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Diatomaceous earth incorporated floating magnetic beads for oil removal on water

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ABSTRACT

Water contamination by oil spills causes severe damage to the environment and human health. Oil adsorbents should be buoyant, effective oil-contact, and regenerable. In this study, diatomaceous-earth (DE)-incorporated magnetic alginate (Alg)-based beads, chemically modified to include phthalic or maleic anhydride (denoted as Alg/DE/Fe₃O₄-PA and Alg/DE/Fe₃O₄-MA), were developed. Internal cavities, formed by cavity-forming agents, made them buoyant. The beads were buoyant and magnetically responsive for 30 days. Maximum oil adsorption capacities of Alg/DE/Fe₃O₄-PA and Alg/DE/Fe₃O₄-MA were 29.7 and 21.0 times their weights, respectively. The pseudo-second order kinetics model ($R^2 \sim 0.999$, $ARE \leq 1.088$, and $\chi^2 \leq 0.041$) and Freundlich isotherm model ($R^2 \sim 0.999$, $ARE \leq 0.1$, and $\chi^2 \leq 3.37 \times 10^{-3}$) were used to determine these values. Both beads were magnetically regenerable for up to 20 cycles. DE pores improved oil adsorption capacity by facilitating intra-bead diffusion of surface adsorbed oil. This was confirmed by micro-structural characterization using surface micrographs, elemental distribution, porosity, thermal stability, crystalline phases, molecular vibrational behavior, and magnetic properties. Scanning electron microscopy and X-ray diffractometry analyses revealed spherical beads with well-distributed Fe₃O₄ spinel on the surfaces. The two adsorbent beads satisfied the criteria for oil-removal from water: buoyancy, high affinity towards various oil types, and magnetic re-collectability.

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1. Introduction

Seawater contamination due to deliberate or accidental oil spills is a global concern. Growing fossil fuel demand has increased oil spill frequency during oil exploration, transportation, refining, and use. In the Deepwater Horizon oil spill

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(the largest recorded oil spill in history) in the Gulf of Mexico (April 20–July 15, 2010), more than 3.19 million barrels of oil were accidentally released into the sea, covering the northern gulf coast by an oil layer (Beyer et al., 2016). Oil residues were detected in the five U.S. Gulf states of Texas, Louisiana, Mississippi, Alabama, and Florida (Thyng, 2019). A large film of low-biodegradable oil, which spreads rapidly over water, is formed during an oil spill. This floating layer is hazardous to marine biota and surrounding ecosystems (Campelo et al., 2021).

Numerous techniques have been developed for oil spill clean-up. Thermal, physical, chemical, and biological techniques, including in-situ burning, using boom-skimmers, dispersants and surfactants, have been used for bioremediation and oil adsorption (Abidli et al., 2020; Bullock et al., 2019; Hoang, 2018; Ke et al., 2021). In-situ burning is a large-scale method that removes oil from water surfaces by direct oil burning (Hoang, 2018). The combustion forms toxic gases, and residues that sink to the seabed, harming marine biota. Additionally, the spread of fire is difficult to control. The boom-skimmer method consists of two stages: localizing oil spills with booms and carrying oil into a reservoir using mechanical devices called skimmers (Wang et al., 2018). This method requires specific expensive tools. Bacterial consortiums can be used for oil bioremediation with limited efficiency. Not all bacterial species degrade oil, and those that do are substrate-specific (Mapelli et al., 2017). Simple and efficient methods for oil spill clean-up and oil separation are scarce. Oil removal from water surfaces using sorbents and sorbent-devices is eco-friendly and cost-effective (Hoang et al., 2021). Several low-cost adsorbents, such as biochar (Gurav et al., 2021), carbon (Yang et al., 2021), aluminosilicate clay (Akpomie et al., 2019), and cellulose-based materials (Chau et al., 2021; Hoang et al., 2018b), have been extensively investigated for oil adsorption. Oil-contaminated water clean-up adsorbents should have buoyancy, high affinity towards different oil types, and be recoverable for long-term applications.

Alginate (Alg) is naturally abundant and can be extracted from brown algae and seaweeds (Hambali et al., 2018; Nizami et al., 2020). It entraps water pollutants (heavy metal ions, basic dyes, and pharmaceuticals) due to its high content of acidic carboxylate groups (Nasrollahzadeh et al., 2021). However, recovery of contaminant-loaded Alg hydrogel from the medium is difficult, limiting its utility as an adsorbent. To overcome this limitation, magnetic iron-based particles of magnetite (Fe_3O_4) have been used (Ali et al., 2021). In this study, magnetic Alg was physically modified with diatomaceous earth (DE), followed by hollow bead-shaping in an ionic crosslinking solution to enhance buoyancy, porosity, surface area, and thermal stability. As reported in a previous publication, NaHCO_3 and CaCO_3 form internal cavities, endowed with magnetic beads, for buoyancy and stability. These properties facilitate oil removal from water surfaces (Sakti et al., 2021). DE filler was used for its physicochemical properties, such as particle size, surface area, porosity, thermal stability, and acid resistance. DE is an eco-friendly adsorbent (Sriram et al., 2020), sensor (Leonardo et al., 2016), catalyst (Đặng et al., 2021), and drug delivery system (Phogat et al., 2021). Although DE-based adsorbents effectively adsorb water pollutants, their sinkable behavior and difficult post-adsorption recovery make investigations challenging, and adsorption information scarce. There are no published reports on oil-contaminated water clean-up using DE-based floating adsorbents.

This study evaluated oil removal from water surfaces using floating DE-incorporated magnetic beads. Chemical modification of attaching acid anhydride groups, by adding phthalic anhydride (PA) and maleic anhydride (MA), was performed to enhance adsorption affinity of the beads towards various oil types, on the surface of various water types. Oil removal tests were performed by varying parameters such as initial pH, contact time, initial oil concentration, oil type, and water type, to evaluate oil clean-up feasibility of Alg/DE/ Fe_3O_4 -MA and Alg/DE/ Fe_3O_4 -PA. Kinetics, isotherms, recoverability, recyclability, floating stability, and magnetically driven properties were also studied. Physical and chemical properties of Alg/DE/ Fe_3O_4 -MA and Alg/DE/ Fe_3O_4 -PA that influence the clean-up performance were evaluated by various techniques. The magnetic, buoyant, and recyclable Alg/DE/ Fe_3O_4 -MA and Alg/DE/ Fe_3O_4 -PA beads facilitated effective, rapid, and eco-friendly oil-contaminated water clean-up.

2. Materials and methods

2.1. Materials

Analytical quality materials and chemicals were used in the experiment and used as received without any further purification, unless otherwise specified. DE and sodium alginate ($\text{Na}(\text{C}_6\text{H}_6\text{O}_6)_n$) were supplied by Wako (Japan). Magnetite (Fe_3O_4 , >90%) was obtained from Kishida Chemical Co., Ltd. (Japan). Acetic acid (CH_3COOH , 98%), acetone ($\text{C}_3\text{H}_6\text{O}$, 99.5%), ammonium hydroxide (NH_4OH , 28%–30%), calcium carbonate (CaCO_3 , 99.5%), hydrochloric acid (HCl, 37%), sodium bicarbonate (NaHCO_3 , 99.8%), and sodium hydroxide (NaOH, 97%) were purchased from Sigma-Aldrich (Germany). Calcium chloride ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 100%), ethanol ($\text{C}_2\text{H}_5\text{OH}$, 97%), hexane (C_6H_{14} , 99%), MA ($\text{C}_4\text{H}_2\text{O}_3$, 99%), and PA ($\text{C}_6\text{H}_4(\text{CO})_2\text{O}$, 99%) were obtained from Merck (Germany). Artificial seawater powder (Marine Art-SF-1) was purchased from Osaka Yakken Co., Ltd. (Japan). Castor oil, coconut oil, olive oil, and vegetable oil were purchased from a local market. Crude oil was obtained from oil drilling in Indonesia. Tap water was collected from our university.

