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Application of natural rubber latex foam as an effective oil absorbent

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Abstract

Oil spills have had catastrophic effects on marine ecosystems. The removal of oil spills from the water surface has then attracted worldwide attention. Natural rubber (NR) is a well-known example of a hydrophobic material, due to its hydrocarbon structure. For this purpose, the potential of NR as an oil absorbent is demonstrated. NR was formed into a cellular structure where oil absorbency can be controlled by their densities. It can be done by varying the foaming volume from 2-8 fold (2×, 4×, 6×, and 8×) the original volume of the beater. Increasing the foaming volume has reduced the densities. Over the variations of densities, the oil absorbencies were 10.58 to 16.76 g/g for crude oil, and 6.56 to 13.18 g/g for diesel oil. This indicated that the absorbent based on NR latex foam provided excellent performance and could be practically used as an oil-absorbent material.

Keywords: *natural rubber latex foam, oil absorbency, foam, environment.*

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1. Introduction

Oil spills in the sea have become a major issue in marine areas. The need for marine oil transportation and the growth of the offshore oil industry are making this problem more serious[1-3]. This also extends to oil spills in workshops or in the industry, particularly when machinery and piping systems are involved. Oil spills have historically been cleaned up using a variety of methods, such as *in situ* burning, mechanical dispersion collection, bioremediation, or by application of absorbent materials[4-6]. A combined approach is the best option because oil collection and remediation are cost-effective and efficient. Thus, finding workable ways to create absorbent materials for oil cleanup is crucial.

Natural rubber latex (NRL) is a fascinating water-based bio-polymer derived from the milky white sap of *Hevea brasiliensis* trees[7]. Natural rubber (NR) consists of long polymer chains that make it durable, flexible, and resistant to abrasion[8,9]. However, NR has many disadvantages, such as poor resistance to oxidation, chemicals, and in particular oils. This is mainly due to the hydrocarbon structure of NR. This has made this raw material interesting to convert to an oil-absorbent material. Scientific and applied research groups are becoming more interested in the nonpolar side chains in NR. The physical characteristics of rubber can be altered by converting the common solid form of rubber into a cellular structure, in order to increase its oil absorbency. Cellular rubber, or rubber foam, has such a structure with air-filled cells. NR foam is a popular option for many applications since it is lightweight, good thermal insulator, and absorbs sounds[10,11]. Additionally, the porosity can improve the rate at which oil penetrates into rubber. NR foam can be made in a number of ways, using either dry or latex form as the main ingredient. In addition, there are tons of other benefits that NR latex foam offers over the synthetic sponge materials.

This is a preliminary study on the potential of NR latex foam to serve as an absorbent for oil in surface water. Here, a series of experiments was designed and the absorption capacities for crude oil and diesel oil were determined. The results are compared with the commercially available oil-absorbent materials polyurethane foam (PUF) and nonwoven polypropylene (PP). These two polymers are representative of cost-effective oil-absorbent materials. Hoang et al.[12] prepared the PU filled with rice straw and found that this material could absorb the oil equal to 12.012 g/g. The capacity was around 3 to 4 times higher than that of material fabricated by pristine PU. Zaro et al.[13] reported on the absorption capacity of the commercial nonwoven PP blanket relative to diesel, petroleum, and lubricant oil in long-term tests, it was found to have a capacity of 5.3, 12.3, and 18.7 g/g respectively. The results of the short and long-term tests did not show a statistical difference in the absorption capacity of the blanket. PU foams can be more flexible than nonwoven PP, semi-rigid, rigid, and have different densities. The use of PU in upholstery composed of reinforced materials with absorption capacity can be more advantageous[14]. Oil absorption and desorption are made possible by the viscoelastic characteristics of foams produced with high open-cell concentrations. However, a lot of absorbents have low oil retention, restricted buoyancy, and inadequate absorption capability[15,16]. If they are administered loosely, they are difficult to regulate and recover. Even if their recovery is facilitated by enclosing them in an outer net or cloth, they frequently become soaked too quickly and may sink. Therefore, the use of PUF and nonwoven PP is very limited by their poor environmental compatibility[17].

