Oils and water absorption behavior of Biduri (*Calotropis gigantea*) fibers

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Abstract. This article reports on the oil and water absorption behavior of Biduri (*Calotropis gigantea*) fibres at room temperature. The material measured is a 100% raw material for Biduri (*Calotropis gigantea*) fibre, from 0.3 g to 1.5 g, and nonwoven structures made from 50/50, 60/40, 70/30, 80/20, and 90/10 mixture of Biduri and polyester. FTIR characterized the Biduri fibre, and the viscosity of used oil measured using Brookfield viscometer. The higher oils absorption at weight 0.3 g, 45.53 g/g, and water absorption at weight 0.3 g, 1.47 (g/g). The higher oil absorption at nonwoven structure showed at 90/10 composition, 42.86 g/g. It showed Biduri fibre is a natural absorbent that exhibits excellent hydrophobic and oleophilic properties.

**Keywords:** *Calotropis gigantea*, water absorption, oleophilic, fibers

1. Introduction

The most significant cause of oil pollution in the oceans by humans is waste that comes from the exploration and processing of oil production and oil storage tanks on ships. Due to the ship's engines and tanks cleaning, oily waste illegally spilled from the vessel. Tank cleaning must be submitted to a licensed waste processing service. However, there are still many parties who are not responsible for processing waste illegally. Spilled oil waste is moved by wind and currents and often stays in motion for days and sometimes weeks. The oil on the surface of the water usually spreads out to form a very thin layer. The size of the affected area will increase over time, and it may not be easy to find some or all of the oil. Oil waste is a hazardous and toxic material (B3). If left unchecked and continuously, the sea waters can be polluted, and environmental damage occurs. Waters contaminated by waste oil must be addressed immediately not to produce more severe marine pollution. One way to handle waste oil in the waters is to use porous materials, i.e., absorbent materials, which are readily available, simple to operate, and cheap [1]. Sorption is a physical method of oils removal in which the process of their absorption take place.

Generally, oil-absorbing materials are made from synthetic materials such as polypropylene fibers, polyester fibers, and polyurethane foam. Still, not a few researchers have developed natural materials that are more environmentally friendly and inexpensive [2]. Polypropylene offers a great absorbency capacity relative to cost. It is lightweight and clean to handle. Materials containing cellulose have high absorption and can be used as absorbents, such as absorbing oil or oil spills. Absorbents from cellulose materials have similar capabilities to synthetic polymer (polypropylene) absorbents. Cellulose materials that have been studied include corn stalk [3], durian skin [4], sawdust [5], kapok fiber [6-7], coir [8], and others.
Indonesia is a country with high biodiversity. Animals and plants can live well and have various types because the sun continues to shine throughout the year. Biodiversity is very beneficial for human life as a source of food, medicine, and industry. However, many plants have not been used optimally, including the Biduri (Calotropis gigantea). This plant is a wild shrub with a height of 3-4 meters and is found in dry areas such as grasslands, low mountain slopes, and sandy beaches[9]. Biduri is similar to kapok in that both fibres are found around the plant seeds rather than extracted from the stem or leaves. Biduri fibres are lustrous and made of a mix of lignin and cellulose. Each fibre is about 2.5 cm long, has a wide lumen (central cavity), and thin walls covered with waterproof wax. The Biduri seeds' fiber is the staple fiber, lustrous, has a cavity in the middle like the cotton fiber. The presence of hollow causes the fibrous to be lightweight and have good insulation properties[10]. Biduri fibers have a waxy coating on the fibers' surface, which has great potential to absorb oil[10].

Other research shows that Biduri fibers have excellent hydrophobic-oleophilic properties and the highest absorption capacities between 22.6 and 47.6 g / g [11]. Also, in the other study showed that soaking in an ethanol solution containing PFOTES for 1 second resulted in a high absorption capacity of 47-87 times its weight [12]. Tu reported that Biduri fiber (Calotropis gigantea) was processed into carbon fiber using pyrolysis resulting in oil absorption of up to 130 g / g [13]. The potential of Biduri fiber is enormous, so further research is still needed. The research aimed to remove waste oil from seawater using Biduri fibres with various variations and different treatments.

2. Materials and Methods

2.1. Materials
The study used Biduri fiber was collected from Bandung, West Java. Polyester fiber and polypropylene pad are purchased from a supplier of synthetic fibers. Polypropylene fiber are sample from PT. Hilon Indonesia. Hydrob FC, demineralized water, NaCl, and NaOH were purchased from a local supplier. Waste oil was collected from local workshops.

