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Purification of Oil-Polluted Sea Water using Carbonized Rice Husk Adsorbent Prepared in a Closed Reactor

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Abstract
The study involves purification of oil polluted sea water using carbonized rice husk (CRH) as adsorbent. Carbonization was carried out in a closed reactor. Important properties of the rice husk (RH) and carbonized rice husk (CRH) were determined using standard methods. Six sets of experiments were carried out with initial oil concentration of 1.35 mg/L and CRH dosages of 0, 6.67, 13.33, 20, 26.67, 33.33 g/L, separately to determine the optimal adsorption dosage and percentage of oil removal. Secondly, five sets of experiments were carried out under the adsorption time of 1 h, 2 h, 3 h, 4 h, and 5 h, with the optimal dosage for adsorption determined from the result of the first experiment to determine the optimal adsorption time. Lastly, another five sets of experiments were carried out under the adsorption time of 0 h, 1 h, 3 h, 5 h, and 7 h with the same optimal dosage for adsorption determined from the result of the first experiment to determine the COD removal. The surface area, bulk density, and thermal conductivity of the RH and CRH were respectively 0.78 and 97.4 m^2/g, 0.1 and 0.19 g/cm^3, and 0.04 and 0.035 W/(m. K). 13.33 g/L was determined as the optimal dosage for adsorption in this study. The oil removed with the optimal dosage was 71.85% at 3 h optimal time, while the total oil removed was 85.01%. The COD removed at this optimal time was 34% while the highest COD removal was 48%. The CRH showed ideal effects in the purification of oil-polluted sea water with excellent adsorption ability for oil in the oil polluted sea water.
Keywords: Oil-polluted seawater, adsorption, carbonized rice husk

1. Introduction

Oil production from fossil fuel, transportation, and utilization has remained amongst the top most viable activities in the world for many years and will continue to remain amongst the top most viable activity in the world until alternative energy (renewable energies are fully developed). Therefore, oil industry has developed so fast over the years that it has become a vital part of the world economy. However, this has made oil pollution amongst the major components of ocean pollution (Junqing and Xiangmao, 2001). Oil pollutants in Nigerian sea waters in particular has been a great challenge to oil industry, government, public health and ecosystems. About 0.6% of annual oil productivity (oil and petroleum products) around the worlds spill into the ocean through different channels. Drilling spills, natural spills at the undersea bottom, leakage and blowouts during exploitation, as well as the frequent occurrence of tanker accidents are among the different channels that lead to marine oil pollution which has become increasingly serious in the recent years and demand critical attention for adequate solution (Hui and Liping, 2007).

Nigeria with vast deposit of coal, crude oil, and natural gas is among the fastest growing economy. However, oil pollution via the different channels is amongst the major problems facing the people, the oil industry in particular and the government in general. The problem of oil pollution is even more serious due to lack of productive conditions, adequate technology, and low public awareness of environmental protection. Junqing and Ming (1999) emphasizes that oil pollution reduces the use value of coastline areas, damages coastal infrastructure, affects the growth of marine life, and even destructs the self-purification ability of the ocean, thereby destroying the marine ecosystem.

Therefore, there is an urgent need to efficiently control and manage marine oil pollution. Some of the general methods used for treatment of oil polluted sea water are physical methods (including fence treatment, skimmers and

oil adsorption materials such as straw, sawdust, etc); chemical methods (including dispersing agent, oil condensation agent and other chemical products); new treatment methods (such as bioremediation technology and combustion method, etc). These treatment methods have one disadvantage or the other. For instance, the reagents from chemical methods are potential sources of secondary pollution. Bacteria employed in bio-remediation technology require carrier, and it is impractical to keep better effect when used in a large area. Adsorption felt can only remove the oil film on the sea surface, while the removal effects of dissolved and emulsion oil are not so ideal, and the material cost is higher (Cui *et al*. 2014). Though adsorption has been proved to be effective in removing oil pollutant from sea water, the material cost of most adsorbent that have been used is high. Cost of adsorbent can be lowered when prepared from low cost and readily available materials such as rice husk (RH) waste.

