



Original article

A Study of Oil Absorbing Capacity of Cellulose-implemented Polyurethane for the Recovery of Oil Spills

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Abstract

Recently, Oil spill incidents from maritime activities and port operation have been causing the serious ocean environment pollution, these problems are said to be the negative effects on the natural environment, social economy, marine species, and human health. Due to the high costs of treating oil spills and oil slick in comparison with a low-income country like Vietnam, many incidents related to the oil spill and oil slick have not been thoroughly processed. Cellulose components from Vietnamese agricultural residues used to produce the absorbent materials are one of the most urgent issues and this is the research object of this work. In this study, two types of structural lengths of cellulose added into PU matrix foam are used to measure how much crude oil, fuel oil, diesel oil and kerosene can be absorbed. The absorbent materials are designed after adding cellulose with 5%, 15%, 25% of mass, respectively. The achieved results show that the oil absorption capacity of PU-cellulose implemented 5% cellulose with 500 μ m of cellulose structure length and 25% cellulose with 3000 μ m of cellulose structure length are highest for crude oil. These study results from this work provide a reasonable price for the protection of the marine environment in the strategies of recovery and treatment of oil spill and oil slick on the seawater surface.

Keywords: PU; cellulose; oil absorption; oil spill and slick; marine environment

1. Introduction

Vietnam has an over 1.000.000 km² of the exclusive economic zone and more than 3.000 large and small islands, the two archipelagos such as Truong Sa and Hoang Sa, the coast stretching over 3.260 km. Vietnamese sea zone has hundreds of wells for exploration of oil, and gas. There is a huge amount of water-mixed-oil, which discharges around 5.600 tons of oil and gas waste into the sea each year, where 20% to 30% of this mixture is a hazardous solid waste without dumping and treating. Furthermore, the pollution of the marine environment also occurs dramatically in ports due to the operation of vessels, ships entering and leaving the port, dredging of canals, and dumping of waste. There are over 160 ports from the North to the South with 20% of the annual growth rate hence the oily water is discharged ever-increasingly. The oil concentration in water at some typical ports in Vietnam in 2016 is shown in Figure 1.

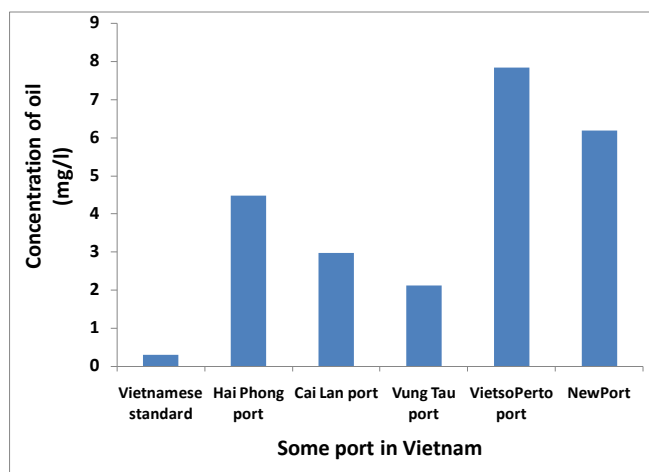


Figure 1: Oil concentration in water at some Vietnamese typical ports in 2016

Figure 1 shows that, the oil concentration in most of the ports has exceeded about 14 times of Hai Phong port, 10 times of Cai Lan port, 7 times mg/l of Vung Tau port, 26 times mg/l of VietsoPerto port, 21 times of New port in comparison with the permitted level of 0.3 mg/l of Vietnamese standard 5943-1995. The oil spill and oil slick prevent oxygen from dissolving into water, so the oxygen content in water is low, the average oxygen content is 3.3-10.9mg/l in the dry season and 1.16-6.1mg/l in rain season in comparison with 13.6-31mg/l of requirement. The oil spill and oil slick are acting much on about 85 of different species of creatures that are facing to the extinction in which more than 70 of creature species have been included in the Vietnamese

Red Book. The polluted marine environment due to oil spills and oil slick has led to the degradation of marine biodiversity, typically coral ecosystems. The sea zone of Vietnam has about 1.122 square kilometers of coral reefs. Each year, more than 50 tons of coral are lost in Quang Binh, Quang Tri, Quang Ninh, and Hai Phong.

