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Impact of Recycled Oleophilic Polyurethane Foams Integrated with Activated Carbon and Silica Enriched-Palm Oil Biomass for Separation of Oil

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Growing awareness of the ecological damage caused by oil spills has driven substantial studies of effective and long-term oil-water separation strategies. This study aims to examine the viability of utilising recycled polyurethane foam (PUF) that has been enfolded with activated carbon and silica obtained from oil palm biomass as an adsorbent for separating oil. The prepared materials, namely AC-PUF and Si-PUF, were then characterised using Fourier Transform Infrared–Attenuated Total Reflectance (FTIR-ATR) and Scanning Electron Microscopy (SEM) to study the presence of critical functional groups and their surface morphologies. Significant parameters, including contact times, agitation speeds, and the initial diesel concentrations, were evaluated to determine the optimum condition for their adsorption capacities. Under the optimised condition, the highest oil adsorption capacity for recycled PUF, AC-PUF and Si-PUF were 25.19 g/g, 26.91 g/g, and 28.89 g/g, respectively. The results showed that the enfolded PUF with non-polar activated carbon and silica improved the effectiveness of recycled PUF in extracting oil via Van der Waal interaction. The enfolded PUF exhibits potential as a viable and efficient approach for addressing oil spills in a sustainable manner. This shows that agricultural waste can be turned into high-value products and encourages an eco-friendlier way to clean up pollution.

Keywords: Activated carbon polyurethane foam (AC-PUF); Silica-coated polyurethane foam (Si-PUF); Polyurethane foam; Adsorption capacity

I. INTRODUCTION

Oil pollution is a common problem that is frequently disputed by society. These environmental disasters often have long-term consequences for the environment, ecology, and socioeconomic activity of the affected region. Oil spills have many origins, including natural oil seepage from the seabed and ocean, drilling in the seabed, leaking oil extraction and transportation equipment, river and sea transport, emergency spills (like when a tanker crashes), road and air transport, petroleum waste from industrial installations, and stormwater from cities (Gołub & Piekutin, 2017). There are two main types of reasons for oil spills: natural and human-caused. Natural oil seepage from the ocean and sea of beds or the release of oil-producing rocks on the ocean floor into the marine environment are two common causes of oil spills (Dhaka & Chattopadhyay, 2021). Additionally, it elucidated that anthropogenic causes can be further categorised into two distinct classifications: inadvertent oil spills and deliberate oil spills. A facile and quick cleanup method is vital since oil spill cases keep increasing yearly.

Many approaches have been developed for oil spill cleanup in open waters, which include in situ burning, mechanical

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extraction, using dispersants, solidifiers, booms, skimmers, and utilisation of oil absorbent. However, most of these methods have limitations in removing oil with their disadvantages of non-portable operation, high-energy, excessive time consumption, and may present secondary pollution (Mokoba *et al.*, 2021; Ozhan & Bargu, 2014; Pinto *et al.*, 2017). Among all the diverse methods available, the absorption method using porous sorbent is the most preferred approach as it could remove and recover oils and many types of organic solvents with high efficiency, inexpensive and negligible secondary pollution involved. Some materials studied to be used as oil adsorbents are wool, vegetable fibres, and exfoliated graphite, which were considered traditional due to their poor properties of not being reusable and inadequate performance in removing oil (Lü *et al.*, 2016).

Polyurethane (PU) foams are a type of cellular material widely applied in a broad range of applications due to their diversity of physical and chemical characteristics, which vary according to formulation and processing. Polyurethane foams occupy the largest polymer foam market share in terms of consumption (Antunes, 2010; Verdolotti *et al.*, 2017). Out of all porous materials, polyurethane foam (PUF) was reported can efficiently adsorb and separate oil due to its properties. Even so, most of the past research did not consider the potential of polyurethane in separating oil and water. This high porosity led to very high absorption capacities resulting in an excellent way to separate oil from wastewater.

