

NOVEL MAGNETIC EGGHELL MEMBRANE FUNCTIONALIZED WITH WASTE PALM FATTY ACID FOR SELECTIVE ADSORPTION OF OIL FROM AQUEOUS SOLUTION

(Novel Magnetik Membran Kulit Telur Berfungsikan dengan Sisa Asid Lemak Sawit untuk
Penjerapan Minyak Terpilih daripada Larutan Aqueus)

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Abstract

Emulsified oil in wastewater is a severe problem and requires extensive treatment before it can be disposed of in a manner that meets environmental regulation standards. One strategy to remove emulsified oil is by utilizing the adsorption process. The goal of this study is to synthesis magnetic eggshell membrane (ESM) functionalized with waste palm fatty acid, (MNP@ESM-WPFA) for the adsorption of oils (lubricating oil). The synthesis procedure involves three steps including preparation of ESM, functionalization of ESM with waste palm fatty acid (WPFA) and magnetization of eggshell membrane (ESM) functionalized with waste palm fatty acid (ESM-WPFA) through co-precipitation method to obtain MNP@ESM-WPFA. The novel adsorbent was characterized using SEM, FTIR, and EDX analyses. SEM analysis revealed the magnetic nanoparticles scattered throughout the porous and fibrous network structure of ESM-WPFA, confirming the successful synthesis of the MNP@ESM-WPFA. Further FTIR analysis on MNP@ESM-WPFA adsorbent revealed the appearance of peaks at 2933 cm^{-1} and 2852 cm^{-1} and 630 cm^{-1} , thus

confirming the presence of the alkyl chain of the waste palm fatty acid (WPFA) and Fe-O band on the surface of MNP@ESM-WPFA. The oil adsorption performance of MNP@ESM-WPFA was optimal at pH 7, treatment time of 50 minutes, and adsorbent dosage of 50 mg. The MNP@ESM-WPFA showed the highest oil adsorption capacity (K) for lubricating oil (4.61 mg/mg), followed by olive oil (2.72 mg/mg), and corn oil (2.00 mg/mg). The MNP@ESM-WPFA adsorbent was also reusable, with a sorption capacity that was maintained after five usage-regeneration cycles.

Key words: Eggshell membrane, waste palm fatty acids, magnetic nanoparticles, adsorbents, oil removal

Abstrak

Minyak teremulsi dalam air sisa merupakan satu cabaran yang serius dan memerlukan rawatan yang meluas sebelum ia boleh dilupuskan dengan cara yang memenuhi piawaian peraturan alam sekitar. Salah satu strategi untuk mengeluarkan minyak emulsi adalah dengan menggunakan proses penjerapan. Kajian ini bermatlamat untuk sintesis membran kulit telur (ESM) magnetik berfungsi dengan sisa asid lemak sawit, (MNP@ESM-WPFA) untuk penjerapan minyak (minyak pelincir). Prosedur sintesis merangkumi tiga langkah termasuk penyediaan ESM, perfungsi ESM dengan sisa asid lemak sawit dan permagnetan membran kulit telur (ESM) berfungsi dengan sisa asid lemak sawit (ESM-WPFA) melalui kaedah kopresipitasi untuk mendapatkan MNP@ESM-WPFA. Penjerap novel ini telah dicirikan menggunakan analisis SEM, FTIR, dan EDX. Analisis SEM mendedahkan nanopartikel magnetik yang bertaburan di seluruh struktur rangkaian berliang dan berserabut ESM-WPFA, mengesahkan kejayaan sintesis MNP@ESM-WPFA. Selanjutnya, analisis FTIR pada penjerap MNP@ESM-WPFA mendedahkan kemunculan puncak-puncak pada 2933 cm^{-1} and 2852 cm^{-1} and 630 cm^{-1} mengesahkan kehadiran rantai alkil WPFA dan Fe-O pada permukaan MNP@ESM-WPFA. Prestasi penjerapan minyak MNP@ESM-WPFA adalah optimum pada pH 7, masa rawatan 50 minit, dan dos penjerap 50 mg. MNP@ESM-WPFA menunjukkan kapasiti penjerapan minyak (K) tertinggi untuk minyak pelincir (4.61 mg/mg), diikuti minyak zaitun (2.72 mg/mg), dan minyak jagung (2.00 mg/mg). Penjerap MNP@ESM-WPFA juga boleh digunak semula, dengan kapasiti penyerapan yang dikekalkan selepas lima kitaran penjanaan semula penggunaan.