2.2. Fabrication of buoyant DE/magnetic Alg composite bead

DE was activated through 3 M HCl solution contact for 2 h, 4000 rpm centrifugation for 15 min, repeated neutralization with demineralized water, and 100 °C oven drying for 12 h. Activated DE (0.25 g) was dispersed in 50 mL 1.5 wt% Alg solution and mixed at 750 rpm for 30 min. Fe_3O_4 , NaHCO_3 , and CaCO_3 (1:1:1) were dispersed in the mixture and stirred

for 30 min. The black viscous suspension obtained was dropped into 300 mL of 5 wt% CaCl₂ solution and stirred for complete gelation. Black beads formed were collected using a magnet, immersed in 300 mL of 6% CH₃COOH solution for 24 h, repeatedly neutralized with water, lyophilized, and labeled as Alg/DE/Fe₃O₄. Alg/Fe₃O₄ was prepared using a similar method, without DE addition.

2.3. Surface functionalization of the composite bead with acid anhydride

Alg/DE/Fe₃O₄ was stirred in acetone overnight before modification. It was refluxed with 0.05 mol L⁻¹ PA in acetone at 55 °C for 3 h. After esterification, the black beads were magnetically recovered and washed with acetone, ethanol, and water. The lyophilized beads were labeled Alg/DE/Fe₃O₄-PA and used for characterization and adsorption tests. Alg/DE/Fe₃O₄-MA was prepared by a similar method, with MA used for modification instead of PA. Bead preparation and modification are shown in Fig. S1.

2.4. Characterization of as-prepared beads

Bead morphology was examined using a FlexSEM-1000 system (Hitachi, Japan) at 5 kV accelerating voltage. Freeze-dried beads were coated with a thin gold layer, using a sputtering system, to enhance electrical conductivity. Elemental composition was determined, and mapping was conducted, using an EDX system. Crystal structure was observed using X-Pert MPD (Philips, Germany) with 1.54443 Å Cu-K_α radiation, 5–90° diffraction angle, and 5 °C min⁻¹ scan step. Thermal degradation was evaluated using a TGA 4000 system (Perkin Elmer, USA). Dried beads (15 mg) were placed inside a crucible and heated under pure N₂ atmosphere, from 30–800 °C, at a heating rate of 5 °C min⁻¹, and 20 mL min⁻¹ N₂ flow rate. Magnetic properties were examined using a VSM-7400 series (Lake Shore, USA), by exposing dried beads (25 mg) to -8–8 kOe magnetic field range, at room temperature. The Brunauer–Emmett–Teller (BET) surface area, pore volume, and pore size distribution were analyzed using an accelerated surface area and porosimetry system (ASAP 2020, Micromeritics, USA). An IRTracer-100 spectrophotometer (Shimadzu, Japan) was used to investigate functional moieties on each bead. Powdered beads were mixed with KBr, and multiple scans were collected at 4 cm⁻¹ resolutions, in the 4000–400 cm⁻¹ wavenumber range. Alg/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA bead sizes were measured using a digital caliper (Krisbow, Indonesia).

2.5. Oil removal and bead regeneration experiments

Coconut oil, vegetable oil, olive oil, and castor oil are widely used, and commonly found in domestic wastewater. Crude oil, an oil-drilling product, can be found in seawater around ports and oil-storage warehouses in Indonesia. Therefore, these oil removals were chosen as objects of research and analysis. Oil clean-up performance by Alg/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA beads, at variable oil–water mixture pH, contact time, and initial oil concentration, have been published previously with a slight modification (Singh et al., 2017; Soares et al., 2017; Yaacob et al., 2018). The synthesized beads (0.015 g) were placed in contact with 15 mL of 66.67 g L⁻¹ oil–water mixture, to investigate the influence of oil–water mixture pH on oil removal. Initial pH of the mixture was regulated using 0.1 mol L⁻¹ HCl and 0.1 mol L⁻¹ NaOH, and the mixture was equilibrated using an orbital shaker at 200 rpm for 2 h. Oil adsorbed by the beads at equilibrium (q_e , g of oil g of beads⁻¹) was calculated using Eq. (1) shown below (Hoang et al., 2018a):

$$q_e = \frac{m_{oil}}{m_0} = \frac{m_{total} - m_{water} - m_0}{m_0} \quad (1)$$

Here, m_{oil} and m_{water} are the masses (g) of adsorbed oil and water, respectively; m_{total} and m_0 are the bead masses (g) before and after adsorption, respectively. Similar protocols were used to examine adsorption kinetics and isotherms, by varying contact time in the range 5–120 min. Initial oil concentration was varied from 40 to 120 g L⁻¹. Experimental setup and tools for examining these variables are presented in Fig. 1.

Regeneration potential was investigated by equilibrating the beads with 15 mL of 100 g L⁻¹ oil–water mixture at 200 rpm for 2 h. The beads were recovered magnetically, and repeatedly washed with n-hexane, methanol, water, and lyophilized before the next cycle. Twenty adsorption–desorption cycles were conducted to assess oil removal performance of the beads. Even though all adsorption experiments were carried out using the same method and under the same conditions, errors in the experiments could not be eliminated. Therefore, all oil adsorption measurements were performed in triplicate to maximize accuracy and reduce experimental errors.

3. Results and discussion

3.1. SEM analysis and EDX mapping

The micrographs show a significant difference between micro-surface and cross-section construction of the synthesized beads (Fig. 2). The surface micrograph showed spherical Alg/Fe₃O₄ beads with a coarse surface. Fe₃O₄ microaggregates were distributed all over the surface, confirming Fe₃O₄ attachment to the Alg matrix. Sieve-shaped DE disks were observed

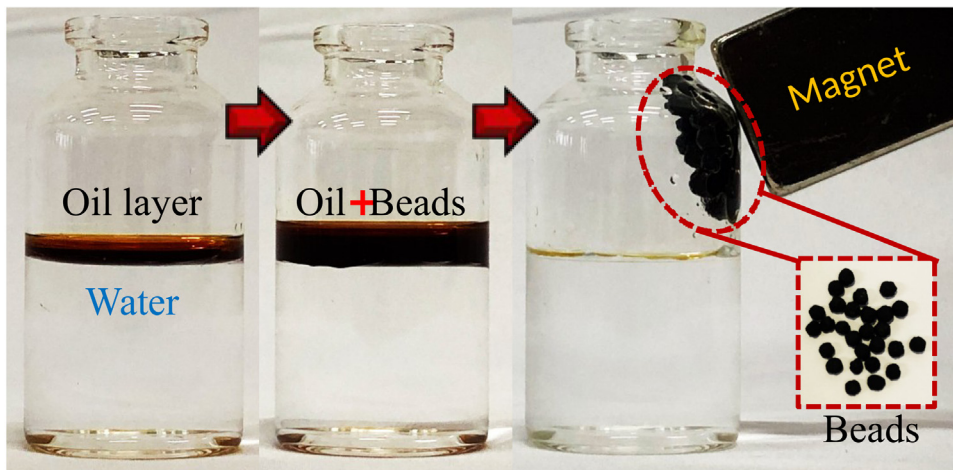


Fig. 1. Experimental setup of oil adsorption by as-synthesized beads.

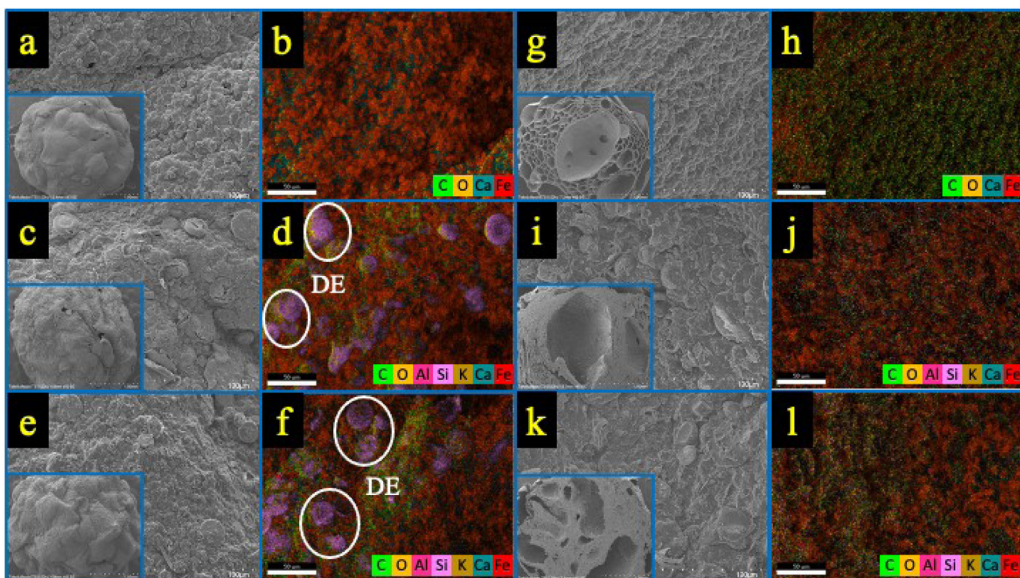


Fig. 2. Surface SEM micrograph (inset: whole bead micrograph) and EDX-mapping of (a,b) $\text{Alg}/\text{Fe}_3\text{O}_4$, (c,d) $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-MA}$ (e,f) $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-PA}$ and cross-section SEM micrograph (inset: whole bead micrograph) and EDX-mapping of (g,h) $\text{Alg}/\text{Fe}_3\text{O}_4$, (i,j) $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-MA}$, and (k,l) $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-PA}$.