Rice husks waste are the major waste products from rice milling process. The world annual paddy production according to the statistical data of Food and Agriculture Organization (FAO) is approximately 582 million tons. Rice husk comprise 22–25% of the rice grain and, therefore about 145 million tons of rice husks residue are produced (Ioannidou and Zabaniotou, 2007). Management and utilization of rice husk, from rice mills, as agricultural wastes are among the greatest challenges facing the government and people in developing country such as ours due to the enormous quantity in which they are produced (Hieu *et al*. 2015). One of the ways in which this rice husk waste can be converted into useful product (wealth) is by carbonization of the rice husk (Obodoeze *et a*l. 2018). The carbonization or pyrolysis of RH at specific temperatures creates a highly porous carbon material with a large specific surface area. Ventikosnp (2002) opined that CRH is a microporous substance whose adsorption ability depends mainly on its physical and chemical structure on the surface having a unique adsorption structure with a large number of unsaturated carbons on its surface. It has high specific surface area containing lots of irregular structure or surface functional structure. This produces a strong molecular field due to the relative function of micro-porous and porous molecular wall, thereby, providing a high-pressure system to physical and chemical changes of molecular adsorption. In addition, the surface of CRH is covered with numerous pores with ease of adsorption and desorption, making it an excellent adsorbent (Yupeng *et al*. 2000).

Carbonized rice husk has been found to be very effective in oil polluted sea water treatment. Carbonized rice husk (CRH) remove oil from oil-polluted sea water primarily through an adsorption process, where oil molecules adhere to the surface of the rice husk. This process is aided by high silicon content in the rice husk which forms a unique spatial grid structure with honeycomb holes, providing numerous sites for oil adsorption. Rice husk when carbonized, becomes hydrophobic (repels water) and oleophilic (attracts oil). These properties enable CRH to selectively adsorb oil rather than the surrounding seawater, allowing it to float on the water surface, absorbing the oil, which can then be skimmed off easily (Zhao *et al*., 2013). Junqing and Ming (1999) used acid-treated rice hull as raw material to investigate the activation process of rice hull adsorbent, which presents evident adsorption effects to polycyclic aromatic hydrocarbons (PAHs) and heterocyclic dyes in water. Also, Malik (2003) confirms optimal adsorption conditions when rice hull adsorbent was used to treat acid dye wastewater and reported adsorption isotherms which met the Languir and Freundlich equations. All this point to the fact that rice husk can serve as an excellent adsorbent. Studies on oil-polluted sea water purification using carbonized rice husk are scarce; however, Zhao *et al*. (2013) achieved oil removal efficiency of 65.3% when oil-polluted water was treated with rice hull adsorbent carbonized under the temperature of 350°C with dosage of 12g. Cui *et al*. (2014) used carbonized RH prepared by open firing (carbonization) of RH waste to purify oil-polluted sea water and reported oil and COD removal of 71% and 35.5% respectively.

Most treatment methods that have been utilized in treatment of oil polluted sea water have one disadvantage or the other. For instance, the reagents from chemical methods are potential sources of secondary pollution. Bacteria employed in bio-remediation technology require carrier, and it is impractical to keep better effect when used in a large area. Adsorption felt can only remove the oil film on the sea surface, while the removal effects of dissolved and emulsion oil are not so ideal, and the material cost is higher (Cui *et al*. 2014). Cost of adsorbent can be lowered when synthesized from clay; however, synthesizing adsorbent in large scale using clay for oil-polluted sea water treatment in industrial scale may lead to environmental problem since the clay mined from earth cannot be adequately replaced. Most researches on treatment of oil-polluted sea water used CRH prepared by open air firing leading to CO_2 emission into the atmosphere; causing other environmental pollutions (problems). Thus, this research is centered on purification of oil-polluted sea water using carbonized rice husk (CRH) obtained by carbonization (firing) of RH waste in a closed reactor, preventing CO_2 emission into the environment.