Kata kunci: membran kulit telur, sisa asid lemak sawit, zarah nano magnetik, penjerap, penyingkiran minyak

Introduction

Emulsified oil wastewater is regarded as a widespread pollutant around the world. Large volumes of wastewater containing oil and grease are increasingly produced due to population growth and fast urbanization. The release of oil-containing wastewater into water bodies causes public concern due to toxicological and aesthetic reasons [1]. Oil effluents come from various sources, including petroleum refineries, rolling mills, chemical processing, manufacturing plants, industrial processing, and municipal wastewaters [2-4]. Oily wastewater can contain fats, oil, cutting fluids, lubricants, edible oil, grease, crude oils, diesel oils, and other hydrocarbons [5]. Emulsified oils are difficult to handle and pose a real problem for treatment because of steric interaction or structural barriers and electrostatic repulsion between oil droplets [6]. Oil emulsions can produce an impermeable layer on the water's surface, decreasing oxygen content in the water and retard the photosynthesis process of aquatic plants [7-9].

The removal of residual oil remains a difficult technological challenge. Gravity separation, flotation, coagulation, ultracentrifugation, and membrane filtration are traditional methods for removing oil from industrial oily wastewater [10, 11]. In recent years, several techniques for handling emulsified oil have been developed, including bacterial degradation [12], electrocoagulation [13], emulsion destabilization [14], filtration [15], and adsorption [16-19]. An ideal adsorbent should have a high adsorption capacity, a fast adsorption rate, high selectivity, and be relatively inexpensive. Although the adsorption process is not extensively employed in treating emulsions, it has several benefits, and it does not always necessitate the use of extra chemicals [20]. Even though most commercially available oil sorbents are made of synthetic polymers with high sorption capacities such as polypropylene and polyurethane, their use releases massive amounts of hard to recover, non-biodegradable plastic into the environment [21].

There are three main types of adsorbents commonly used to remove oily pollutants: organic sorbents

(activated carbon, sawdust, straw, butyl rubber, carbon-based product), inorganic sorbents (kieselguhr, organoclay, bentonite, vermiculite), and synthetic sorbents (polyvinyl chloride, polyurethane, nylon fibres) [17]. Recently, biomaterials have attracted a great deal of attention as a prospective adsorbent material due to their abundance, environmental friendliness, and ability to be manufactured from renewable sources [22]. Various studies have been carried out to evaluate the preparation of bio-sorbents (sugarcane bagasse, coffee grounds, eggshell membranes, lignocellulosic material) as well as their properties and efficacy in removing various organic contaminants [22]. One of which, the eggshell membrane (ESM), comprises of a porous network of insoluble fibrous proteins with a high surface area that could be exploited as a sorbent for various types of pollutants in wastewater [23, 24]. ESM contains disulfide bonds, causing it to be impermeable to water, thus giving it an advantage in specific applications [25]. However, ESM consists of hydrophilic functional groups such as hydroxyl, amine, and carboxylic acid that limits its use as adsorbent for hydrophobic pollutants, especially oil contaminants [26, 27]. To overcome this problem, the surface of ESM can be coated with a hydrophobizing agent.

Waste palm fatty acid can be a potential hydrophobic modifier on the surface of ESM. High amounts of free fatty acids can be produced in waste palm cooking oil through a hydrolytic process that occurs in the presence of oil and water from food. The chemical structure of free fatty acids, which consists of long alkyl chains, makes them an excellent hydrophobizing agent to tune the hydrophilic surface of ESM for selective adsorption of oil from aqueous. Waste palm cooking oil was selected because it is easily available and economically viable. In this study, a novel magnetic eggshell membrane (ESM) functionalized with waste palm fatty acid, (MNP@ESM-WPFA) was synthesized for the removal of lubricating oil. Magnetic Fe_3O_4 nanoparticles dispersed onto ESM functionalized with waste palm fatty acid can facilitate the rapid separation of oil from water while boosting the adsorption capability of ESM-WPFA. As the utilization of MNP@ESM-WPFA has not been reported yet for environmental applications,

therefore, it is a good idea to explore its potential as adsorbent for treatment and decontamination of organic pollutants, particularly oil. Therefore, this work focuses on analyzing the performance of MNP@ESM-WPFA in removing lubricating oil. The influence of adsorbent dosage, pH of the solution and contact time on the removal process was thoroughly investigated. Subsequently, the adsorption capacity and reusability of MNP@ESM-WPFA were evaluated.

Materials and methods

Chemicals

Sulphuric acid was purchased from Fischer Scientific (New Hampshire, USA). Sodium sulphate (anhydrous), while *n*-hexane, ethanol, ammonium iron(II) sulphate and iron trichloride hexahydrate were obtained from Merck (Darmstadt, Germany). All solvents used for the synthesis were of analytical grade. Deionized water was used throughout the study.

Instrumentation

Surface morphological analysis of MNP@ESM-WPFA was carried out using Hitachi SU8220 Scanning Electron Microscopy (Oxford Instrument, Oxfordshire, UK) that is equipped with x-ray energy dispersion (Carl Zeiss, Germany). FTIR spectra were recorded on Perkin Elmer FTIR spectrophotometer (Massachusetts, USA) with a crystal diamond universal ATR sampling accessory at room temperature. All spectra were recorded in the wavenumber range of 4000 to 450 cm^{-1} . Wettability analyses were performed using the Theta Lite 100 and Theta Lite 101 contact angle measurement instruments (Attension, Espoo, Finland).