This study focused on the use of NR latex foam as oil absorbent material. To optimize oil absorbency, NR latex foams with various foam densities were prepared. This is because the foam density of the foam reflects the proportions of rubber and gas phases in the cellular structure. It is believed that the optimum oil absorbency can be obtained by tuning the proportions of these two phases. The ability of oil to penetrate throughout the cellular structure can be optimized by preparing various foam densities. The results obtained from this study will be used to support further experiments for improving oil absorbency by applying other key factors. This is considered a proof of concept study for preparing oil-absorbent materials based on scraps from raw rubber production.

2. Experimental Details

2.1 Materials

The high ammonia centrifuged latex with 60% dry rubber content (or 60% HA) was acquired from Yala Latex Industry, Co., Ltd., Thailand. The quality of the latex was tested, and it had total solids content (TSC) of 61.4%, dry rubber content (DRC) of 60%, alkalinity of 0.53%, volatile fatty acids (VFA) content of 0.06%, and mechanical stability time (MST) of 875 sec. The other additives namely 20% potassium oleate, 50% zinc diethyl dithiocarbamate (ZDEC), 50% zinc 2-mercaptobenzothiazole (ZMBT), 50% zinc oxide (ZnO), 50% Wingstay L, 50% sulfur, 15% diphenylguanidine (DPG), and 50% sodium silicofluoride (SSF), were supplied by Siamnavakam Co. Ltd., Thailand. Polyurethane foam (PUF) was supplied by 3M Thailand Co. Ltd., Thailand. The density of PUF is 0.0170 ± 0.0008 g/cm^3 with an average cell size of 304.62 ± 79.24 µm. Nonwoven Polypropylene (PP) was purchased from Benetech Products Co. Ltd., Thailand. The average fibrous diameters of Nonwoven PP is 19.42 ± 4.48 µm.

2.2 Preparation of NR latex foam

The formulation for lab-scale production of NR latex foam is displayed in Table 1. The components in dispersion or emulsion forms were incorporated alongside the HA latex. The Dunlop process was used to prepare NR latex foam. First, a cake beater was filled with a specific amount of NR latex (HA-type), and ammonia was gradually evaporated for 3 min. Subsequently, 20% potassium oleate, 50% ZDEC, 50% ZMBT, 50% Wingstay™ L, and 50% sulfur were added. The beating speed was increased and then maintained such that the desired foam volume was achieved at 5 mins. From this step onwards, the foaming volumes varied depending on the experimental design. Figure 1 illustrates the foam-forming behaviour of NR latex foam during beating. The foaming volumes varied from 2× to 8× the original volume of total batch.

Table 1. Compounding ingredients for preparing the NR latex foam.

Ingredient	Amount (phr)
60% HA	100
20% Potassium oleate	2.0
50% ZDEC	1.0
50% ZMBT	1.0
50% Sulfur	2.5
50% Wingstay L	1.5
15% DPG	1.2
50% ZnO	2.0
20% SSF	1.2

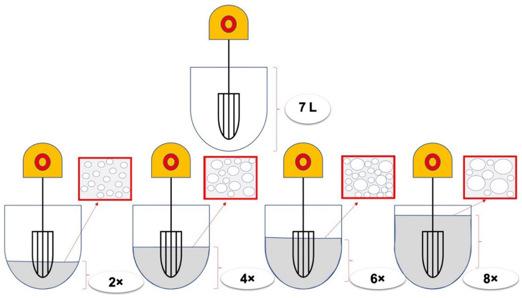


Figure 1. Illustration of preparing NR latex foams to various desired foaming volumes.

Next, 50% DPG and 50% ZnO were added, followed by quick addition of the gelling agent (20% SSF) and the mixture was beaten for another 1 min. Finally, the un-gelled foam was immediately poured into an aluminium mould and allowed to be set for 2 mins at ambient temperature. The gelled foam was cured for 45 mins at 100°C in a hot air oven. To remove soap and unreacted substances, the dried foam was thoroughly cleaned with water after removal from the mould. The cured NR latex foam was washed and dried for 4 hrs in a hot air oven at 80°C.

2.3 Oil absorbency

The test samples were cut to 1.5 cm × 1.5 cm × 1.0 cm dimensions. This equals a volume of 2.25 cm^3. Each sample was then weighed before immersion in crude oil for 15 min. After that, the equilibrium swelling was determined by measuring the development of swelling uptake or oil absorbency. The sample was weighed on a scale after gentle removal of extra oil from surfaces by tapping with a filter paper, in order to estimate the amount of swelling. The oil absorbency was calculated by the Equation 1.

$$\text{Oil absorbency} = \frac{W_a}{W_b} \tag{1}$$

where W_a is the weight of absorbed oil, and W_b is the weight of the sample. The unit of oil absorbency is g/g, representing the mass of oil absorbed per 1 g of sample. In this study, PUF and nonwoven PP were also tested for oil absorbency. This was done to enable a comparison with commercially available products.