on the $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-MA}$ and $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-PA}$ micro-surfaces, confirming homogeneous blending of DE. Compared to $\text{Alg}/\text{Fe}_3\text{O}_4$, $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-MA}$ and $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-PA}$ had rougher (with many wrinkles) surfaces with more pores, facilitating rapid intra-bead diffusion. An oval-shaped cavity was observed in the cross-sectional micrographs of $\text{Alg}/\text{Fe}_3\text{O}_4$, $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-MA}$, and $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-PA}$, due to the reaction of NaHCO_3 (as a porogen inside beads) with acetic acid. To maintain its spherical structure after NaHCO_3 dissolution by acetic acid, CaCO_3 was added as an internal crosslinking agent. Alg reacted with Ca^{2+} , a product of CaCO_3 dissolution, to form a rigid structure through ionotropic gelation.

Ca distribution on the outer and inner surfaces of the synthesized beads indicated complete ionotropic gelation. EDX mapping confirmed the presence of Fe on both outer and inner surfaces of $\text{Alg}/\text{Fe}_3\text{O}_4$, indicating successful entrapment of Fe_3O_4 in the Alg matrix (Fig. 2). DE presence in $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-MA}$ and $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-PA}$ was confirmed by observing Si (the major component of DE) distribution. CO_2 formation during NaHCO_3 and CaCO_3 dissolution forced inner DE to the outer surface, causing higher Si detection on the outer surface of $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-MA}$ and $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-PA}$, than the inner surface. $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-MA}$ and $\text{Alg}/\text{DE}/\text{Fe}_3\text{O}_4\text{-PA}$ surface contained 23%–27% Si, facilitating intra-bead diffusion. Small quantities (<5%) of DE minor components, Al and K, were also detected. Si, Al, and K were not detected on the $\text{Alg}/\text{Fe}_3\text{O}_4$ surfaces.

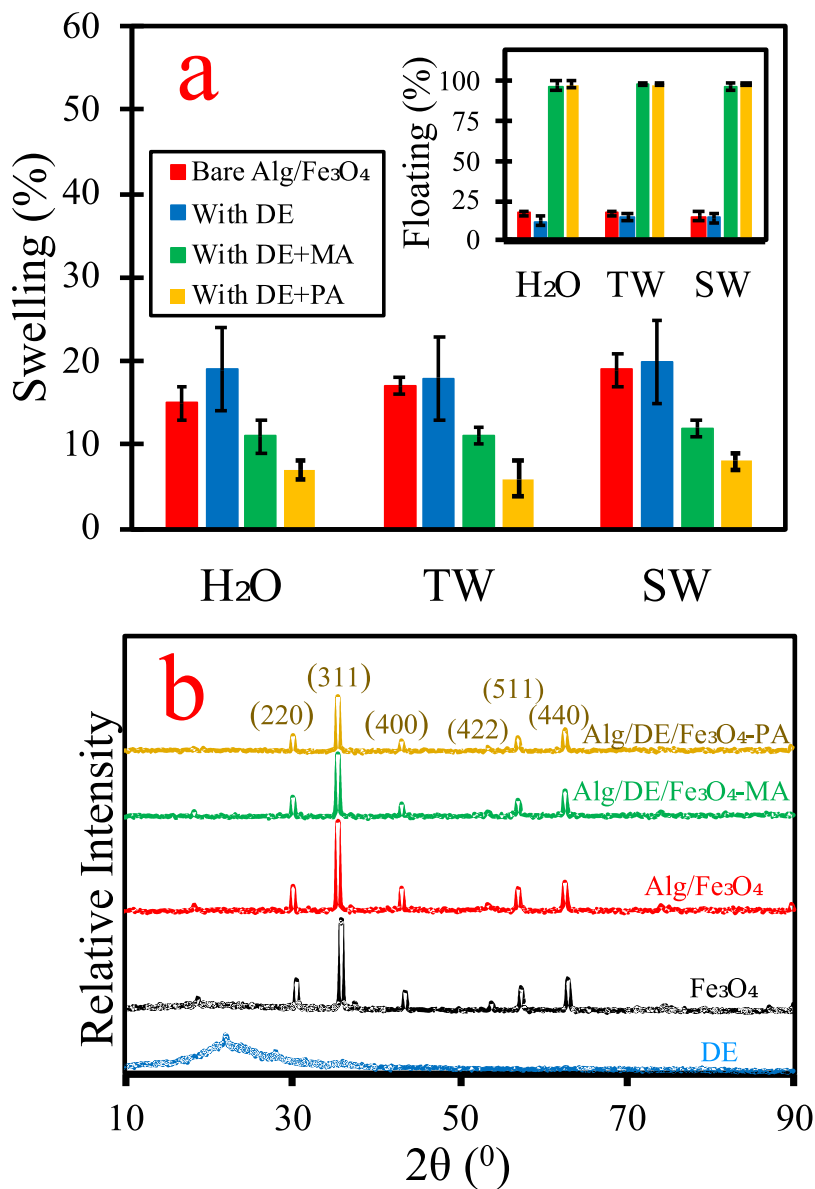


Fig. 3. (a) Swelling property (inset: self-floating property) in demineralized water, tap water and seawater and (b) XRD diffractogram of Alg/Fe₃O₄, Alg/DE/Fe₃O₄, Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA.

3.2. Physical dimensions, swelling, and buoyancy

Alg/DE/Fe₃O₄-PA and Alg/DE/Fe₃O₄-MA were observed to slightly increase in size, with unmodified shape, on DE entrapment and PA/MA attachment (Table 1). Penetration of water molecules into the gel bead-matrix through DE pores caused swelling. After MA/PA modification, bead surface hydrophilicity decreased, reducing water penetration into the gel matrix, decreasing the swelling ratio. Spherical shape enabled multi-directional oil diffusion into Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, and both beads floated on demineralized water, tap water, and seawater, maintaining buoyancy for up to 30 days (Fig. 3(a)). Thus, high contact occurred between water-surface oils and Alg/DE/Fe₃O₄-MA, or Alg/DE/Fe₃O₄-PA.

3.3. XRD analysis

As shown in Fig. 3(b), sharp peaks observed in the Fe₃O₄ XRD pattern at 2θ values of 30.46°, 35.84°, 43.50°, 53.98°, 57.38°, and 62.92° corresponded to (220), (311), (400), (422), (511), and (440) plane reflections (Choochian et al., 2019; Sakthi Sri et al., 2020) of cubic Fe₃O₄ (JCPDS card No. 79-0418), respectively. For DE, amorphous and quartz phases of

Table 1Physical dimension, textural and magnetic properties of Alg/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA.

Magnetic bead	Component	Average bead size ^a (mm)	S _{BET} ^b (m g ⁻¹)	V _T ^b (cm ³ g ⁻¹)	D _p ^b (nm)	H _c ^c (G)	M _s ^c (emu g ⁻¹)	M _r ^c (emu g ⁻¹)	Mr/Ms
Alg/Fe ₃ O ₄	Alginate, Fe ₃ O ₄	2.81 ± 0.11	15.476	0.017	4.161	130.450	41.489	5.313	0.128
Alg/DE/Fe ₃ O ₄ -MA	Alginate, Fe ₃ O ₄ , Diatomaceous earth, Maleic anhydride	3.01 ± 0.21	24.740	0.038	4.931	118.440	29.008	3.484	0.120
Alg/DE/Fe ₃ O ₄ -PA	Alginate, Fe ₃ O ₄ , Diatomaceous earth, Phthalic anhydride	3.14 ± 0.19	35.058	0.045	5.832	120.440	23.094	2.850	0.123

^aAverage bead size was measured using a digital caliper (n = 100).^bS_{BET}, V_T and D_p were obtained from N₂ adsorption-desorption analysis.^cH_c, M_s, and M_r were measured using a vibrating sample magnetometer (VSM).

