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The Potential of Activated Carbon from Durian (*Durio Zibenthius*) Peel Waste As An Adsorbent Of Oil Spill

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Abstract

Oil exploration activities, such as ship loading and unloading, can lead to oil spills at sea, as can oil spills and tanker collisions. An affordable method for dealing with these spills is adsorption using activated carbon derived from bio-waste, such as durian peel. This study aims to evaluate the adsorption efficiency of durian peel-activated carbon on oil spills. The carbonization of durian peel was carried out at 300 °C and activated with KOH at 5, 20, 35, and 50% (w/v). Durian peel activated carbon with 35% KOH showed the best results for moisture content (5.442%), ash content (4.457%), bound carbon content (72.050%), and iodine adsorption (896.033 mg/g), according to SNI 06-3730-1995. FTIR measurements revealed that KOH activation did not induce significant changes in the wavenumbers of functional groups, but did increase the pore diameter, as confirmed by SEM. Its adsorption efficiency with a batch system on crude oil in simulated and real seawater reached 90.182% and 90.091%, respectively. These results suggest that activated carbon from durian peel has excellent potential as an oil adsorbent. This would increase its utility value and reduce the accumulation of biomass waste.

Keywords: activated carbon, adsorbent, durian peel, oil spill, seawater

Introduction

Based on data from the Ministry of Marine Affairs and Fisheries of Indonesia, there have been cases of oil spills due to pipeline leaks that occurred in Balikpapan in 2018 (Setiawan & Jatmiko, 2018). Other cases occurred in 2020 at Pulau Seribu and in 2022 at Cilacap, resulting in marine pollution and a pungent odor that disrupted the activities of the surrounding community (Ubaidillah & Ramadhan, 2020; Zain & Belarminus, 2022). In other countries, cases of marine pollution due to oil spills have also occurred in Thailand and the Peruvian region, resulting in enormous losses to nature and wildlife and being considered the most significant ecological disaster ever experienced (Buakamsri, 2022). Oil spills not only directly harm marine ecosystems and organisms but also threaten the lives of coastal communities that rely heavily on fish resources and marine services for their livelihoods.

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Currently, several techniques are available for addressing oil spills at sea, including physical, chemical, natural, and biological methods, which are selected based on the composition of the crude oil, weather conditions, the amount spilled, the sensitivity of the location, and the toxicity of the chemicals. The limitations of various oil spill handling techniques, such as bioremediation, phytoremediation, and natural attenuation, include high costs, variability in oil composition, and the limited availability of suitable microorganisms or plants (Sharma et al., 2024). One low-cost method that is still under development is the use of adsorbents. Among the various types of adsorbents, including synthetic adsorbents and inorganic materials, activated carbon is the most environmentally friendly adsorbent derived from natural fibers (Noviasari et al., 2017; Suhas et al., 2016).

Several types of natural biomass have been used to make oil-adsorbing activated carbon, including corn husks and palm fronds. However, their iodine adsorption capacity does not yet comply with Indonesian National Standard (SNI) 06-3730-1995, which requires a minimum capacity of 750 mg/g (Rios et al., 2024; Yuwita et al., 2024). Another material that can be used as a source of activated carbon is carbonized durian peel waste, with carbon-rich content as much as 80.6% (Tambun et al., 2024). By 2022, durian peel waste in Indonesia is expected to reach 1.2 tons, which is not being optimally utilized (Anggraini et al., 2024). Therefore, processing durian peel can increase the economic value of waste.

Previous research has shown that chemical activation using a 1:0.5 ratio of potassium hydroxide (KOH) produces high-quality activated carbon of durian peel due to its lower activation temperature, higher yield, and well-defined microporous structure (Chandra et al., 2009). KOH activation exfoliates and layers the carbon surface, creating numerous micro and macro pores, which significantly enhance the surface area, making it ideal for applications like adsorption, energy storage, and catalysis (Amelia et al., 2024; Lestari & Nasra, 2022).

In previous research, activated carbon derived from durian peel has been utilized as an adsorbent for metals, such as iron (Febriansyah et al., 2015) and lead(Lestari & Nasra, 2022), dye adsorbent of methylene blue (Hanum et al., 2017) and organic compounds such as bisphenol (Lazim et al., 2015) and tofu waste (Ridhayanti & Rusmini, 2020). In the previous study, KOH was used in a 1:0.5 ratio, whereas in this study, it was used at a lower concentration.

