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Magnetically Modified Carbonized Plant-Derived Sorbents for Efficient Oil Adsorption and Recovery

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Abstract

The elimination of organic pollutants and oil spills remains one of the major environmental challenges of our time. Among the various remediation techniques, the sorption method is considered the most efficient for removing oil from water surfaces. In this study, organophilic carbon-based oil adsorbents were synthesized via carbonization of plant-derived raw materials, peanut shells and walnut wood, in an argon atmosphere at 600 °C. Additionally, magnetite-modified carbonized composites of these materials were prepared. The adsorbents were characterized using scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), Brunauer–Emmett–Teller (BET) surface area analysis, and Barrett–Joyner–Halenda (BJH) pore size distribution. FTIR spectra confirmed the presence of Fe–O bonds, indicating successful incorporation of magnetite within the composites. The formation of magnetite in the pores of the carbon matrix resulted in a reduction of specific surface area, from 1696.20 m^2/g to 1053.47 m^2/g for peanut shell carbon, and from 1533.60 m^2/g to 1033.91 m^2/g for walnut wood carbon. Oil adsorption studies revealed adsorption capacities of 3.8 g/g and 3.6 g/g for the carbonized peanut shell and walnut wood, respectively, and 4.3 g/g and 4.8 g/g for their corresponding magnetite composites. The enhanced oil adsorption performance of the magnetic composites is attributed to an increased proportion of macropores (50–200 nm), which facilitate oil uptake in emulsion systems.

Keywords: Oil adsorption; Carbonization; Peanut shell; Walnut wood; Magnetite composite.
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1. Introduction

Environmental pollution caused by organic contaminants has emerged as one of the most demanding issues of times. Oil spills among these stand out as acute, complex threats to earthly, freshwater and marine environment alike, their effect goes beyond the immediate slick to chain reaction on biodiversity, biogeochemical cycles and human health. According to existing assessments, from 1970 to 2017 over 5.7 million tonnes of oil entered the world's oceans through accidents and spills.[1] This statistic contradicts the inordinate harm even driblets of oil can cause: a thin film on the water surface, wound down to monomolecular thickness, is sufficient for oxygen transfer to be restricted in the water column and thus dis tort aquatic respiration. In addition, transmission of light into water is significantly altered by these surface films, diminishing photosynthetic activity in phytoplankton and submerged macrophytes and thereby altering food-web dynamics and declining primary productivity. Similarly, chronic ecological risks develop long after the visible spill has been cleaned, as many of the hydrocarbons released including persistent polycyclic aromatic hydrocarbons (PAHs) and other heavy oil fractions bioaccumulate in aquatic organisms and biomagnify through trophic levels. The environmental fate and behavior of spilled oil are still ambiguous in many contexts, as confirmed by modern literature. A detailed review in 2024 highlights how oil influences not only marine life but also the soils, vegetation and wetlands adjacent to the spill sites, highlighting the multifaceted nature of hydrocarbon pollution.[2] Concurrently, a 2025 synthesis of Asian marine disasters laid emphasis on how spills can provoke cross-boundary ecological and socio-economic outcomes and are not merely local events, worsened by weak governance and climate-driven changes in current and wind regimes.[3] These insights strengthen the necessity of developing sorbents and remediation technologies that can deliver an expeditious and extensive response. Compounding the problem, the vertical and horizontal transport of spilled oil is strongly impacted by weather, sea currents, waves and oil

