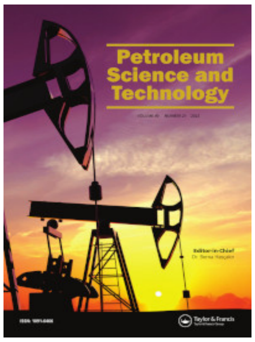

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Flexible polyurethane foam with improved oleophilic and hydrophobic properties for oil spill cleaning

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ABSTRACT

Three chemical compounds (A1, A2, and A3) with long alkyl chains (C5, C8, and C10) were used in this work to improve the oleophilic and hydrophobic properties of blank flexible polyurethane (FPU) cubes for oil spill clean-up. Water sorption was observed to be reduced by almost 50% when compared to blank FPU, while crude oil sorption of modified FPU cubes was raised by A1 = 37.16, A2 = 42.68, and A3 = 45.94 g/g, as well as diesel fuel sorption by A1 = 36.72, A2 = 39.80, and A3 = 41.68 g/g. In the water-oil system, the sorption capacity of FPU cubes modified with three new oleophilic organic compounds was increased by A1 = 43.47, A2 = 44.49, and A3 = 45.15 g/g for crude oil spills and by A1 = 34.07, A2 = 34.16, and A3 = 36.05 g/g for diesel fuel spills, compared to blank FPU cubes of 27.29 and 22.28. The findings suggest that the modified FPU cubes could be utilized to clean up oil spills successfully.

KEYWORDS
crude oil; diesel fuel; hydrophobic; modified fpu; oil spill; oleophilic; sorption capacity

1. Introduction

The oil spills, the leakage of toxic organic materials, and unexpected accidents have all inflicted severe environmental and ecological harm. An oil spill occurs when liquid petroleum is released into the environment by vehicles, pipelines, or ships. Since the discharge of industrial oily effluent and the incidence of oil spill accidents have resulted in environmental contamination in recent years, oil/water separation and oil absorption have been hot research subjects. Dispersants, booms, skimmers, sorbents, and bioremediation are some of the methods used to treat oil spills (Wang et al. 2021). Absorption offers the advantages of high efficiency, ease of operation, and high absorbability among these approaches because of their potential to efficiently separate oil and organic contaminants from the marine ecosystem. Oil collection utilizing hydrophobic and oleophilic materials

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has recently attracted considerable interest (Kim et al. 2018; Wang et al. 2019).

Synthetic organic sorbents such as polymeric ones (polyurethane, polypropylene, polyethylene, and cross-linked polymers) are the most commonly used commercial sorbents in oil spill clean-up due to their oleophilic and hydrophobic properties (Pyo and Chang 2021; Birlik and Aygül 2019; Liu et al. 2021) because of their effervescent, abrasion-resistant, and insulating characteristics (Li et al. 2021).

Superhydrophobic and superoleophilic features were observed in the as-made modified polyvinyl alcohol (PVA) sponge. In addition, the modified PVA sponge could extract oil from water under pump pressure with good separation capacity, efficiency, and reusability. A simple and environmentally friendly synthetic approach was developed to successfully prepare the silylated PVA sponge with superhydrophobicity and superoleophilicity. The original PVA structural integrity was preserved in the silylated PVA sponge. The silylated PVA sponge possessed superhydrophobicity and superoleophilicity due to the silylation of the skeleton surface and such distinctive nano micro substructures (Chen et al. 2019).

The marine ecosystem and the environment are both threatened by oil spills. As a result, the demand for oil recovery is increasing, as is the search for effective hydrophobic or oleophilic sorbents for oil-water separation. A superhydrophobic SA/PPy/MF sponge from polypyrrole (PPy) encapsulated melamine-formaldehyde (MF) sponge treated with stearic acid (SA) was produced. Furthermore, the MF sponge is a suitable sorbent because of its commercial availability, low cost, lightweight, and robustness, all of which are desirable qualities in a high-performance adsorbent.

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To modify the surface of MF, two-step processes are used: first, a low concentration of DA is used to build the hydrophobic reaction platform, and then acyl chloride is used as a hydrophobic agent because of its high reactivity, low toxicity, and affordability. As a result, this technique of preparation has the advantages of being time-saving, low toxicity, low cost, high oil absorption, low energy consumption, and high reusability (Shui et al. 2020).