Therefore, a review on the impacts of oil spills and oil slick on the environment and creature lives as: seabirds and long-living animals, even temporary or permanent damage of foreshore habitats or marine habitats; damage or smothering of intertidal vegetation and biota; impacts on tourism and entertainment activities; economic loss (Husseien et al., 2009). Some reasons increase the oily content in the water such as industrial development (Srinivasan and Viraraghavan, 2010), petroleum transportation (Al-Majed et al., 2012), maritime operation, oil refining industry, people habit and other... There were many developed techniques that include physicochemical or biological methods (Pokonova, 1993), for recovering, removing, treating the oil spill and slick from the water surface. These methodologies apply different techniques, including micro-filtration, filters or ultra-filtration, a chemical dispersant, osmosis methods, based-gravity, biology separations (Campos et al., 2002; Hoang and Chau, 2018; Hoang et al., 2018; Al-Jeshi and Neville, 2008; López-Vazquez and Fall, 2004). However, adsorption can probably be a potential method for polluted component recovery, removal, a treatment that is involved in the oil and oil processing products (Cybulski and Trawczynski, 2004). Oil absorption materials are categorized into two main types which are natural organic or inorganic, synthetic adsorbent materials (Qi et al., 2011). Bagasse, husk, sawdust, activated carbon, and peat is said to be absorption materials originated from nature (Brandão et al., 2010; Cambiella et al., 2006; Ahmad et al., 2005; Hoang et al., 2018).

For artificial adsorbent materials, which are made with polyurethane (PU), showed that an increase in absorption capacity of the oil when being implemented suitable ingredient. The obtained oil absorption was 46.98g/g of diesel and 41.42g/g of kerosene. This can be done by adjusting parameters of PU, such as monomer ratios, polymerization time or initiator (Li et al., 2013). Besides, the highest oil capacity of modified PU-A also was showed with 58.25 g/g of crude oil (Tanobe et al., 2009). It is found that when adjusting polyurethane

foams by adding extra 3% cloisite 20A nanoclay weight, 16% and 56% of efficiency of oil cleanup and removal, comparing with pure polyurethane foams, the result given was similar to that found by Nikkhah et al. (2014) and Phan, et al. (2017).

The aim of this research is to choose an ideal PU foams by adding cellulose or natural organic materials. The result of this article is a big step in producing oil absorption materials based on polyurethane foam and cellulose that is made from available materials in Vietnam aiming at recovering oil spill and oil slick to protect the marine environment.

2. Materials and methods

2.1. Materials

Cellulose: Vietnam is one of largest rice exporter in the world since it has a great potential for biomass from rice straw and husk, and this can be proven by an annual productivity approximately 42-45 million tons. The amount of biomass is obtained from the remaining parts of the rice is approximated to be 64% of Vietnam's biomass resources, which is equal to 150 million tons of straw and husk combined. In this respect, rice husk and straw are concentrated mainly in the Mekong Delta, Northern Delta, and South Central Coast. In 2015, Vietnam's rice output reached over 45 million tons and among them, the collected rice husk and straw is about 5.5-7 million tons, the remaining is released into the environment. Besides, among of the short-time industrial crops, sugarcane is one of the full-potential trees with 564.300 tons of productivity by 2015, however about 29% of sugarcane mass is bagasse, it is meaning that 163.674 tons of bagasse are discharged into the environment. Therefore, recycling rice husk and straw, bagasse resources does not only bring economic and social benefits but also has important implications for environmental protection. The properties of husk, straw, and bagasse are given in Table 1.

Table 1: Composition of husk, straw, and bagasse in Vietnam

Composition	Husk (% mass)	Straw (% mass)	Bagasse (% mass)
Cellulose	35-45	56-60	40-52
Hemicellulose	17-22	18-24	20-28
Lignin	25-32	14-17	18-24
Others	5-11	4-8	3-7

Table 1 shows that the percentage of cellulose in husk, straw, and bagasse is the most. The cellulose is a high-molecular-weight compound with $(C_6H_{10}O_5)_n$ of the chemical formation. To obtain cellulose from biomass (husk, straw, and bagasse...etc.), catalysis NaOH (10-20% of mass compared to biomass) was used at 120-160°C in 3-5 hours.