However, pristine PUF has several drawbacks, exhibiting hydrophilic properties that could interfere with the selective adsorption of oil from water and have low mechanical durability that cannot withstand harsh chemical environments (Yuan *et al.*, 2017). Thus, it is recommended to carry out modifications on the surface of PUF to further improve the performance in adsorbing oil, together with overcoming these drawbacks. In producing composites with enhanced properties, PUF possessing a wide, porous structure, is considered an ideal material to be modified and incorporated with other substances (Xie *et al.*, 2020; Zhou *et al.*, 2020). Silica is a suitable candidate to enhance PUF due to its high porosity with large surface areas, low density, and possessing good hydrophobic properties, as exhibited by previously studied silica aerogel (Sun *et al.*, 2015; Wang *et al.*, 2012). Besides, carbon-based materials have also been shown to be effective at adsorbing a variety of pollutants, including heavy metal ions, oil separation from water, and organic colours (Mallakpour & Behranvand, 2020). To date, the enfolded activated carbon and silica from palm oil biomass to the recycled PUF has yet to be discovered. Hence, this study aims to investigate the contributions of non-polar activated carbon and silica nanoparticles enriched from palm oil biomass in assisting the oil adsorption activity of recycled PUF using UV-VIS. The proposed methodology involves the fabrication of recycled polyurethane foam (PUF) incorporated with activated carbon polyurethane foam (AC-PUF) and silica-coated polyurethane foam (Si-PUF) through a mild functionalisation process. The assessment of the contact time, agitation velocity, and initial diesel concentration as variables that influence the adsorption capacity of oil onto polyurethane foam (PUF), activated carbon-PUF (AC-PUF), and silicon-PUF (Si-PUF) The findings of this study will provide an insight into the potential of recycled PUF and green sources of AC and Si as cost-effective and efficient adsorbents for water remediation technologies.

II. MATERIALS AND METHOD

A. Chemical and Materials

The chemicals that used was anhydrous ethanol (CH_3CH_2OH, ≥ 99.97%, Merck), diethyl ether ($(C_2H_5)_2O$, Fisher Chemical), ascorbic acid ($C_6H_8O_6$, Sigma Aldrich), sodium hydroxide (NaOH, Sigma Aldrich), hydrochloric acid (37%) was purchased from J.T. Baker and 1.0 L Diesel Shell Fuel-Save. Silica microparticles, SiO_2, were extracted from oil palm frond leaves. At the same time, activated carbon was extracted from empty fruit bunchs. The PUF used was obtained and recycled from the waste of automotive seat cushions.

B. Pre-treatment of PUF

In extracting PUF from automotive waste, the PUF was cut into cubes of sizes approximately 1 cm × 1 cm × 1 cm. Then, the PUF was soaked in ethanol overnight to remove any impurities present on its surface. The soaked PUF was

removed from the beaker, allowed to drain for 1 hour, and dried in a vacuum oven overnight at 60 °C. The treated polyurethane foam was then placed in a desiccator to be preserved until used.

C. Preparation of Adsorbents

1. Preparation of AC-PUF

Extracted AC from empty fruit bunch (EFB) was introduced by (Ibrahim, 2019), and the method used to prepare AC-PUF was adapted with slight modification from a previous study (Anju & Renuka, 2020). Freshly carbonised empty fruit bunches (EFB) were crushed with KOH pallets at a weight ratio of 1:1 to improve the porosity of the carbon-based EFB. After that, the EFB-KOH paste was calcined at 800 °C for 2 hours. The resulting black carbon powder was extensively rinsed with 1.0 M hot HCl and hot distilled water until pH 67% was reached, then dried at 110 °C to yield AC-derived EFB. Then, activated carbon was dissolved in deionised water, and the pH of the dispersion solution was adjusted to pH 7 using sodium hydroxide (NaOH). Then, to treat PUF, Ascorbic Acid was added to the solution and stirred for 5 hours. The AC-PUF was then filtered, rinsed with deionised water, and dried overnight in an oven at 30 °C oven.

2. Preparation of Si-PUF

Meanwhile, the preparation of the Si-PUF composite was referred from a previous study with minor modifications (De Nino *et al.*, 2021). Extracted silica nanoparticles were prepared by subjecting oil palm frond to acid treatment using a method described in the literature (Onoja *et al.*, 2018). In this work, 50.0 g of powder oil palm leaves (OPL) was dispersed in 500.0 mL of 1.0 M HCl (37%) and heated in an oil bath equipped with a magnetic stirrer. The dispersed solution was heated to 100 °C and constantly stirred for 2 hours at 400 rpm. Then wash the dispersed powder solution with distilled water several times until the pH solution becomes neutral (pH 7). Then, filter the dispersed powder, dry it in an oven at a temperature of 80 °C for 24 h, and label it as oil palm leaves treated (OPLT). The OPLT samples will then be subjected to thermal treatment using a furnace to guarantee the removal of organic compounds. The silica crucibles were calcined in a preheated furnace at 600 °C for 14 hours with a ramping time of 5 hours and a hold time of 9 hours at a rate of 2 °C/min, resulting in a white colour silica. Then, PUF cubes were added into the homogenous mixture of 200 mL of diethyl ether and 2 g of prepared silica nanoparticles. The mixture was stirred for five hours at a temperature of 25 °C by using a magnetic stirrer with a speed of 700 rpm. Then, the Si-PUF composite was washed and filtered with diethyl ether removing silica microparticles that did not adsorb to the PUF surface. The Si-PUF was then allowed to drain and dried again in the oven for two days at 50 °C.