A PU sponge is a type of porous and hydrophilic polymer that has excellent sorption capacity, low density, and easily scalable fabrication processes. However, it usually absorbs both water and organic chemicals, simultaneously, which limits its application for selective oil separation from water with high efficiency. Given the above, it is necessary to change the hydrophilicity of polyurethane sponges such that they become highly hydrophobic and highly oleophilic, allowing for continuous absorption and removal of oil contaminants from water with high separation capacity (Hailan, Ponnamma, and Krupa 2021; Li, Liu, and Yang 2012).

The current study aims to improve the oleophilic and hydrophobic properties of PU foams by modifying the surface with three different organic compounds having long-chain alkyl groups and good crude oil and diesel fuel affinities. The sorption characteristics of the blank and modified flexible polyurethane (FPU) cubes were thoroughly tested and compared in water, crude oil, and diesel fuel as well as in water-oil systems.

2. Materials

The Italian Arix Company made the flexible polyurethane sponge (FPU). It was cut into one-cm3 cubes. The cubes were rinsed well with ethanol before use. The FPU surface was treated with N^1-acryloyl-N^7-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl) acryloyl) phenyl) dodecane diamide, which was labeled as FPU-A1, FPU-A2, and FPU-A3. For detailed information and characterization of the structure of the A1, A2, A3, and modified FPU (see the supplementary data). The modified FPU with three different alkyl chains was used as an oleophilic and hydrophobic sample for spill cleanup. The Zubair Field Operation Division Company (ZFOD) supplied the Iraqi crude oil and diesel fuel.

3. Methods and experimental work

3.1. Sorption capacity test

The ASTM F726-17: Standard Test Method for Sorbent Performance of Adsorbents was used to establish the method for measuring the sorbent oil and water sorption capacity (ASTM F726-17, 2017).

3.2. Oil sorption experiment

Crude oil or diesel fuel (50 mL) was poured into a 100 mL beaker for oil sorption testing. After weighing the sorbent and recording the value, it was immersed in the oil. In general, the sorbent was removed after 30 min ± 4 min of immersion and left to drain for 30 s ± 3 s. The saturated sorbent was then transferred and weighed into a pre-weighed weighing bottle (Li, Liu, and Yang 2012). The following equation was used to calculate the sorbent's oil sorption:

$$Oil\ sorption\ (g/g) = \frac{(S_t - S_o)}{S_o} \quad (1)$$

where S_o is the sorbent's original dry weight and S_t is the sorbent's weight after crude oil or diesel fuel has been absorbed.

3.3. Water sorption tests

The sorbent was weighed before being placed in a 100 mL Erlenmeyer flask filled with 50 mL of deionized water for water sorption testing. The flask was capped and then agitated for 15 min ± 20 s in a shaker (150 rpm). Allow 2 minutes for the contents of the flask to settle. The sorbent was removed and allowed to drain for 30 s ± 3 s before being weighed in a pre-weighed weighing bottle (Li, Liu, and Yang 2012). The following equation was used to determine the water sorption:

$$Water\ sorption\ (g/g) = \frac{(S_{wt} - S_o)}{S_o} \quad (2)$$

where S_o is the sorbent's initial dry weight and the sorbent's wet weight after water sorption is S_{wt}.

3.4. Water–oil system sorption tests

In the water-oil system sorption tests, 4.0 g crude oil and diesel were placed in a 100 mL Erlenmeyer flask with 50 mL of deionized water. The oil film had a thickness of 2–3 mm. To begin with, the sorbent was weighed and placed in an Erlenmeyer flask. After that, the flask was placed in a shaker (150 rpm) for 30 min. ± 20 s. Allow 2 minutes for the contents of the flask to settle. The sorbent was then transferred to an Erlenmeyer flask and extracted with petroleum ether several times (boiling range of 30–60 °C). The sorbent was removed and squeezed out, releasing the absorbed petroleum ether, which was treated with anhydrous sodium sulfate until no agglomeration occurred, and then the Erlenmeyer flask was covered for 30 minutes to dehydrate. Following that, the filtrate was collected in a dry beaker with a constant weight after filtration. To evaporate petroleum ether, the beaker was placed in a 65 ± 5 water bath, then placed in a 65 ± 5 °C drying oven until it attained constant weight. Finally, the following equation was used to compute the oil sorption in the water-oil system