2. Materials and methods

Rice husks used in this study were obtained from one of the rice mills in Omor, Anambra Sate Nigeria. The rice husk was carbonized in a flow Reactor of 5000cm^3 capacity at 600^{o}C. The device Scanning Electron Microscope (SEM)-JED-2300 Analysis Station (JEOL) were used to obtain elemental composition of initial raw material (rice husk) and the products of carbonization. Surface area was determined using Brunaur-Emmett-Teller (BET) method according ASTM D3663-20 (2020). Fourier-transform infrared spectroscopy (FTIR) was used to determine the functional group. The *thermal conductivity* was measured using a TPS2500S *instrument* (Hot Disk thermal constant analyzer) while bulk density was determined according to the method described by Amidon and Mudie (2017).

Six mixtures of oil polluted sea water (750 ml, pH: 6.83) and different quantity of CRH (0, 5, 10, 15, 20, 25 g) were mixed in 1000 ml Erlenmeyer flasks, and allowed to absorb statically for 72 hours at ambient (room) temperature and Table 1 present the different quantity of CRH (g) and CRH dosages (g/L). The mixture in each flask were separately filtered through the glass sand core funnel G4 (0.45 micron) and the oil concentrations in the resulting water were measured using UV spectrophotometry. Also, five mixtures of oil polluted sea water (750 ml) and CRH (10 g) in 1000 ml Erlenmeyer flasks were prepared and allowed to absorb statically. The mixture in each flask were separately filtered through the glass sand core funnel G4 (0.45 micron) and the oil concentrations in the resulting water were measured using UV spectrophotometry for 0 h, 1 h, 3 h, 5 h, 6 h, respectively. Lastly, five mixtures of oil polluted sea water (750 ml) and 10g of CRH were mixed in 1000 ml Erlenmeyer flasks and allowed to absorb statically for 0 h, 1 h, 3 h, 5 h, 7 h, respectively. The mixture in each flask were separately filtered through the glass sand core funnel G4 (0.45 micron) and the COD concentrations in the resulting water were measured using alkaline potassium permanganate.

Table 1: Six mixtures of oil polluted sea water (750 ml, pH: 6.83)

Parameters	**Values**					
Different quantity of CRH (g)	0	5	10	15	20	25
CRH dosages (g/L)	0.00	6.67	13.33	20.00	26.67	33.33

3. Results and Discussion

The elemental composition of initial raw material (rice husk) and the products of carbonization are shown in Table 2, while the main physical indicators of the CRH are presented in Table 3. From Table 2, it can be seen that rice husk compose mainly of C, O, H, and Si. After carbonization, there were significant decrease in the amount of H, O, and N, while there is significant increase in amount of C, Si, and other metals. It can be seen from Table 3 that the surface area of the rice husk increased significantly from 0.78 (m^2/g) to 97.4 (m^2/g) after carbonization. This implies that carbonization increases the adsorption property of the rice husk thus, making the carbonized rice husk a good adsorbent. The bulk density of carbonized rice husk (0.19 g/cm^3) is slightly higher than that of the raw rice husk (0.1 g/cm^3) due to the fact that carbonization burns off organic volatile matter while retaining the silica structure, generally resulting in a more compact material with a higher bulk density (Fetene and Tikuneh, 2025). The thermal conductivity of the CRH (0.035) is slightly lower than that of the raw RH (0.04). This is due to the fact that carbonization process creates a more porous but stable structure, similar to charcoal, that enhances insulating capabilities (Official Origin, 2025).