Preparation of magnetic eggshell membrane functionalized with waste palm fatty acid:

Preparation of ESM

Raw eggshells were collected from a local shop and rinsed with deionized water to minimize the deterioration of the eggshell membrane (ESM). The ESM were then carefully removed from the eggshells and rinsed with deionized water again. This is followed by a drying process for 1 hour at 70 °C in the oven, after which the ESM were crushed and sifted using a 30 nm sieve to produce consistently sized powder particles.

Preparation of ESM-WPFA

The ESM-WPFA was prepared in accordance with a previously reported work by Sathasivam and Haris [28] but with minor modifications. Briefly, 2.00 g of ESM powder was mixed with 8.00 g of free fatty acid from waste palm cooking oil (WPCO) and three drops of concentrated sulphuric acid was used as a catalyst. This mixture was dispersed in 20 mL of *n*-hexane for 15 minutes at 60 °C. The obtained ESM-WPFA was then washed with excess *n*-hexane and air-dried.

Preparation of MNP@ESM-WPFA

The MNP@ESM-WPFA preparation was also slightly modified from the reported work by Rozi et al. [29]. In summary, 2.00 g ESM-WPFA, 3.17 g FeCl₂.4H₂O, and 7.57 g FeCl₃.6H₂O were mixed in 320 mL deionized water. The mixed solution was agitated for 1 hour at 80 °C. The reaction mixture was then rapidly infused with 40 mL of aqueous ammonia. The resulting mixture was stirred for another hour before allowed to cool to room temperature. The precipitated MNP@ESM-WPFA was washed five times with hot water until the filtrate becomes neutral, after which it was separated using magnetic decantation.

Batch adsorption studies

A certain amount of MNP@ESM-WPFA was mixed with 3 mL lubricating oil in 20 mL water at room temperature for 30 minutes according to the method of Rozi et al. [29]. Using an external magnet, the MNP@ESM-WPFA was separated from the water

surface after several minutes. The procedure is illustrated in Figure 1. The adsorption capacity was determined by weighing the sample before and after introducing it to the lubricating oil. Oil adsorption capacity *K* was calculated using Equation 1.

$$K = \frac{m_2 - m_1}{m_1} \quad (\text{eq. 1})$$

Where *m*₁ represents the weight before adsorption and *m*₂ represents the weight after adsorption.

In this experiment, several parameters such as contact time, adsorbent dosage, and pH of the solution were investigated for their influence in the removal of lubricating oil. Adsorption experiments were carried out in batches, with different conditions of the parameters which includes adsorbent dosage (10, 20, 30, 40, 50, and 60 mg), contact time (10, 20, 30, 40, 50, and 60 minute), and pH of the solutions (pH 2, 4, 7, 8, and 10). This is to determine the best conditions for the removal of lubricating oil using MNP@ESM-WPFA. The optimum conditions obtained from adsorption of lubricating oil were adopted to examine the performance of MNP@ESM-WPFA for removal of several oils including olive and corn oil. Experiments were performed in triplicate for each parameter. After adsorption, the MNP@ESM-WPFA adsorbent was rinsed with *n*-hexane to desorb the lubricating oil and then dried for 24 hours at 90 °C. The regenerated MNP@ESM-WPFA adsorbent was reused up to 5 times. The oil adsorption capacity of the regenerated adsorbent towards the removal of lubricating oil was again measured to determine its reusability.

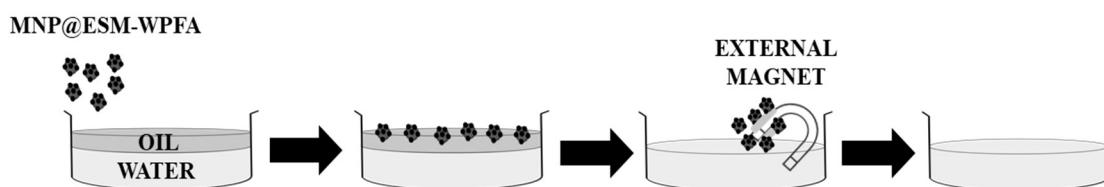


Figure 1. The schematic illustration of oil adsorption using magnetic adsorbent

Results and Discussion

Characterization

The FTIR spectra of ESM, ESM-WPFA, and MNP@ESM-WPFA are illustrated in Figure 2(A-C). Figure 2(A) shows the most significant peaks for ESM at 3429 cm⁻¹, 2933 cm⁻¹, 2852 cm⁻¹ and 1647 cm⁻¹. The

broad peak at 3429 cm⁻¹ can be assigned to the N-H and O-H stretching vibrations. The peaks at 2922 cm⁻¹ and 2852 cm⁻¹ are attributed to asymmetric and symmetric stretching of a methylene group (=CH₂) respectively, while the band at 1647 cm⁻¹ represents the stretching of the carbonyl group (C=O). In Figure 2(B), the high intensity of infrared bands at 2925 cm⁻¹ and 2855 cm⁻¹

for ESM-WPFA correspond to an alkane stretching, proving that the alkyl chain of the WPFA has been successfully functionalized onto the ESM surface. In

Figure 2(C), a new prominent band appears at 630 cm^{-1} due to the Fe-O group confirming the presence of Fe_3O_4 particles in MNP@ESM-WPFA.