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type, so that predicting the full ecological footprint is far from negligible.[4] For example, lighter oil fractions can be dispersed by waves into the water column or sink to the seabed, while heavier residuals may persist as submerged oil mats for years, slowly releasing toxic compounds and degrading ecosystem function. Field evidence from 2025 confirms that episodic spill events still trigger long-lasting contamination of sediments and wildlife, including coral, benthic invertebrates and large marine vertebrates.[5] In short, oil spills are not only acute disasters amenable to short-term clean-up; they often set off a cascade of persistent, hard-to-reverse impacts that demand proactive, durable remediation approaches. Various methods have been proposed to solve the problem of water purification from oil and oil products, such as burning oil on the surface of the water,[6,7] separating oil from water with the help of mineral sorbents,[8,9] polymers,[10,11] microorganisms,[12,13] wool and other absorbing materials.[14-17] However, burning oil on the water surface produces a large smoke plume and viscous sludge in water, and the spilt oil needs to be at least 3–5 mm thick to initiate and support the combustion process.[18] Many superhydrophobic sorbent materials are obtained by additional efforts to modify their surfaces.[19-21] Polymers, organic compounds, dispersed particles, and acids and alkalis can be used as modifiers. Remediation of oil-contaminated soils using bacteria and plants requires a long period, which is not commonly used to treat oil contamination. Among the methods of water treatment from oil, the sorption method is the simplest, most efficient, and environmentally friendly approach, but there are certain requirements for sorbents. They should have high sorption capacity, mechanical strength, chemical stability, and be cheap and readily available.[22-24] These requirements are well met by carbonized materials derived from plant biomass, which provide an abundant and renewable source of precursors for the fabrication of sorbents with high porosity and hydrophobicity. Bio-based sorbents have drawn significant attention due to their low cost, biodegradability, and the possibility of surface tuning through physical and chemical activation. The literature describes a large number of sorbents obtained by carbonization of various parts of plants and wastes of plant food products.[25-29]

Recent advances show that agricultural residues such as rice husks, coconut shells, sawdust, and corn stalks can be transformed into biochars with tailored surface area and pore structures suitable for oil adsorption.[30,31] Since the affinity of the adsorbate to the adsorbent surface is of great importance in the sorption process, those types of plant waste whose fruits are characterized by a high-fat or lignocellulosic content are of particular interest, as they yield carbon materials with high surface activity and hydrophobic character after carbonization. In addition, the physicochemical structure of biomass-derived carbon depends strongly on the activation temperature, atmosphere, and precursor composition, allowing tunable porosity and surface chemistry for selective adsorption of hydrophobic pollutants.[32-34] On the other hand, it is no less important to enhance the functionality of such sorbents by imparting magnetic properties, which enable easy recovery and reuse after the adsorption process. The inclusion of magnetic particles into the structure of carbonized sorbents could fasten their separation from water.[35] Synthesis of magnetite within the pores of carbonized materials combines the high adsorption capacity of biochar with the magnetic separability of Fe_3O_4 nanoparticles.[36] Such magnetic biochar composites have become an effective and sustainable strategy for the removal of dyes, ions, and oil from water.[37] Several studies conducted show that Fe_3O_4-modified carbon materials exhibit increased pore accessibility, enhanced surface roughness, and improved wettability balance, thereby improving oil sorption efficiency.[38,39] Additionally, the use of these magnetic sorbents can be regenerated several times through mild solvent washing and magnetic recovery, which improves their practical deployment and cost-effectiveness in large-scale oil spill response.

In this regard, our current study aim to create oil sorbents using walnut tree branches and peanut shells, as well as their magnetic composites. The availability of these plants materials as agro-wastes, their relatively high lignin and fatty compound content, and their capacity to form porous carbon structures when heated motivated our choice of these plants materials. To establish their potential efficient, magnetically retrievable oil sorbents for environmental remediation application, the resulting solvents were characterized using Brunauer-Emmett-Teller (BET) surface area analysis, Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and their oil adsorption behavior.

2. Materials and methods

2.1 Preparation of carbonized sorbents

The carbonized sorbents were prepared using the peanuts shells and walnut tree branches as biomass precursors. There peanuts shells were cut into pieces of 0.3-1.0 cm whereas the walnut branches with diameter ranging from 0.5-0.9 cm were cut to cylindrical segments of 1.0-1.5 cm before being chopped. Both materials were thoroughly washed with deionized water and oven-dried at 80-90 ºC. The dried materials were treated with 4 wt.% of NaOH solution for 24 h at room temperature (25 ºC) to enhance porosity and remove impurities. They were then washed with deionized water until the pH was neutralized and dried once again at 80-90 ºC. Carbonization was conducted at 600 ºC for one hour in an argon atmosphere with an argon flow rate of 3 L min^{-1}. The subsequent carbonized materials were reheated at 600 ºC after chemically activating the KOH at a mass ratio of 2:1 (KOH: carbon). Before usage, the activated carbons were rinsed with

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deionized water until they were dried and neutral.