D. Characterisation of PUF, AC-PUF and Si-PUF

The functional group in the adsorbents was determined using Attenuated Total Reflectance Fourier Transform Infrared Spectrophotometer Thermo Nicolet 6700 (ATR-FTIR). The IR spectra are going to be determined using internal reflections (evanescent waves) within an adsorbent and diamond crystal. No sample preparation is needed as ATR allowed direct analysis of the adsorbent in the form of a solid, and the frequency ranging between 450 to 4000 cm^{-1} was applied. The surface morphology of the adsorbents was identified using a Scanning Electron Microscope TM3030 Plus Hitachi Tabletop Microscope (SEM) that operated at 15kV of voltage. The surface wettability of adsorbents was determined by simple alternative water contact angle measurement. The angle of the droplet is measured by taking the points on the droplet that are simultaneously in contact with the surface of the adsorbents. Samples of the adsorbents were placed on a flat surface, and a drop of deionised water was dropped on the adsorbents by using the pipette. Images were taken with a smartphone camera by using the autofocus macro lens settings.

E. Batch Studies on Oil Removal

The batch studies were carried out to investigate the adsorption reaction of oil onto PUF, AC-PUF and Si-PUF by varying the value of contact time, agitation speed and initial diesel concentration. It was able to determine the optimum values for the adsorbents because each of the parameters had an influence on the adsorption capacity. The adsorption mass was constant at 0.5 g throughout the entire study. The adsorption capacity performance was evaluated by putting

the weighed prepared adsorbent into 50 mL of diesel in a conical flask and then shaking it with a chemical shaker using studied parameters at room temperature. After that, the adsorbent was removed from the solution and drained for 5 minutes. After adsorption, the adsorbent was weighed again and then squeezed, obtaining the diesel adsorbed. The amount of diesel extracted was weighed.

F. Data Analysis

In data analysis, the capacity of oil adsorption by polyurethane foam was calculated by using the following equation:

The equation will be used to calculate the adsorption capacity (g/g):

$$Adsorption\ capacity, q = \frac{M_d}{M_0} \quad (1)$$

Where Md is the mass of the extracted diesel, and M0 is the mass of the PUF before adsorption.

The oil adsorption percentage Pa (%) by polyurethane foam was calculated by using the following equation:

$$Pa\ (\%) = \frac{O_a}{O_t} \times 100 \quad (2)$$

where Oa is the quantity of the adsorbed oil after the process, and Ot is the initial weight of the oil in water.

III. RESULT AND DISCUSSION

A. Characterisation of AC-PUF and Si-PUF

Polyurethane is a long-chain linear polymer that has a molecular backbone containing a carbamate group or indicated as urethane (NH-CO_2), as shown in Figure 1.

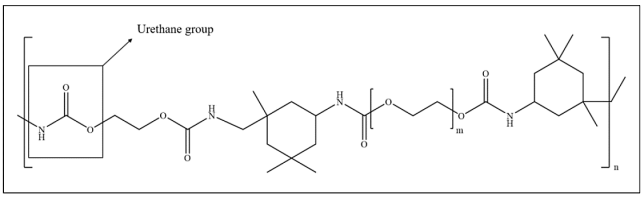


Figure 1. Structure of Polyurethane

The physical characteristics of PUF were slightly different after being coated with activated carbon and silica. Ethanol-treated PUF exhibits brown yellowish colour, as shown in Figure 2(a). However, the colour turns black after coating with activated carbon (Figure 2(b)), indicating that the AC particles are homogeneously dispersed onto the PUF surface. Similar to Si-PUF (Figure 2(c)), the faded light yellowish colour revealed the presence of fine white silica particles filled in the pores of PUF.

