http://dx.doi.org/10.18623/rvd.v22.n4.3590

ADSORPTION CAPABILITY OF MAGNETIC BIOCHAR DERIVED FROM LONGAN PEEL FOR DIESEL OIL REMOVAL FROM WATER

CAPACIDADE DE ADSORÇÃO DO BIOCARVÃO MAGNÉTICO DERIVADO DA CASCA DE LONGAN PARA REMOÇÃO DE ÓLEO DIESEL DA ÁGUA

Article received on: 8/15/2025
Article accepted on: 10/17/2025

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The authors declare that there is no conflict of interest

Abstract
Oil spills pose significant threats to marine ecosystems. This study aims to assess the adsorption capacity of magnetic biochar derived from longan peel (M/BLP), produced at various pyrolysis temperatures using FeCl3-treated longan peel, as an adsorbent for removing diesel oil from water. The M-BLP was synthesized through a slow pyrolysis method at temperatures ranging from 400 to 600 °C and characterized using FTIR, XRD, BET surface area analysis, XRF, and SEM. The results showed that M-BLP had numerous small holes, repelled water, and could be separated using magnets, making it a suitable choice for cleaning up oil spills and helping the environment. We analyzed the adsorption capacity of M/BLP for diesel oil removal, considering the adsorbent dose, pH level, salinity, and contact time. Increasing the M/BLP dosage, contact time, and salinity significantly improved the sorption capacity; however, variations in pH did not have a significant effect on adsorption. Moreover, the M/BLP adsorbent had good stability and magnetic separation.

Resumo
Derramamentos de petróleo representam ameaças significativas aos ecossistemas marinhos. Este estudo teve como objetivo avaliar a capacidade de adsorção de um biochar magnético derivado da casca de longan (M/BLP), produzido em diferentes temperaturas de pirólise utilizando casca de longan tratada com $FeCl_3$, como adsorvente para a remoção de óleo diesel da água. O M/BLP foi sintetizado por meio de um método de pirólise lenta em temperaturas que variaram de 400 a 600 °C e caracterizado por FTIR, XRD, análise de área superficial BET, XRF e MEV. Os resultados mostraram que o M/BLP possuía numerosos poros microscópicos, repelia a água e podia ser separado por meio de ímãs, tornando-o uma opção adequada para a limpeza de derramamentos de petróleo e para a preservação do meio ambiente. Analisamos a capacidade de adsorção do M/BLP para a remoção de óleo diesel, considerando a dose do adsorvente, o nível de pH, a salinidade e o tempo de contato. O aumento da dosagem de M/BLP, do tempo de contato e da salinidade melhorou

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Keyword: Magnetic Biochar. Longan Peel. Diesel Oil. Adsorption. Pyrolysis.

significativamente a capacidade de adsorção; no entanto, as variações de pH não apresentaram efeito significativo na adsorção. Além disso, o adsorvente M/BLP apresentou boa estabilidade e separação magnética.

Palavras-chave: *Biochar Magnético. Casca de Longan. Óleo Diesel. Adsorção. Pirólise.*

1 INTRODUCTION

Oil spills cause significant threats to marine and coastal ecosystems, depending on the specific environment. Moreover, hydrocarbon pollution adversely affects human health and marine ecosystems. The continual release of hydrophobic contaminants into aquatic ecosystems, particularly diesel oil from industrial activities, transportation, and accidental spills, profoundly affects both aquatic life and human health. Diesel oil comprises a complex mixture of hydrocarbons, including both aromatic and aliphatic compounds, which can disrupt ecological balances and pose risks to marine life [1]. Conventional remediation techniques, including bacterial degradation [2], adsorption with magnetic materials [3] and polymers [4], application of biosurfactants [5], and sorbent devices [6], may involve expensive and resource-demanding procedures. Consequently, there is an increasing need for sustainable, economical, and environmentally friendly methods for reducing diesel oil pollution in water.

A potential approach to attaining a sustainable resolution for hydrocarbon contamination in aquatic environments involves the creation of novel adsorbents derived from inexpensive raw materials that can effectively extract oil from water. Biochar has recently gained popularity as an adsorbent for oil spill removal [7]. Biochar is a carbon-rich material generated in oxygen-limited environments via slow and fast pyrolysis processes. It is derived from diverse carbon-based biowastes, including woody biomass, crop residues, animal carcasses, and biosolids. Biochar represents a potentially innovative technology for the removal of contaminants from water or aqueous solutions, characterized by unique properties such as porosity, a large surface area, and a negative surface charge, which contribute to its effectiveness in decontaminating water from both organic and inorganic pollutants [8,9]. However, the high cost of commercial biochar, attributed to expensive sources and the need for regeneration and reactivation procedures, limits its widespread application. Longan is a significant fruit in Thailand, with

substantial production and export volumes. The export processing of longan generates significant residue, with longan peel frequently discarded or incinerated as waste, amounting to hundreds of tons annually due to insufficient comprehensive utilization. Longan peel, characterized by a high carbon, oxygen, and hydrogen content, is an effective precursor for biochar production due to its abundance and availability [10]. Exhausted biochar adsorbents may include significant quantities of pollutants. Therefore, when utilizing biochar-based adsorbents for the treatment of natural water bodies, it is essential to establish a method for the retrieval of pollutant-laden adsorbents from aqueous solutions to prevent secondary contamination. This technique can effectively facilitate the commercialization of biochar-based adsorbents [11].