Polyurethane: A polymer compounds are used to surround unsaturated polyesters, epoxies, and phenolics (Wilson et al., 1992). Polyurethanes are produced when isocyanate $R-(N=C=O)_n$ start to chemically react with a polyol (polyol) $(R'-(OH)_n$ in the catalyst or in the UV beam (Marc et al., 2014). Therefore, the properties of isocyanates and polyols have a significant effect on polyurethane properties. In-crosslinking polyurethanes (PU) consist of a three-dimensional network and may be considered as a giant molecule. Polyurethanes are included the segments with chemical structure $-(O-R-O)-(CONH-R_1-NH-CO)-(O-R_2-O)-(CO-NH-R_3-NH-CO)-$, with R, R₁, R₂, R₃ of organic groups (Stirna et al., 2006). Because of low density and high porosity, the PU foams are considered as a good material for oil absorption. However, due to PU molecular contains not only poly-hydroxyl compounds (-OH), and poly-amino compounds (-NH_x) with poly isocyanates ($R-N=C=O$), but also it contains ether (-CO-), ester (COO-), carbamate (R₁OCONR₂R₃), and amide (R_nX(O)_xNR₂, X may be C, S, P) groups, hence PU absorb both of oil and water. In this work, PU with 0.978 of porosity provided by China is implemented with 5%, 15%, 25% of cellulose, respectively. These obtained products are used to test the ability to absorb oil. The densities of cellulose-implemented-PU (CIP) are given in Table 2, and Figure 2 shows the difference in the structure of PU and Cellulose implemented PU.

Table 2: Density of CIP with different cellulose mass

Properties	CIP5	CIP15	CIP25
Density (g/cm ³)	0.295	0.289	0.284



Pristine PU



5% of 500µm of cellulose implemented PU



15% of 500µm of cellulose implemented PU

25% of 500µm of cellulose implemented PU



5% of 3000µm cellulose implemented PU



15% of 3000µm of cellulose implemented PU



25% of 3000µm of cellulose implemented PU

Figure 2: Parcels of PU and cellulose implemented PU (CIP)

2.2. Measurement method of oil absorption

2.2.1. Preparation for experiment

The method corresponding to ASTM F726-99 is used to determine the sorbent capacity of used oil and water. In order to evaluate the absorption capacity of CIP materials, petroleum products, including crude oil, fuel oil (FO), diesel oil (DO), and kerosene with varying densities were utilized as test subjects. This is because fuel oil (FO), diesel oil (DO) are the most widely used energy source for ships and vessel fleet. Crude oil is one of the most popular products exploited on oil rigs in Vietnam and kerosene is mainly used for cooking in the Mekong Delta. These fuels regularly float on the surface of the river, port in the Mekong Delta, so it can be used as a test subject. Table 3 shows the properties of crude oil, fuel oil (FO), diesel oil (DO), and kerosene measured in Vietnam.

Table 3: Density of oil

Properties	Unit	Crude oil	FO	DO	Kero-sene
Density, ρ	kg/m ³	937	890	850	810
Surface tension, σ	mN/m	31.7	28.8	26	22.5

2.2.2. Measurement method

Absorbent material (AM) samples are prepared for evaluation of absorption capacity of oil. For purpose mentioned above, the experimental description has shown that 1 gram of absorbent material was immersed into the glass of 250ml liquid fuel. Figure 3 depicts the experiment was performed at room temperature, ambient pressure in ten minutes without any application of external forces as it may affect the oil absorption capacity

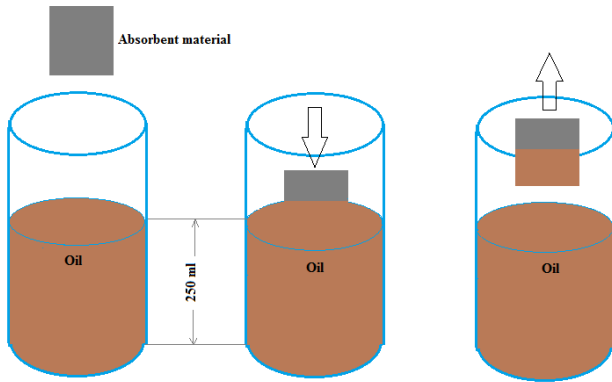


Figure 3: Measurement method of oil absorption capacity

The equation to calculate oil absorption capacity of experimental materials is the ratio of absorbed oil weight (m_{oil}) to cellulose-implemented-PU (m_{CIP}) weight, also known as PU (m_{PU}). A scale with an accuracy of three decimals is used to weigh two experimental samples. The weight of absorbed oil is calculated by Eq.1.