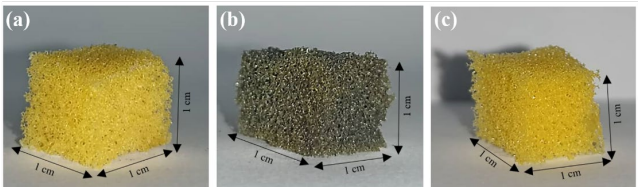


Figure 2. Photograph of (a) PUF, (b) AC-PUF and (c) Si-PUF

ATR-FTIR spectra of PUF, AC-PUF and Si-PUF are shown in Figure 3. The absorption bands and peaks that are typically observed in polyurethane foam are N-H stretching (3300 – 3310 cm^{-1}), C-O stretching (1090 – 1095 cm^{-1}), C=N groups (1533 – 1535 cm^{-1}) and the C-H stretching (2856 – 2970 cm^{-1}). In comparing the spectra of PUF before and after treatment with ethanol, the resulting peaks were similar, with no apparent change to the wavenumber and strength of the peaks. The IR spectra of AC-PUF show a similar pattern as PUF with a slight difference in the broadness and intensity of absorbance that became weaker and shifted to a lower wavenumber. The broadness is related to the variability of the carbonyl environments, and it proves the AC-polymer relationship (Keshavarz *et al.*, 2015). Meanwhile, The Si-PUF spectrum showed a decrease in the strength of all peaks in comparison to the PUF. This was displayed by a very weak and broader N-H peak at 3300.98 cm^{-1} and a C=N peak at 1531.99 cm^{-1}. These diminished effects could be explained by a very strong hydrogen bond formed due to the interactions between the urethane chain and silica microparticles during the immobilisation process (De Nino *et al.*, 2021).

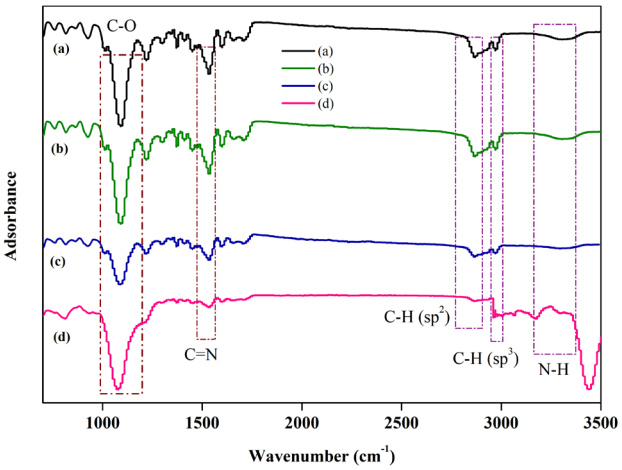


Figure 3. FTIR spectra of (a) untreated PUF, (b) PUF after being treated with ethanol, (c) AC-PUF and (d) Si-PUF

B. SEM Analysis

The micrograph of PUF and both modified PUF with silica and activated carbon are shown in Figure 4. SEM was used for morphological study, which revealed the entire surface coating of the polyurethane composites made with silica and activated carbon. Image 4(a) shows that the PUF display smooth and highly porous surface morphology. Meanwhile, Figure 4(b) shows rougher surface morphology with the presence of white particles agglomerated on the side of PUF pores. This revealed that the silica nanoparticles successfully coated onto the PUF surface and may provide an additional active site for adsorption activities. Whereas Figure 4(c) for AC-PUF shows agglomerated particles scattered on the PUF surface, indicating the existence of AC, which enhanced the roughness of the PUF surface, which assists the foam in firmly holding the adsorbed oil (Keshavarz *et al.*, 2015).

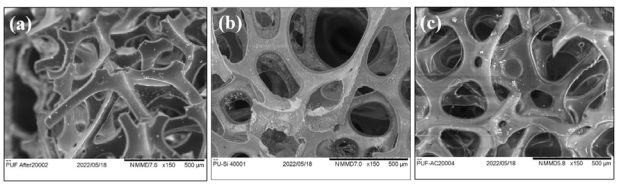
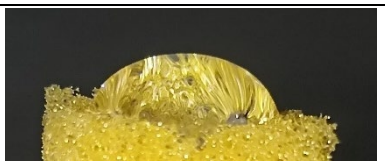
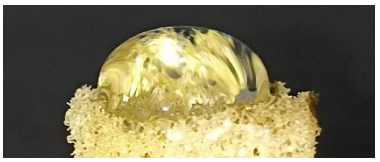