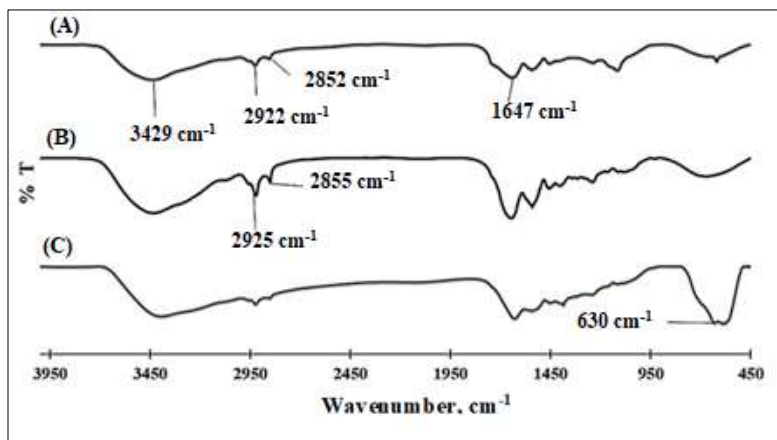


Figure 2. FT-IR spectra of (A) ESM (B) ESM-WPFA and (C) MNP@ESM-WPFA

The morphologies of ESM, ESM-WPFA and MNP@ESM-WPFA (Figure 3(A-C)) was examined by using scanning electron microscopy. The image of ESM shows that the virgin ESM has porous and fibrous surface (as circled in Figure 3(A)). The porous and fibrous structure of ESM-WPFA (Figure 3(B)) was

found to be smoother after functionalization of waste palm fatty acid (WPFA). Small particles scattered throughout the structure of ESM-WPFA (Figure 3 (C-D)) are the magnetic nanoparticles proving the successful synthesis of MNP@ESM-WPFA.

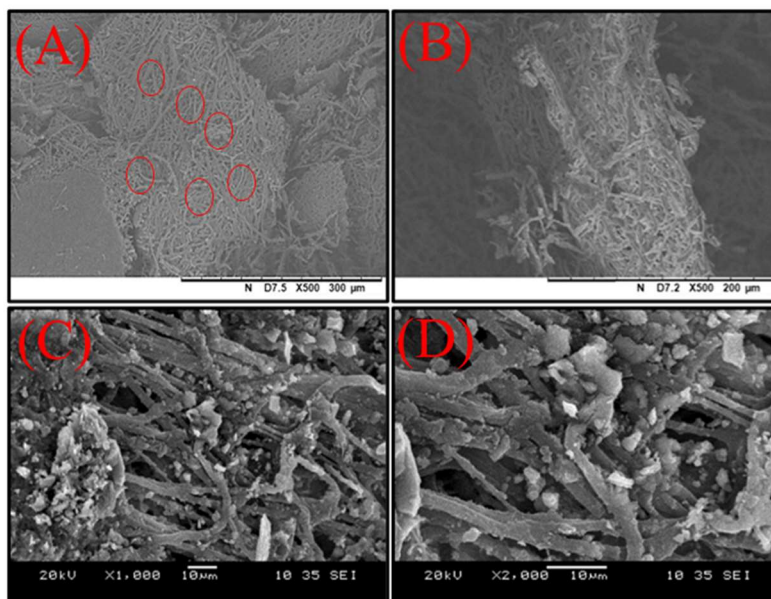


Figure 3. Morphological surface structure of (A) ESM (B) ESM-WPFA under 500× and MNP@ESM-WPFA under magnification of (C) 1000× (D) 2000×

The elemental composition of MNP@ESM-WPFA was examined using energy-dispersive X-ray spectroscopy (EDX) analysis. Figure 4 shows that MNP@ESM-

WPFA have an iron (Fe) content of 32%, carbon (C) content of 26%, nitrogen (N) content of 28%, and oxygen (O) content of 14%.