$$h = \frac{m_{oil}}{m_o} = \frac{m_{total} - m_o}{m_o} \tag{1}$$

Where:

m_{oil} is mass of absorbed oil

m_{total} is the total mass after pulling the CIP

m_o is mass of dry CIP

However, because of the influence of oil density, added surface treatment components in each study, hence using the ratio of m_{oil}/m_o shows the difference in determining the parameters during the process of oil absorption. To overcome this disadvantage, we can use the ratio of volumetric absorption such as (v_{oil}/v_{AM}) and this ratio is shown in Eq.2.

$$\frac{v_{oil}}{v_{AM}} = \left(\frac{m_{oil}}{\rho_{oil}}\right) / \left(\frac{m_{AM}}{\rho_{AM}}\right) = h \frac{\rho_{AM}}{\rho_{oil}} \tag{2}$$

ρ_{AM} is the density of the absorbent material. In this study, absorbent material samples are PU foam with 0.3 g/cm^3 of density and CIP. CIP includes CIP5, CIP15, CIP25 (PU implemented with 5%, 15%, 25% of cellulose, respectively).

Using the ratio of volumetric absorption may be able to compare exactly the performance of absorbing oil spill for different samples with assuming that oil spill or oil slick is not the cause of swelling of the AM, as well as the surface treatments do not affect the volume of the AM. In this case, the maximum volume of oil is considered as the volume of porosity (V_p). Hence, volumetric absorption ratio (v_{oil}/v_{AM}) may be determined as Eq.3 with the relative density of AM (ρ_{rAM}) (Pinto et al., 2016).

$$\frac{v_{oil}}{v_{AM}} = \frac{V_p}{\rho_{AM}} = \frac{1 - \rho_{rAM}}{\rho_{rAM}} \tag{3}$$

According to Eq.3, some results plotted in Figure 4 are shown about the relationship between the ratio $\frac{v_{oil}}{v_{AM}}$ characterized the capacity of oil absorption and the porosity of AM.

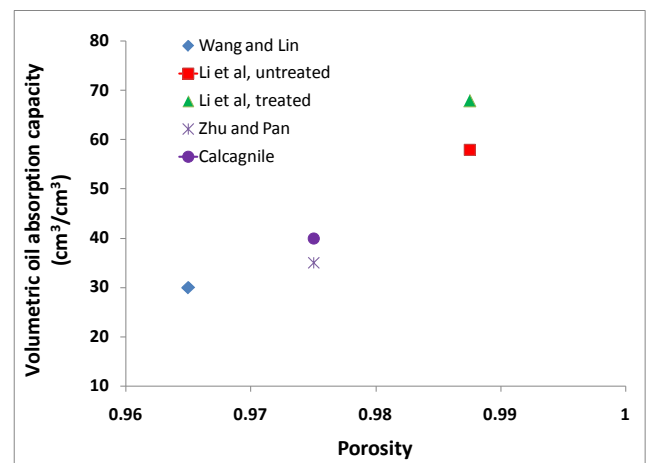


Figure 4: Some results of past researches for the capacity of oil absorption with PU foams

Based on theoretically, the capacity of maximum volumetric absorbed oil ($(v_{oil}/v_{AM})_{max}$) is correlated directly with the percentage of spaces that allow the oil to fill and the retainable capacity of the oil inside the porous structure. The increase in the filling, taking oil into the porous structure can be promoted in case of maintaining two conditions such as an interconnected porous structure and mechanism of driving the oil. Moreover, once the oil is pushed into the pores and then retained with a capillary pressure

presence (ΔP) (Zhu et al., 2013) that is calculated as Eq.4.

$$\Delta P = (4\sigma_{oil} \cdot \cos\alpha)/d \tag{4}$$

where:

σ_{oil} is the oil surface tension

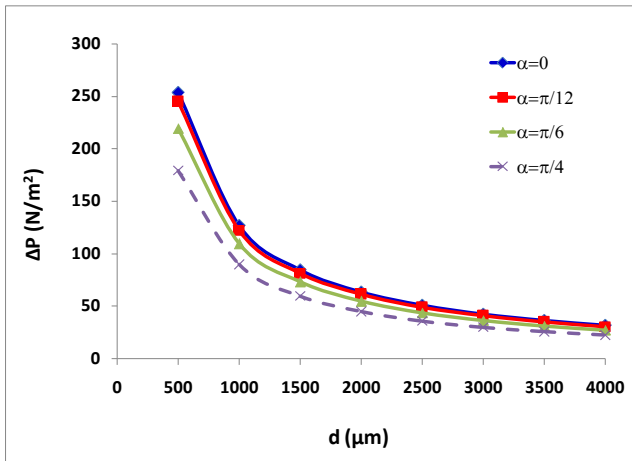
α is considered as the contact angle between oil molecular and the AM surface

d is the pore diameter

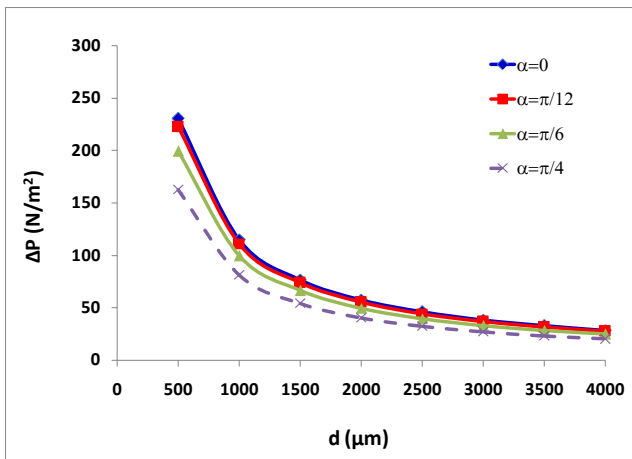
3. Results and discussion

3.1. Capillary pressure

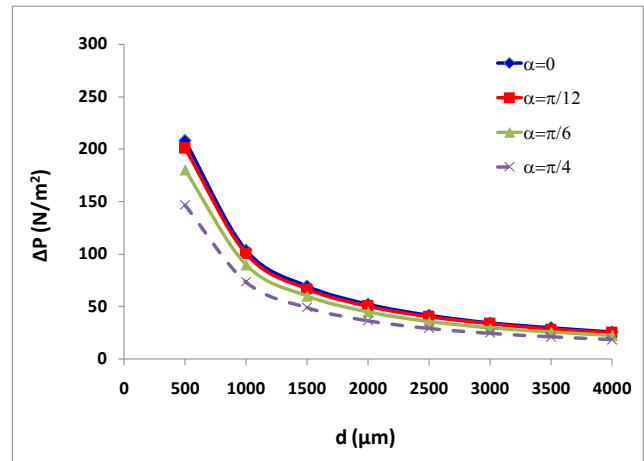
Capillary pressure (ΔP) is inversely proportional to pore diameter, oil surface tension, and contact angle. Meaning that the capacity for filling, taking the oil can be used in determining oil surface tension and the structural pores. Moreover, there is difficulty because of appearances of the small diameter of pores, and large oil surface tension. Figure 5(a-d) demonstrates the relationship between ΔP and σ_{oil} , α and d



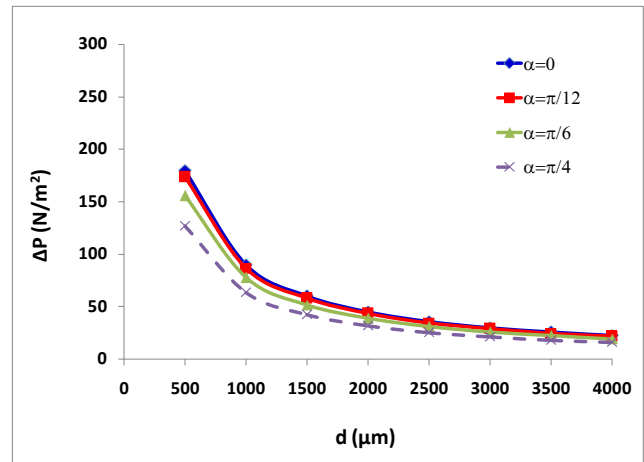
(a): ΔP of a crude oil-CIP ($\sigma_{crude\ oil} = 31.7\text{mN/m}$) for ranging from 500 to 4000 μm of pore diameters with different contact angles