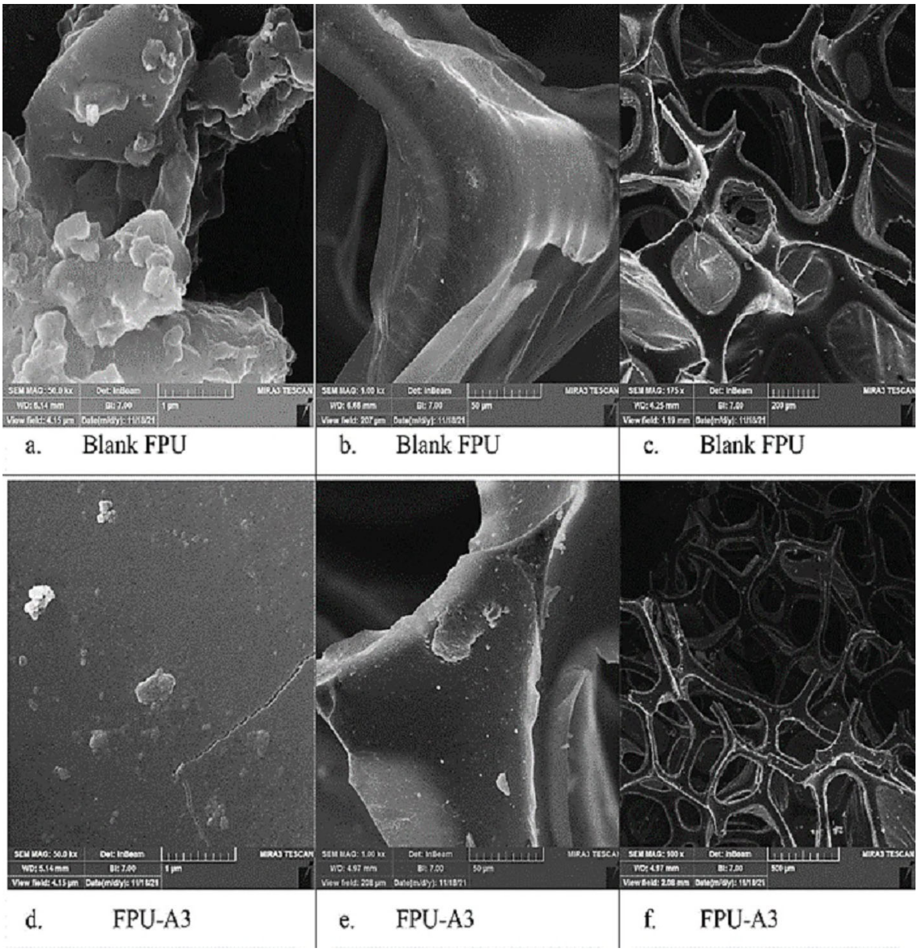

Figure 1. SEM micrographs of the blank FPU cubes (a–c) and modified FPU cubes (d–f).

(Li, Liu, and Yang 2012):

$$Oil\ sorption\ (g/g) = \frac{(M_t - M_o)}{S_o} \tag{3}$$

where M_o denotes the constant weight beaker, M_t denotes the weight of the constant weight beaker containing absorbed oil, and S_o denotes the sorbent's initial dry weight.

4. Results and discussion

4.1. Scanning electron microscopy (SEM)

Scanning Electron Microscopy (SEM) was used to analyze and study the morphological aspects of the blank FPU surface and the modified FPU surface. The SEM images of the blank FPU show that the pentagonal dodecahedron cell structure of the blank FPU cubes is not ideal.

Table 1. The crude oil sorption capacity of the blank FPU cubes and modified FPU.