2.2 Preparation of magnetic composites

Fe_3O_4 nanoparticles were incorporated into carbonized sorbents to synthesize the magnetic composites. 20 g of dried plant material was dispersed into 100 mL of deionized water and stirred for1 hour at 25 ºC while mixing 1 mol L^{-1} $FeCl_3$. To enhance the magnetic formation, 1 mol L^{-1} $FeCl_2$ solution was added dropwise with 15% of ammonia present, adjusting the pH to 9.3-9.5.

2.3 Oil adsorption experiments

For adsorption studies, crude oil obtained from the Munaily field (Atyrau region, Kazakhstan) was used. The oil obtained had a density of 867.2 kg.m^{-3} and contained 36.0 wt.% resins, 1.3 wt.% paraffin, and 0.3 wt.% sulfur. An oil-water combination (oil/water mass ratio = 3:1) was mixed with 1 g of adsorbent and stirred for 1 hour at 25 ºC. The sorbent was then removed, and the residual oil content was measured gravimetrically. The oil adsorption capacity (A, g g^{-1}) was calculated using:

A = $(m_1\text{-}m_2)\ M^{-1}$, where M is the mass of the adsorbent (g), and m_1 and m_2 are the masses of oil before and after adsorption (g).

The oil recovery efficiency (R, %) was determined as: R = $(m_1\text{-}m_2)$ 100, where m_1 and m_2 are the masses of oil before and after adsorption (g).

2.4 Characterization

The chemical and structural of the magnetic and carbonized sorbents were analyzed using the Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM). After grinding the samples to particles smaller than 1 mm, the samples are coated with a thin gold layer to enhance its conductivity and then the surface morphology was analyzed on a JSM-6490LA scanning electron microscope (JEOL, Japan). The FTIR spectrometer (Shimadzu, Japan) is used to identify the functional groups present on the surfaces of the sorbent. For each measurement, KBr pellets containing finely ground sorbent powder were prepared, and spectra were recorded within the wavenumber range of 3500–500 cm^{-1}.

3. Results and discussion

The surface morphologies of the carbonized sorbents derived from peanut shells and walnut tree branches were examined using scanning electron microscopy (SEM), as presented in Fig. 1 (a–f). A well-developed porous framework typical of biochars produced from lignocellulosic biomass is exhibited my both materials. The carbonized peanut shell (Fig. 1a, b) displays a network of large, elongated pores ranging from approximately 7.9 µm to 51.0 µm, originating from the natural cellular channels of the peanut hull structure. These pores originate from the peanut hull structure's intrinsic cellular channels. During adsorption, these networked macropores help to promote oil diffusion and penetration. As a result of its denser lignocellulosic matrix, the sorbent generated from walnut trees (Fig. 1c, d) exhibits smaller and more irregular pore geometries, with pore diameters ranging from 0.6 µm to 11.1 µm. Increased surface reactivity is probably a result of the partial structural collapse of cellulose and hemicellulose components during carbonization, as evidenced by the rough, fragmented texture seen on the carbonized surface. Different granular deposits of Fe_3O_4 nanoparticles are visible scattered throughout the carbon matrix after magnetite is added and carbonized (Fig. 1e, f). Many open macropores are still visible despite some magnetite particles partially blocking smaller pores, indicating that overall porosity was unaffected by the impregnation process. The presence of magnetite contributes