SiO₂ showed a broad peak centered at 22.14°, and a sharp peak at 28.20°. Crystalline quartz is commonly present in DE (Mota dos Santos and Cordeiro, 2021). After co-entrapment of Fe₃O₄ and DE in Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, only peaks associated with Fe₃O₄ were detected in the diffractogram. This phenomenon is observed for Fe₃O₄ entrapment in other materials (Ewis et al., 2020; Li et al., 2020; Nuryono et al., 2016). This could be due to distribution of crystalline Fe₃O₄ on Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA surfaces, as confirmed by SEM analysis. Amount of entrapped Fe₃O₄ was confirmed to be higher in Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, than DE, by EDX mapping.

3.4. Thermal stability

TGA and DTG curves of Fe₃O₄, Alg, Alg/Fe₃O₄, Alg/DE/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA, are presented in Fig. 4(a–b). No decomposition was observed for Fe₃O₄ over the entire temperature range examined, because of its high thermal stability. In contrast, the thermograms of bare Alg beads and composite beads exhibited multistage degradation, with 205.65, 273.05, and 737.37 °C T_{max} values, respectively, corresponding to multi-step decomposition. Mass degradation at temperatures below 200 °C is associated with release of physically bound water molecules (Rigueto et al., 2021). In the first stage (below 200 °C), the bare Alg beads and Alg/Fe₃O₄ exhibited 9% and 7.78% mass losses, respectively, while Alg/DE/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA lost less than 5% of their masses. In the second degradation stage (200–350 °C), caused by combustion of organic material, 38.32% bare Alg beads were degraded at T_{max} (273.05 °C) through polysaccharide ring splitting (Metin et al., 2020). In contrast, Alg/Fe₃O₄ and Alg/DE/Fe₃O₄ exhibited 14.52% and 12.24% mass losses, respectively, indicating suppression of decomposition, and enhancement of thermal stability on introduction of Fe₃O₄ and DE into the Alg matrix. Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA experienced 14% mass loss due to combustion of Alg matrix and MA/PA groups. In the final stage, 26.22% bare Alg bead mass was retained as an inorganic residue from the carbonization process (da Silva Fernandes et al., 2018), while the residual masses of Alg/Fe₃O₄, Alg/DE/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA, were 66.24%, 72.71%, 64.86%, and 64.02%, respectively. This indicated improvement of Alg bead thermal stability by more than 2.4-fold on introduction of Fe₃O₄, DE, MA, and PA. By comparing their residual masses to that of Alg/DE/Fe₃O₄, the amount of MA and PA attached to the surface of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA were found to be 7.85% and 8.69%, respectively.

3.5. Texture and pore distribution

As presented in Fig. S2(a–c), the N₂ adsorption/desorption isotherms were type IV, according to International Union of Pure and Applied Chemistry (IUPAC) classification, indicating mesoporous nature of Alg/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA (Song et al., 2019). A mesoporous H4 type hysteresis loop could be observed in the isotherms at P/P⁰ values of 0.21–0.86, 0.35–0.8, and 0.25–0.81 for Alg/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA, respectively. Owing to DE on the surfaces, an H4 type hysteresis loop, associated with the presence of slit-like pores, was more evident in Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, than in Alg/Fe₃O₄ (Song et al., 2021). Textural parameters and pore structures determined by N₂ adsorption/desorption experiments are listed in Table 1. Owing to higher porosity, Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA could generate more active sites for oil removal than Alg/Fe₃O₄.

3.6. Magnetic responsiveness of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA

Magnetic responsiveness determined recovery of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA from oil–water mixtures. As shown in Table 1 and Fig. S2(d), M_s value of Alg/Fe₃O₄ was lower than that of bare Fe₃O₄ (75.7 emu g⁻¹). DE entrapment and anchoring of magnetically non-responsive PA or MA, further reduced M_s values in Alg/DE/Fe₃O₄-PA and Alg/DE/Fe₃O₄-MA by 2.6-fold and 3.2-fold, respectively, compared to bare Fe₃O₄. Encapsulation effects, such as the presence of magnetically non-responsive Alg, DE, PA, and MA, caused this reduction. Similar phenomenon, on coating magnetic cores by different magnetically non-responsive matrices in similar materials, has been previously reported (Narita et al., 2019; Nuryono et al., 2020, 2014; Sakti et al., 2020, 2015; Armedya et al., 2019; Nuryono et al., 2014; Fahmi et al., 2020).

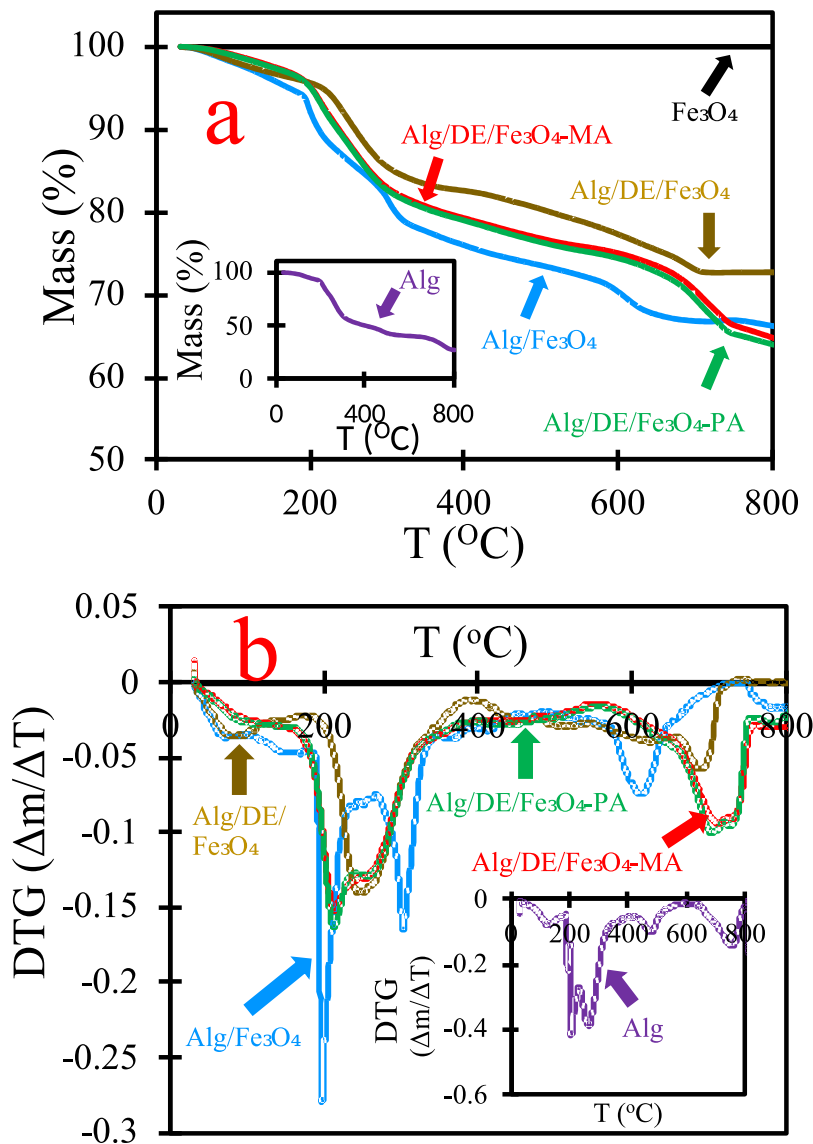


Fig. 4. (a) TGA thermogram (inset: thermogram of non-magnetic alginate) and (b) DTG thermogram (inset: thermogram of non-magnetic alginate) of Alg/Fe₃O₄, Alg/DE/Fe₃O₄, Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA.