Fe_2O_3 particles serve as information storage ferrofluids and are also considered potential candidates for biomolecule imaging, sensing, and renewable energy applications. Moreover, Fe_2O_3 serves as an efficient adsorbent for a range of chemical substances, including heavy metals, organic dyes, and antibiotics [12,13]. Furthermore, post-usage magnetic retrieval of depleted iron oxide particles facilitates the recycling of the contaminant-laden adsorbent. Still, Fe_2O_3 nanoparticles have high surface energy because of strong van der Waals forces. This makes them more likely to combine in water, which lowers their surface area and adsorption capacity and raises their costs. A biochar/Fe_2O_3 composite may be synthesized to acquire qualities unattainable by the individual components alone [11].

This study investigated the feasibility of utilizing magnetic biochar derived from longan peel (M/BLP) as an adsorbent for the removal of diesel oil from wastewater. The textural and structural properties of M/BLP concerning adsorption were analyzed using advanced techniques. The adsorption performance of M/BLP for the removal of diesel oil was assessed by examining the influence of various M/BLP adsorbent types, adsorbent dosage (0.005–0.020 g), initial pH (5–9), salinity (500–2,000 mg/L), and contact time (20–240 minutes) on the removal efficiency of diesel oil from water.

2 MATERIALS AND METHODS

2.1 Materials and chemical reagents

Ferric chloride hexahydrate ($FeCl_3.6H_2O$; analytical grade 99%) was acquired from Sigma-Aldrich. Diesel oil (commercial grade) was purchased from Shell Company of Thailand Limited. All chemical solutions were prepared using deionized (DI) water from RCI Labscan, which was also used to rinse and clean the samples. Longan peel, a common agricultural residue material, was collected from Rim Rong Housewives Community Enterprise, Makhuea Chae Subdistrict, Mueang District, Lamphun Province, Thailand.

2.2 Preparation of adsorbents

2.2.1 Preparation of biochar from longan peel (BLP)

This experiment utilized longan peel residue as the raw material for biochar production. The longan residue was first washed several times with deionized water and subsequently dried at 110 ºC for a duration of 2 days. The dried samples were ground and sieved to achieve a particle size of less than 10 mm. This material underwent pyrolysis at temperatures of 400 and 600 °C for 4 hours, with a heating rate of 5 °C/min, utilizing a tube furnace. The biochar produced from longan residues was designated as BLP-400 and BLP-600, respectively. Finally, they were stored in the desiccator.

2.2.2 Preparation of magnetic biochar from longan peel (M/BLP)

A Biochar/Fe_2O_3 composite was synthesized using a ferric chloride ($FeCl_3$) solution to impregnate longan peel powder, followed by pyrolysis at elevated temperatures. The $FeCl_3$ solution was prepared by dissolving 40 g of $FeCl_3.6H_2O$ in 60 mL of deionized water. The longan peel powder from section 2.2.1 was immersed in the prepared $FeCl_3$ solution for 2 hours. The mixture was subsequently dried at 80 °C for 2 hours in air. The pretreated biomass underwent pyrolysis in a furnace at temperatures of 400 and 600 °C for 4 hours, with a heating rate of 5 °C/min. The Biochar/Fe_2O_3 composite

generated from pyrolysis was carefully crushed and sieved to a particle size of 0.5–1 mm. The biochar produced from longan residues was designated as M/BLP-400 and M/BLP-600, respectively. The samples were subsequently washed with deionized water multiple times, oven-dried at 80 °C, and stored in a desiccator before use.

2.3 Characterization of adsorbents

Fourier-transform infrared spectroscopy (FTIR) was utilized to identify functional groups in the composites, employing a Perkin-Elmer spectrometer with the KBr technique. Sixty-four scans were averaged for each spectrum, covering the range of 400-4000 cm^{-1} at a resolution of 4 cm^{-1}.

Structural information about the materials was acquired through powder X-ray diffraction (XRD) analysis utilizing an Empyrean 3 Panalytical diffractometer with Cu Kα radiation at an X-ray power of 40 kV and 40 mA.

N_2 adsorption-desorption measurements were performed at -196°C utilizing an Anton Paar Nova 600 to determine the textural properties of the materials. The sample weight, initially approximately 40 mg, was precisely measured following pretreatment at 150 ºC for 2 hours.

X-ray fluorescence analysis (XRF) was done with an M4 Tornadoplus Bruker spectrometer to measure the amount of iron in the samples.

The morphology of the sample was assessed using scanning electron microscopy (SEM). The SEM images were captured using a TESCAN VEGA3 scanning electron microscope functioning at 10 kV. The samples on carbon tape were examined with a gold coating applied.