FPU Cubes	S_O (g)	$S_{C.O}$ (g)	Sorption capacity (g/g)
Blank FPU	0.0916	2.7536	29.06
FPU-g-A1	0.095	3.6255	37.16
FPU-g-A2	0.1166	5.094	42.68
FPU-g-A3	0.1185	5.563	45.95

Figure 1 exhibits different magnifications of the SEM images of the FPU sponges before and after being grafted (Peng et al. 2014). The surfaces of the blank FPU sponges are smooth and flat (Figure 1a–c). Furthermore, Figure 1d–f have shown that the surface modification caused certain destruction of the cell structure/backbone of the FPU cubes (Li, Liu, and Yang 2012). The micrographs also reveal the presence of thin cellular wall films within the foams, which could increase their surface area and facilitate oil sorption even more. In the low magnification (Figure 1a) and high magnification (Figure 1b, c) SEM images, there were no visible micro-scale protrusions or spherical structures spread over the surfaces.

In contrast, the surface of the treated sponge exhibited random roughness, as indicated in the low magnification SEM images in Figure 1d–f. Moreover, many tiny spherical particles are distributed on the sponge, as indicated by the high-magnification SEM image, and Keshawy, Farag, and Gaffer 2020, made the same observation. The SEM images demonstrated that the modification with the new monomers (A1, A2, and A3) resulted in the formation of spherical particles on the surface. The spherical particles were very important to the hydrophobicity of the sponges, just like surface protrusions are to the hydrophobicity of lotus leaves (Peng et al. 2014).

4.2. Crude oil sorption capacity

Different alkyl chain lengths were used to examine the effect of the modified FPU hydrophobicity. Furthermore, a modified FPU foam with hydrophobic groups of different alkyl chain lengths (C5, C8, and C10) for (A1, A2, and A3) compounds was synthesized (Chen et al. 2021).

The crude oil sorption capacity was calculated using Eq. (1). This experiment data was an average of three experiments. The results obtained for the sorption capacity of blank FPU and modified FPU cubes with the three different organic compounds (A1, A2, and A3) are listed in Table 1 and shown in Figure 2. Their absorption capacities were ranked as follows: FPU-A3 > FPU-A2 > FPU-A1. This ranking indicates that the alkyl chain length of the compounds plays an important role in the capacity of the modified FPU foam for crude oil absorbency.

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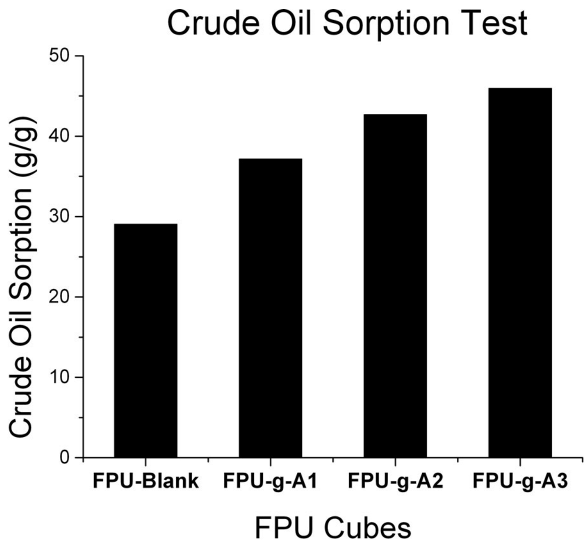

Figure 2. The crude oil sorption capacity of the blank FPU cubes and modified FPU cubes.

Table 1 and shown in Figure 2. Their absorption capacities were ranked as follows: FPU-A3 > FPU-A2 > FPU-A1. This ranking indicates that the alkyl chain length of the compounds plays an important role in the capacity of the modified FPU foam for crude oil absorbency. Because of the rising hydrophobicity properties, increasing the number of (CH2) groups in the alkyl chain length resulted in better sorption capacity (Gao et al. 2017; Fukuhara et al. 2021).

4.3. Diesel fuel sorption capacity

Equation (1) was used to calculate the diesel fuel sorption capacity. Table 2 and Figure 3 show the results obtained for the diesel fuel sorption capacity of blank FPU and modified FPU cubes with three distinctive organic compounds (A1, A2, and A3).

4.5. Water sorption capacity

Equation (2) was used to calculate the water sorption capacity. Table 3 and Figure 4 show the results obtained for the water sorption capacity of blank FPU and modified FPU cubes with three distinctive organic compounds (A1, A2, and A3).

Figures 3 and 4 show that modified FPU cubes performed better in absorbing crude oil and diesel fuel than blank FPU cubes. This implies that the hydrophobic properties of FPU cubes are enhanced by adding long-chain alkyl groups to the FPU backbones. The sorption capacity of

Table 2. The diesel fuel sorption capacity of the blank FPU cubes and modified FPU.