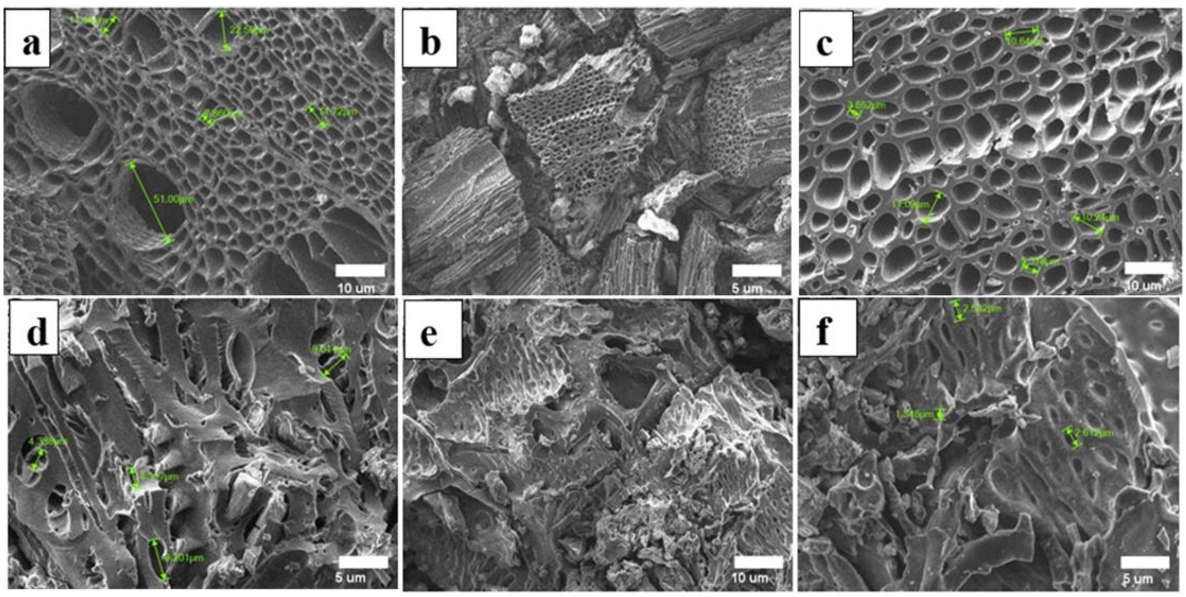

Fig. 1: SEM image of carbonised sorbents: peanut shell (a), composite peanut shell-magnetite (b, c), walnut tree (d), composite walnut tree-magnetite (e, f).

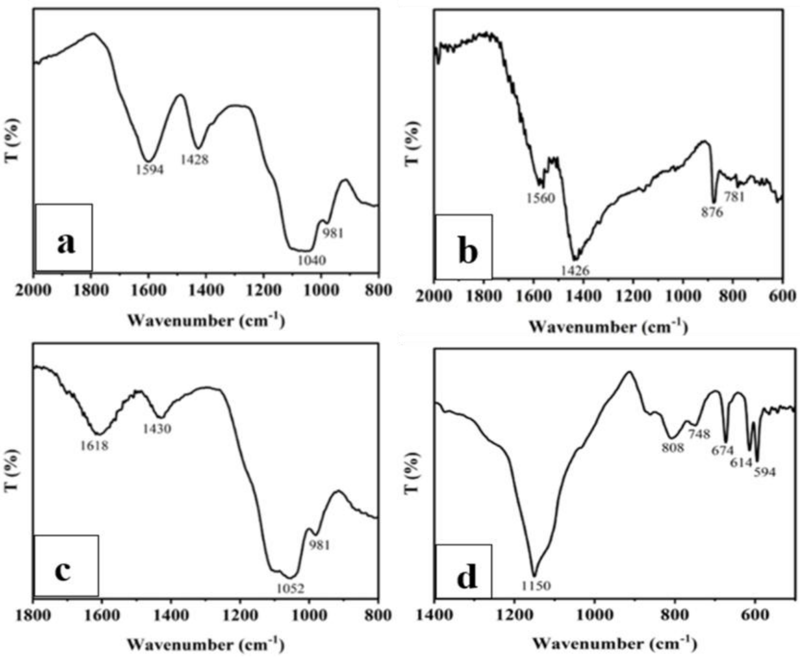

Fig. 2: FTIR spectra of carbonised sorbents: peanut shell (a), composite peanut shell-magnetite (b), walnut tree (c), composite walnut tree-magnetite (d)

to increased surface roughness and heterogeneity, characteristics that can improve the sorbent's interfacial interaction with hydrophobic oil droplets. Overall, the SEM observations confirm that both peanut shell– and walnut tree–based sorbents retain a hierarchical porous structure after carbonization, with magnetite-modified composites exhibiting a well-integrated dual morphology, porous carbon channels combined with evenly distributed magnetic domains, favorable for enhanced adsorption and facile magnetic recovery.