Table 2: Elemental composition of rice husk and carbonization products

Materials	**Contents (% wt.)**								
	C	H	O	N	K	Si	Ca	Mg	Al
Rice husk	39.21	5.41	46.01	0.39	1.28	7.01	0.29	0.16	0.07
Carbonization products	43.11	0.02	29.34	0.04	1.58	25.01	0.40	0.30	0.09

Table 3: Main physical and chemical indicators of the CRH

Parameters	**Rice husk (RH)**	**Carbonized rice husk (CRH)**
BET Surface area (m^2/g)	0.78	97.4
Bulk density (g/cm^3)	0.1	0.19
Thermal Conductivity (W/(m. K)	0.04	0.035

Figure 1 and Figure 2 present the variation of oil concentration with dosage of carbonized rice husk (g/L) and the profile of oil removal with adsorption time. It can be observed from Figure 1, that oil concentrations decrease rapidly from initial oil concentration of sea water (1.35mg/L) to 0.39 mg/L under the CRH dosages of about 13.33 g/L. Further increase in CRH dosages after 13.33g/L dosage yielded little decrease in the oil concentration until the lowest oil concentration of 0.33 mg/L was reached at 33.33g/L under the experimental conditions.

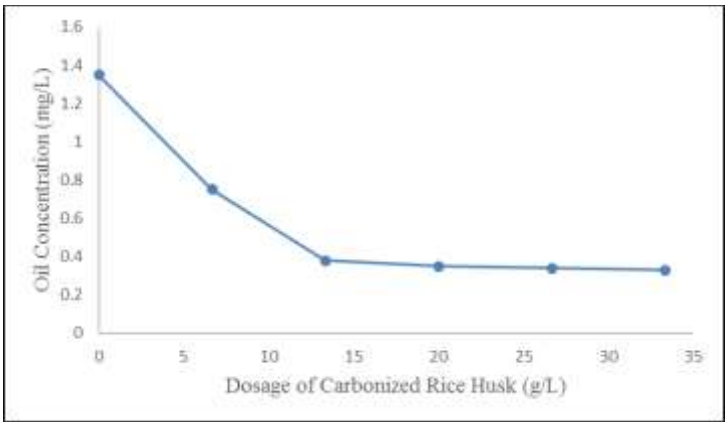


Figure 1: Profile of Oil Concentration after adsorption

When the dosages of CRH range from 0 to 13.3 (13.33) mg/L, the oil removals increase from 0 to 71.85%. As the dosages of CRH increases from 13.33 to 33.33 mg/L, the oil removal only improves 13.16%, getting the highest removal of 85.01%. Hence, the CRH dosage of 13.33 mg/L is the optimal choice from economic point of view.

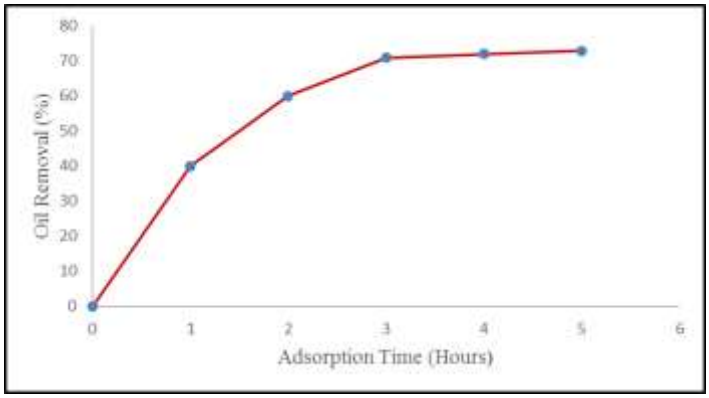


Figure 2: Oil removal with adsorption time

Figure 3 illustrate the variation in oil concentration of the oil polluted sea water with time under the CRH adsorption. Concentration of oil decreased significantly during the initial time (0 - 3 hours). Then after 3-hour adsorption time, there seems to be little decrease in the oil concentration until the 5-hour adsorption time was reached. This implies that adsorption efficiency increases rapidly during the first hour and then stabilizes after 3-hour adsorption time at maximum oil removal of 71.85%. After 3-hour adsorption time, there was no significant increase in oil removal (decrease in polluted sea water oil concentration), rather, a little increase in oil concentration was observed due to desorption process. This implies that 3-hour adsorption time is the optimum adsorption time under the experimental condition.