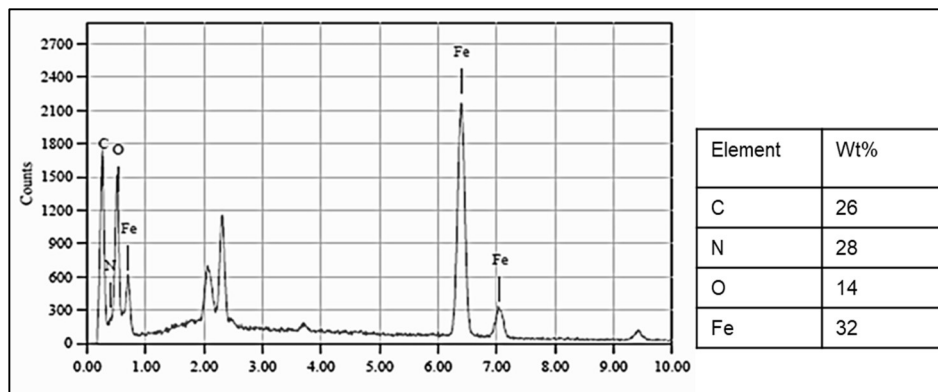


Figure 4. EDX spectrum of MNP@ESM-WPFA

The buoyancy of the synthesized nanosorbent was checked by placing it on a surface of water for 24 hours. Remarkably, the MNP@ESM-WPFA was found to float on water (Figure S1 (A) and (B)) , suggesting the fabricated adsorbents possess high buoyancy. In addition, The contact angle (CA) of MNP@ESM-WPFA was measured to be 123.6° (Figure S1 (C)) showing this adsorbent exhibits highly hydrophobic, which may be exploited for the adsorption of oil pollutants on the surface of water.

The optimization of the parameter for efficient oil removal:

Effect of treatment time

The effect of treatment time on oil adsorption using MNP@ESM-WPFA was studied within the range of 10

to 60 minutes. Figure 5(A) shows that the K value increase significantly until 50 minutes, after which it reaches a plateau (at time >50 min). As the treatment time increases, so does the mixing cycle; as the oil droplets have more time to break up and shrink in diameter (emulsification), resulting in a larger interfacial area for adsorption on the MNP@ESM-WPFA [30]. The curve shows that the maximum adsorption capacity was reached at 50 minutes of treatment. The highest K value obtained at 50 minutes of treatment is due to greater existence of interfacial surface of MNP@ESM-WPFA for adsorption, while the K value plateau after 50 minutes is due to the saturation of the surface of MNP@ESM-WPFA. Thus, 50 minutes was chosen as optimum treatment time for subsequent studies.

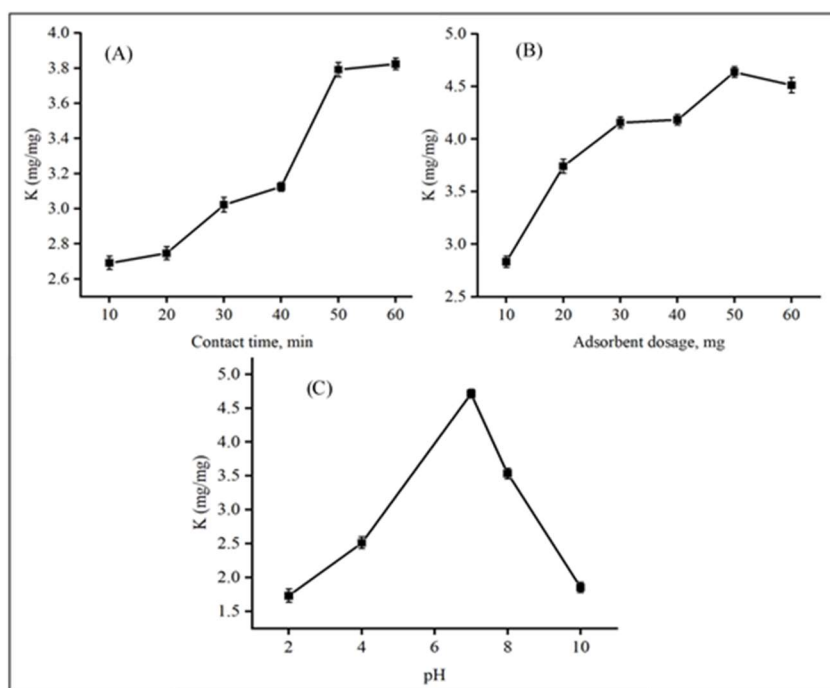


Figure 5. The effect of (A) contact time (B) adsorbent dosage (C) pH on oil adsorption capacity. The error bar represents the relative standard deviations of 3 measurements

Effect of adsorbent dosage

A batch experiment was carried out to determine the effect of adsorbent dosage (10 to 60 mg) on adsorption capacity. Figure 5(B) shows the relationship between adsorption capacity and the MNP@ESM-WPFA adsorbent dosage. Initially, the adsorption capacity increases together with increase of adsorbent dosage from 10 to 50 mg. The maximum adsorption capacity was reached when the dosage was 50 mg. After 50 mg, the adsorption capacity value decreased slightly, likely due to self-aggregation of the adsorbent which reduces the available sites for oil adsorption [31]. The optimum dosage for oil uptake using MNP@ESM-WPFA was thus set at 50 mg.