The chemical functionalities of the carbonized peanut shell and walnut tree–based sorbents were analyzed using Fourier transform infrared (FTIR) spectroscopy, as shown in Fig. 2 (a–d). The FTIR spectrum of the carbonized peanut shell (Fig. 2a) exhibits characteristic absorption peaks at 1594 cm^{-1}, 1428 cm^{-1}, 1040 cm^{-1}, and 981 cm^{-1}. The bands at 1594 cm^{-1} and 1428 cm^{-1} correspond to C–H stretching vibrations and possible N–H bending of protein residues, while the absorption at 1040 cm^{-1} can be attributed to C=O stretching of carbonyl and carboxylic groups originating from carbohydrates and fatty acids. The minor band near 981 cm^{-1} may indicate the presence of C–O–C linkages of polysaccharide backbones. After magnetite incorporation (Fig. 2b), the peanut-shell composite shows peak shifts to 1560 cm^{-1} and 1426 cm^{-1}, suggesting interaction between Fe_3O_4 and the oxygen-containing surface groups of the carbon matrix. New absorption bands at 876 cm^{-1} and 781 cm^{-1} appear, corresponding to the Fe–O stretching vibrations of magnetite, confirming successful magnetic modification of the carbon framework.

Similarly, the FTIR spectrum of the carbonized walnut tree sorbent (Fig. 2c) displays bands at 1618 cm^{-1}, 1430 cm^{-1}, 1052 cm^{-1}, and 981 cm^{-1}, which are associated with C=C stretching of aromatic structures, C–H deformation of aliphatic chains, and C=O stretching of cellulose-derived carbonyl groups. Upon magnetite synthesis (Fig. 2d), these characteristic peaks shift and broaden, with a new prominent band emerging at 1150 cm^{-1}, reflecting alterations in surface oxygen functionalities due to iron oxide deposition. Additional absorption features at 808 cm^{-1}, 748 cm^{-1}, 674 cm^{-1}, 614 cm^{-1}, and 594 cm^{-1} correspond to Fe–O lattice vibrations of magnetite,[36] further verifying its successful incorporation within the carbon matrix.

The carbonized sorbents made from peanut shells and walnut tree branches, along with their magnetic composites, were tested for oil adsorption behavior at 25 °C. The resulting isotherms are shown in Fig. 3 (a–b). Both materials have classic Type II adsorption isotherms, which are marked by a plateau zone after an initial sharp climb. The strong affinity of the sorbent surface for hydrophobic oil molecules and the abundance of active sites are responsible for the quick rise in adsorption at low oil dosages. The plateau area shows that the sorbent surface has been saturated and that adsorption equilibrium has been reached. As illustrated in Fig. 3, the carbonized sorbents (CS) demonstrate maximum oil adsorption capacities of 3.8 g/g for the peanut shell–based sample and 3.6 g/g for the walnut tree–based sample. The corresponding magnetic composites (CSM) show enhanced performance, with adsorption capacities of 4.3 g/g and 4.8 g/g, respectively. The improvement in adsorption after magnetite incorporation can be attributed to an increase in surface roughness and the creation of additional interfacial sites that promote oil droplet adherence and capillary uptake. For biochar-based sorbents made from agricultural wastes, the observed adsorption capabilities are in line with published data; they typically range from 2.5 g g^{-1} to 11.2 g g^{-1} for oil & petroleum compounds.[36,37] Through surface heterogeneity