3.7. Oil removal performance of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA

3.7.1. Effect of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA dosage and initial pH

As illustrated in Fig. 5(a), 0.25 g L⁻¹ Alg/DE/Fe₃O₄-PA demonstrated higher adsorption capacity (19.2 g g⁻¹) than Alg/Fe₃O₄ and Alg/DE/Fe₃O₄-MA. At higher adsorbent dosage (1 g L⁻¹), it exhibited 1.5-fold increase in adsorption capacity, due to the presence of more vacant adsorption sites. However, increasing adsorbent dosage further (> 1 g L⁻¹) caused insignificant change in oil adsorption due to reduction in contact area of oil with Alg/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA, making fewer vacant sites and pores accessible.

Fig. 5(b) depicts oil adsorption efficiency as a function of pH, at optimum adsorbent to oil-water volume ratio. Oil adsorption changed negligibly with pH variation, indicating predominantly non-electrostatic forces in interfacial interactions of oil with Alg/Fe₃O₄, Alg/DE/Fe₃O₄-MA, and Alg/DE/Fe₃O₄-PA. At pH 6, considered to be the optimum pH, adsorption efficiency exhibited 9.5-fold increase in Alg/DE/Fe₃O₄-PA compared to Alg/Fe₃O₄. Phthalic benzene ring π - π interactions in Alg/DE/Fe₃O₄-PA enhanced adsorption efficiency by amplifying non-electrostatic interactions. Oil adsorption was not conducted under highly basic conditions to avoid saponification of the oil layer by OH⁻, and to prevent bead rupture and DE release by Ca(OH)₂, formed by reaction of Ca²⁺ (crosslinker) with excess OH⁻.

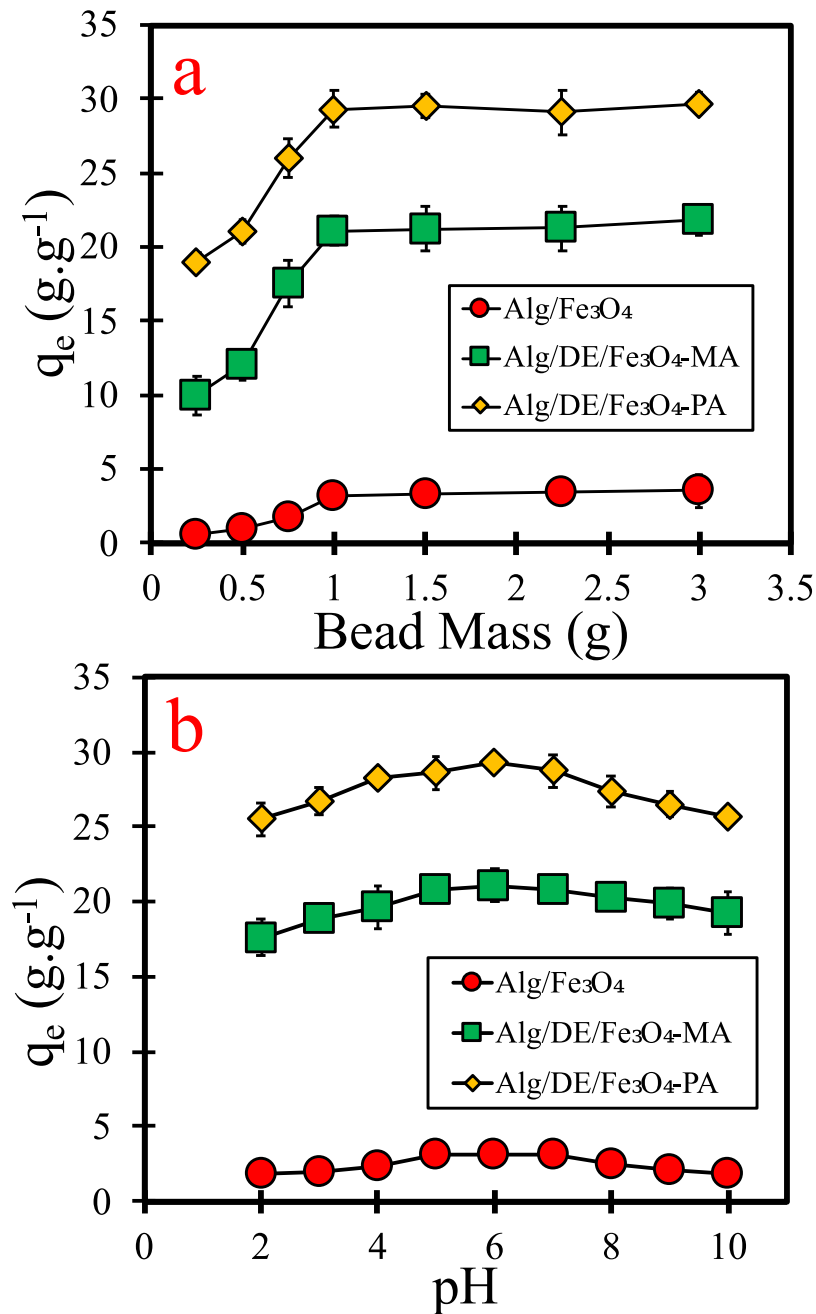


Fig. 5. Effect of (a) Alg/Fe₃O₄, Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA dosage (condition: C₀: 66.67 g L⁻¹, pH: 6, t: 2 h, T: 25 °C, and n: 3) and (b) initial pH on removal of crude oil (condition: C₀: 66.67 g L⁻¹, bead dose: 1 g L⁻¹, t: 2 h, T: 25 °C, and n: 3).

3.7.2. Influence of contact time on oil removal

Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA adsorbed oil up to 8.7 and 12 times their weights in the first 5 min, respectively, and oil adsorption increased exponentially with increasing contact time (Fig. 6(a)). This could be due to attachment of oil to the available active sites and pores of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA. Gradually, most active sites and pores were occupied by oil, and equilibrium, indicated by a plateau region in the adsorption curve, was attained within 30 min. After equilibrium, oil adsorption by Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA became constant. The as-prepared beads attained oil-removal equilibrium within a short time, indicating their applicability in large-scale systems.

