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# Preparation and Characterization of Carbonized Coconut Shell and Zeolite Adsorbents Blend

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**ABSTRACT:** Industrial waste water poses a serious threat to humans, animals and sustainability of the environment. The study investigated the preparation and characterization of carbonized coconut shell and zeolite adsorbents blend. Data obtained show that the blend has the following properties. Bulk Density (0.63±0.01); Moisture (3.12±0.03); pH (10.23±0.01); Iodine (124.68±0.07). From the study, the bulk density property of the ratio 1:1 blend of zeolite /carbonized coconut shell is higher compare to the others, implying better flow consistency and packaging quality of its material. Overall, the lesser the moisture and higher the ash content of an adsorbent, the more fixed and compact will the adsorbent carbon chains be and the better its adsorption properties.

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Environmental problems caused by industrial waste waters are very serious (Mekuria *et al* 2021). Industrial waste waters are colored effluents discharged from fabric, paint, paper, leather, and other industries (Medfu *et al* 2020) which are major sources of aquatic pollution. The presence of heavy metals even in low concentrations is quite dangerous (Kumari and Kumar, 2023). Removal of these wastes is often difficult due to their non-biodegradable and recalcitrant nature (Shrestha *et al*., 2021). The decolorization of industrial waste waters using adsorption technique is superior considering its numerous advantages. Techniques for removal of heavy metals from industrial waste waters include biodegradation, chemical oxidation, membrane separation and adsorption (Chai *et al.*, 2021). Adsorption's benefits include cost-effectiveness, convenience of operation, material reuse, high removal efficiency, and the absence of by-products, which make adsorption generally accepted. Molecular sieves, polymers and metal-organic frameworks, activated carbon zeolites, and other adsorbents are widespread. These materials have a large specific surface area and adsorption capacity that may be adjusted. (Lu and Astruc 2020). A large number of researches have reported the potential of carbonized coconut shell, as well as zeolites as effective adsorbents for removing various pollutants (such as dyes, polystyrene, volatile organic compounds, tetracycline, heavy metals and others) from waste waters (Al-Jubouri and Holmes 2020). For instance, recent studies have reported on removal of cobalt ions from aqueous solution using zeolite and zeolite-based carbon composites (Machida et al 2005), adsorption of Cu(II) and Pb(II) ions onto activated carbon (Stahelin et al 2020), and the use of coconut shell-based activated carbon adsorbent for removal of benzene and toluene from synthetic automotive gasoline (Li et al 2022). Notably, Li et al. (2022) used Fe-N-Co - modified biochar to study the adsorption of rhodamine B from wastewater. According to the study, coconut shell was combined with $FeSO_4.7H_2O$ and urea to make biochar using a once-through pyrolysis

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technique at 500$^0$C. Carbon materials from coconut shell have been reported to have larger specific surface area and developed pore structures (Kaur et al 2023). Commercially activated carbon possess high surface area of 500-2000m$^2$/g (El-Bery et al 2022). Examples of carbonaceous materials used in research include cocoa shell, pineapple waste, sour cherry stones, olive waste cake, sugarcane bagasse, orange peels, coconut shells, and others. Hence, the objective of the research was to examine the preparation and characterization of carbonized coconut shell and zeolite adsorbents blend.

## MATERIALS AND METHOD

*Materials:* Materials used in this experiment include: coconut shell, deionized water, and zeolite.

*Equipment:* A muffle furnace, pH meter, mechanical grinder, sieve, crucible, desiccator, stainless steel beaker were used during the experiment.

*Procedures:* Water effluent samples were first collected from the Ologbo flow station at Uhunwonde Local Government Area of Edo State and transported immediately to the Department of Chemical Engineering laboratory, University of Benin, Benin City for sample treatment and analysis.

*Carbonization Process:* To reduce moisture content, the coconut shell was cleaned with deionized water and dried for 48 hours at 110 $^0$C. After drying, the samples were crushed and sieved to a size range of 1-2mm. Following that, the coconut shell was carbonized in a furnace at 600 $^0$C at a rate of 30 $^0$C/mins for 2 hours.

*Preparation of adsorbents:* After carbonization, it was allowed to cool, then grinded with a mechanical grinder to form powder. The powder was sieved to size fraction and then stored in teflon nylon bag. The zeolite was brought out from the reagent bottle and measured into a crucible and put in an oven at 110 $^0$C for 1 hour. Immediately, after 1 hour it was brought out and placed in the dessicator and allowed to cool before being stored in a teflon bag. Part of the carbonized coconut shell was mixed with part of the zeolite in a stainless steel beaker with the weight ratio of zeolite/carbonized coconut shell equal to 1:1. The blend samples were placed in an oven at 110 $^0$C for 1 hour. The product was cooled in a desiccator with a drying agent for 30 minutes before being placed in a furnace for 20minute at 700 $^0$C. The furnace was turned off and the zeolite/carbonized coconut shell blend was removed from the furnace and put in a dessicator and allowed to cool. It was then grinded and packed in a teflon bag. The produced adsorbents were then characterized and use for treatment of produced water.

*Characterization:* The prepared adsorbents were characterized for physical and chemical functional properties. The physical properties analysed for the adsorbents in this studies include moisture content, pH, and bulk density; while the chemical functional properties include: Iodine number using standard methods.