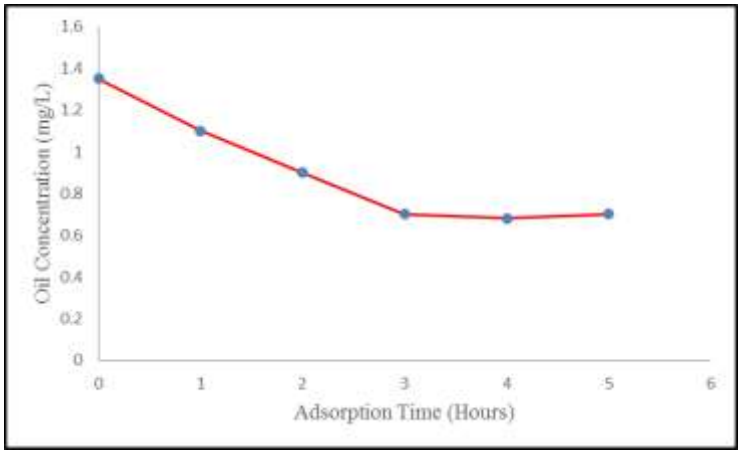


Figure 3: Oil concentration with adsorption time

Figure 4 shows the variation of COD concentration with time. It can be observed from the figure that concentration of COD decreased from 3.45 mg/L to 2.3 mg/L during the first 3 hours adsorption, corresponding 33.33% of removal efficiency. Then it decreased slowly, getting the lowest value of 1.8 mg/L and highest removal efficiency of 47.83%.

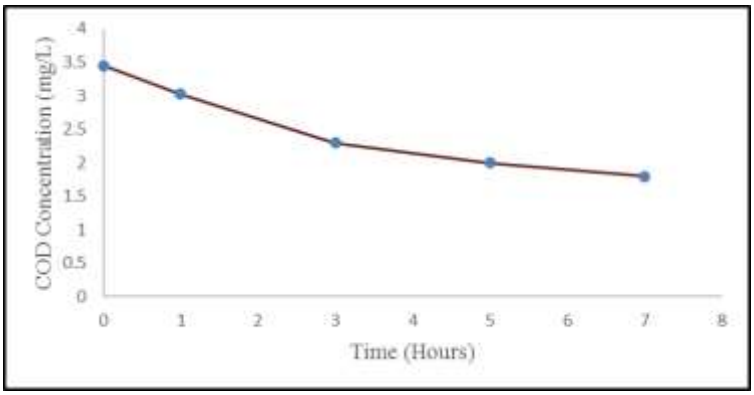


Figure 4: COD concentration with adsorption time

Figure 5 present COD removal efficiency with adsorption time. It can be observed from this figure also that there was a significant increase in COD removal in the first 3-hour adsorption time. After the first 3-hour, COD removal then began to increase gradually with time, reaching 48% at 7 hours.