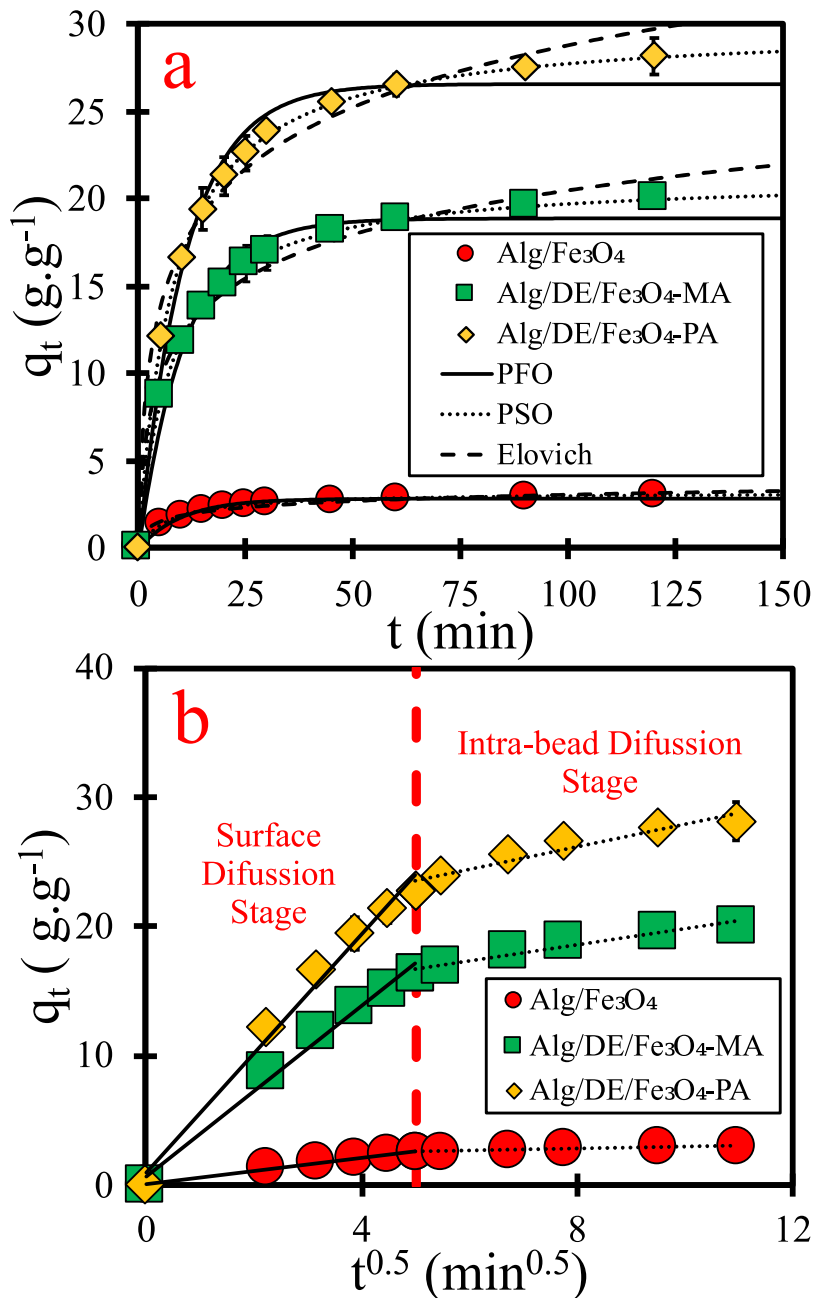


Fig. 6. (a) The non-linearized plot for PFO, PSO and Elovich kinetics model and (b) The linearized plot for IPD kinetics model (condition: C_0 : 66.67 g L⁻¹, bead dose: 1 g L⁻¹, pH 6, T: 25 °C, and n: 3).

3.7.3. Evaluation of kinetics

Kinetics analysis (Fig. 6(a) and Table S1) revealed that oil removal by Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA did not follow the pseudo-first order and Elovich models, because both models produced low R^2 and high χ^2 and ARE values. The PSO model revealed a better fit to experimental data, with R^2 close to unity and lower χ^2 and ARE values. This indicated that oil removal by Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA could be analyzed by the PSO model. Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA were confirmed to be porous by SEM images and N₂ adsorption-desorption isotherms. Role of diffusion in their oil removal mechanism was assessed using the intraparticle diffusion (IPD) kinetics model. Fig. 6(b) shows that the IPD curve did not exhibit a single linear plot over time. It exhibited two segments, representing two different diffusion mechanisms. The first segment represented diffusion of oil to the external surface of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, passing through the boundary layer. This rapid surface-diffusion caused sharp increase in oil adsorption. Slow oil

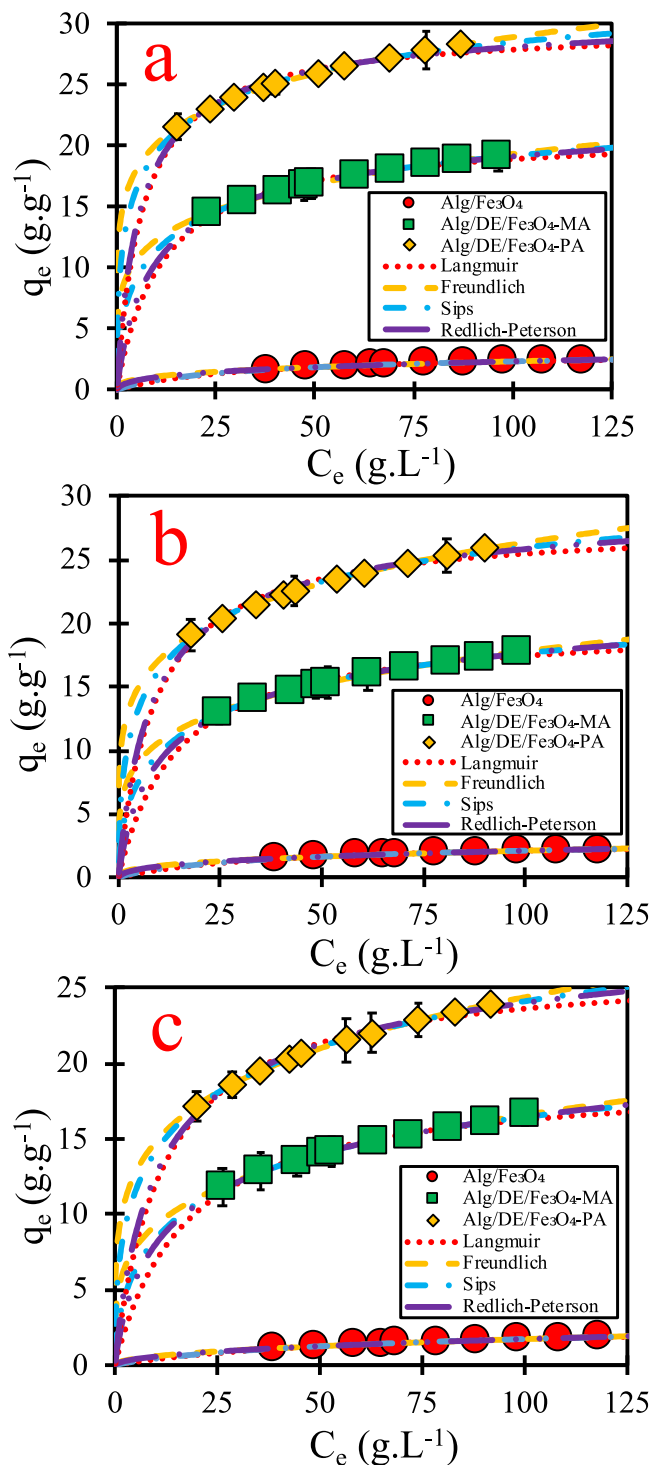


Fig. 7. Equilibrium Langmuir, Freundlich, Sips and Redlich–Peterson isotherms for removal of crude oil on deionized water surface by Alg/Fe₃O₄, Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA at (a) 25 °C, (b) 35 °C, and (c) 45 °C (condition: bead dose: 1 g L⁻¹, pH 6, t: 2 h, and n: 3).

adsorption, controlled by intra-bead diffusion, was represented by the second segment. In this segment, diffusion occurred inside DE pores, on Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA surfaces, resulting in lower value of K_{2-IPD} than K_{1-IPD} . As presented in Table S1, the K_{2-IPD} of Alg/DE/Fe₃O₄-PA was approximately 10 times higher than that of Alg/Fe₃O₄, due to

differences in surface morphology and pore profile, confirmed by SEM and N₂ adsorption–desorption analysis, respectively. Alg/Fe₃O₄ had a less porous surface, inhibiting rapid IPD of oil.

3.7.4. Influence of initial oil concentration

Significant increase in oil adsorption was observed at low initial oil concentration due to concentration gradient between the bulk oil–water mixture and Alg/DE/Fe₃O₄-MA, or Alg/DE/Fe₃O₄-PA surfaces (Fig. 7(a)). Increase of initial oil concentration intensified collisions between oil and Alg/DE/Fe₃O₄-MA or Alg/DE/Fe₃O₄-PA, leading to an increase in oil adsorption. At equilibrium, the active sites and pores of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA were occupied, making oil adsorption constant. Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA showed 6.7-fold and 9.5-fold increase in Langmuir q_{\max} in comparison to Alg/Fe₃O₄, respectively, indicating contribution of DE, MA, and PA to oil-molecule accommodation during adsorption.