FPU Cubes	S_O (g)	S_{DES}. (g)	Sorption capacity (g/g)
Blank FPU	0.1082	3.0587	27.26
FPU-g-A1	0.0996	3.7572	36.72
FPU-g-A2	0.0954	3.8926	39.80
FPU-g-A3	0.1107	4.7253	41.68

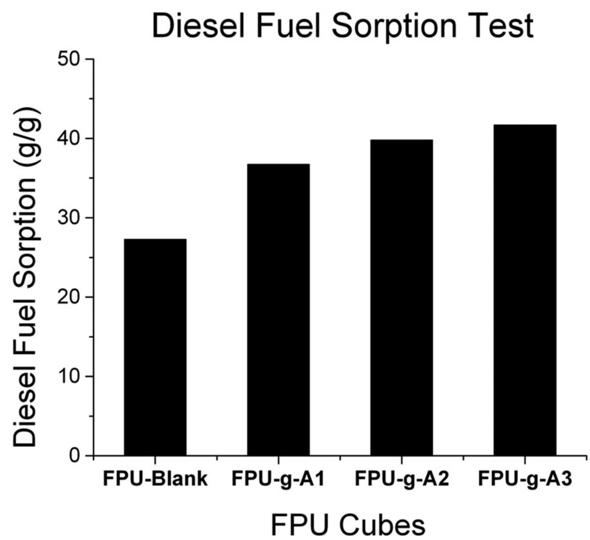

Figure 3. The sorption capacity of diesel fuel of the blank FPU cubes and modified FPU cubes.

Table 3. The water sorption capacity of the blank FPU cubes and modified FPU.

FPU Cubes	S_O (g)	S_W (g)	Sorption capacity (g/g)
Blank FPU	0.1033	1.9674	18.05
FPU-g-A1	0.0987	0.8656	7.77
FPU-g-A2	0.0968	0.8329	7.60
FPU-g-A3	0.1096	0.9405	7.58

modified FPU cubes is indicated in the following order: FPU-g-A3 > FPU-g-A2 > FPU-g-A1.

Long aliphatic non-polar groups (-CH_2), which are hydrophobic, improved the modified FPU cubes' sorption capacity for crude oil and diesel fuel more than the blank FPU cubes, which can be explained by the modified FPU cubes having higher oleophilic and hydrophobic features (Peng et al. 2014). A modest difference in the absorption ratio between the modified FPU cubes and the crude oil was observed in both crude oil and diesel fuel sorption, as shown in Tables 1 and 2, respectively. This may be attributed to the increase in the length of the alkyl chain from five carbon atoms in A1 to eight carbon atoms in A2 to ten carbon atoms in compound A3. This may be seen clearly in Table 4 and Figure 5 for the water, crude oil, and diesel fuel sorption capacities of blank FPU and modified FPU cubes.

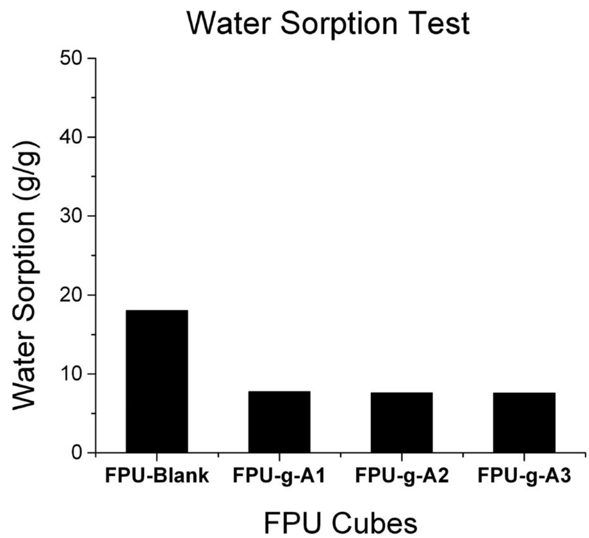

Figure 4. The water sorption capacity of the blank FPU cubes and modified FPU cubes.

Table 4. The water, crude oil, and diesel sorption capacities of the blank FPU cube and modified FPU cubes.