The reusability test of the material was also conducted by repeating the test at each absorption cycle. Before repeating the steps, ensure that the sample is clean for each reuse cycle. After each cycle, record the weight of oil absorbed to calculate the oil absorption capacity of the absorbent material across multiple cycles.

2.4 Measurement of physical properties

The foam density of the foam was calculated as mass per volume. Initially, 1.0 × 1.0 × 1.0 cm^3 cubes of NR foam were made. Each sample was weighed and its mass per volume was determined. Compression–deflection test was done according to ASTM D575. A sample of dimensions 10.0 × 10.0 × 2.5 cm^3 was prepared for this testing. A universal testing machine (Tinius Olsen, H10KS, Tinius Olsen Ltd., Surrey, UK) was used to compress the specimen between its parallel metal plates until the thickness had decreased by 25% with the cross-head speed of 50 mm/min. The load was promptly read and recorded. The same specimen was cycled through the test multiple times until the load measurements did not vary by more than 5%. The force per area was recorded. Compression set was measured according to ASTM D395. A representative sample of dimensions 5.0 × 5.0 × 2.5 cm^3 was used for testing. The test specimen was compressed to 50% of its original thickness. The load was released after 70 h and the thickness was measured after 30 min at room temperature. The compression set calculation based on the Equation 2.

$$Compression\ set\ = \left[\frac{t_0 - t_1}{t_0 - t_s} \right] \times 100 \quad (2)$$

where t_0 is the original thickness, t_1 is the thickness of the specimen after the specified recovery period, and t_S is the thickness of the spacer bar used. An average of five replicates is reported for each type of sample.

2.5 The optical images of the samples

The cellular structure of the samples was screened using a light microscope at 40× magnifications. The image was then captured and imported to the Image J software to measure the cell size and fibrous diameters.

3. Results and Discussion

3.1 Physical properties of NR latex foam

The oil absorbency of a NR latex foam depends on two main factors: the physical and chemical characteristics of the foam. Here, the focus was more on the physical characteristics. The density of the foam was manipulated, since increasing the air phase will both increase the number of cells and porosity of the NR latex foam. Controlling the density of foam was simply done by fixing the foaming volume achieved by beating. The relationship of foaming volume with density of NR latex foam is shown in Table 2, where increasing foaming volume reduces density of the foam. This is because the proportion of air phase increased with the foaming volume. The porosity of the foam increased accordingly. Various densities of the foam can be achieved by varying the foaming volume during whipping in the cake beater. This method can be done easily without adding more foam promoter or blowing agent from fatty acid soap. This is not similar to other works that control the density by varying the foam promoter or blowing agent from fatty acid soap. For instance, Suethao et al.[18] reported that the chemical blowing agent also affected the morphology of the foam where reducing the blowing agent showed more interconnected foam structures with smaller cell sizes than those of the control sample.

Table 2. Foam density, compression set and compressive strength of NR latex foams prepared to various foaming volumes.