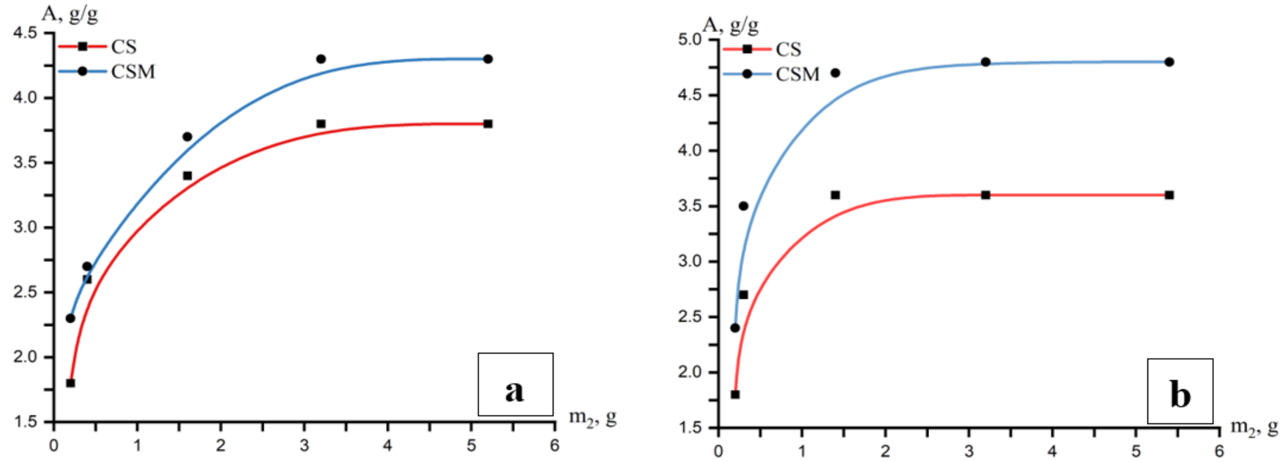

Fig. 3: Isotherms of oil adsorption on the surface of carbonized on the base of peanut shell (a) and walnut tree (b) (T = 25 °C).

Table 1: Data on adsorption of oil and on the surface of known carbonised sorbents.

Adsorbents	Adsorption capacity (g/g)	Ref.
Carbonized pine sawdust	11.2	[16]
Carbonized walnut sawdust	5.4	[16]
Carbonized barch	4.5	[16]
Carbonized leaf	2.8	[16]
Carbonized peat	0.4-0.5	[27]
Carbonized corn stalk	6.4	[28]
Carbonized potato peels	72	[29]
Sawdust-oleic acid	4.5	[37]
Carbonised rubber Seed Kernels	0.89	[38]

and magnetic cluster formation, magnetite appear to promote improved oil retention even if they may partially occupy pore spaces. The findings imply that a good compromise between porosity, hydrophobicity, and recyclability is offered by the complementary interaction of magnetite domains and porous carbon networks.

Table 1 lists the reported oil adsorption capacities of various carbonized sorbents derived from plant sources. The adsorption performance of these biochars is significantly influenced by the raw precursor and activation method. Carbonized pine sawdust has the highest capacity of 11.2 g g^{-1} among the sorbents discussed, but natural sawdust and peat have lower adsorption values ranging from 0.4 to 2.5 g g^{-1}, which represent their restricted porosity and surface functioning. Modified sorbents, such as carbonized maize stalk (6.4 g/g) and sawdust treated with oleic acid (4.5 g/g), have improved capacities, indicating that surface modification and biomass composition positively affect oil uptake. Notably, carbonized potato peels display an exceptionally high adsorption value of 72 g/g, attributed to their highly porous structure and abundant hydrophobic functional groups, whereas rubber seed kernel–based carbon shows comparatively lower efficiency (0.89 g g^{-1}). Overall, the data highlight the diversity in oil sorption performance among bio-derived carbons and underscore the critical role of feedstock selection and carbonization conditions in optimizing sorbent effectiveness.

As presented in Fig.4 (a–b), the oil recovery efficiency of the carbonized sorbents (CS) and their magnetic composites (CSM) were assessed to determine their separation performance and reusability. The results demonstrate that both sorbents have high oil recovery percentages; for the magnetite-modified samples, the greatest value was 95%. Strong interfacial contacts between the oil phase and the hydrophobic carbon surfaces are confirmed by the good recovery (>90%) shown by both sorbents at lower oil concentrations (m_1 = 2–3 g). However, the recovery efficiency gradually declines as the oil load rises, most likely as a result of retained oil residues partially blocking pores and saturation of available adsorption sites.