FPU cubes	Water sorption test (g/g)	Crude Oil Sorption test (g/g)	Diesel fuel sorption Test (g/g)
Blank FPU	18.05	29.06	27.26
FPU-g-A1	7.77	37.16	36.72
FPU-g-A2	7.60	42.68	39.80
FPU-g-A3	7.58	45.95	41.68

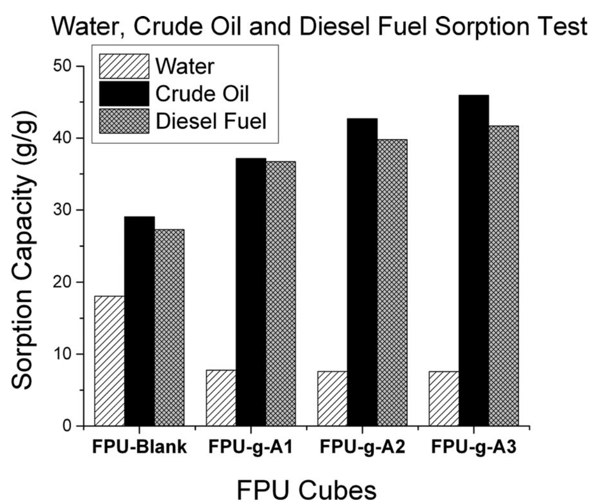

Figure 5. Water, crude oil, and diesel fuel sorption capacities of the blank FPU cube and modified FPU cubes.

The length of the alkyl chain increases the hydrophobicity of the polymer because of the interaction by the van der Waals force between oil and polymer and the polymer absorption of oil. This longer hydrophobic tail could

Table 5. The crude oil sorption capacity of the blank FPU cube and modified FPU cubes (water–crude oil system sorption tests).

FPU cubes	S_O (g)	M_O (g)	$M_{C.O}$ (g)	Sorption capacity (g/g)
Blank FPU	0.1033	43.2935	46.11	27.29
FPU-g-A1	0.1058	43.2935	47.89	43.47
FPU-g-A2	0.1082	43.2935	48.11	44.49
FPU-g-A3	0.1055	43.2935	48.06	45.15

have a greater van der Waals force of attraction with oil, which can lead to the reduction of the interfacial tension between oil and water (Dib et al. 2021). In addition, the driving force in the present system is intermolecular forces between highly hydrophobic sponge and non-polar oil-in-water droplets, which is the London force. Through the absorption of oil droplets on the hydrophobic sponge, the total surface energy of the system decreases because the oil-water interfacial area decreases (Khosravi and Azizian 2015).

The amount of water, crude oil, and diesel fuel adsorbed on modified FPU cubes could be controlled by varying the hydrophobic alkyl chain length in the sorption test. These findings suggest that grafted PU cubes could be useful adsorbents for oil spill cleanup (Aydin and Bulbul Sonmez, 2015). The length of alkyl chains influences the hydrophobicity of materials. The longer alkyl chain protects against water better than the shorter alkyl chains. When it comes to the alkyl chain, having fewer carbon atoms is not enough to make the surface hydrophobic enough to reject water without leaving a watermark. The hydrophobicity of the chain increases as it becomes longer (Baig and Kammakakam 2021).

4.6. Water–oil system sorption tests

The oil sorption in water–crude oil systems and water–diesel fuel systems is calculated using Eq. (3). Tables 5 and 6 present the obtained sorption capacity results, and Figures 6 and 7 show the sorption of water–crude oil and water–diesel fuel systems by blank FPU and modified FPU cubes, respectively.

Oils were collected from the surface of the water by simply placing modified flexible polyurethane (FPU) cubes on the surface of the crude oil-water mixture and the diesel fuel-water mixture. The sponge absorbs crude oil and diesel fuel. This test resulted in the quick absorption of crude oil and diesel fuel and separation from the water phase by removing the sponge (Visco et al. 2021). The results obtained imply that the modified sponge material was developed to show higher sorption capacities as shown in the tables and figures above. On the other hand, the results revealed that the hydrophobic surfaces have less interaction with water because of non-polar functional groups at the surface. In general, the length of the alkyl

Table 6. The diesel sorption capacity of the blank FPU cubes and modified FPU cubes (water–diesel oil system sorption tests).