The quality of activated carbon is assessed according to SNI 06-3730-1995 standards, including moisture content, ash content, bound carbon content, and iodine adsorption capacity. Its adsorption efficiency is then tested under various conditions, including different contact times, adsorbent masses, and adsorbate concentrations. The durian peel-derived activated carbon is also characterized using FTIR spectroscopy and SEM to analyze its functional groups and surface morphology, offering insights into its potential applications.

Materials and Methods

The research utilized various tools, including an oven (Memmert), a furnace, glassware, a pH indicator, and a porcelain cup. Materials included durian peel waste (*Durio zibenthinus)*, crude oil spill samples from the waters off Lampung Beach and chemicals such as potassium hydroxide (Merck), sodium chloride (Merck), potassium iodide (Merck), iodine, sodium thiosulfate (Merck), potassium dichromate (Merck), 1% amylum indicator, concentrated hydrochloric acid (Merck), and distilled water.

Dried, carbonized durian peels, furnace-treated at 300°C for 3 hours, were mashed in a mortar. The activation process was carried out by soaking the carbon in 5%, 20%, 35%, and 50% KOH solutions (w/v) for 24 hours, then filtering and neutralizing it with hot distilled water. The activated carbon was then dried using an oven at 110°C (Irnameria, 2020).

The quality of activated carbon was evaluated by analyzing moisture, ash, volatile matter, bound carbon content, and iodine adsorption. For iodine adsorption analysis, 50 mL of 0.1 N iodine solution was added to 0.5 g of activated carbon. The solution was filtered, then 10 mL of the filtrate was titrated with 0.1 N sodium thiosulfate, followed by the addition of a few drops of 1% starch indicator, until the blue color disappeared (Ridhayanti & Rusmini, 2020). Equation 1 is used to calculate the iodine adsorption capacity, where V, N, fp, and *a* represent volume (mL), concentration (N), dilution factor,

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and carbon active mass (g), respectively. The quality of the synthesized activated carbon was then compared with the SNI 06-3739-1995 standards to assess its performance and suitability.

$$Iodine\ adsorption\ \left({}^{mL}/_{g}\right) = \frac{V_{I_2} - \frac{V_{Na_2S_2O_3}\ x\ N_{Na_2S_2O_3}}{N_{I_2}}\ x\ 126{,}9\ x\ fp}{a} \quad (1)$$

To understand its physical and chemical properties, small samples of the activated carbon were analyzed using a Scanning Electron Microscope (Phenom Pro-X) and a Fourier Transform Infrared (FTIR) spectrometer (Bruker Alpha). FTIR testing was conducted by creating a homogeneous pellet from an activated carbon sample. KBr at a ratio of 1:100 and then measuring the absorption at 4000–400 cm^{-1}. For SEM testing, the sample holder was inserted into a gold plating machine and placed under an electron microscope with 2000x magnification.

The optimum conditions of activated carbon adsorbent were tested on the variation of adsorbate mass (5; 7.5; 10; 12.5; and 15 g), contact time (60, 90, and 120 minutes), and adsorbent mass (0.5; 1; 2; 3 and 4 g) in 3.5% (w/v) NaCl media, using analytical balance (Ohaus). The optimization results are used as a reference condition when applying oil adsorption to seawater. A total of 4 g of adsorbent was weighed and then added to 100 mL of seawater and 5 g of crude oil. After 30 minutes, the adsorbent was filtered, and its adsorption efficiency was calculated using equation 2:

$$adsorption\ efficiency\ (\%) = \frac{(w_o - w_e)}{w_o}\ x\ 100 \quad (2)$$

where w_o is the initial mass of adsorbate (g) and w_e Is the final mass of adsorbate (g).

Results and Discussion

Quality of Durian Peel Activated Carbon

In this study, a relatively low carbonization temperature of 300 °C was used to produce activated carbon from durian peel. Carbonization temperatures above 700 °C and high concentrations of potent chemical activators reduce the specific surface area of activated carbon, eliminating heteroatoms and thereby decreasing the number of functional groups. For improved adsorption capacity, soft materials should be carbonized at temperatures below 600 °C to preserve functional groups and maintain a higher surface area (Karume et al., 2023).