2.4 Adsorption experiments

We conducted batch adsorption experiments at various concentrations of diesel oil in water. 50-mL Erlenmeyer flasks serve as containers for batch adsorption processes. The adsorbents were immersed in a beaker with 20 mL of a diesel oil-water mixture, maintaining an oil-to-water ratio of 1:10 mL. The system was stirred at 200 rpm using a magnetic stirrer. All experiments are conducted at a constant room temperature. Contact time ranged from 20 to 240 minutes, depending on the experimental design. After

centrifugation, the adsorbents were separated from the mixture, and the oil in the solution was measured. The ability of M/BLP to remove diesel oil was tested by looking at how different types of M/BLP adsorbents, the amount used (0.005–0.020 g), the starting pH (5–9), salt levels (500–2,000 mg/L), and the time they were in contact (20–240 minutes) affected how well diesel oil was removed from water. The sorption capacity was calculated using Equation 1. Each run is conducted three times, and the average values were reported.

$$\text{Sorption capacity} = \frac{\text{Mass of adsorbent adsorbed} - \text{Mass of adsorbent}}{\text{Mass of adsorbent}} \quad (1)$$

3 RESULTS AND DISCUSSION

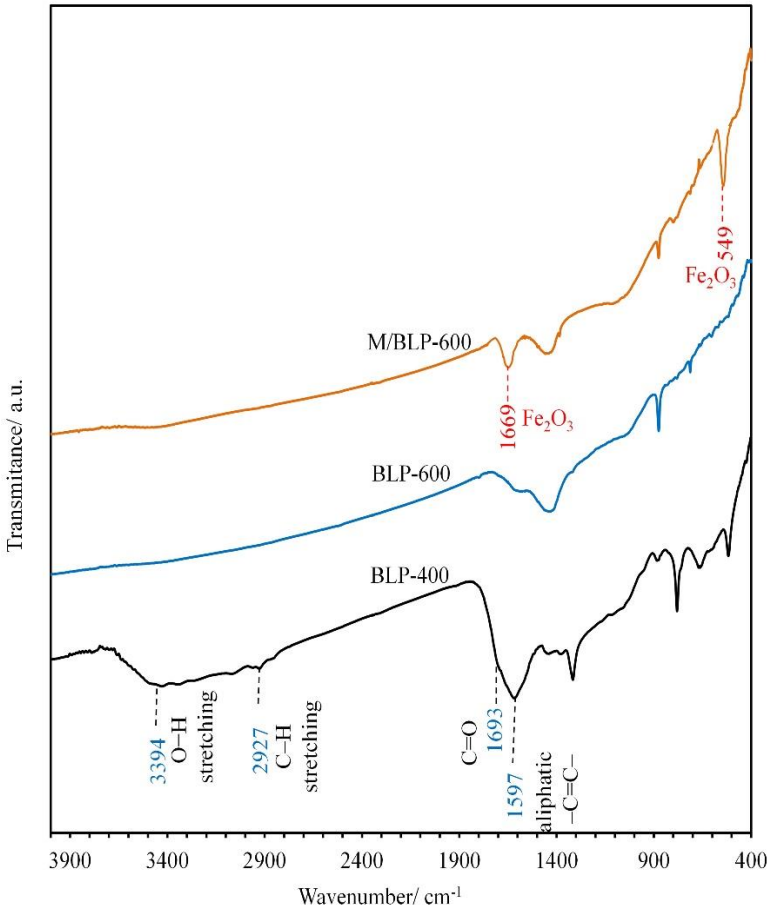
3.1 Adsorbent characterization

Figure 1 shows the FTIR spectra of biochar made from longan peel, including BLP-400, BLP-600, and M/BLP-600. The FTIR analysis indicates a reduction in the functional groups of biochar samples as pyrolysis temperature increases. The rapid loss of volatile compounds occurs due to the acceleration and intensification of carbonization at elevated pyrolysis temperatures. The FTIR spectra of biochar samples showed a wide peak at 3394 cm^{-1}, which is related to the stretching of O−H bonds in alcohol and phenol groups. The intensity of this peak was observed to be strong at lower pyrolysis temperatures; however, it disappears at higher temperatures. The characteristic C–H stretching vibration of the alkyl structure within the aliphatic group is observed at 2927 cm^{-1}. The peak at 1693 cm^{-1} indicated carbonyl bonds (C=O) stretching associated with carboxylic groups or conjugated ketones. The stretching vibrations of the aliphatic –C=C– are observed at 1597 cm^{-1} [14]. Figure 1 illustrates that the intensity of all peaks diminishes as pyrolysis temperature increases. At elevated temperatures (600 °C), the presence of aliphatic functional groups in the biochar is negligible. Aliphatic structures are known to reform into aromatic structures, leading to an increased presence of phenolic and ether groups. Moreover, at high temperatures many C=C bond breakages take place due to availability of adequate energy. Therefore, at higher temperature, due to extensive carbonization, formation of graphite-like structures of the biochar occurred which shows

less intense peaks [14]. In addition, the Biochar/Fe_2O_3 composite (M/BLP-600) observed the sharp peaks at 549 and 1668 cm^{-1}, related to Fe_2O_3 [15].