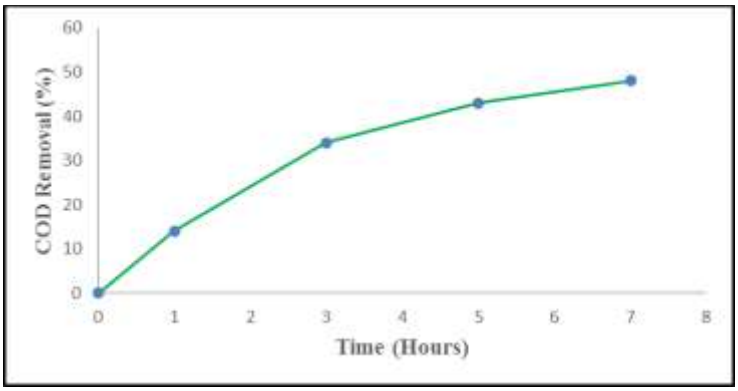


Figure 5: COD removal efficiency with adsorption time

The FTIR spectrum of carbonized rice husk is presented in Figure 4. From the figure, it can be seen that the 3451 cm^{-1} broad band indicates the presence of -OH groups of Si-OH and Si-O-Si-OH. The deformation vibration of water molecules (d-H_2O) is indicated by the peak observed at 1634 cm^{-1}. The bands at 1055 cm^{-1} and 816 cm^{-1} are attributed to the Si-O-Si stretching, while the sharp peak at 530 cm^{-1} is attributed to O-Si-O bending vibration indicating absence of organic matter.

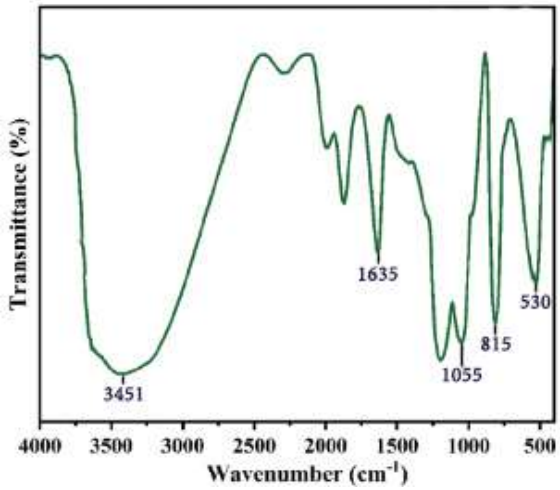


Fig. 3 FTIR spectrum of carbonized rice husk (CRH)

4. Implication of the finding

The results have shown that the thermal processing is an effective method to make the agricultural wastes a most effective material for marine environmental remediation. Carbonization makes up large bulk of fixed carbon and silicon content, reducing the amount of volatile organics. This structural change greatly increases the specific surface area, thus multiplying the available number of binding sites for oil capture. The above adsorption tests show that an optimum dosage of the adsorbent can remove a considerable amount of the oil within a few hours, while being both economical and efficient, and also reduces the chemical oxygen demand. Such changes in structure are crucial for maximizing the time it takes for fluid to be sorbed, which is essential for creating scalable systems that use nature to purify the environment when designing safe, environmentally friendly filtering systems.

Although these results promote the development of pollution management, it is necessary to have the overall picture of the production systems when introducing new processing technologies. Starch hydrolysis profiles (Okpalanma et al., 2024a) and composite blending from alternative resources (Aniemena et al., 2024) are often used to guide evaluation of raw biomaterials which are used as materials. Besides, thermopreservation mechanisms and compositional stability can be correlated with lipid oxidation pathways for long storage periods (Nwagbo et al., 2020). Likewise, applying a treatment to eliminate organic matter from the raw residues is similar to the customized cooking or fermentation processes that transform functional matrices to a stable, valuable end product (Chude et al., 2021; Chude et al., 2023; Okpalanma et al., 2024b; Okpalanma et al., 2025).

5. Conclusion

Finally, the carbonized rice husk (CRH) prepared by using the closed reactor process from agricultural waste is proved to be an excellent and efficient adsorbent in the removal of oil polluted seawater. Controlled thermal treatment significantly enhances the structure and material behaviour to effectively capture both the soluble and emulsion oil fractions. The carbonization is done in a closed system, which ensures an effective reduction of environment emissions and at the same time an increase of the porosity and surface area of the carbonized material. The material thus obtained shows a perfect combination of high affinity for oil and excellent desorption properties, both essential for sustainable water treatment in realities. In conclusion, this study presents a viable, cost-effective, and environmentally friendly solution for marine environment protection, and demonstrates how to turn the natural agricultural wastes into a high-performance filtering medium to solve key issues in marine environment pollutions.

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