A comparison between the materials indicates that the walnut tree–based magnetic sorbent consistently outperforms its peanut shell counterpart, achieving higher recovery rates at all tested oil masses. This enhanced recovery is attributed to the combination of a well-developed pore structure and the magnetic properties introduced by Fe_3O_4 nanoparticles, which facilitate both efficient oil desorption and easy magnetic separation from water.

The combined effect of capillary and hydrophobic forces explains the remarkable recovery performance. While the linked pores physically capture oil droplets through capillary suction, the CH groups on the carbon surface interact with the oil's nonpolar hydrocarbon chains. Crucially, adding magnetite improves sorbent performance by raising surface

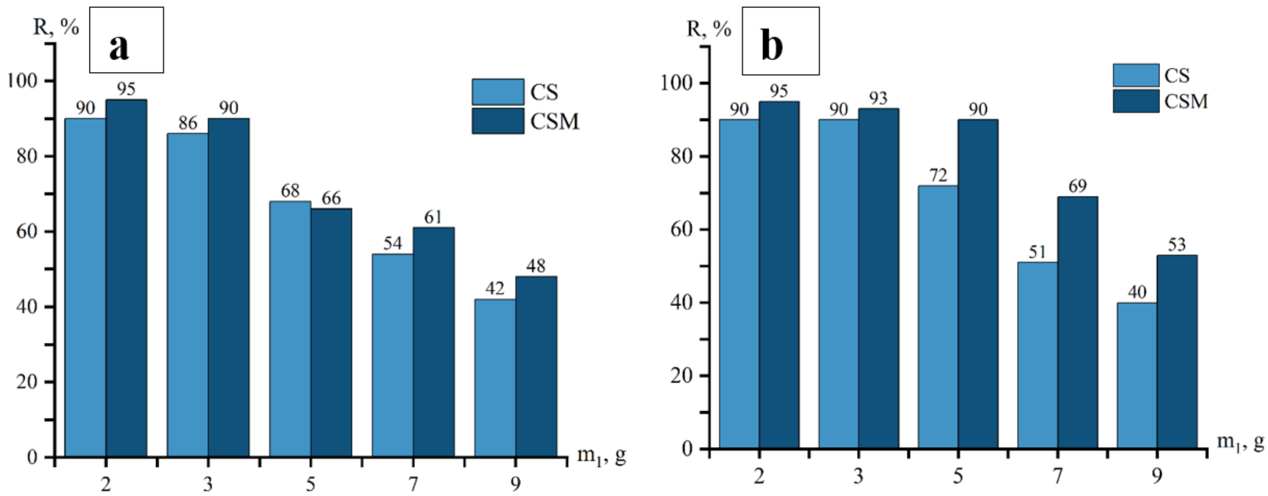

Fig. 4: Oil recovery efficiency (R, %) of carbonized sorbents (CS) and magnetic composites (CSM) derived from (a) peanut shell and (b) walnut tree at 25 °C.

Table 2: The BET analysis data on specific surface area of carbonised sorbents.

Adsorbents	Specific surface area (m^2/g)	Total pore volume (cm^3/g)	Average pore diameter (nm)
Peanut	1053.47	4.26	16.18
Peanut-magnetite composite	1696.20	5.89	13.90
Walnut tree	1033.91	3.48	13.46
Walnut tree-magnetite composite	1533.60	5.41	14.12

roughness and facilitating quick magnetic retrieval, not by impeding oil adsorption. These results demonstrate that magnetically modified carbonized biomass sorbents are interesting options for real-world oil spill cleanup because they provide the simultaneous benefits of high adsorption efficiency and simple recovery.