Figure 1

FTIR spectra of biochar from longan peel BLP-400, BLP-600 and M/BLP-600 composite.

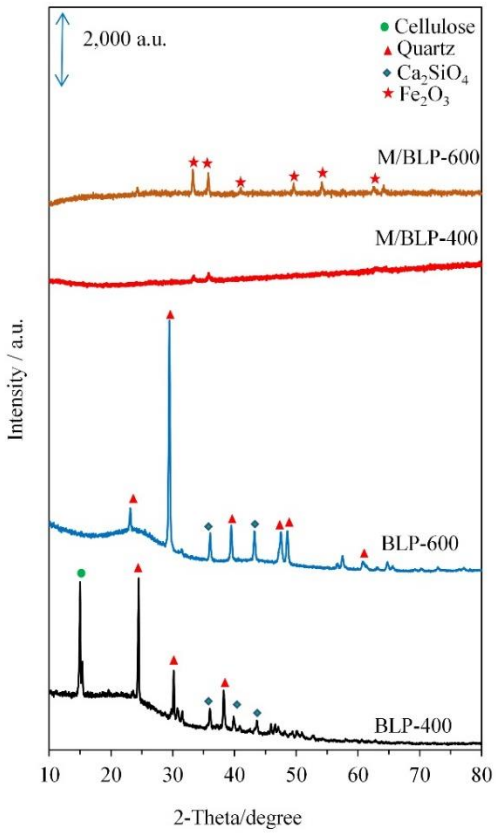


XRD analysis was conducted to investigate the crystalline and amorphous structures of the synthesized adsorbents. Figure 2 presents a comparison of the XRD patterns for BLP-400, BLP-600, M/BLP-400, and M/BLP-600 biochar. The XRD spectra of BLP materials exhibited an amorphous peak at 2θ values of approximately 10−25°. The XRD pattern of BLP-400 showed that the peaks around 15° matched the specific diffraction peaks of cellulose, which correspond to the crystal planes of (004). After pyrolysis, cellulose and hemicellulose were decomposed and transformed during heat treatment, and the diffraction peaks here disappeared (BLP-600), indicating that the cellulose structure was destroyed in a high temperature environment. A group of diffraction peaks with 2θ values of 23.47°, 29.46°, 39.55°,47.74°, 49.53° and 61.28° were

the characteristic diffraction peaks of the quartz phase. It also showed that the silicon species in the BLP-400 and BLP-600 in the carbon-based structure in the form of quartz phase [16]. In addition, calcium silicate (Ca_2SiO_4) was also found at 2θ at 36.02°, 39.45°, 42.96° and 43.39° in BLP-400 and BLP-600.

Figure 2

XRD analysis of biochar from longan peel BLP-400, BLP-600, M/BLP-400 and M/BLP-600 composite.



Whereas the XRD results of Biochar/Fe_2O_3 composite (M/BLP-400 and M/BLP-600) exhibited new six diffraction peaks around 33.3°, 35.6°, 43.2°, 49.8° 57.2° and 62.9° compared to initial biochar, which corresponded to the characteristic of Fe_2O_3 [17]. Moreover, the M/BLP-600 composite revealed stronger and sharper diffraction peaks than the M/BLP-400. This should be due to the amount of Fe_2O_3 incorporated in biochar structure.

The surface area and pore volume of biochar are critical factors influencing the adsorption capacity for the removal of diesel oil from water. The BET surface area and pore volume of biochar samples are presented in Table 1. The BET surface areas and pore

volumes of biochar were significantly enhanced with increasing pyrolytic temperature. The biochar produced at 600 °C exhibited a surface area of 19.01 m^2/g and a pore volume of 0.0408 cm^3/g. Conversely, biochar produced at 400 °C demonstrated a surface area of 16.81 m^2/g and a pore volume of 0.0371 cm^3/g. The increase in surface area results from the removal of carbon mass as volatile matter from the biomass surface, leading to the formation of pores in the structure of the resulting biochar. However, Biochar/Fe_2O_3 composite (M/BLP-400 and M/BLP-600) exhibited a lower textural property than pristine biochar due to the Fe_2O_3 incorporated into the carbonaceous structure of biochar.

Table 1

Physicochemical properties of the adsorbents.

Sample	S_{BET}[a] (m^2 g^{-1})	V_t[b] (cm^3 g^{-1})	% Fe [c]
BLP-400	16.81	0.0371	*n.d.*
BLP-600	19.01	0.0408	*n.d.*
M/BLP-400	12.58	0.0337	6.2
M/BLP-600	16.22	0.0351	8.1

[a] BET surface area
[b] Total pore volume
[c] Determined from XRF

Figure 3 presents the magnetic separation of biochar from longan peel BLP-400, BLP-600, M/BLP-400 and M/BLP-600 composite in water (salinity = 500 mg/L) at 30 min. It was found that BLP-400 and BLP-600 were well dispersed in saline water and after 30 minutes, BLP-600 precipitated at the bottom more than BLP-400. In addition, neither biochar exhibits the magnetic separation property. Whereas the biochar/Fe_2O_3 (M/BLP-400 and M/BLP-600) composites precipitated at the bottom after 30 min and exhibited the magnetic separation property. However, the M/BLP-600 composited displayed the magnetic separation property more than the M/BLP-400 corresponded to the amount of Fe content the biochar/Fe_2O_3 composites (Table 1).