The quality of activated carbon, according to SNI-06-3730-1995, is assessed through tests on water content, ash content, activated carbon content, and iodine adsorption. This study compares the quality of unactivated carbon (NA) with that of potassium hydroxide-activated carbon to assess the impact of activation. As an activator, KOH reacts with carbon, filling it and forming pores that increase the activated carbon's surface area (Azizah & Rachman, 2023). During the activation, there will be an intercalation process of potassium metal ions into the carbon network, with the following reaction (3-6) (Tambun et al., 2024):

$$4KOH + C \rightarrow K_2CO_3 + K_2O + 2H_2 \quad (3)$$
$$K_2CO_3 + 2C \rightarrow 2K + 3CO \quad (4)$$
$$K_2CO_3 \rightarrow K_2O + CO2 \quad (5)$$
$$K_2O + C \rightarrow 2K + CO \quad (6)$$

The results of the quality analysis of durian peel-activated carbon are summarized in **Table 1**. All moisture content of KOH-activated carbon met the SNI 06-3730-1995 standards, while that of NA exceeded. Using a 50% KOH solution produces activated carbon with the lowest moisture content. KOH, as a hygroscopic activator, can bind water molecules to activated carbon, thereby increasing its pore size and surface area. Lower moisture content improves the quality of activated carbon, enhancing

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its ability to absorb gases or liquids, making it a more efficient adsorbent for various applications (Legiso et al., 2020). Additionally, the ash content decreased to 4.55% as the KOH concentration increased.

Table 1. Durian peel activated carbon quality based on SNI 06-3730-1995

Parameters	NA	Activated with KOH (AK)				Reference*
		5%	20%	35%	50%	
Water content (%)	15.75	14.07	9.95	5.44	2.30	≤ 15%
Ash content (%)	13.87	8.77	6.03	4.55	5.34	≤10%
Carbon-bound content (%)	65.11	66.20	65.82	72.05	71.81	≥ 65%
Volatile content (%)	21.02	25.03	28.15	23.40	22.85	≤ 25%
Iodine Adsorption (mg/g)	532.99	748.37	775.87	896.03	855.89	≥ 750 mg/g

Note: NA= non-activated, AK= activated with KOH
*SNI 06-3730-1995

The bound carbon content is influenced by ash content, volatile substances, the cellulose and lignin in organic materials, which can be converted into carbon. These factors are also affected by the activator used during the activation process, ultimately determining the quality and effectiveness of the activated carbon produced (Lestari & Nasra, 2022). The optimal bound carbon content is achieved with a 35% KOH concentration, corresponding to 72.05%. The decrease in bound carbon content at the highest KOH concentration (50%) is a result of pore damage on the surface of the carbon (Maulina & Iriansyah, 2018).

Volatile substance levels are organic components contained in activated carbon and have not entirely evaporated during the carbonization process (Li et al., 2019). Based on the research conducted, only KOH-activated carbon at 35% and 50% concentrations meets SNI 06-3730-1995.

The iodine adsorption capacity was chosen as one of the determinants of activated carbon quality because it can show the adsorption capacity of activated carbon through the overall adsorption ability, and illustrates whether or not the activation process is successful in increasing the surface area (Lubis et al., 2020). In this study, the base activation of KOH increases iodine adsorption to a maximum concentration of 896.03 mg/g. As explained by Erlina et al (2015), a higher concentration of KOH (≥ 50%) resulted in decreased adsorption capacity due to its saturated state.

Characteristics of Durian Peel Activated Carbon

Functional Group Analysis Results

Activated carbon typically exhibits absorption patterns with O-H bond types (3500-3200 cm^{-1}), C=O (1820-1600 cm^{-1}), and C-O (1200-900 cm^{-1}) bonds. The activation process on activated carbon will form C=C bonds that show an increase in carbon content at 1500-1400 cm^{-1} and C-H groups from alkane compounds at 3000-2850 cm^{-1} (Suhas , et al., 2016).

The FTIR spectrum of durian peel activated carbon shows several absorption bands at wave numbers of 3417, 1618, and 1027 cm^{-1}, which align with previous study results (Hayu et al, 2021). The O-H functional group on KOH-activated carbon experienced a shift in wavenumber compared to that without activation, from 3439 cm^{-1} to 3457 cm^{-1}. This is due to lignin breakdown by OH^- ions from the KOH activator. Activation of activated carbon with 35% KOH did not significantly alter the spectrum, but it did increase the absorption intensity, as shown in **Figure 1**.

The functional groups, like C-O, C=O, C-H, and O-H bonds, come from cellulose, hemicellulose, and lignin compounds contained in durian peel-activated carbon that has not been fully carbonized (Suhas et al., 2016). A detailed comparison of the wave numbers of the functional groups is provided in **Table 2**.

