carbon from waste PET bottles, a Fourier Transform Infrared (FTIR) analysis was performed on the resulting product to confirm the successful conversion of PET into carbon. Figure 1 presents the FTIR spectrum obtained from this analysis, which provides evidence of the functional groups present and validates the formation of carbon-based material.

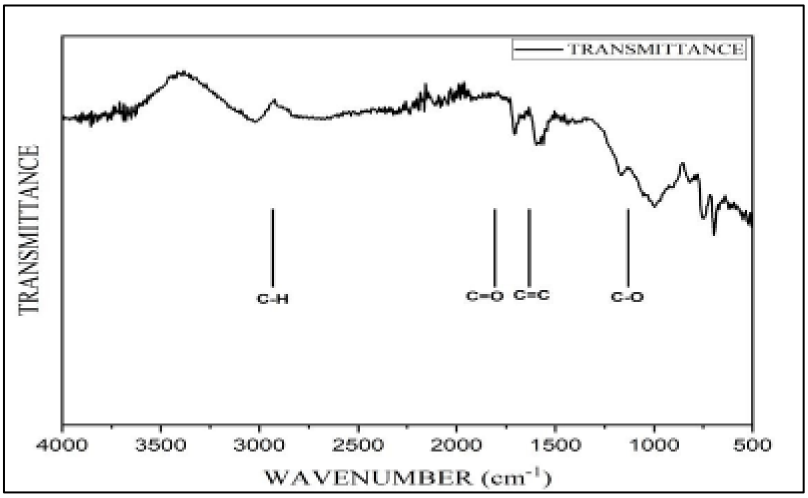

Figure 1 FTIR spectra for carbon sample

The FTIR spectrum of the carbon samples synthesized from waste PET bottles provides insights into the functional groups present on the carbon material surface. The peak display at 2923 cm^{-1} corresponds to the stretching vibration of aliphatic C–H bonds, which suggests the presence of residual hydrocarbon chains or incomplete decomposition of the PET during pyrolysis. The peak displays at 1791 cm^{-1} and 1622 cm^{-1} indicates the presence of carbonyl groups (C=O), which could arise from the oxidation of the carbon material and then aromatic (C=C) bonds, which are characteristic of graphitic or aromatic structures formed during the carbonization process. It reflects the development of sp^2 hybridized carbon domains in the material. Finally, the peak at 1140 cm^{-1} corresponds to the stretching vibration of C–O bonds, likely originating from oxygenated functional groups such as ethers or alcohols on the carbon surface. These groups could form during synthesis.

The FTIR spectrum shows that the carbon samples contain a variety of functional groups, including aliphatic hydrocarbons (C–H), oxygenated groups such as carbonyl (C=O) and ether/alcohol (C–O), as well as aromatic structures (C=C). These results indicate that while the PET bottles were effectively carbonized, the resulting carbon retains some oxygen-containing surface groups. These surface functionalities can improve the material's chemical reactivity and make it more effective for applications such as the adsorption of contaminants from water. Additionally, the presence of these groups provides opportunities for further surface modification to tailor the material for specific uses.

3.2. Characterization of Sample Water

The water sample was collected from the *Wewe River* in July 2024. To identify the concentration of lead in the sample water, an atomic adsorption spectroscopy (AAS) test was conducted, and the result is shown in Table 1.

Table 1 Concentration of lead in sample water

Element	Concentration (mg/l)
Lead	0.4963

The concentration of lead (pb) in the sampled water was measured at 0.4693 mg/l, which significantly exceeds the maximum permissible limits for lead in water as established by the World Health Organization (WHO) and the Environmental Protection Agency (EPA). These regulatory bodies recommend a maximum allowable concentration of 0.01 mg/l (WHO) and 0.015 mg/l (EPA), indicating that the water is contaminated with lead to a level that poses serious health risks and requires treatment. Contamination levels higher than the maximum allowable concentration levels have also been reported in other Ghanaian rivers [18].