2.2. Seawater preparation
Artificial sea water was used to imitate the condition of oil spills on seawater with a concentration of 3.5 wt. % was prepared. Seventy grams of NaCl are dissolved in a beaker containing 2000 ml of demineralized water. NaCl solution and demineralized water were filtered to remove suspended matter.

2.3. Treatment of the sorbent
Mercerization of the absorbent is carried out using a NaOH solution. The alkaline mercerization treatment is one of the most frequently used organic absorbent modification methods in the literature due to its simple and effective manner. The fiber was added to the NaOH solution (2%) in a glass flask and shaken at room temperature for 1 hour. After the treatment is complete, the obtained fibers are dried and then oven-dried at 105°C for 3 hours. Biduri fibers were added with Hydrob FC water, and oil proofing agent soaked for 1 hour and dried in an oven at 105°C for 3 hours.

2.4. Methods
Density is the mass of a unit volume of oil, usually expressed as grams per milliliter (g/ml). The density of an oil sample in g/ml, is measured using pycnometer. The Biduri fibers are cleaned and separated from the seeds, then weighed and put into plastic rollers of known dimensions. Prepare a tube-shaped plastic container, enter the artificial seawater then pour the used oil until it reaches a layer of 10 mm. Put the roller filled with fiber into the oil in the container with a specific contact time. Remove and drain on wire mesh for 5 minutes, then weigh. The Biduri nonwoven is obtained from a mixture of Biduri fibers with polyester fibers and Biduri fibers with polypropylene. Nonwoven is made on a hot-press machine with a temperature of 130-140°C for 5 minutes. Nonwoven is cut to a specific size, then weighed and put in a container filled with oil and seawater. Let it sink and remove it after reaching the desired contact time. Drain the Biduri nonwoven for 5 minutes and weigh it.
Absorption capacity calculated using Eq. (1) below:

\[ q = \frac{(m_a - m_i)}{m_i} \]  

(1)

where \( q \) is the absorption capacity (g/g), \( m_a \) is the weight of sample fibers after the absorption and \( m_i \) is the initial weight of sample fibres before absorption.

3. Results and Discussion

The density of most oils ranges from 0.7 to 0.99 g/ml. In this research, density waste oil is 0.877 g/ml, oils typically float on water. The density of sea water is 1.03 g/ml, even heavier oils will usually float on it.

3.1. Properties of fibre

Biduri fiber was characterized using attenuated total reflectance Fourier Transform Infrared (FTIR) summarized in table 1.

| Spectra         | Description                |
|-----------------|----------------------------|
| 3384, 2898, 1377, 1319, 1163, 1052 | Cellulose                  |
| 1734, 1427, 1247 | Hemicellulose and lignin   |

The main characteristic of an oil-absorbing sorbent is the high oil absorption capacity. Besides, the oil must be firmly bound by the sorbent, but it can still be recovered quickly, and the sorbent can be reused. For oil spills in water, the oil sorbent must be hydrophobic and have good buoyancy. Hydrophobic is defined as a low ability to absorb water. The lower the ability of a sorbent to absorb water, the higher its hydrophobicity. In the case of an oil spill in water, there will be competition between water molecules and oil molecules to bind to the sorbent. Hydrophobic sorbents tend to bind to oil molecules and repel water molecules, while the good buoyancy properties make it easier to separate sorbents already bound with oil from water. The structure of Biduri fibers is composed of cellulose.

The absorption of water by the lignocellulosic material depends on the number of free -OH groups present in the fiber. The reduction in water absorption capacity is thought to occur due to a decrease in the free -OH group on the lignocellulose, thereby reducing hydrogen bonds with water molecules. In this process, it is estimated that the compound that plays a role in cellulose because lignin is naturally hydrophobic.