The oil adsorption process by durian peel-activated carbon showed an increase in absorption intensity, with the emergence of new peaks at 2924 and 1458 cm^{-1}. Both absorption bands correspond to the wave numbers of alkane C-H groups (sp^3) and aromatic groups (Suhas et al., 2016). Aromatic functional groups, or so-called unsaturated hydrocarbons with a ring-like shape, are often found in

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crude oil. This allows the emergence of new absorption bands in aromatic groups on activated carbon used as an adsorbent for oil spills.

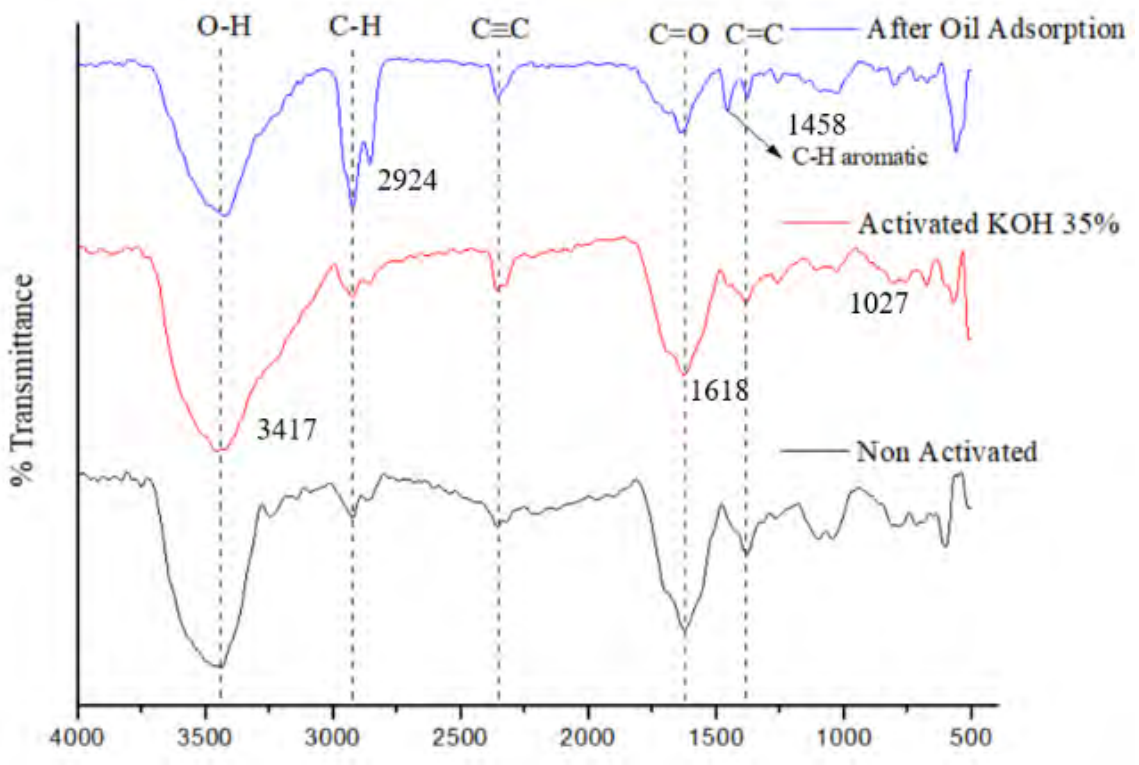


Figure 1. FTIR spectrum of durian peel activated carbon

Table 2. Comparison of functional groups of durian peel activated carbon

Functional group	Wavenumber (cm^{-1})			
	NA	AK35	After adsorption	Reference (Parmita et al, 2020)
O-H	3439	3457	3427	3100-3488
C-H asymmetric	2926	2924	2924	2920-2990
C≡C	2357	2359	2356	2361
C=O	1626	1627	1639	1739
C-O	1046	1032	1029	1054
C=C	1385	1385	1379	1430-1625
C-H aromatic	-	-	1458	

Surface Morphology Analysis Results

There are differences in morphology on the surface of AC before and after activation. The size and number of pores on the sample after KOH activation (2b) are larger than before the activation (2a). Activation treatment with KOH results in the widening of existing micropores as well as the formation of new micropores, which are marked in red lines in **Figure 2b**. This will increase surface area and pore volume. KOH solution, which is a strong base, will form pores on the carbon surface by removing hydrocarbon compounds (tar) or other impurities (Karume et al., 2023).