Figure 4. SEM analysis of (a) PUF, (b) Si-PUF and (c) AC-PUF

C. Contact Angle of AC-PUF and Si-PUF

In determining the hydrophobic properties of the prepared PUF composites, the water contact angle of the sample surfaces with water was measured, as displayed in Table 1. It was observed that the contact angle value for the PUF was 57°. These values indicated that the PUF exhibits surface hydrophilicity. Meanwhile, the contact angle recorded for AC-PUF and Si-PUF were increased to 62° and 74°, respectively. Assuming that a higher contact angle indicates greater hydrophobicity, it could be explained that the modifications made on the PUF by immobilising AC or Si on PUF surfaces have shown increasing hydrophobicity of the foam. This would result in higher selectivity and interactions by the prepared PUF composites in removing oil.

Table 1. The surface contact angle of PUF, AC-PUF and Si-PUF

Sample	Contact angle	
PUF		57°
AC-PUF		62°
Si-PUF		74°

D. Batch Adsorption Studies

1. Effect of contact time

An optimum contact time can predict the mechanism of the removal process and the efficiency of the absorbent to remove the oil (El-Gamal *et al.*, 2015). Using different contact time (15, 30, 45 and 60 minutes) with an agitation speed of 200 rpm, the effect on the adsorption capacity of diesel using the adsorbents were studied. The optimal contact time between absorbents and oil was established by analysing the evolution of the water and oil uptakes with the contact time. Figure 5 shows that the AC-PUF demonstrated the highest oil adsorption capacity with a value of 25.38 g/g compared to PUF and Si-PUF, which were 25.19 g/g and 23.87 g/g, respectively. The interaction between the oleophilic surface of the adsorbent with oil causes the interpenetration of the oil molecules into the adsorption sites available in the pores of the sorbent. The trend in the graph

increases up to 45 minutes and slightly decreases after that. This is because there are less amount of unoccupied surface sites available for diesel adsorption, as most of these sites have been saturated with diesel molecules. Thus, a further increase in contact time would no longer cause an increase in the sorbent adsorption capacity.

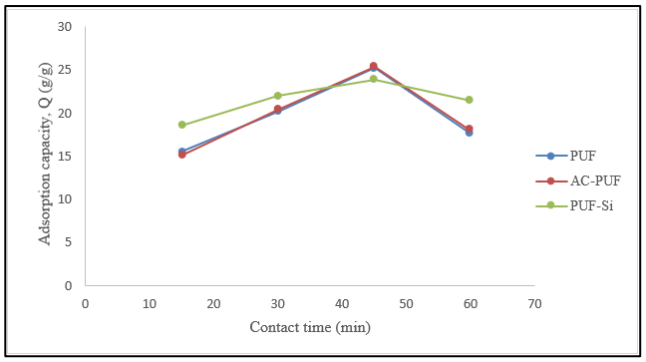


Figure 5. Effect of contact time on adsorption capacity for PUF (blue), AC-PUF (red) and PUF-Si (green)

2. *Effect of agitation speed*

Agitation speed is an important parameter to optimise the adsorption capacity. Sufficient agitation speed is required to facilitate the mass transfer processes, hence increasing the adsorption activity to reach an equilibrium state. The effect of different agitation speeds on the adsorption capacity was studied using 50 mL oil shaken with the sorbent at a speed between 100 to 250 rpm at an optimum contact time of 45 min, as shown in Figure 6. The adsorption capacity was found to increase when shaken at speed from 100 to 200 rpm. It was found that the Si-PUF had the highest adsorption capacity, which is 28.89 g/g, as the adsorbent was able to mix with the oil molecules more homogenously at a faster speed of agitation. Therefore, this allowed more rapid interaction between the adsorptive sites of the sorbent with the oil molecules leading to higher adsorption. It was followed by AC-PUF, which has the second highest adsorption capacity, 26.91 g/g and 21.78 g/g for PUF. Nevertheless, the significant decrease in adsorption capacity observed after the equilibrium could be assumed was due to the oil molecules not having enough time to penetrate the adsorbent surfaces at a very high agitation speed causing the inefficient sorbent.