3.2. Effect of sorbent weight

The effect of absorbent weight on oil absorption was carried out in a weight range of 0.3 to 1.5 g after 10 minutes of contact time with 10 mm oil film thickness using waste oil and hanging for 5 minutes, as shown in Fig. 1. Based on Figure 1, it can be seen that the addition of sorbent weight from 0.3 to 1.5 g decreased the absorption capacity of Biduri fiber from 45.53 g/g to 18.22 g/g and decreased the absorption efficiency from 97.89% to 94.75%. The addition of fiber weight at a constant volume will increase the fibers' density so that the space between the fibers gets smaller, and oil does not enter the fibers.
3.3. Effect of Pre-treatment
The effect of the comparison of treatment results on oil absorption is presented in Fig. 3. Absorption of Biduri fibers without treatment resulted in the absorption of 24.91 g/g, while the absorption power of Biduri fibers from 2% NaOH mercerization decreased to 17.75 g/g. The decrease can occur because lignin has been reduced in the Biduri fiber. After all, it dissolves with NaOH. Meanwhile, the literature states that lignin is a supporter of oil absorbents.
In the pre-treatment, using the Hydrob FC waterproof agent resulted in lower absorption. The waterproof agent envelops the fiber and enters the cavity in the middle of the fiber so that oil cannot stick to the fiber's surface. However, this needs to be proven by morphological tests using SEM. In this study, the morphological test has not been carried out.

![Figure 3](image_url)  
**Figure 3.** Oil absorption capacity at different pre-treatment

### 3.4. Effect of contact time

The contact time effect was made with five different time variations: 5, 10, 30, 60, and 90 minutes. The absorption power of Biduri can be seen in Fig. 4. At the contact time of 5 minutes, the absorption capacity was 17.17 g/g. The absorption capacity increased at the contact time of 10 minutes and reached a peak of absorption power at the contact time of 30 minutes, namely 24.91 g/g. After 60 minutes and 90 minutes, there was no increase in absorption and even more stable because the fiber has started to saturate not to absorb oil anymore.

![Figure 4](image_url)  
**Figure 4.** Oil absorption capacity at different contact times

### 3.5. Biduri nonwoven

For efficiently handling, Biduri fibers are made into a nonwoven as in commercial products. The Biduri nonwoven consists of Biduri fibers and polyester fibers, with the composition of Biduri fibers being 50, 60, 70, 80, and 90%. The absorption capacity and efficiency of various Biduri fibers and polyester fibers nonwoven are shown in Figure 5.
Figure 5. Oil absorption capacity and efficiency at different composition

The absorption capacity of the Biduri nonwoven to oil at the composition of 50% was 36.76 g / g. The composition's addition to 60% resulted in 36.90 g / g absorption, and 70% composition resulted in 37.03 g / g absorption. The increase in absorption capacity from 50% to 70% has only a small difference. At the 80% composition, the absorption capacity increased to 39.52 g / g, and at the 90% composition, the oil absorption rate was 42.86 g / g, which was the highest oil absorption in this study. At 80% composition, the absorption rate increased to 39.52 g / g, and at 90% composition, the oil absorption rate was 42.86 g / g. The highest oil absorption occurred in the 90% composition of the Biduri nonwoven because the arrangement of cellulose fibers, Biduri, was more significant than polyester fibers. The Biduri fiber, which has a cavity in the middle, is a place that can be used as a trap.

In this research, Biduri and polypropylene nonwoven were also made with a composition ratio of 50:50. The absorption capacity of Biduri-polypropylene nonwoven was 29.27 g / g, and absorption efficiency was 96.70%. Compared to the absorption capacity of 50% Biduri-polyester nonwoven, the absorption capacity of Biduri-polypropylene nonwoven is lower so that the variations in other compositions do not follow suit. It is assumed that the overall absorption capacity will be lower than that of Biduri-polyester nonwoven.

3.6. Comparison with commercial products.
The commercial product commonly used is polypropylene fiber compared to a Biduri membrane with a composition of 70%, as in Table 2.

Table 2. Comparison Biduri nonwoven and Polypropylene Pad (Commercial Product)

| Product          | Gramasi (g/m²) | Capacity (g/g) |
|------------------|----------------|----------------|
| Biduri Nonwoven  | 179.34         | 18.79          |
| Polypropylene Pad| 178.39         | 12.40          |

Biduri-polyester nonwoven is manufactured with every effort to approach the gramation of the polypropylene absorbent material. The absorption capacity of the Biduri-polyester nonwoven was 18.70 g/g and higher than that of polypropylene pad, which only reached 12.4 g / g. This is very exciting and wonderful because the Biduri fiber can outperform commercial materials.
4. Conclusion
This study concludes that Biduri fibers have a better absorption performance than commercial polypropylene materials. Additional treatment NaOH and Hydrofob agent does not help the absorption of oil. The best composition for the manufacture of Biduri membranes with the best absorption is 90% Biduri fibers and 10% polyester fibers. This results show a wider applications of Biduri fibers.

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