3.7.5. Temperature dependence of isotherm models

Quality parameters from Langmuir, Freundlich, Sips, and Redlich–Peterson isotherm fittings were compared. The Freundlich parameter demonstrated a better fit for oil adsorption by Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA at 25 °C, with R² values close to unity, $\chi^2 \leq 3 \times 10^{-4}$, and ARE ≤ 0.0995 (Fig. 7(a–c) and Table S2.). Freundlich isotherm models revealed multilayer oil adsorption on Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA heterogeneous surfaces. The surfaces were heterogeneous due to DE pores and MA/PA attachment, as indicated by SEM images, N₂ adsorption–desorption profiles, and FTIR spectra. The homogeneous Alg/Fe₃O₄ surface adsorbed oil in the monolayer mode, as described by the Langmuir isotherm having R², χ^2 , and ARE values of 0.9997, 6.94×10^{-5} , and 0.1375, respectively. The introduction of DE and MA/PA altered the synthesized adsorbent surface from homogeneous to heterogeneous, changing oil adsorption mechanism. Similar oil adsorption by Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA was observed at elevated temperatures (Table S2). Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA oil adsorption adhered to the Freundlich isotherm model at all studied temperatures (25–45 °C), while adsorption by Alg/Fe₃O₄ was best interpreted using the Langmuir model. The Langmuir q_{\max} value of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA decreased with temperature increase. This demonstrated influence of temperature on interfacial behavior of oil and the synthesized adsorbents. Decrease in oil viscosity on temperature elevation increased oil solubility and decreased Langmuir q_{\max} values. Furthermore, non-electrostatic interaction between oil and Alg/DE/Fe₃O₄-MA or Alg/DE/Fe₃O₄-PA weakened with temperature increase, allowing easy oil detachment from the adsorbent pores and surfaces during post-adsorption recollection. Similar reduction in Langmuir q_{\max} values with increasing temperature have been reported in previous publications (Mahmoud, 2020; Raj and Joy, 2015). Comparison of q_{\max} values of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA with Alg/Fe₃O₄, at all studied temperatures, confirmed that introduction of DE and MA/PA suppressed the reduction of Langmuir q_{\max} .

3.8. Mechanism of oil removal

Oil removal mechanism was investigated by FTIR spectrophotometry of fresh and oil-loaded Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, as shown in Fig. S3(a–d). In the FTIR spectrum of Fe₃O₄, several peaks were observed at 3665, 576, and 451 cm⁻¹, associated with O–H vibrations of adsorbed water, tetrahedral sites of Fe–O stretching, and octahedral sites of Fe–O stretching vibrations, respectively (Shahrashoub and Bakhtiari, 2021). After Fe₃O₄ incorporation, new peaks were observed at 1622 and 1419 cm⁻¹, corresponding to Alg C=O stretching (Lv et al., 2017). A weak peak at 1012 cm⁻¹ was assigned to the Alg pyranose ring C–O group. DE presence in Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA was detected by peaks at 1091 and 802 cm⁻¹, associated with stretching vibration of siloxane (Si–O–Si) (Song et al., 2021, 2019). The attachment of MA and PA on the surface of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, respectively, was confirmed by a peak at 1720 cm⁻¹, assigned to MA/PA ester carbonyl group asymmetric stretching. A peak at 1595 cm⁻¹ was attributed to C=C in MA/PA. A peak at 2850 cm⁻¹, assigned to C–H stretching vibrations, indicated ester bond formation (Yadav et al., 2019).

Crude oil, castor oil, coconut oil, vegetable oil, and olive oil consist mainly of hydrocarbons; therefore, related peaks were observed in the FTIR spectra. Peaks at 2910 and 2850 cm⁻¹, associated with C–H asymmetric and –CH₂– symmetric stretching vibrations, respectively, were observed in all the oil spectra (Neves and Poppi, 2020). A peak at 1745 cm⁻¹, attributed to C=O stretching vibration of the fatty acid ester group, was observed in the FTIR spectra of edible oils, such as castor oil, coconut oil, vegetable oil, and olive oil (Jamwal et al., 2021). This peak was absent in the FTIR spectrum of crude oil (mainly composed of aliphatic and aromatic structures of carbon and hydrogen). A peak at 1456 cm⁻¹ was assigned to C–H bending of –CH₂– or –CH₃ (Tarhan, 2020). After oil adsorption, the above-mentioned peaks were observed in the FTIR spectra of oil-loaded Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, indicating adsorbed oil molecules. A slight shift to lower wavenumbers could be observed for C=O and C–H vibrations of edible oils after adsorption, indicating physical attachment of the oil (Ewis et al., 2020). Interactions (π – π) facilitated by DE pores, between oil and MA/PA moieties of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, could be responsible for this attachment.

3.9. Practical feasibility analysis

Oil spills can occur in various aquatic environments. The components of simulated seawater are listed in Table S3. To evaluate applicability of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA in oil-contaminated water clean-up, various water types were used as adsorption test media. Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA exhibited similar oil adsorption, regardless of water type used. Oil adsorption was slightly higher from seawater than demineralized or tap water. Screening effect could be responsible for this phenomenon. High concentration of counter ions in seawater, such as Na⁺, Mg²⁺, and Ca²⁺ can cause Alg matrix-surface neutralization, diminishing adsorbent-oil electrostatic repulsion. Furthermore, oil solubility in high-salinity seawater is low, leading to many effective collisions of oil with Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA, enhancing oil adsorption (Diraki et al., 2019).

Oil adsorption by Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA increased in the following order: crude oil < olive oil < vegetable oil < coconut oil < castor oil. This could be explained by oil density. Liquid pollutants such as high-density oils can easily accumulate in adsorbents. Thus, the adsorption of high-density oil was relatively higher than that of low-density oil. The relationship between oil density and adsorption for Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA is illustrated in Fig. S4(a). Differences in adsorption capacities of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA towards various oil types were negligible, indicating their wide applicability.

Fig. S4(b–d) shows the reusability of Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA for removal of various oil types from different aqueous media. Reusability of the as-synthesized adsorbents, obtained by comparing oil adsorption before and after 20 adsorption cycles, decreased in the following order: Alg/DE/Fe₃O₄-PA > Alg/DE/Fe₃O₄-MA > Alg/Fe₃O₄. After 20 adsorption cycles, Alg/DE/Fe₃O₄-PA exhibited up to 95% adsorption capacity, regardless of oil or water type, while Alg/Fe₃O₄ lost more than 15% of its adsorption capacity. Thus, DE, MA, or PA introduction improved reusability and adsorption capacity of the synthesized beads. As presented in Table S4, Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA exhibited higher adsorption than previously reported magnetic adsorbents. Several reported Fe₃O₄-based adsorbents have higher M_s and shorter t_{eq} values due to their small sizes. However, small size enables facile oxidation of Fe₃O₄ to Fe₂O₃ after several adsorption–desorption cycles, especially in high-salinity seawater, weakening magnetic properties, making post-adsorption recovery difficult, and causing secondary pollution. The Alg matrix in Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA protected Fe₃O₄ from oxidation. Both Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA maintained their buoyancy over several oil adsorption–desorption cycles. Therefore, the as-synthesized Alg/DE/Fe₃O₄-MA and Alg/DE/Fe₃O₄-PA beads meet the criteria for effective and eco-friendly oil removal from aquatic environments.

4. Conclusions

This study reports the synthesis of magnetically driven Alg/DE composite beads, modified with PA/MA, for oil-contaminated water clean-up. High buoyancy of beads allowed intensive contact with aqueous oil films, removing oil within 30 min, following the PSO model ($R^2 \sim 0.999$, $\chi^2 \leq 0.041$, and ARE ≤ 1.088). Owing to the presence of DE molecules and PA/MA groups, the modified magnetic beads exhibited high adsorption capacity towards different oils, with maximum adsorption capacity values up to 29.7 times their weights, described by the Freundlich model, with $R^2 \sim 0.999$, $\chi^2 \leq 3.37 \times 10^{-3}$, and ARE ≤ 0.1 . Oil adsorption was pH independent and extremely effective in deionized water, tap water, and seawater. Owing to high M_s values, confirmed by VSM data, the as-prepared beads were magnetically recoverable and regenerable over 20 oil adsorption–desorption cycles. Unique features of the magnetically driven PA/MA-modified Alg/DE composite beads demonstrated their potential as efficient and eco-friendly adsorbents for oil-contaminated water clean-up. Future research could include real-time analysis of the as-synthesized beads in environmental applications, evaluating their environmental effects, and biodegradability. Combination of the composite beads with other techniques, such as floating booms or oil-degrading mechanisms, could also be investigated for cost-effective solutions to mitigate the adverse effects of a large-scale oil spill.

CRedit authorship contribution statement

Satya Candra Wibawa Sakti: Conceptualization, Methodology, Investigation, Writing – original Draft. **Nindayu Indrasari:** Investigation. **Rizki Ainuna Wijaya:** Investigation. **Mochamad Zakki Fahmi:** Investigation, Funding acquisition. **Alfa Akustia Widati:** Investigation, Funding acquisition. **Hwei Voon Lee:** Investigation. **Takahiro Fujioka:** Writing – review & editing. **Nuryono:** Conceptualization. **Chun-Hu Chen:** Writing – review & editing, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary material related to this article can be found online at <https://doi.org/10.1016/j.eti.2021.102120>.