Effect of pH

The effect of the treatment pH on the oil adsorption process is presented in Figure 5(C). The adsorption process of oil on MNP@ESM-WPFA was found to be highly pH-dependent. The adsorption capacity rapidly increased from 1.73 to 4.71 mg/mg when the solution pH was increased from 2 to 7. However, at pH above 7 (pH 8-10), the adsorption capacity slowly decreased

until it reaches 1.86 mg/mg at pH 10. This phenomenon can be explained by the changes in the number of free protons in the solution and the stability of the oil emulsion [6]. In acidic conditions, the residual amine, carboxyl, and hydroxyl groups on the surface of MNP@ESM-WPFA becomes protonated, increasing the surface's cationic property and thus improving the adsorbent's hydrophilicity [29]. Additionally, in acidic conditions, as the acidity increased (from pH 7 to pH 2), the oil emulsion began to destabilize, causing clusters to form and larger oil droplets to form [30]. As a result, the interfacial area available for adsorption on MNP@ESM-WPFA decreases. On the other hand, the deprotonation of residual amine, carboxyl, and hydroxyl from ESM-WPFA in alkaline solution causes the adsorbent surface to become more anionic, reducing the oleophilic property of MNP@ESM-WPFA [29] and thus its oil adsorption capability. Thus pH 7 was selected as the optimum pH for oil uptake using MNP@ESM-WPFA.

The adsorption capacity of various oils

The adsorption capacities (K) of MNP@ESM-WPFA for lubricating oil, olive oil and corn oil were also

determined, and the results are shown in Table 1. MNP@ESM-WPFA was found to have the highest K value for lubricating oil, at 4.6 mg/mg, followed by olive oil (2.7 mg/mg) and corn oil (2.0 mg/mg). The difference in oil adsorption capacity is attributed to the

difference in the viscosity of the oils [32]. The adsorption capacity increases with oil viscosity due to improved oil adhesion onto the adsorbent surface [33-35].

Table 1. Adsorption capacities and viscosity values for studied oils. (Amount of MNP@ESM-WPFA = 50 mg, contact time = 50 min, and pH = 7)

Type of Oils	K, (mg/mg)	Viscosity, (mm ² /s)	Density, (kg/m ³)	Surface Tension, (mN/m)
Lubricating Oil	4.6	157.7	767.0	30.3
Corn oil	2.0	34.9	918.5	31.6
Olive oil	2.7	43.2	916.0	31.9

Reusability of MNP@ESM-WPFA

For practical application purposes, reusability is a crucial feature of any adsorbent. Here, the reusability study was carried out by repeating the lubricating oil adsorption experiment five times using the same adsorbent. Between each repeated runs, the adsorbent was cleaned with *n*-hexane and dried in an oven at 70 °C for one hour. Each experiment run used 50 mg MNP@ESM-WPFA, pH 7, and 50 min treatment time. The performance for five successive cycles is shown in Figure 6. Evidently, the regenerated MNP@ESM-WPFA can be recycled many times as it can adsorb oil 4.18 times its own weight even after five cycles.

Therefore, the synthesized adsorbent MNP@ESM-WPFA can be considered recyclable and stable. The high hydrophobic/oleophilic property also contributes to the excellent recyclability of the adsorbent. This is because when hydrophobicity and oleophilicity are introduced to a sorbent, it tends to concentrate and interact with the oil phase medium, hence, transforming it to a semisolid phase via Van der waals forces [36]. This makes it easier for desorption. This characteristic suggests that as-prepared MNP@ESM-WPFA shows a great potential application in multifunctional wastewater purification.

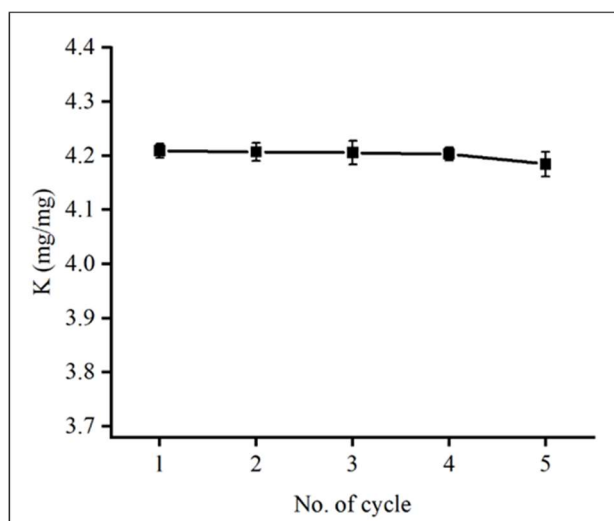


Figure 6. Oil-adsorption capacity of MNP@ESM-WPFA at different cycles. The error bar length accounts for the relative standard deviations for 3 measurements