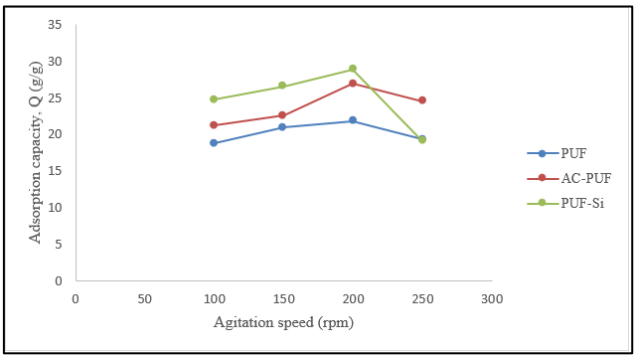


Figure 6. Effect of agitation speed on adsorption capacity for PUF (blue), AC-PUF (red) and PUF-Si (green)

3. *Effect of initial diesel concentration*

The adsorption capability of the adsorbents was tested for various initial diesel concentrations (10, 20, 30, 40, 50 g/L). Figure 7 shows that the highest rate of adsorption of diesel was at 30 g/L with 15.17 g/g for PUF and at 30 g/L with 17.43 g/g for Si-PUF, while for AC-PUF at 40 g/L at 20.03 g/g. This can be proved that the initial oil concentration affected the amount of adsorption for oil removal. The highly hydrophobic interaction of AC-PUF made the absorbent remove more oil from the water, and the amount of oil absorbed into the absorbent was highly dependent on the available sites of the adsorbent surface (Zabi *et al.*, 2021).

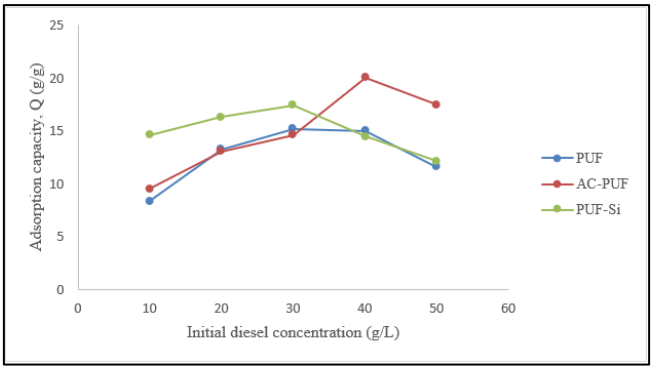


Figure 7. Effect of initial diesel concentration on adsorption capacity for PUF (blue), AC-PUF (red) and PUF-Si (green)

E. *Comparison of Oil Removal Efficiency*

Oil removal efficiencies from water using PUF and modified PUF, which are AC-PUF and Si-PUF, were compared. The adsorption capacities and the percentage of oil adsorption capacities under optimum conditions for all types of adsorbents are the factors considered in this comparison (contact time: 45 min; agitation speed: 200 rpm). Based on

Table 2 shows that Si-PUF has the highest adsorption rate, followed by AC-PUF. This behaviour may be due to the silica and activated carbon particles coating the surface of PUF, providing a larger surface area for diesel to be captured in the rougher pores. It could also be assumed that the better oil adsorption performance in PUF-Si was contributed by the hydrophobic properties of the silica, SiO_2, that has been successfully coated on the surface of the sponge.

Table 2. The maximum adsorption capacity of adsorbents

Types of adsorbents	Adsorption capacity (g/g)
PUF	25.19
AC-PUF	26.91
Si-PUF	28.89

IV. CONCLUSION

Two abundant natural products from enriched-palm oil biomass, silica and activated carbon, were coated with polyurethane foam to enhance adsorption capacity in removing oil. The results demonstrated that the modified adsorbents' performances are significantly improved over the starting polyurethane. Under the optimum parameters observed (contact time: 45 minutes, agitation speed: 200 rpm, and initial diesel concentration: 30 g/L), the result shows that the modified Si-PUF is the most promising adsorbent in removing oil since it has the highest adsorption capacity with 28.89 g/g followed by AC-PUF with 26.91 g/g and PUF 25.19 g/g and 26.91 g/g respectively. Then, a reusability test was performed, and it was decided to only use polyurethane foam once due to the high cost and additional handling process if we reuse those polyurethane foam. Given the impracticality of reusing polyurethane foam due to cost and the need for additional handling processes, it is imperative to investigate viable regeneration techniques for the modified adsorbents. Enhancing the economic viability and sustainability of these adsorbents through developing efficient regeneration methods will increase their attractiveness for real-world applications.

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