(b): ΔP of a crude oil-CIP ($\sigma_{crude\ oil} = 28.8\text{mN/m}$) for ranging from 500 to 4000 μm of pore diameters with different contact angles



(c): ΔP of a crude oil-CIP ($\sigma_{crude\ oil} = 6\text{ mN/m}$) for ranging from 500 to 4000 μm of pore diameters with different contact angles



(d): ΔP of a crude oil-CIP ($\sigma_{crude\ oil} = 22.5\text{ mN/m}$) for ranging from 500 to 4000 μm of pore diameters with different contact angles

Figure 5: ΔP of an oil-CIP as a function of pore diameters and contact angles

Figure 5 (a-d) shows there is a reduction of ΔP , meanwhile CIP pore diameters increase. However, the oil absorbent capacity of CIP is determined and evaluated not only by the surface tension of the oil spill but also by the CIP structure such as the connectivity and the diameter of the pores. Ordinarily, the more the oil absorption efficiency increase, the more the pores size, especially, maximum oil absorption efficiency can be reached if the pore diameter is less than 1000 μm . This can be explained that if the ΔP value in each pore is strong enough, it can fill the oil completely into the porous structure of CIP, oil spills can be preserved before recovery and

extraction by mechanical equipment. Besides, reducing the pore diameter results in a significant increase of ΔP , shown in Figure 5, the ΔP of CIP samples with 500 μm of the pore diameter is six times higher than that of CIP samples with 3000 μm of the pore diameter.

3.2. Oil absorption capacity

3.2.1. Effect of the cellulose structure length on oil absorption

Figure 6 and Figure 7 show the relationship between the cellulose structure length such as 500 μm , 3000 μm and oil absorption capacity

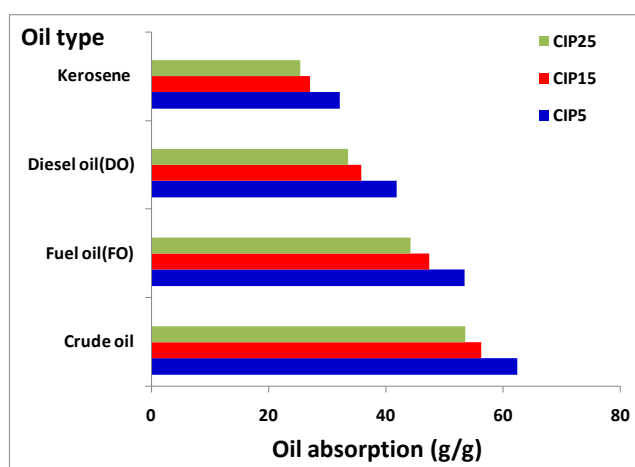


Figure 6: Oil absorption capacity of CIP with 500 μm of cellulose structure length

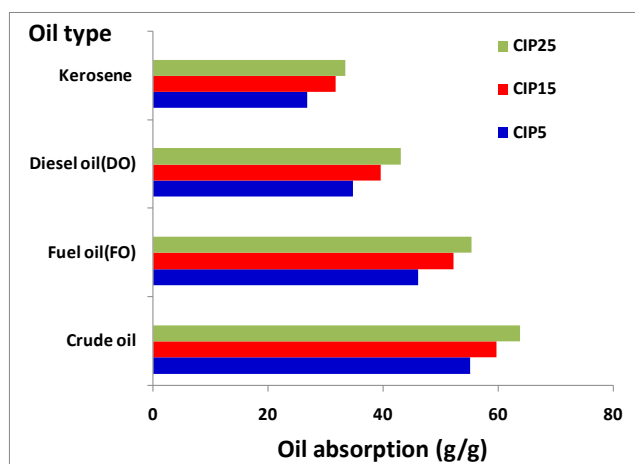


Figure 7: Oil absorption capacity of CIP with 3000 μm of cellulose structure length