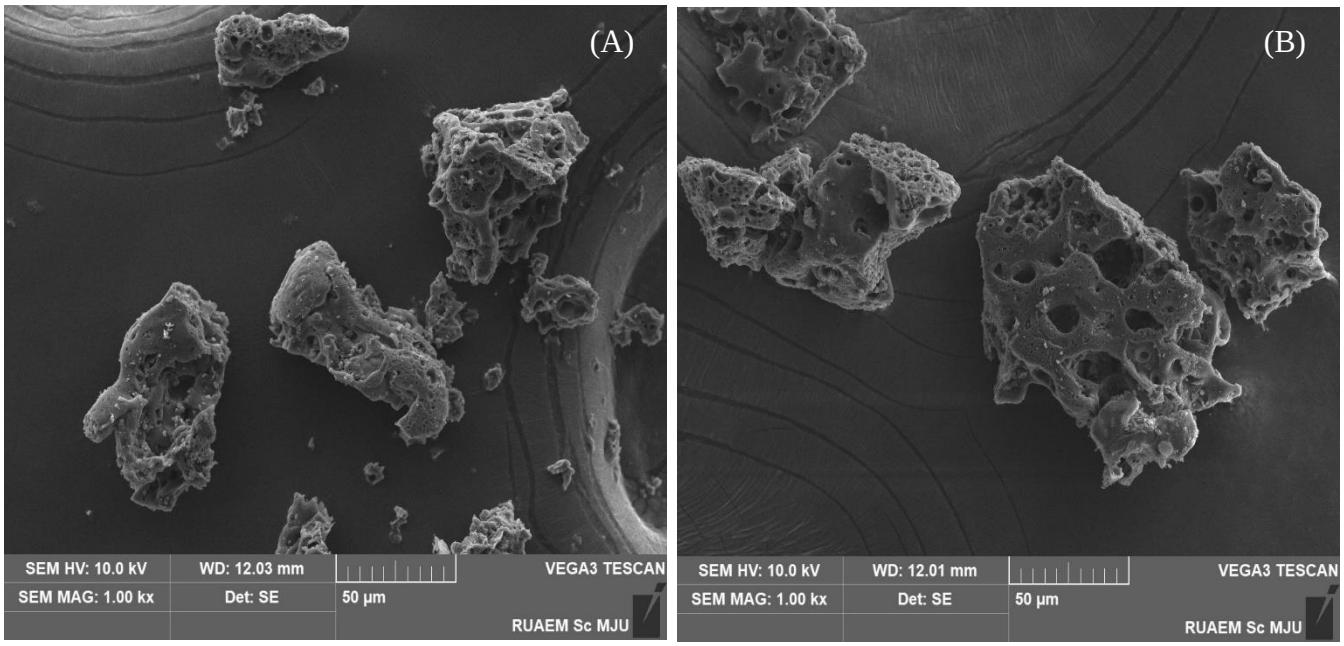
Figure 3

The magnetic separation of biochar from longan peel BLP-400, BLP-600, M/BLP-400 and M/BLP-600 composite.

In addition, the morphology of the BLP-600 and M/BLP-600 was shown in representative SEM images in Figure 4. They revealed a small particle (~90 – 120 μm) with a porous structure, as shown in Figure 4A, B. However, the M/BLP-600 displayed a larger particle size than that of the initial BLP-600 material. This result corresponded to the textural properties of them as shown in Table 1 and suggested that Fe_2O_3 deposited onto the biochar structure resulted in an enhancement of particle size.

Figure 4

SEM images (1,000 x magnification) of the (A) BLP-600 and (B) M/BLP-600 composite.