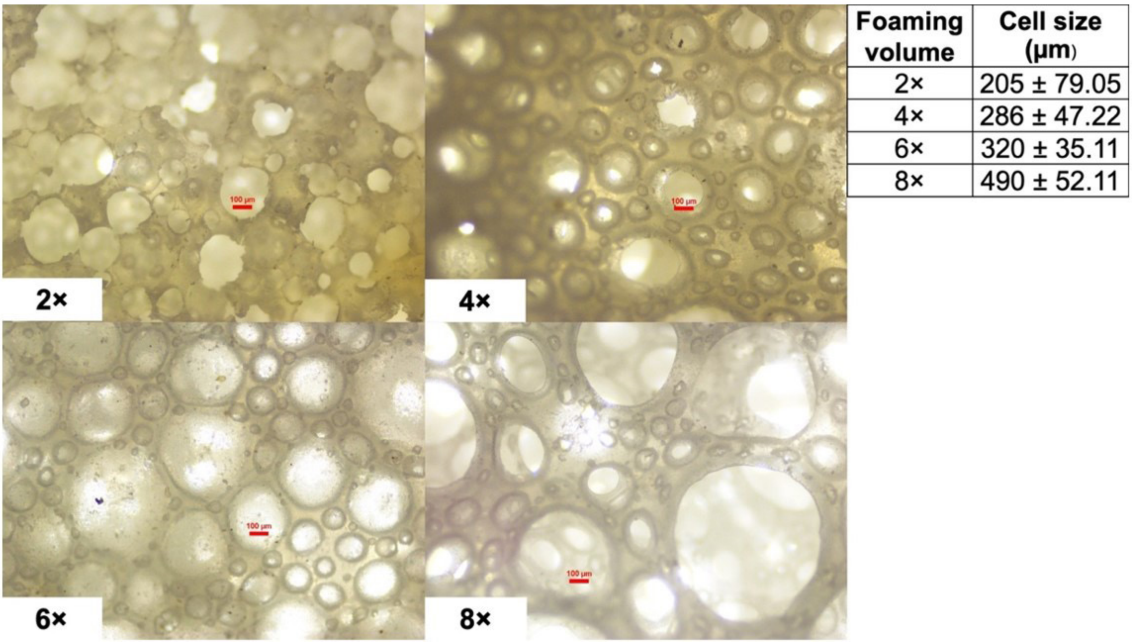
Foaming volume (ϕ)	Foam Density (g/cm^3)	Compression set (%)	Compressive strength (MPa)
2×	0.1888 ± 0.0099	62.68 ± 1.93	0.38 ± 0.02
4×	0.1752 ± 0.0098	62.15 ± 1.24	0.31 ± 0.02
6×	0.1567 ± 0.0083	59.37 ± 1.62	0.23 ± 0.01
8×	0.1296 ± 0.0099	58.12 ± 1.11	0.18 ± 0.01

Figures 2 and 3 show the characteristics and sizes of the sponge cells obtained for each foaming volume tested, before and after thermal aging. In the samples before thermal aging, roughly uniform distributions of cell sizes were found at low foaming volumes. However, the cell size of the foam tends to increase with foaming volume. On increasing the fraction of air phase by beating, the wall separating adjacent bubbles could collapse, so that they combined to a larger sized foam cell. This resulting increased cell size is associated with increased porosity of the foam. The porosity is expected to be coupled with the interfacial area of the solid and gas phases, and also porosity contributes to the permeability of the foam to oil. This is an important factor contributing to the efficiency of oil absorption.

The compression set and compression–deflection stress are also listed in Table 2 for NR latex foams prepared to various foaming volumes. The compression set indicates the ability of the sample to elastically recover its original shape after prolonged compressive deformation. Low values mean that the material has rebounded nearly to its original thickness, and there is very little residual deformation. Here, the foaming volume affected the compression set, which decreased with the foaming volume. Larger foaming volume increased the cell size and air diffusion through the sample. This also helps recovery of the shape after compression. As for the compressive strength, it also decreased with the foaming volume, so a lesser force was required to deform a bulkier more porous sample. Generally, the compressive strength of a foam depends on its cell size distribution, along with the number of cells that is associated with porosity. In this experiment, the foam density was varied affecting compressive strength.

3.2 Oil absorbency of NR latex foam

Figure 3 depicts oil absorbency versus foaming volume for the samples. It was observed that the oil absorbency increased up to 6× foaming volume, and then decreased: the maximum value was 16.76 g/g. This suggests that initially increasing the cell size facilitated penetration of oil and consequent swelling of the rubber. However, when the cell size is excessive, there are less rubber-air interfaces and the too large pores cannot hold the liquid in place, so less penetration and retention of oil is observed at an excessive



Foaming volume	Cell size (µm)
2×	205 ± 79.05
4×	286 ± 47.22
6×	320 ± 35.11
8×	490 ± 52.11

Figure 2. Optical images at 40× magnification of natural NR foams prepared to various foaming volumes, taken before aging.