*Moisture Content:* 2 g of dried adsorbent was weighed and placed in a pre-dried crucible in a 150 $^o$C oven to dry for 3 hours. The crucible was removed after drying, cooled in a desiccator, and weighed (ASTMD2867). The amount of moisture in percentage was computed as follows:

$$Mc = \frac{B - F}{B - G} \times 100$$

Where: B = mass of crucible plus original sample in gram; F = mass of crucible plus dried sample; G = mass of crucible

*Bulk Density:* The bulk density of the samples was calculated by filling a graduated cylinder (10ml) with dry adsorbent to the top graduation, gently tapping the cylinder on a table, weighing the cylinder with the sample, and then dividing the weight of the sample by the volume of the cylinder.

$$Bulk\ density = \frac{Mass\ of\ dry\ sample}{Volume\ of\ cylinder} \times 100$$

*pH:* 1 g of adsorbent was weighed and placed in a beaker. The samples were mixed for one hour with 10 ml of distilled/deionized water. The pH of the samples was measured using a pH meter once they had stabilized.

*Iodine determination:* 1 g of the adsorbent was weighed and placed in a clean, dry 250 ml Erlenmeyer flask with a glass stopper. In each flask holding the adsorbent, 10 ml of 5% hydrochloric acid solution was measured and spun until the material was moist. The flask was put in a fume hood on a hot plate and allowed to boil. The flasks were taken off the hot plate and allowed to cool to room temperature. Each flask received 100 ml of 0.1N iodine solution. Stopper each flask and violently shake the contents for one minute. Each liquid was poured into a beaker after being filtered. Titrated with standardized 0.1 N Sodium thiosulphate solution until the solution is light yellow. 2ml of starch indicator solution was added, and the titration with sodium thiosulphate solution was repeated until one drop produced a colorless solution. For each sample, the volume of sodium thiosulphate used was recorded (ASTMD 4607-94, 2006).

$$Iodine\ number\ (In) = \frac{(C-Q)}{C} \times \frac{VM}{W} \times 126.91$$

Where: C=volume of thiosulphate solution required for blank titration; Q= thiosulphate solution volume used for sample free aliquot: W= used sample weight: M= Iodine solution concentration: V= the volume of thiosulphate used in the titration.

# RESULT AND DISCUSSION

*Characterization of Carbonized Coconut Shell, Zeolite and 1:1 ratio blend Zeolite/Carbonized Coconut Shell Adsorbents:* Activated carbons are carbonaceous solids with significant interior surface areas and well-developed porous structures. Activated carbon may be made from a variety of materials, mostly biomass, using high-temperature carbonization and activation techniques. The characterization of carbonized coconut shell, zeolite and 1:1 ratio blend zeolite/carbonized coconut shell adsorbents is presented in Table 1

**Table 1:** Carbonized coconut shell, zeolite and 1:1 ratio blend zeolite/carbonized coconut shell adsorbents from Triplicate Analyses and Standard Deviation.

| Parameters | Carbonized coconut shell | Zeolite | Carbonized coconut/Zeolite blend (ratio 1:1) |
|---|---|---|---|
| Bulk Density | 0.56±0.01 | 0.42±0.01 | 0.63±0.01 |
| Moisture | 4.36±0.07 | 2.38±0.04 | 3.12±0.03 |
| Ph | 6.84±0.01 | 11.62±0.01 | 10.23±0.01 |
| Iodine | 97.24±0.02 | 107.17±0.03 | 124.68±0.07 |

Table 1 shows that the three different Adsorbent materials has moisture content 4.36% for carbonized coconut shell, 2.38% for zeolite and for ratio 1:1 blend zeolite/carbonized coconut shell has moisture content 3.12% respectively. Bulk density was found to be 0.56, for carbonized coconut shell; 0.42 for zeolite while for the ratio 1:1 blend Zeolite/carbonized coconut shell was found to be 0.63 respectively. The pH value of the adsorbents was found to be 6.84 for the carbonized coconut shell, 11.62 for the zeolite while for the ratio 1:1 zeolite/carbonized coconut shell respectively. The pH value of the carbonized coconut shell falls within the acceptable pH ranges from 6-8. The iodine number was determined as an approximate measure of the surface area and were found to be 97.24 mg/g for carbonized coconut shell, 78.75 mg/g for Zeolite, while for the ratio 1:1 zeolite/Carbonized coconut shell it was found to be 124.68 mg/g respectively. For the surface area, which was calculated as the reciprocal of the iodine number was found to be $2.00x10^{-2}$, for carbonized coconut shell, $1.00x10^{-2}$ for zeolite while for the ratio 1:1 zeolite/carbonized coconut shell was found $1.00x10^{-3}$ respectively. This indicate that there was better evolution and development of pore in the ratio 1:1 zeolite/carbonized coconut shell compared to that of the Carbonized coconut shell.

*Conclusion:* This study has established that adsorbents are viable alternative for the treatment of produced water. This study has also established that blended adsorbent has shown flow consistency and packaging, which indicate better treatment quality. It can be concluded that all three adsorbents can be considered a low cost option for treating industrial effluents for toxic hydrocarbon removal before discharge into the environment. The effective treatment of produced water for the removal / reduction in the parameters determined is among the most important issues for many industrialized countries.

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