Figure 2c shows the changes in the external surface of the AC that has adsorbed the oil. The porosity of the AC is significantly reduced, with a denser and wavier surface, as in the study by Rofikoh (2024). This indicates the efficient use of the porous adsorbent's active sites during oil adsorption.

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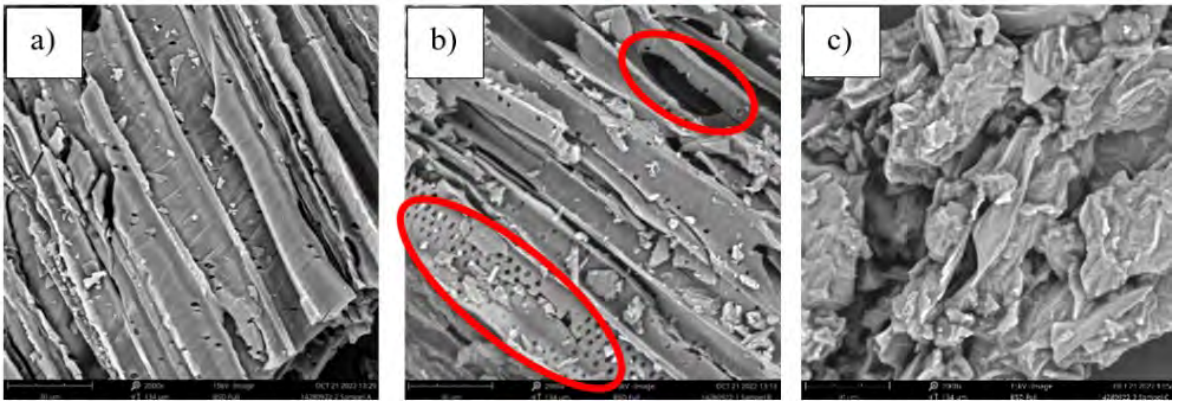


Figure 2. SEM analysis results of activated carbon (a) before activation (NA), (b) after 35% KOH activation (AK35), and (c) after adsorption (AA)

Optimum Conditions for Oil Adsorption

The adsorption capacity of 35% KOH-activated carbon was tested over variations in adsorbate mass, contact time, and adsorbent mass. Activated carbon can adsorb organic compounds due to its active groups, including oxides. The interaction that occurs between activated carbon as an adsorbent and adsorbate is very complex because there are various functional groups in activated carbon, such as hydroxyl groups, carboxyl groups, and oxide groups (Ferdian et al., 2022).