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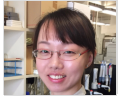


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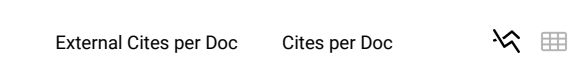
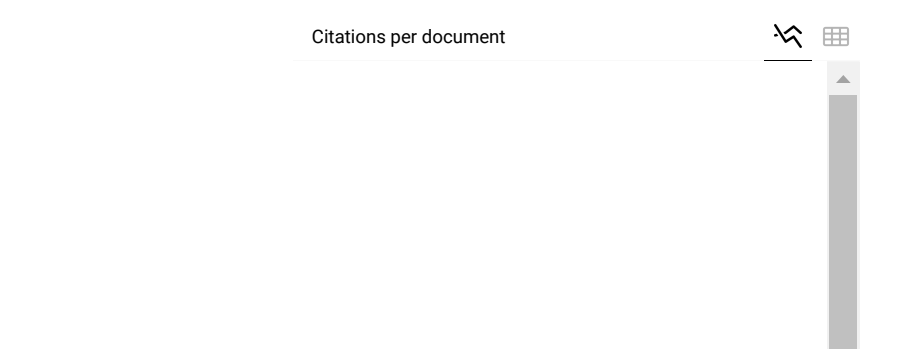
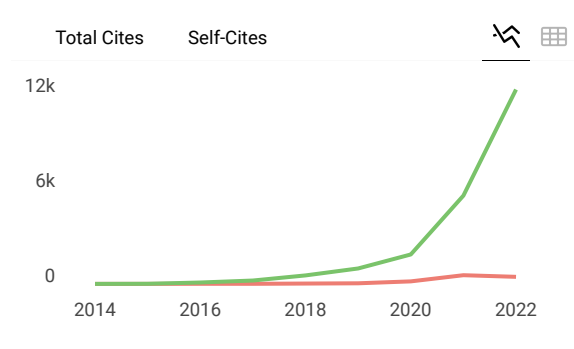
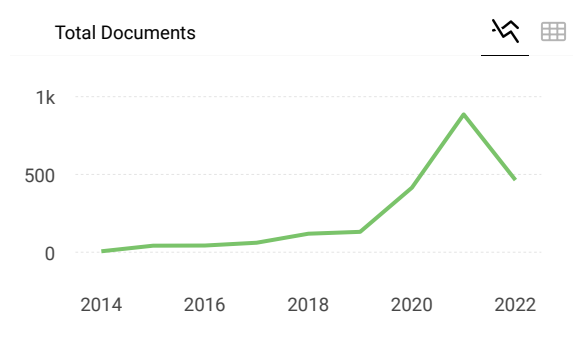
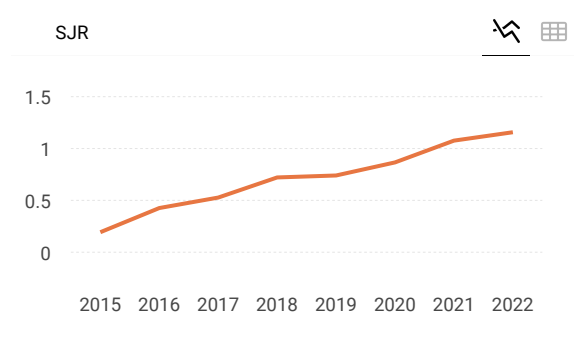
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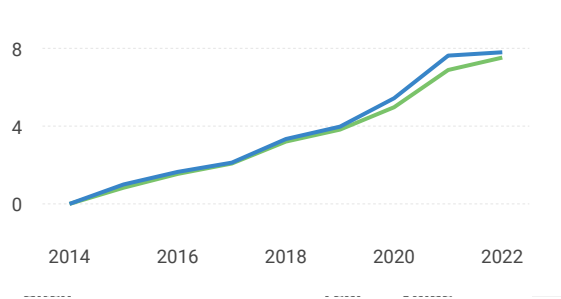
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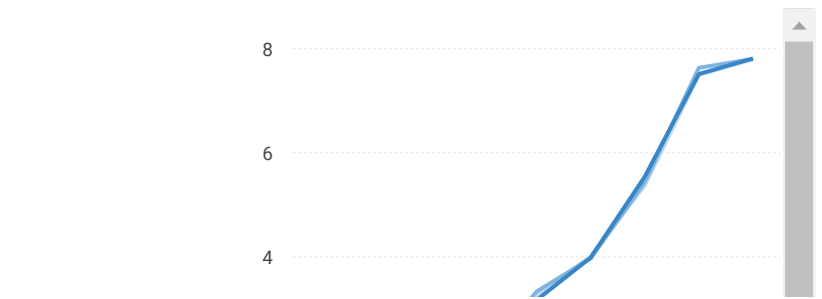
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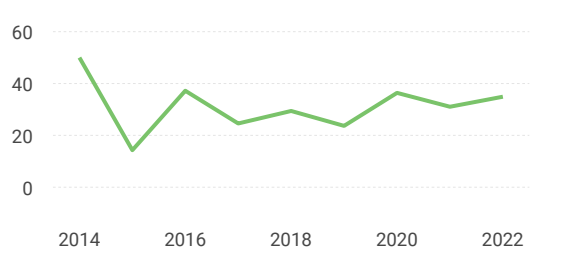


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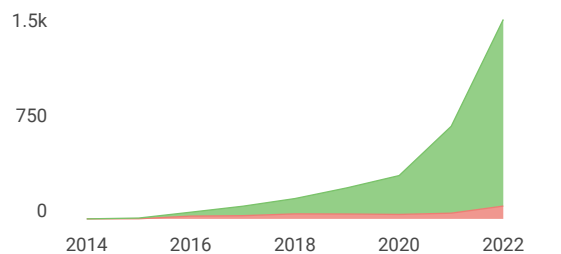
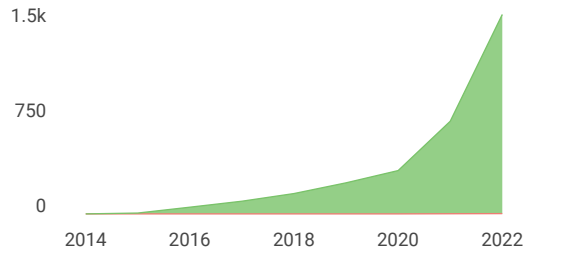
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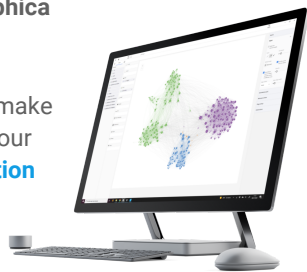
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Fwd: Confirming submission to Environmental Technology & Innovation

1 message

SATYA CANDRA <satya.sakti@fst.unair.ac.id>
To: Mochamad Fahmi <m.zakki.fahmi@fst.unair.ac.id>

Sat, May 6, 2023 at 10:31 AM

Sent from my iPhone

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Date: 10 November 2021 12.44.55 GMT+7
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Subject: Confirming submission to Environmental Technology & Innovation
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Diatomaceous earth incorporated floating magnetic beads for oil removal on water

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Confirm co-authorship of submission to Environmental Technology & Innovation - reminder

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Reply-To: Environmental Technology & Innovation <support@elsevier.com>
To: Mochamad Zakki Fahmi <m.zakki.fahmi@fst.unair.ac.id>

Wed, Oct 20, 2021 at 4:30 PM

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Journal: Environmental Technology & Innovation
Title: Diatomaceous earth incorporated floating magnetic beads for oil removal on water
Corresponding Author: Dr. Satya Candra Wibawa Sakti
Co-Authors: Nindayu Indrasari; Rizki Ainuna Wijaya; Mochamad Zakki Fahmi, Dr.; Alfa Akustia Widati, Dr.; Hwei Voon Lee, Dr.; Takahiro Fujioka, Assoc. Prof; Nuryono Nuryono, Prof.; Chun-hu Chen, Prof.
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