FPU Cubes	S_O (g)	M_O (g)	$M_{C.O}$ (g)	Sorption capacity (g/g)
Blank FPU	0.1033	43.2935	45.59	22.28
FPU-g-A1	0.1058	43.2935	46.89	34.07
FPU-g-A2	0.1082	43.2935	46.98	34.16
FPU-g-A3	0.1055	43.2935	47.09	36.05

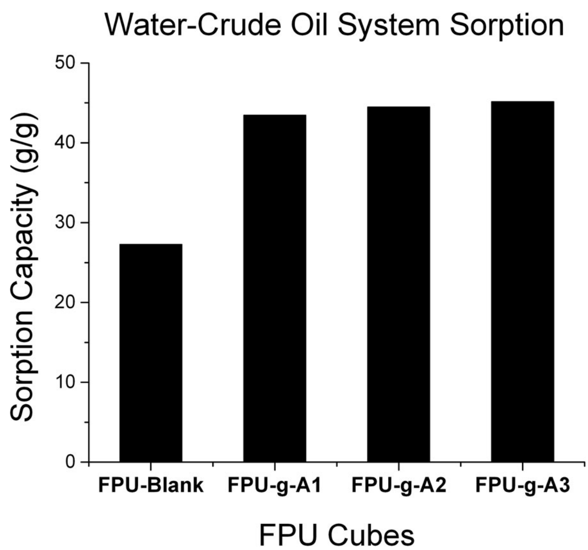

Figure 6. The crude oil sorption in water–crude oil system tests of the blank FPU cube and modified FPU cubes.

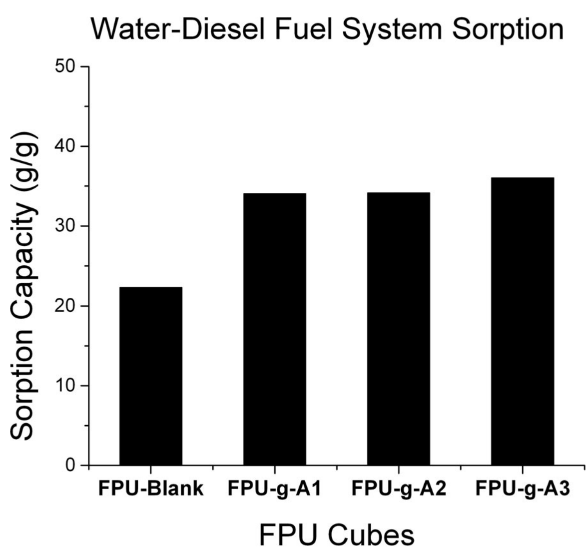

Figure 7. The diesel fuel sorption in water–diesel fuel system sorption tests of the blank FPU cube and modified FPU cubes.

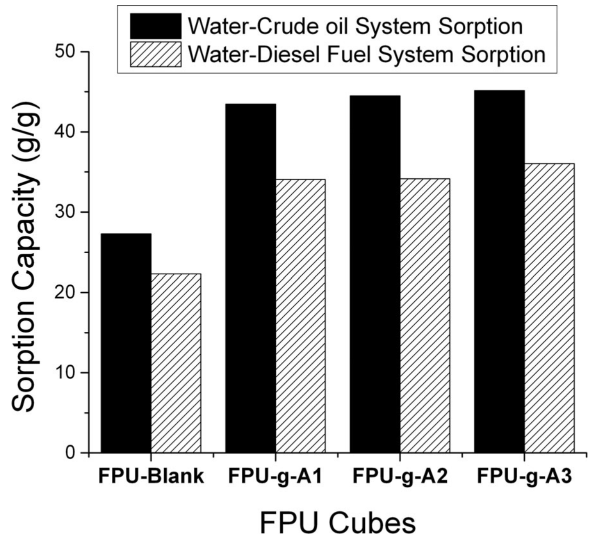

Figure 8. Crude oil and diesel fuel sorption capacity of the blank FPU cube and modified FPU cubes.

chains affects the hydrophobicity; by increasing the length of an alkyl chain, the surface would be more hydrophobic (Glser and Weitkamp 2008).