3.3. Final Concentrations of Lead

After the adsorption experiment using the three sets of adsorbents under varying contact times, the resulting water samples were analysed using Atomic Absorption Spectroscopy (AAS) to determine the final concentrations of lead (Pb) in each sample. The results, summarized in Table 2, show the final concentrations of lead in the water for the control, 5 wt.% expanded graphite membrane, and 10 wt.% expanded graphite membrane at each contact time.

Table 2 Final concentrations of water in each adsorbent at each contact time

Time (min)	Control (mg/l)	5wt% (mg/l)	10wt% (mg/l)
5	0.1657	0.1321	0.0236
10	0.1544	0.1041	0.0193
15	0.1915	0.1011	0.0106
20	0.1119	0.0921	0.0095
30	0.1025	0.1084	0.0136
60	0.2015	0.1534	0.0184

The results show that the adsorbent containing 10 wt.% expanded graphite consistently performed better than the 5 wt.%. expanded graphite membrane and the control (no adsorbent). Across most contact times, the lead concentration in water treated with the 10 wt.% membrane was significantly reduced, with several values falling below the maximum allowable lead concentration in water set by the World Health Organization (WHO) (0.01 mg/l) and the Environmental Protection Agency (EPA) (0.015 mg/l) [8].

In particular, the 10 wt.% membrane achieved the lowest lead concentrations, with a final concentration of 0.0095 mg/l at 20 minutes, below the WHO and EPA thresholds. In contrast, the 5 wt.% membrane showed moderate adsorption efficiency, with lead concentrations ranging between 0.0921 mg/l and 0.1534 mg/l, higher than the allowable limits. The control, lacking any adsorbent, exhibited minimal reduction in lead concentration, maintaining levels between 0.1025 mg/l and 0.2015 mg/l, far exceeding the regulatory standards.

These results highlight the enhanced adsorption capacity of the 10 wt.% expanded graphite membrane, attributed to the higher concentration of active adsorption sites and improved surface area. The 5 wt.% membrane, while moderately effective, demonstrated less efficient removal of lead, suggesting that increasing the expanded graphite content enhances the membrane's adsorption performance. Overall, the 10 wt.% expanded graphite membrane proved to be the most effective in reducing lead contamination to safe levels within shorter contact times.

3.4. Adsorption Efficiencies

To evaluate the performance of the adsorbents, the adsorption efficiency (%) was calculated at each time interval using the initial lead concentration in the sample water (0.4693 mg/l) and the final concentrations obtained after the adsorption experiment. The adsorption efficiency was computed using the formula:

$$\text{Adsorption Efficiency (\%)} = \frac{C_i - C_f}{C_i} \times 100$$

Where:
C_i = Initial lead concentration (0.4693 mg/l)
C_f = Final lead concentration (mg/l)

Table 3 Adsorption Efficiencies of Control, 5wt% and 10wt% Adsorbents

Time	Control (%)	5wt% (%)	10wt% (%)
5	64.7	71.9	94.9
10	67.1	77.8	95.9
15	72.0	78.5	97.7
20	76.2	80.4	97.9
30	78.2	76.9	97.1
60	57.1	67.3	96.1

Table 3 demonstrates the adsorption efficiency trends for the control, 5 wt.%, and 10 wt.% expanded graphite membranes. The 10 wt.% membrane consistently achieved the highest efficiencies at all contact times, confirming its superior lead adsorption capacity compared to the 5 wt.% membrane and the control. The data highlights the potential of the PVDF-expanded graphite composite membranes, particularly the 10 wt.% concentration, for effective lead remediation from contaminated water. These values were used to plot a graph of adsorption efficiencies against contact time, as shown in Figure 2.