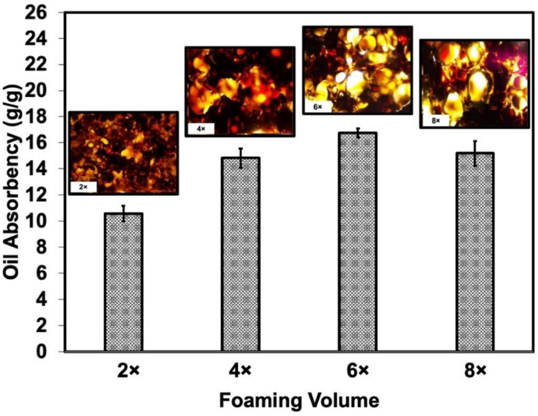


Figure 3. Crude oil absorbency and their optical images after absorption (40× magnification) of NRlatex foams prepared to various foaming volumes.

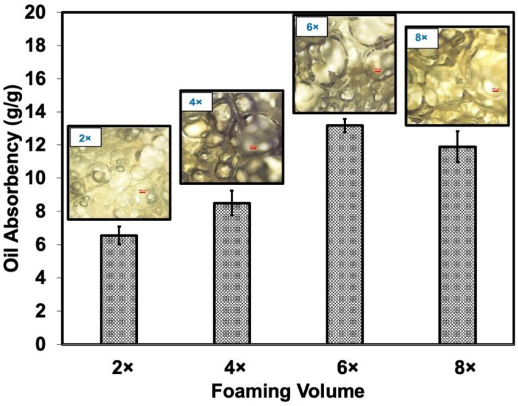


Figure 4. Diesel oil absorbency and their optical images after absorption (40× magnification) of NR latex foams prepared to various foaming volumes.

foaming volume. Previous work by Ratcha et al.[19], who prepared oil-absorbent materials based on NR and NR-g-PMMA, indicates that cellular structure of foam affects its oil absorbency. This is further explained in a study by Lee et al.[20], which stated that the swelling of NR foam is influenced by the cell structure and bulk density, and a lower bulk density was associated with a larger swelling uptake. Figure 4 also shows the expansion of cell size after absorption of crude oil. Clearly, it can be seen that the oil was dispersed throughout the sample (see the dark areas) especially when the foaming volume was high.

Figure 4 shows the absorbency of diesel oil by the various NR latex foams. This is similar to what was found for crude oil. The foaming volume affected the absorbency, and 6× foaming gave the highest oil absorbency of 13.18 g/g, while for 8× case the absorbent decreased. The diesel oil is a specific type of oil derived from crude oil, although it contains hydrocarbons as do other types of petroleum oils[21,22]. Diesel primarily contains straight-chain and branched-chain alkanes, which is not similar to crude oil. Crude oil contains many types of hydrocarbons including alkanes that are saturated hydrocarbons without double bonds, alkenes that have one or more double bonds, and aromatic hydrocarbons with a ring-like structure, such as benzene[23,24]. In general, NR is very compatible with aromatic hydrocarbons, and this may be responsible for the higher oil absorbency observed for crude oil than for diesel.

In addition to this, it can be observed from optical images after immersion in diesel oil, that the cell size was greatly expanded and some were completely swollen (see the embedded images in Figure 4). Figure 5 also shows the physical appearances of NR latex foam cubes before and after immersion in crude oil and diesel oil. Clearly the samples have been expanded by the absorption of oil.

3.3 Material comparison, reusability and simulation

Based on the previous results, it can be concluded that the NR latex foams prepared to various foaming volumes provided remarkable oil absorbencies. The NR latex foam prepared to 6× foaming was selected for further experiments, comparing it with two commercially available products namely PUF and nonwoven PP. Figure 6 shows the oil absorbencies of NR latex foam, PUF, and non-woven PP. It was observed that NR latex foam presents significantly higher absorbency than PUF or non-woven PP, in this rank order. The compatibility with oil of the solid material is the main factor affecting the oil absorbency. NR latex foam may be more compatible with the oil than PUF or PP. Another possible reason is the physical form of each material. For example, the non-woven PP is fibrous and may be less permeable to oil, while the cellular materials NR latex foam and PUF had cell size and cell characteristics favorable to oil absorption. Furthermore, PUF and nonwoven PP might be efficient in oil spill remediation, but they are based on synthetic materials. Consequently there are concerns about residues and contamination from the use of such materials. Figure 7 shows optical images of PUF and nonwoven PP. The average cell size of PUF measured from Image J software is 304.62 ± 79.24 μm while the average fibrous diameters of Nonwoven PP is 19.42 ± 4.48 μm. It was also observed that there was less change in the size and diameters of both PUF and nonwoven PP after oil absorption. These indicate that after oil absorption, no expansion has occurred, suggesting less strong interactions with the oil than in NR latex foam. The nonwoven PP showed only slight amounts of oil that were absorbed, apparently due to its comparatively low porosity (pore volume in proportion to bulk volume).