The SEM images, Figure 1, showed that the crude oil and diesel fuel were stored in the pores formed by the interconnected skeleton of the sponge, exhibiting a high oil absorption capacity. When sponges were used as absorptive materials for cleaning crude oil and diesel fuel on the water surface, they demonstrated significant oil-absorption capacity and selectivity by combining unique wettability and high porosity (Visco et al. 2021).

4.7. The impact of oil type

Figure 8 shows the effect of oil type on oil sorption capacity using water-crude oil and water-diesel fuel systems. The following order is observed for the oil sorption capacity values: FPU-g-A3 > FPU-g-A2 > FPU-g-A1. The maximum sorption capacity values for crude oil and diesel fuel are recorded, with the former being the best. With increasing oil viscosity, the adherent forces between the adsorbent and the oil surface increase, resulting in increased oil adsorption (Eldin et al. 2016). The sponges' high porosity accounts for the sponges' great oil absorption capability. The attraction forces between the oil and the treated sponge were used to store oil in the pores. The sponge had many holes in it, which allowed the water to pass through.

The sponges' great porosity is largely responsible for their excellent oil absorption capability. The attraction forces between the oil and the modified sponge allowed oil to be stored in the pores. The sponge had many holes in it, which allowed the oil to flow upward to a higher level. These holes may be thought of as approximate capillaries, and the entire structure could be thought of as capillary walls (Peng et al. 2014).

The differentiation in the sorption capacity of crude oil and diesel fuel in their water systems may be attributed to viscosity differences between crude oil and diesel fuel that are higher than the viscosity of water (Jalal, Khalaf, and Hussein 2021). The competition between water and crude oil at the sponge's surface is low enough for crude oil to reach the surface. Therefore, crude oil wins out, while the competition between water and diesel is increased, resulting in a drop in diesel absorption relative to when each liquid is alone.

5. Conclusions

The current study aims to improve the oleophilic and hydrophobic properties of PU foams by modifying the surface with three different organic compounds having long-chain alkyl groups and good crude oil and diesel fuel affinities. The sorption characteristics of the blank and modified flexible polyurethane (FPU) cubes were thoroughly tested and compared in water, crude oil, and diesel fuel as well as in water-oil systems.

The oil sorption of modified flexible polyurethane (FPU) cubes by three different long-chain alkyl groups were enhanced. The modified FPU cubes are applied to clean up spilled oil. It was found that the greater the length of the chain, the greater the non-polarity, and therefore the hydrophobic and oleophilic qualities increase. As a result, the modified FPU cubes can be placed in the following order: FPU-g-A3 > FPU-g-A2 > FPU-g-A1 in terms of oil absorption capability. The improved FPU-g-A1, FPU-g-A2, and FPU-g-A3 have increased the oil sorption capacity of crude oil by 37.16, 42.68, and 45.94 g/g, and diesel fuel by 36.72, 39.80, and 41.68 g/g, respectively. Whereas, the water sorption capacity of the flexible polyurethane (FPU) cubes was lowered from 18.04 g/g for blank FPU to 7.77, 7.60, and 7.58 g/g for FPU-g-A1, FPU-g-A2, and FPU-g-A3, respectively, after FPU surface modifications. This may be attributed to the van der Waals force interaction between oil and polymer; the length of the alkyl chain increases the polymer's hydrophobicity, and the polymer's oil absorption increases. For these liquids, all of the grafted FPU foam cubes had a very high swelling capacity. The length of alkyl chains influences the hydrophobicity of materials. The longer alkyl chains protect against water better than the shorter alkyl chains. When it comes to the alkyl chain, having fewer carbon atoms is not enough to make the surface hydrophobic enough to reject water without leaving a watermark. The hydrophobicity of the chain increases as it becomes longer.

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Nomenclature and Abbreviation List

PU	polyurethane
FPU	flexible polyurethane
A1	N^1-acryloyl-N^7-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl) acryloyl) phenyl)heptane diamide
A2	N^1-acryloyl-N^{10}-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl) acryloyl) phenyl)decane diamide
A3	N^1-acryloyl-N^{12}-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl) acryloyl) phenyl)dodecane diamide
S_o	sorbent's original dry weight
S_t	sorbent weight after crude oil or diesel fuel has been absorbed
S_{wt}	wet weight of sorbent after water sorption
M_o	container's constant initial weight
M_t	container constant weight containing absorbed oil