Figure 6 shows that the oil absorption capacity of CIP for all experimental oil as cellulose structure length is 500 μm will increase while decrease percentage of cellulose in PU foam. The maximum weight of absorbed oil is 62.4 g/g for CIP5. For crude oil, the oil absorption capacity of CIP5 is 9.93% higher than that of CIP15 and 14.26% than that of

CIP25. For FO, the oil absorption capacity of CIP5 is 11.23% higher than that of CIP15 and 17.22% than that of CIP25. For DO, the oil absorption capacity of CIP5 is 14.35% higher than that of CIP15 and 19.61% than that of CIP25. For kerosene, the oil absorption capacity of CIP5 is 15.57% higher than that of CIP15 and 20.87% than that of CIP25. The reduction of oil absorption happens because the surface tension and density of DO, FE and crude oil are larger than that of kerosene. Besides, Figure 5 shows the smaller ΔP for a kerosene-CIP in comparison with ΔP of DO, FO, crude oil with CIP. Moreover, a small length of cellulose results in decreasing the porosity and oil absorption capacity as increasing the percentage of cellulose.

However, while the length of cellulose is 3000 μm , the oil absorption capacity is proportional to the increase in the percentage of cellulose. From Figure 7, it can be seen that the oil absorption capacity of CIP25 is 13.48% higher than that of CIP5, and 6.42% than that of CIP15 for crude oil. For FO, the oil absorption capacity of CIP25 is 16.78% higher than that of CIP5, and 5.77% than that of CIP15. For DO, the oil absorption capacity of CIP25 is 19.25% higher than that of CIP5, and 8.12% than that of CIP15. For kerosene, the oil absorption capacity of CIP25 is 19.76% higher than that of CIP5, and 5.09% than that of CIP15. The above-mentioned result shows that, although the percentage of cellulose increases but the ratio of porosity also increases, it results in increasing the oil absorption capacity. Thus, with 500 μm of cellulose length, the CIP5 is the best of oil absorption capacity (CIP5_500); with 3000 μm of cellulose length, the CIP25 is the best of oil absorption capacity (CIP25_3000). The oil adsorption capacity of this new sorbent is relatively high, it is higher than oil adsorption capacity of Azolla plant (Amin et al., 2015), or several vegetable fibers (Annunciado et al., 2005). Moreover, this oil adsorption capacity of this new sorbent even is around 4 times higher than that of rice straw or 2 times higher than that of polyurethane (Hoang et al., 2018).

3.2.2. Effect of oil surface tension on the absorption capacity

The impacts of the surface tension petroleum products on the CIP absorption capacity are shown in

Figure 8.

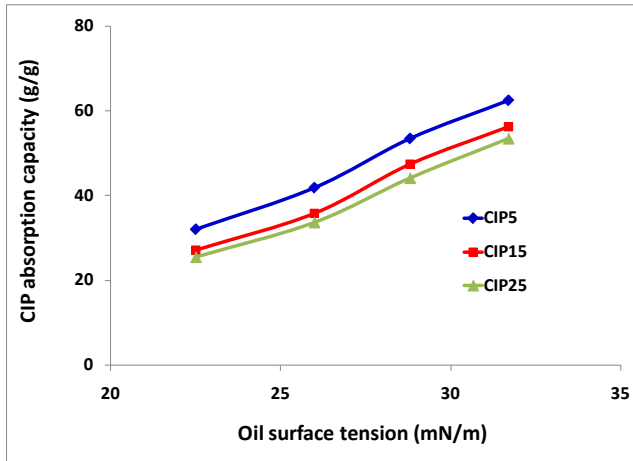
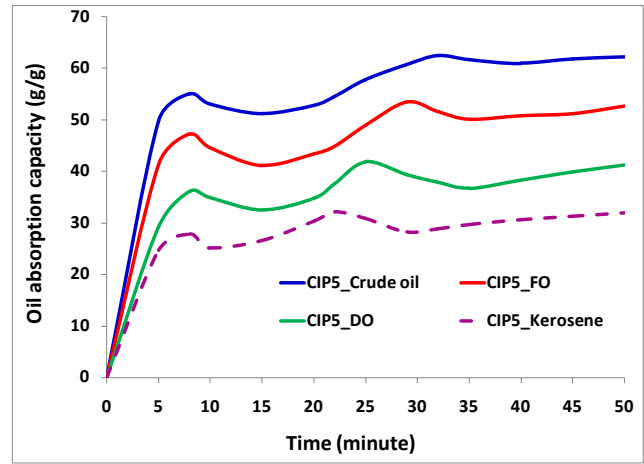