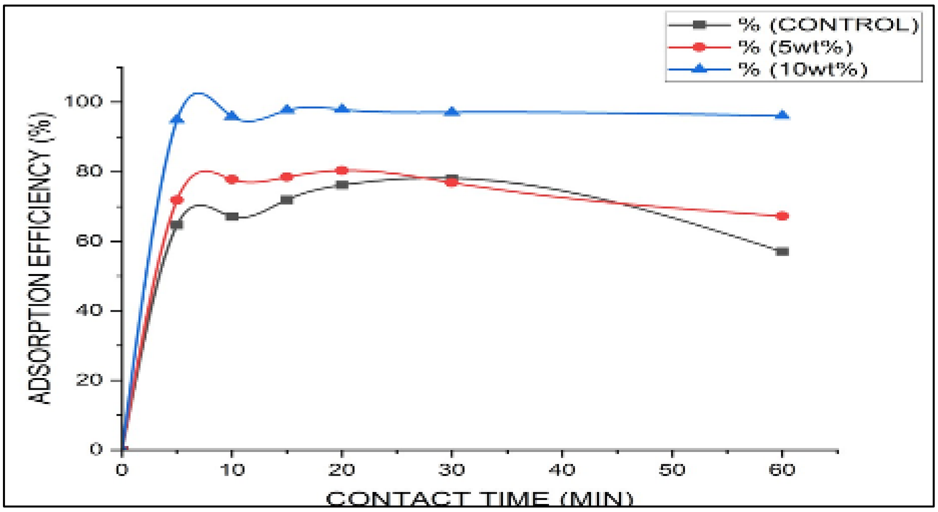

Figure 2 A plot of adsorption efficiency against contact time

The data presented in Figure 2 demonstrate that the 10 wt.% expanded graphite composite adsorbent had the highest adsorption efficiency, followed by the 5 wt.% composite, with the control (adsorbent without filler) exhibiting the lowest efficiency. This trend highlights the enhanced adsorption capabilities imparted by the addition of expanded graphite.

At the initial stages of the adsorption process, the efficiencies for all adsorbents increased with contact time. This can be attributed to the high availability of active adsorption sites on the adsorbent surfaces, allowing many lead particles to bind efficiently.

After 30 minutes, the adsorption efficiency of both the 5wt% filler adsorbent and the control adsorbent began to decrease. This resulted from saturation of adsorption sites present in the mentioned adsorbents. This meant little or no lead particles could be further adsorbed, accounting for the reduction in the efficiency values. Further exhibition of this behaviour eventually leads to desorption, which involves the release of adsorbed lead particles back into the water samples. This leads to a further decline in adsorption efficiency over time.

In contrast, the 10 wt.% expanded graphite composite adsorbent exhibited consistently high adsorption efficiency throughout the contact time. This superior performance is attributed to the higher concentration of expanded graphite in the composite, which provides a significantly greater number of active adsorption sites. The increased surface area and pore volume facilitated a higher lead adsorption capacity, effectively preventing saturation and desorption within the tested time intervals.

The results show that the 10 wt.% expanded graphite composite adsorbent offers the best performance for lead removal due to its enhanced adsorption capacity and stability over time. This makes it the most effective option among the tested adsorbents for treating lead-contaminated water.

3.5. Characterization Of Best Performing Membrane (Adsorbent)

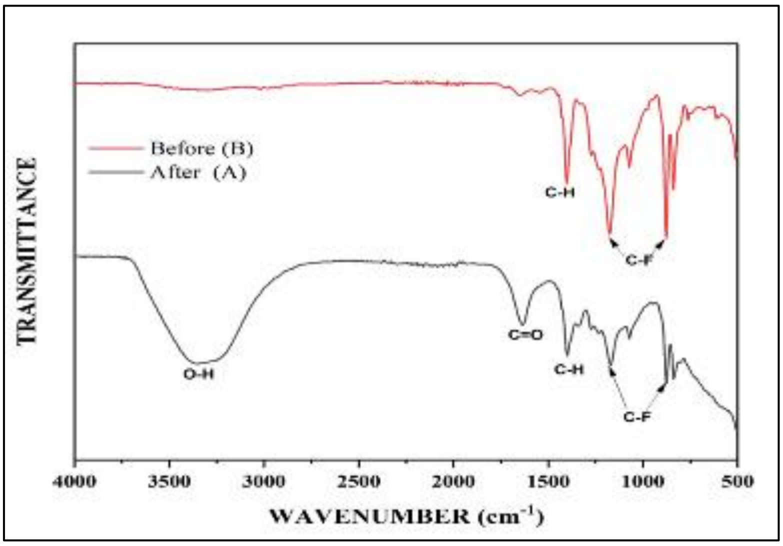

Figure 3 FTIR Spectra of 10wt% Adsorbent Before and After Adsorption

To understand the changes in chemical composition and structure resulting from the adsorption process, Fourier Transform Infrared (FTIR) spectroscopy was conducted on the best-performing adsorbent, the 10 wt.% expanded graphite composite membrane, both before and after adsorption. The resulting spectra are presented in Figure 3.