Reusability was also tested in this study, and the results are shown in Figure 8. The values of absorbency from the first to fifth absorption cycles of NR latex foam were found to be 16.76, 6.38, 5.49, 5.1, and 4.75 g/g for crude oil, while

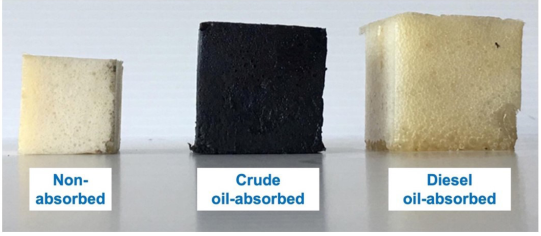


Figure 5. Physical appearances of sample cubes before and after absorbing oil.

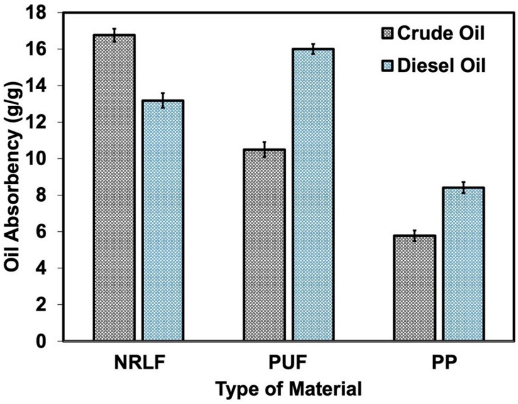


Figure 6. Comparison of oil absorbencies of NR latex foam, polyurethane foam (PUF), and nonwoven polypropylene (PP), with both crude and diesel oils.

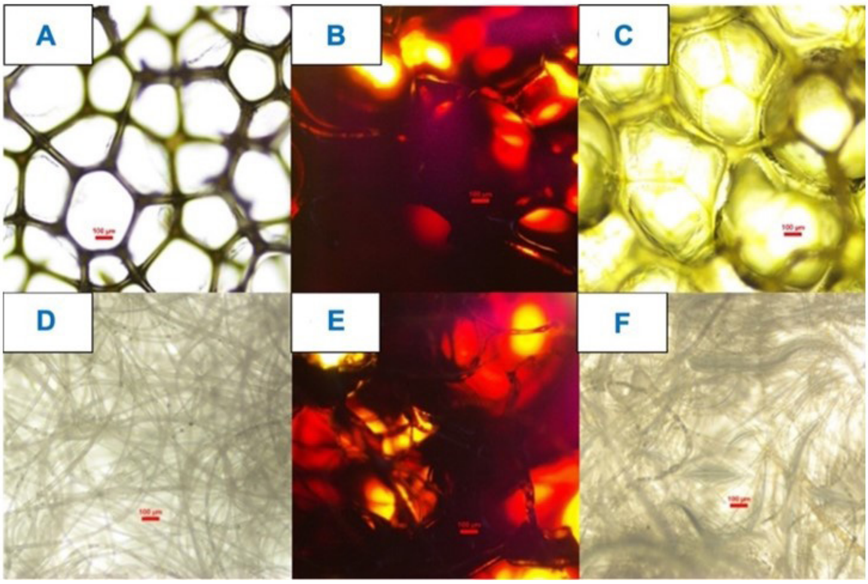


Figure 7. Optical images at 40× magnification of PUF before (A) and after absorption of crude oil (B) and of diesel oil (C); and of PP before (D) and after absorption of crude oil (E) and diesel oil (F).

for diesel oil they were similarly 13.18, 5.02, 4.32, 3.95, and 3.74 g/g. Meanwhile, the values of absorbency from the first to fifth absorption cycles of PUF were found to be 10.51, 8.31, 7.62, 7.25, and 6.90 g/g for crude oil, while for diesel oil they were similarly 16.04, 12.40, 11.32, 11.05, and 10.49 g/g. However, the reusability test for nonwoven PP was unable to be tested due to its limited absorption. Nonwoven PP absorbed only one cycle and cannot be further reused.