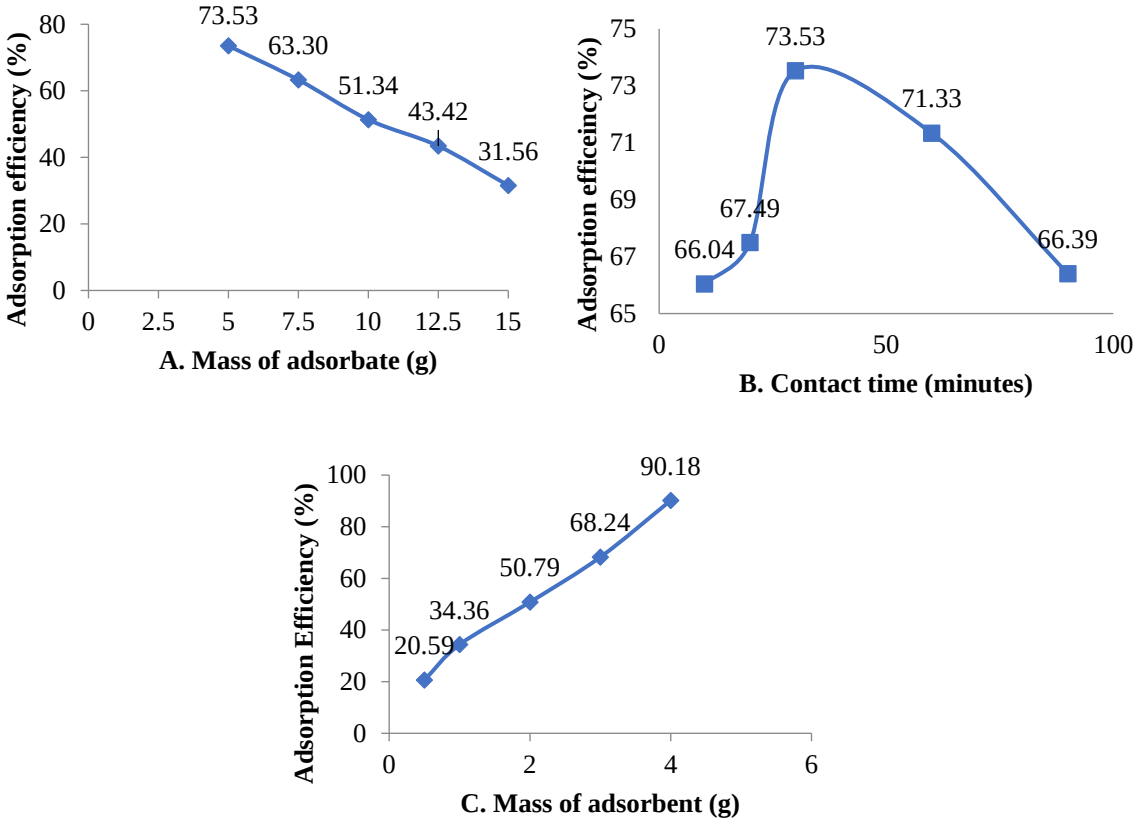


Figure 3. Oil adsorption efficiency by durian peel activated carbon at various variations: (a) mass of adsorbate, (b) contact time, (c) mass of adsorbent

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The variation of crude oil adsorbate mass aims to determine the adsorption efficiency of varying adsorbate mass with a fixed adsorbent mass. **Figure 3a** illustrates that the adsorption efficiency decreases as the mass of adsorbate in solution increases. An adsorbate mass of 5 g can be adsorbed quickly on the surface of activated carbon up to 73.5%, while larger masses cannot be absorbed optimally because the active sites of the adsorbent are already at the saturation point (Sastrawidana, 2022).

Adsorption of durian peel-activated carbon was also carried out over contact times ranging from 10 to 90 minutes. Based on the test results in **Figure 3b**, the adsorption process from minutes 10 to 30 increased to 73.3%. According to Abel et al (2020), the increase in adsorption occurs because there are still many vacant active sites on the adsorbent surface, as well as attractive forces between the oil molecules and the adsorbent. However, the contact time variation curve does not yet indicate an equilibrium state. After 30 minutes, the adsorption efficiency begins to decrease because an excessively long contact time between the adsorbent and adsorbate can cause the adsorbent to become saturated, leading to the release of the adsorbate that has become entangled (Panda et al., 2017). A similar situation was observed in previous research by Sugiharto (2020), where a decrease in the % of dye adsorption was observed due to a reduction in the available surface area, which was covered by a layer of dye on the adsorbent surface.

The adsorption efficiency continues to increase with increasing activated carbon mass, as observed in **Figure 3c**. Increasing the mass of the adsorbent causes an increase in the surface area of the adsorbent, allowing higher contact between adsorbent particles and adsorbate molecules (Nsi et al., 2017). The greater the amount of adsorbent used, the more active its surface is. At a contact time of 30 minutes, 4 g of adsorbent produced the highest adsorption efficiency of 90.2%.

Optimum conditions for the adsorption of durian peel-activated carbon in seawater were used to demonstrate that the adsorbent can be applied under actual environmental conditions. Test results for samples using real seawater, with 4 g of activated carbon and 5 g of crude oil as adsorbate, showed an adsorption efficiency of 90.091%. These results demonstrate the utility of durian peel as an activated carbon, showing that biomass waste can be repurposed as an adsorbent for oil spills.

Conclusion

The KOH-activated carbon produced with 35% and 50% concentrations has met the SNI 06-3730-1995 standards. The 35% KOH-activated carbon exhibited the best quality, with a moisture content of 5.44%, an ash content of 4.45%, a bound carbon content of 72.05%, and an iodine adsorption capacity of 896.03 mg/g. The activation process enhanced the carbon pore diameter, improving its adsorption properties without significantly altering its structure. Oil spills can be adsorbed by durian peel-activated carbon adsorbents, as evidenced by the appearance of a new absorption band for aromatic groups at 1458 cm^{-1}. The optimal adsorption condition occurred at a contact time of 30 minutes with an adsorbent mass of 4 g, yielding an adsorption efficiency of 90.182%.

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