The surface textural properties of the carbonized and magnetic sorbents were determined using the Brunauer–Emmett–Teller (BET) method, and the results are summarized in Table 2. The specific surface areas of the carbonized peanut shell and walnut tree sorbents were 1053.47 m^2/g and 1033.91 m^2/g, respectively. Upon magnetite incorporation, these values increased to 1696.20 m^2/g for the peanut-based composite and 1533.60 m^2/g for the walnut-based composite. The total pore volumes likewise increased from 4.26 cm^3/g and 3.48 cm^3/g for the pristine carbons to 5.89 cm^3/g and 5.41 cm^3/g for the corresponding magnetic composites. The average pore diameters ranged between 13.46 nm and 16.18 nm, placing these materials within the mesoporous domain. The observed increase in pore volume and surface area following magnetite loading implies that the Fe_3O_4 particles were evenly distributed throughout the internal channels, encouraging the creation of new interparticle voids rather than obstructing the ones that already existed. Similar results have been documented for magnetically modified biochars made from carbons sourced from straw and *Trachycarpus fortunei* fruit seeds, where high-temperature carbonization and chemical activation greatly enhanced the surface area from 29 m^2/g to values above 1000 m^2/g.[35-39] Alkali activation, the controlled carbonization temperature (600 °C), and the precursor composition all work together to improve pore growth and carbon ordering, which accounts for the high surface areas of the resulting adsorbents. Conversely, the slightly smaller pore diameters in the magnetic composites indicate partial pore filling by magnetite nanoparticles. BJH pore distribution analysis further supports this trend, showing a substantial increase in the proportion of macropores (50–200 nm) after magnetite synthesis, from 57.68 % to 87.67 % for peanut shell–based and from 81.52 % to 97.28 % for walnut tree–based sorbents.

These structural modifications are consistent with the improved oil adsorption and recovery performance observed for the magnetic sorbents. The walnut tree–magnetite composite, in particular, exhibits the highest adsorption efficiency, likely due to its hierarchical pore architecture and mechanically robust surface morphology, as seen in the SEM micrographs (Fig. 1). The walnut-derived sorbent possesses shallow, well-distributed cavities capable of retaining oil droplets more effectively than the tubular, open-pore structure of the peanut shell–derived carbon. Overall, both the surface area and the geometrical configuration of the pores play decisive roles in governing oil adsorption behavior, demonstrating that the synthesis of magnetite within biochar frameworks can serve as an effective surface modification strategy for tuning adsorption capacity and recyclability.

4. Conclusion

This study shows how peanut shells and walnut tree branches may be successfully carbonized at 600°C in an argon atmosphere to create effective bio-based oil sorbents. In situ magnetite incorporation is then used to create magnetic composites. According to FTIR spectroscopy, the effective synthesis of magnetite was demonstrated by the emergence of distinctive Fe–O absorption bands in the 808–594 cm^{-1} region. SEM showed that both carbonized materials had a well-developed porous structure that allowed for efficient oil adsorption. The highest oil adsorption capabilities were 3.8 g/g for carbon produced from peanut shells and 3.6 g g^{-1} for carbon generated from walnut trees. After magnetite alteration, adsorption capabilities rose to 4.3 g/g and 4.8 g/g, respectively. Furthermore, the magnetic composites attained up to 95% of the oil recovery efficiency, exhibiting their strong potential for practical oil spill remediation. High specific surface areas of 1696.20 m^2/g for the peanut shell–based and 1533.60 m^2/g for the walnut tree–based adsorbents have been revealed by BET surface analysis, with average pore diameters in the 13–16 nm range. Magnetite incorporation slightly minimized surface area but improved structural stability and sorption performance through increased surface roughness and enhanced capillary oil retention. Overall, the results demonstrate that sustainable sorbents with high adsorption and recovery efficiencies are yielded by the synergistic combination of hierarchical porosity and magnetic functionality. The study confirms that carbonized plant-based materials, particularly walnut tree–magnetite composites, represent promising, low-cost candidates for ecologically friendly oil spill cleanup and wastewater treatment applications.

Acknowledgments

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not applicable.

CRediT Statement

Haoran Zhang: Contributed to the conceptualization of the study and Data analysis; **Sagdat Tazhibayeva:** Methodology and Scientific supervision; **Bakyt Tyussyupova:** Conducted experimental investigations and Data processing; **Yerdos Ongarbayev:** Worked on data visualization and Verification of analytical accuracy; **Kuanysh Tastambek**: Handled manuscript preparation and Text editing; **Adilkhan Seipiyev:** Participated in data collection and Computational assessments; **Kuanyshbek Musabekov**: Supervised the project and provided interpretation of the results.

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