It is interesting to note that the oil absorbency of NR latex foam decreased dramatically in the second absorption cycle. As expected, part of the rubber was swollen and destroyed during the first absorption cycle. This decreased the efficiency in the next absorption cycle. However, the rate of decrease in oil absorbency was much less from the second cycle onwards, which is acceptable when considering the level of absorption. While the absorbency initially dropped drastically, the material remains practically functional for several cycles of reuse. The constant level of absorption is believed to be due to the physical absorption of NR foam. Nevertheless, there was a gradual reduction in the oil absorbency of PUF over the absorption cycle. As can be seen from Figure 7, there is less destruction of the cellular structure of PUF after being absorbed by crude and diesel oils. Therefore, the oil efficiency was still presented over the absorption cycles.

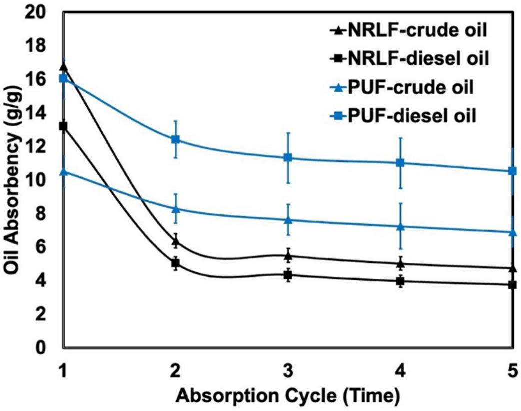


Figure 8. Reusability tested with crude and diesel oils, of NR latex foam when prepared to 6× foaming volume and PUF as absorbents.

To highlight the selectivity for oil over water, Figure 9 shows the oil removal efficiency of NR latex foam in oil-water dispersion. Obviously the hydrophobicity of NR latex foam greatly influenced oil-water separation. The high selectivity for oil is an advantage that also enables the recovery of oil by simply squeezing. This can be done rapidly, and in this laboratory study, the oil was completely removed from seawater within 15 mins. In contrast, PUF had non-selective absorption of both oil and water, as polyurethane is polar by its character, matching water in this aspect.

To check whether NR latex foam really performs better than PUF, a simulation test was done, and an image captured during the test is shown in Figure 10. The NR latex foam has the special characteristic of selectively absorbing oil on the water surface, while PUF has no selectivity by type of liquid. Therefore, the latter collected more water, which reduced the amount of oil absorbed. As a result, the NR latex foam is effective as an absorbent in the removal and collection of oil residues off of the water surface, in case of an oil spill. In summary, NR latex foam is a better-performing oil absorbent than the commercially available products tested.