Figure 8: Relationship between the absorption capacity of CIP and oil surface tension

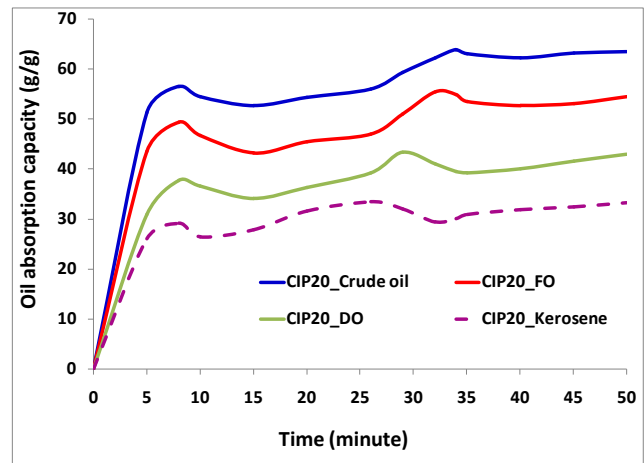
Figure 8 shows the increase of CIP absorption capacity and the surface tension of petroleum products, owing to the fact that fewer petroleum products were retained in the CIP pores. The result shown in Table 3 depicts kerosene with the lowest surface tension, density ($\sigma = 22.5 \text{ mN/m}$, $\rho = 810 \text{ kg/m}^3$) have low absorption for the CIP, while the crude oil surface tension and density ($\sigma = 31.7 \text{ mN/m}$, $\rho = 937 \text{ kg/m}^3$) have higher absorption. These results are the same as the study of Peacock et al. (2005). Hence, the above results prove that oil absorption capacity depends on not only the cellulose percentage, cellulose length, but also the petroleum product type.

3.2.3. Effect of time on oil absorption

Contact time between oil type and CIP surface is listed as one of the factors that affect the absorption capacity of CIP. From Figure 9a and Figure 9b, it is can be seen that, after increasing the oil absorption capacity for CIP with increasing in contact time on the first 8 minutes for CIP5_500 and 9 minutes for CIP25_3000, the equilibrium occurs.



(a) Effect of contact time on oil absorption of CIP5_500



(b) Effect of contact time on oil absorption of CIP25_3000

Figure 9: Effect of contact time on oil absorption

Moreover, the curve in Figure 9 includes two phases such as rapid and slow one. This trend can be explained that absorption starts with bigger pores and porosities absorbing the petroleum products, subsequently the oil molecular penetrated into the micro-pores/porosities until equilibrium steady state is reached. The time of equilibrium for crude oil, FO, DO and kerosene on CIP5 with 500 μm (CIP5_500) of cellulose length are 32, 29, 25, and 22 minutes, respectively. Furthermore, the time of equilibrium for crude oil, FO, DO and kerosene on CIP25 with 3000 μm (CIP25_3000) of cellulose length are 34, 32, 29, and 26 minutes, respectively.

4. Conclusions

The experimental results show that CIP5_500 and CIP25_3000 are maximum oil absorption materials with 62.4 g/g for CIP5_500 and 63.8 g/g for CIP25_3000. However, both of above absorbent materials, the absorption capacity for crude oil is highest and for kerosene is lowest. The average time for oil absorption to stabilize for CIP25_3000 is 40.8 minutes/63.13 grams

of absorbed crude oil and for CIP5_500 is 40.4 minutes per 61.78 grams of absorbed crude oil. Therefore, it may confirm that CIP5_500 or CIP25_3000 can be used for overcoming the oil spill and slick on the sea face. Besides, using the agricultural residue such as husk, baggage or straw to recovery the cellulose and implement cellulose into the PU foam will bring many big benefits to the environment.

In the next research, the authors will carry out designing, fabricating the equipment taking CIP plate in order to recover oil spill and slick at some sea areas or port.

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