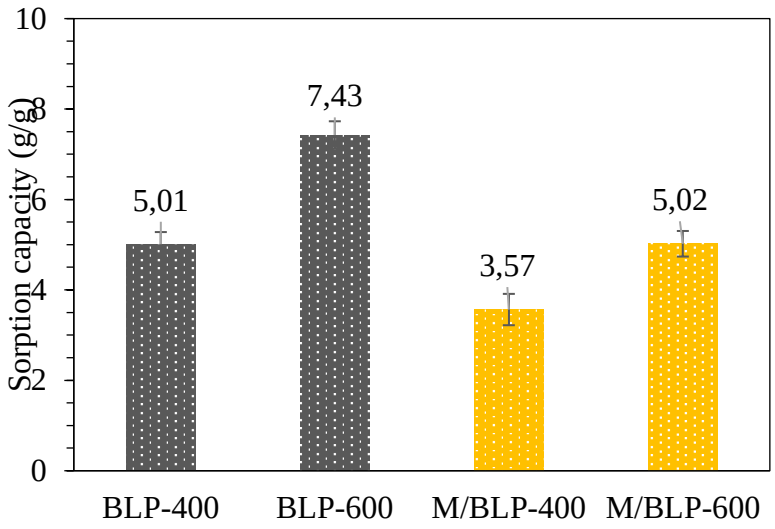


3.2 Effect of solution environmental conditions

All biochar derived from longan peel materials were evaluated as an adsorbent for a diesel oil-water mixture at a ratio of 1:10 mL. The adsorption capacity of diesel oil of these samples is presented in Figure 5 (adsorbent dose = 0.005 g; diesel oil/water ratio = 1:10 mL; t = 240 min; agitating speed = 200 rpm; pH= 7 and room temperature). The biochar made at higher pyrolysis temperatures showed a better ability to adsorb diesel oil (BLP-400 < BLP-600), which is linked to the BET surface area and pore volume shown in Table 1. The biochar mixed with Fe_2O_3 (M/BLP-400 and M/BLP-600) showed a lower ability to remove diesel oil compared to the original biochar. The results showed that the textural properties of the adsorbent influence the highest adsorption capacity for diesel oil removal. However, the M/BLP-600 showed a high magnetic separation property in water (Figure 3). Therefore, M/BLP-600 was a suitable adsorbent for use in further studies.

Figure 5

Effect of type of adsorbents on diesel oil removal.

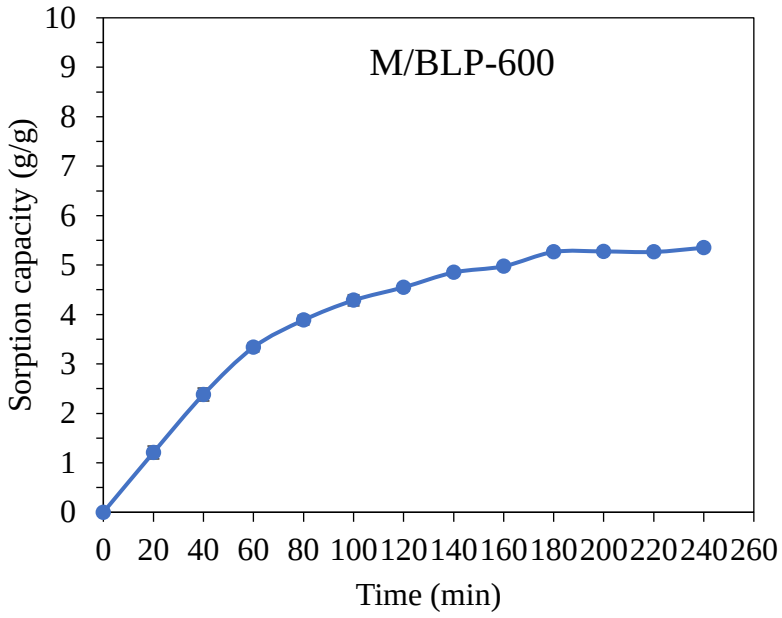


The M/BLP-600 adsorbent was used to study how long it takes for adsorption to happen, as shown in Figure 6)adsorbent dose = 0.005 g; diesel oil/water ratio = 1:10 mL; time = 20-240 min; stirring speed = 200 rpm; pH = 7; room temperature). The findings indicated that the contact time necessary to achieve adsorption equilibrium was approximately 180 minutes. The contact time necessary to achieve adsorption equilibrium

was approximately 240 minutes. The results showed that these conditions allowed M/BLP-600 to reach its capacity limit.

Figure 6.

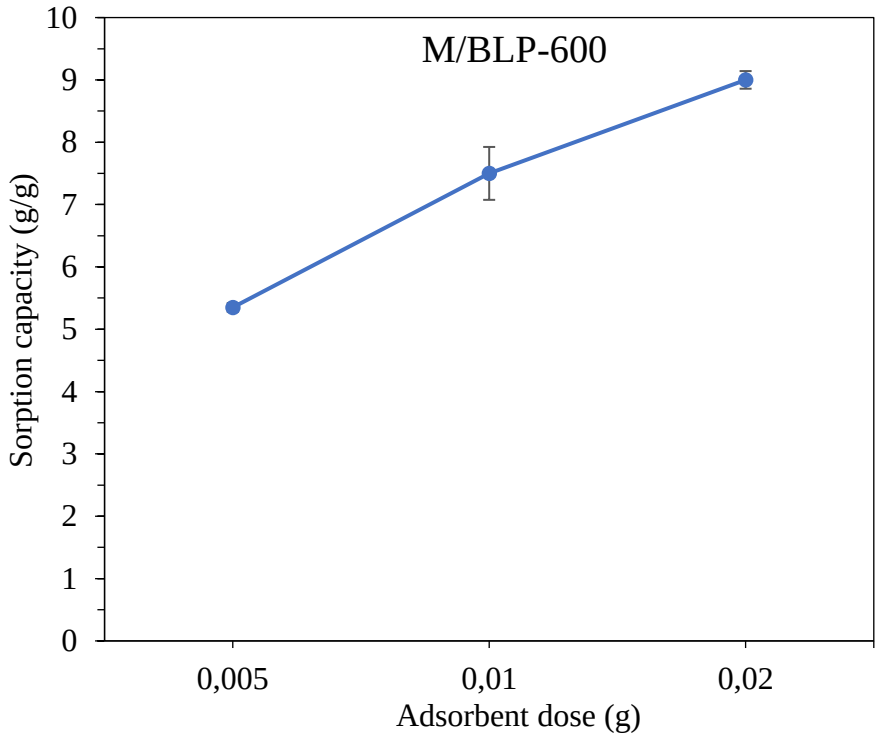
Effect of contact time on diesel oil removal.