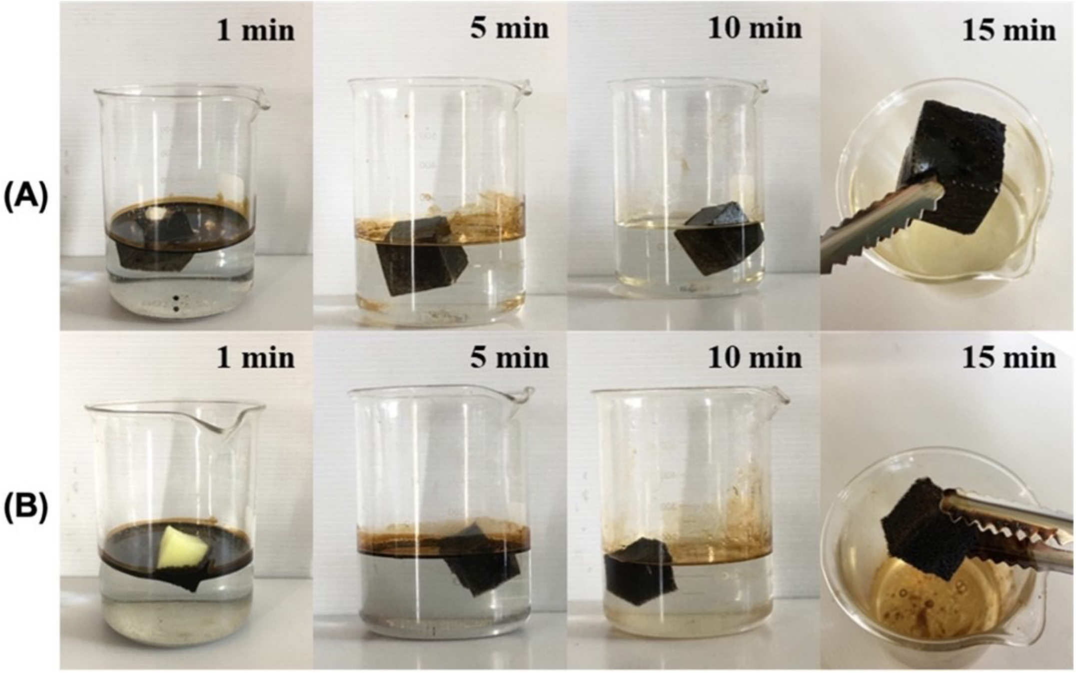


Figure 9. Physical appearances of samples after absorbing oil for 15 min: NR latex foam (A), and PUF (B).

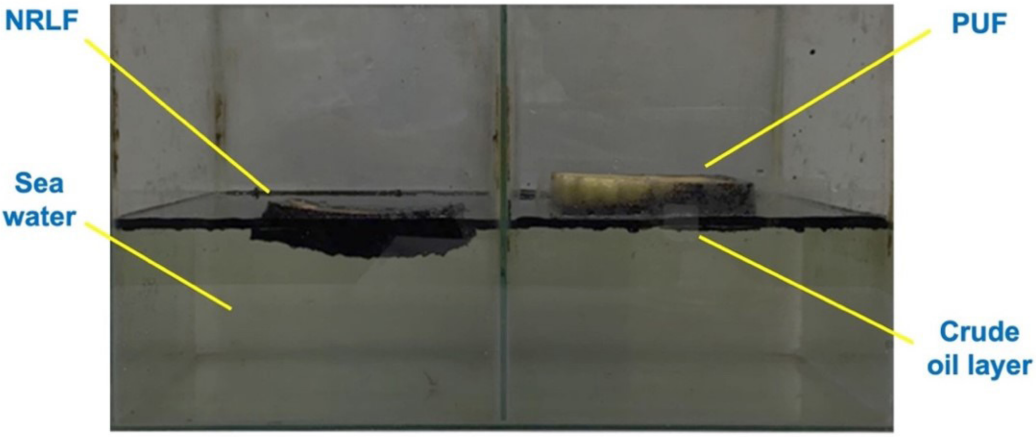


Figure 10. Physical appearances of sample after absorbing crude oil floating on sea water: NR latex foam on left, and PUF on right.

4. Conclusions

Oil-absorbent materials based on NR were successfully prepared in this study, namely NR latex foams made with the Dunlop process. To control the foam density (associated with cell structure) and assess its effects on oil absorbency, the foaming volume was manipulated by adjusting the rotation speed of the beater. It is clear that foaming volume greatly influenced oil absorbency, whether tested with crude or diesel oil. Increasing the speed of the beater initially tended to open the cell structure and provide remarkable improvements in the oil absorbency of the NR latex foam. Oil absorbency of NR latex foam prepared at 6× foaming volume was near optimal. Due to the hydrophobicity of NR, as oil absorbent the NR latex foam was more selective, favoring oil over water, than the other tested absorbents. NR latex foam can be applied to remove spilled oil either from the water surfaces or from a dispersion with water. Cell size and cell structure can be further manipulated via the compounding formulation by the use of foaming agents and other additives. Using a stable and precise beating speed range will give consistent cell characteristics.

5. Author's Contribution

- **Conceptualization** – Nabil Hayeemasae
- **Data curation** – Abdulhakim Masa; Nabil Hayeemasae
- **Formal analysis** – Abdulhakim Masa; Nureeyah Jehsoh
- **Funding acquisition** – Nabil Hayeemasae
- **Investigation** – Abdulhakim Masa; Nureeyah Jehsoh
- **Methodology** – Nabil Hayeemasae
- **Project administration** – Nabil Hayeemasae
- **Resources** – Abdulhakim Masa; Nabil Hayeemasae
- **Software** – NA
- **Supervision** – Nabil Hayeemasae
- **Validation** – Abdulhakim Masa; Nabil Hayeemasae
- **Visualization** – Abdulhakim Masa; Nabil Hayeemasae
- **Writing – original draft** – Abdulhakim Masa; Nabil Hayeemasae
- **Writing – review & editing** – Nabil Hayeemasae

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