Analysis of Oil-Loaded MNP@ESM-WPFA

The surface of lubricating oil loaded MNP@ESM-WPFA was evaluated using FTIR, SEM and EDX analyses. Figure 7 depicts the FTIR analysis of MNP@ESM-WPFA with and without oil loading (A-B). When comparing the original MNP@ESM-WPFA (Figure 7(A)) spectrum to the oil loaded MNP@ESM-

WPFA spectrum (Figure 7(B)), it is observed that the intensity of $\text{-CH}_2\text{-}$ group bands at 2853 cm^{-1} and 2923 cm^{-1} are substantially higher for oil loaded MNP@ESM-WPFA. This result proves that a significant amount of oil adsorption occurred at the hydrophobic tails of waste palm fatty acid on the surface of MNP@ESM-WPFA.

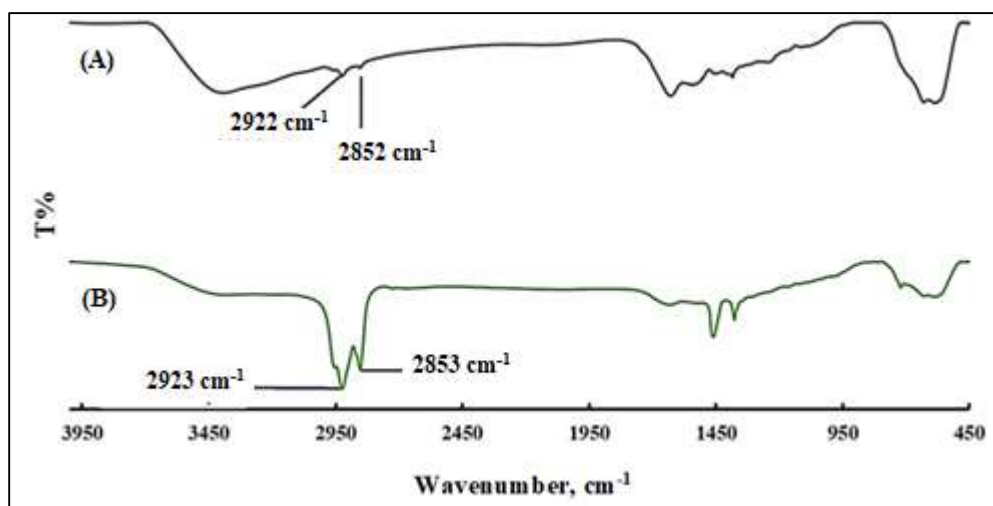


Figure 7. FTIR spectra of (A) MNP@ESM-WPFA and (B) lubricating oil loaded MNP@ESM-WPFA

Figure 8 shows the SEM images of MNP@ESM-WPFA before and after the lubricating oil adsorption process. The SEM images of oil-loaded MNP@ESM-WPFA (Figure 8(C-D)) show substantial differences from the unloaded MNP@ESM-WPFA (Figure 8(A-B)). The oil loaded MNP@ESM-WPFA surface was covered with a layer of oily mud-like material, which is the adsorbed oil molecules.

Elemental analysis reveals that the mass content of C (33%) in the oil-loaded MNP@ESM-WPFA (Figure 9(B)) is notably higher than the percentage of C discovered in the unloaded MNP@ESM-WPFA (26%) (Figure 9(A)). These results align with that obtained from the FTIR analysis for oil loaded MNP@ESM-WPFA (Figure 7(B)) indicating that a higher carbon content causes a stronger band intensity for the alkane group in oil-loaded MNP@ESM-WPFA.