The adsorbent dose determined the amount of biochar available for adsorption, determining the process's effectiveness as shown in Figure 7 (adsorbent dose = 0.005-0.020 g; diesel oil/water ratio = 1:10 mL; t = 240 min; agitating speed = 200 rpm; pH= 7 and room temperature). A low dose of adsorbent (0.005 g) might initially lead to decreased removal efficiency because of insufficient surface area for adsorption. Increasing the dosage improved removal efficiency. The adsorption capacity of diesel oil rose from 5.35 to 9.02 g/g. The addition of biochar increased surface area and the availability of adsorption sites for diesel oil molecules, resulting in enhanced removal capacity from water.

Figure 7

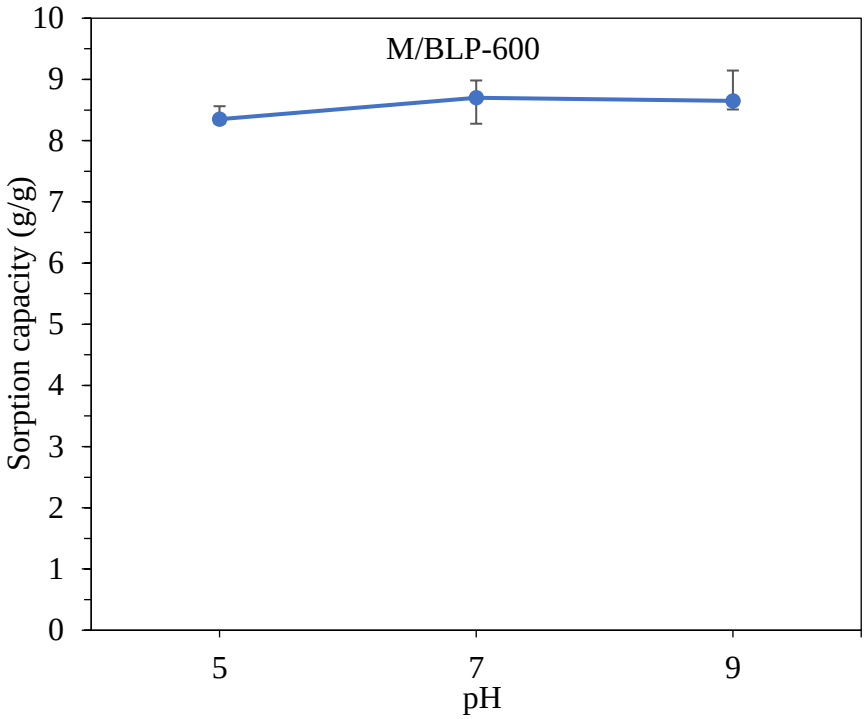
Effect of adsorbent dose on diesel oil removal.



The sorption capacity of M/BLP-600 changed in response to pH level as shown in Figure 8 (adsorbent dose = 0.020 g; diesel oil/water ratio = 1:10 mL; t = 240 min; agitating speed = 200 rpm; pH= 5-9 and room temperature). The maximum sorption capacity was observed at pH 7, indicating that this pH level was optimal for adsorption efficiency, whereas the minimum sorption capacity was recorded at pH 5. However, the variations in sorption capacity at different pH levels did not demonstrate a significant difference. The concave downward trend depicted in Figure 8 suggested that further increases in pH beyond 7 did not lead to any additional improvement in sorption capacity. The pH affected the charge properties of the M/BLP-600 adsorbent. Modifying these charges allowed pH to affect the adsorption behavior of M/BLP-600. At reduced pH levels, a notable interaction was observed between M/BLP-600 and diesel oil, resulting from beneficial charge interactions. Charge interactions may become saturated as pH rises above a particular threshold, which would reduce the rate at which sorption capacity increases [18]. The observed saturation effect might elucidate why the sorption capacity at pH 9 returned to the level observed at pH 5.

Figure 8

Effect of pH on diesel oil removal.