3.6. Before Adsorption

The FTIR spectrum of the membrane, before adsorption, revealed characteristic peaks associated with its composite components, particularly PVDF and expanded graphite:

- 3018 cm^{-1}: A peak corresponding to C–H stretching vibrations, confirming the presence of aliphatic groups in PVDF.
- 2019 cm^{-1}: A peak indicative of C=O stretching vibrations, attributed to carbonyl groups within the PVDF matrix.
- 1655 cm^{-1}: A peak associated with C=C stretching vibrations, characteristic of the graphite component within the composite.
- 1401 cm^{-1}: C–H bending vibrations, further validating the presence of PVDF.
- 1232 cm^{-1} and 1170 cm^{-1}: Peaks assigned to C–F stretching vibrations, signifying the presence of fluorine atoms in the PVDF structure.
- 876 cm^{-1}: A C–F bending vibration, reinforcing the presence of fluorinated bonds in the membrane.
- 600 cm^{-1} and 508 cm^{-1}: Peaks corresponding to C–H bending vibrations, linked to the graphite component.
- 478 cm^{-1} and 430 cm^{-1}: C–F bending vibrations, further confirming the presence of fluorinated species. [19].

The observed peaks align with known functional groups of PVDF and graphite, confirming the integrity of the composite structure before adsorption.

3.7. After Adsorption

The FTIR spectrum after adsorption revealed distinct changes, indicating the adsorption of lead and the interaction of the membrane with water molecules:

- 3350 cm^{-1}: A broad peak attributed to O–H stretching vibrations, signifying water adsorption onto the membrane surface.
- 1634.28 cm^{-1}: A peak associated with C=O stretching vibrations, which indicates polar bonds formed due to the complexation of lead ions during adsorption.
- 1399.71 cm^{-1}: A peak corresponding to C–H bending vibrations, confirming the presence of carbon within the composite.

- 1232.20 cm^{-1} and 875.65 cm^{-1}: Peaks corresponding to polar C–F bonds, indicating the retained presence of fluorinated groups in the membrane structure. [19].

The spectral changes confirm the adsorption of lead onto the membrane surface, evidenced by the appearance of new peaks (e.g., O–H and C=O) and shifts in existing peaks. The results suggest that the 10 wt.% expanded graphite composite effectively interacts with lead ions and retains its structural integrity post-adsorption. This chemical evidence supports the membrane's high adsorption efficiency and potential for water treatment applications.

4. Conclusion and recommendation

The research objectives were successfully accomplished, demonstrating that PVDF-expanded graphite composite membranes synthesized from waste PET bottles can effectively remove lead from contaminated water systems. Incorporating expanded graphite into the PVDF matrix produced composite membranes with enhanced adsorption properties through the synergistic combination of PVDF's chemical stability and flexibility with graphite's high adsorption capacity and conductivity. The 10 wt.% expanded graphite composite membrane exhibited superior performance, achieving significantly higher lead removal efficiency than the 5 wt.% membrane and the control. This was attributed to the increased availability of active adsorption sites provided by higher graphite content, which also mitigated issues of site saturation and desorption observed in the other adsorbents. The FTIR analysis confirmed the structural integrity of the composite membranes and highlighted changes in functional groups post-adsorption, providing further evidence of successful lead interaction and removal. The results validate the effectiveness of the composite membranes in addressing heavy metal contamination and underscore their potential as a sustainable water treatment solution. Additionally, this project demonstrated a dual environmental benefit by contributing to water purification while addressing plastic waste management. The upcycling of waste PET bottles into functional composite membranes represents a step forward in achieving cleaner water resources and reducing environmental plastic pollution. These findings provide a foundation for further research and optimization of composite membranes for broader applications in water treatment.

Compliance with ethical standards

Acknowledgments

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Disclosure of conflict of interest

No conflict of interest has been declared by the authors.

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