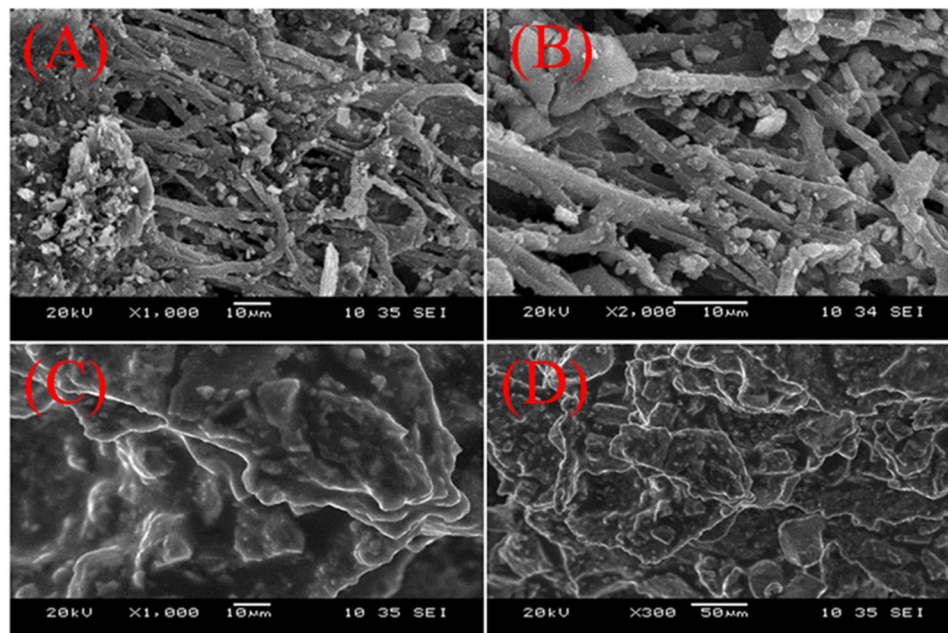


Figure 8. Morphological surface of MNP@ESM-WPFA under different magnifications (A) x 1000 (B) x 2000 and lubricating oil loaded MNP@ESM-WPFA under different magnifications (C) X 1000 (D) X 300

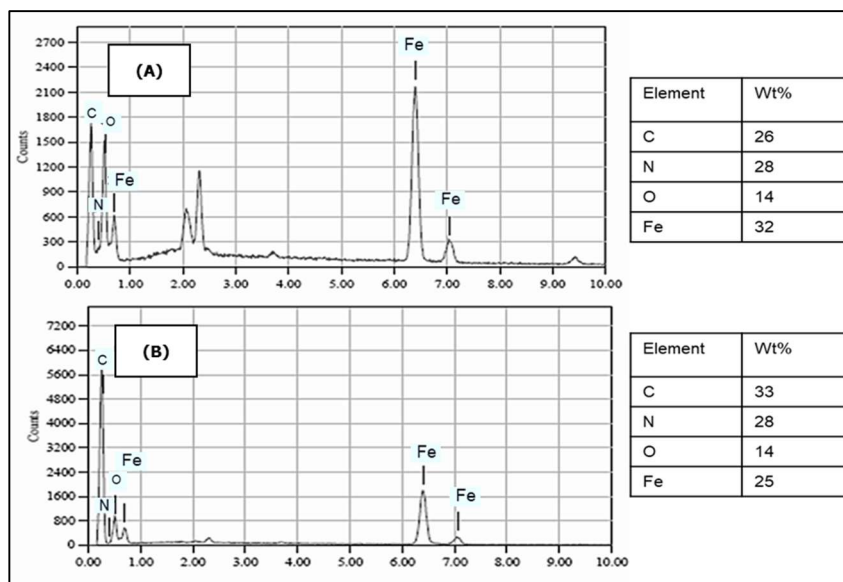


Figure 9. EDX spectra of (A) MNP@ESM-WPFA and (B) Oil Loaded MNP@ESM-WPFA

Comparison of MNP@ESM-WPFA with other adsorbents

Table 2 summarizes and compares this work with previous works reported in literature. MNP@ESM-WPFA shows the highest adsorption capacity among the

published works reported in the table, suggesting the potential of MNP@ESM-WPFA usage in water remediation. Furthermore, the proposed approach offers numerous other benefits, such as simplicity, ease of removal, and operation.

Table 2. Comparison to oil adsorbents from the literature

Adsorbent	Emulsified Oil Studied	Sorption Capacity (mg/mg)	References
MNP-FA	Lubricating oil	3.50	[29]
ACF-PO	Lubricating oil	3.03	[37]
Fe ₃ O ₄ @PS	Motor oil	3.00	[38]
Fe ₃ O ₄ @PVP	Lubricating oil	3.63	[39]
MNP@ESM-WPFA	Lubricating oil	4.61	This study

Conclusion

This present study shows that the synthesized MNP@ESM-WPFA can be potentially used for the removal of oil from an oil-water system. SEM, FTIR, and EDX analyses confirmed the successful synthesis of MNP@ESM-WPFA. The oil adsorption performance of MNP@ESM-WPFA was optimum at pH 7, 50 minutes treatment time, and 50 mg adsorbent dosage. MNP@ESM-WPFA is versatile as it can adsorb several types of oil, which are, for this study, lubricating oil, corn oil and olive oil. The MNP@ESM-WPFA has an adsorption capacity of 4.6 mg/mg for lubricating oil, 2.0 mg/mg for corn oil, and 2.7 mg/mg for olive oil. The MNP@ESM-WPFA can also be reused up to 5 times without losing efficiency. FTIR, SEM, and EDX analyses proved that the oil was adsorbed by MNP@ESM-WPFA, as evidenced by the intense peaks attributed to the oil in the FTIR spectrum, the mud-like covering seen in the SEM images, and the increase in C content during the EDX analysis. It is suggested that the oil droplets adsorbed on MNP@ESM-WPFA interacts with the hydrophobic tails of waste palm fatty acid via hydrophobic interaction. This study opens new possibilities for developing high-efficiency, recyclable demulsifiers for emulsified oil wastewater.

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