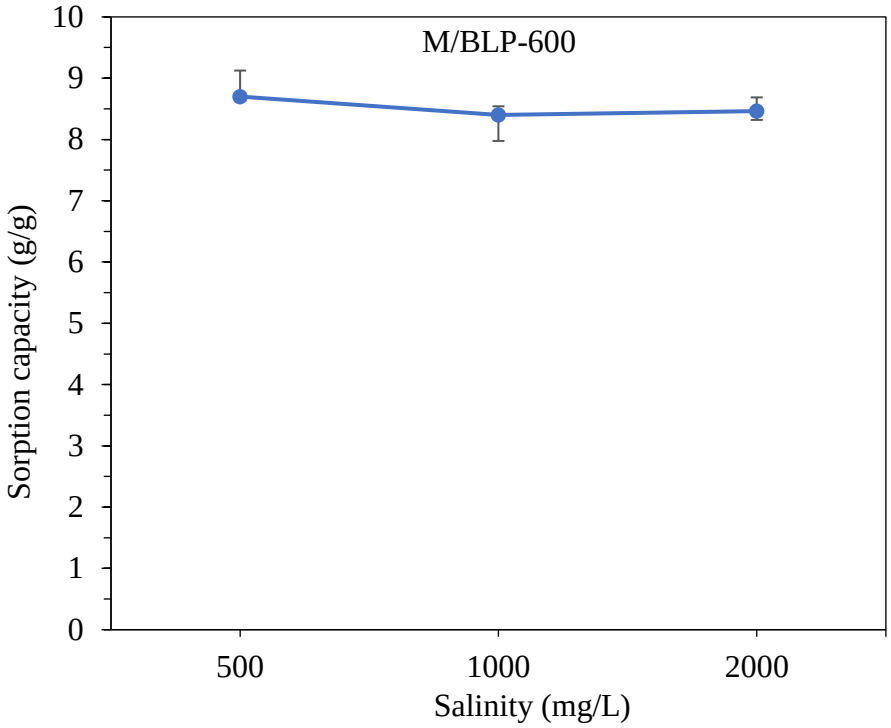


The studies employed salinity levels of 500, 1,000, and 2,000 mg/L (adsorbent dose = 0.020 g; diesel oil/water ratio = 1:10 mL; t = 240 min; agitating speed = 200 rpm; pH= 7 and room temperature). Figure 9 demonstrates the minimum sorption capacity at 500 mg/L and the maximum capacity at 2,000 mg/L of salinity. Increased salinity might improve the absorption capacity of biochar due to various factors. Salinity could change the surface charge and functional groups of biochar, which affected how it interacted with diesel oil molecules. Alterations in surface chemistry could affect adsorption capacity. Longan peel provided the material, which had a substantial surface area. It contained functional groups capable of interacting with ions in the salt solution via electrostatic forces. The amount of ions in water could change how thick the layer around biochar particles was, which in turn affected whether oil molecules were pulled towards or pushed away from the adsorbent. These variables might affect the sorption capacity of biochar [18]. Also, leftover longan peel was used to make biochar, which had different surface functional groups like hydroxyl (−OH) and carboxyl (−COOH) groups. These functional groups played a significant role in oil absorption. When using biochar, the salinity levels

might influence the ionic strength associated with oil adsorption. The presence of nonpolar functional groups contributed to hydrophobic interactions. Salinity effects might alter the pH and ionic strength of the solution, thereby impacting on the accessibility and availability of functional groups. Variations in salinity within the solution might alter the surface chemistry of biochar, thereby affecting its oil absorption capacity.

Figure 9

Effect of salinity on diesel oil removal.



4 CONCLUSIONS

In this study, magnetic biochar from longan peel was prepared in different pyrolysis temperature and used as adsorbents to remove diesel oil from water. The pyrolysis conditions were found to have significant impact on biochar structure. Inorganic minerals were found in this biochar such as SiO_2 and Ca_2SiO_4. An increasing pyrolysis temperature to produce biochar resulted in almost no aliphatic functional groups would be present in the biochar but enhanced the BET specific surface area and pore volume. In addition, the biochar/Fe_2O_3 composite displayed good magnetic separation property from the water. The adsorption results indicated that the magnetic biochar made from longan

peel heated to 600 °C (M/BCL-600) was effective in removing diesel oil. The maximum adsorption capacity for diesel oil was determined to be 9.02 g/g. The optimal environmental conditions for maximizing diesel oil removal were identified as follows: pH was set at 7; the adsorbent dose was 0.002 g per 20 mL; salinity levels were maintained at 2,000 mg/L; the duration is 240 minutes; and the agitation speed is 200 rpm. However, this material could be further studied for application in the real sea water to remove diesel oil.

ACKNOWLEDGMENTS

The authors are appreciative of the financial assistance provided by the Thailand Science Research and Innovation (TSRI), as stated in the annual government statement B.E. 2563. The authors gratefully acknowledge all staff of Industrial Chemistry Innovation Program, Faculty of Science, Maejo University and Montfort College for supporting the facilities to this study.

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Authors' Contribution

Both authors contributed equally to the development of this article.

Data availability

All datasets relevant to this study's findings are fully available within the article.

How to cite this article (APA)

Thammasit, C., Pripwai, N., Mungthong, P., & Nuntang, S. (2025). ADSORPTION CAPABILITY OF MAGNETIC BIOCHAR DERIVED FROM LONGAN PEEL FOR DIESEL OIL REMOVAL FROM WATER. *Veredas Do Direito*, 22(4), e223590. https://doi.org/10.18623/rvd.v22.n4.3590