Removal of Total Hydrocarbon from Oilfield Produced Water using Activated Carbon prepared from Coconut Shells

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ABSTRACT: The adsorption capacity of coconut shell activated carbon as adsorbent for the removal of total hydrocarbon from produced water was investigated. The physico-chemical properties of the produced water were determined and the adsorbent was characterized in terms of pH and other surface characteristics. The effects of adsorption process variables on the adsorption capacity of the activated carbon for the removal of total hydrocarbon was evaluated and optimized using the response surface methodology. The adsorption process variables considered and their range of values are adsorbent dosage of 0.4 to 1.0 g, contact time of 20 to 60 mins and temperature of 30 to 50°C. The physicochemical properties of the produced water before treatment revealed that produced water was contaminated with a high concentration of hydrocarbon and other contaminants. Elemental composition showed that the adsorbent contained predominantly CaO (73.22%) and P2O5 (20.59%). The BET surface area, pore volume and pore size of the adsorbent were found to be 689.406 m²/g, 0.266 cm³/g and 2.153 nm respectively. The maximum amount of THC adsorbed in the adsorption process was found to be 1.068,451.73 mg/g. This maximum was achieved using an adsorbent dosage of 0.40 g, a contact time of 60 mins at a temperature of 30°C. The high and close R-squared values of the experimental and predicted values of the THC adsorption indicate reasonable agreement. The high amount of THC adsorbed showed that coconut shell activated carbon is efficient for the treatment of wastewater streams laden with hydrocarbon.

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Keywords: Produced water; coconut shell activated carbon; adsorption; total hydrocarbon content; optimization

Numerous research in recent years has taken into account concerns about monitoring water quality. Freshwater resources on Earth are few, and multiple forms of contamination brought on by numerous human activities are continually threatening their purity (Liu et al., 2017). Consequently, there is a need to protect water resources to stop pathogenic organisms and hazardous substances from contaminating them. Oil and gas exploration generates a sizable amount of liquid waste, akin to many industrial processes. Produced water is the name for this liquid waste (PW). Produced water is the water that is brought to the surface during the extraction of oil and gas from subterranean deposits. The environment (both on land and in the sea) has been polluted by hydrocarbons in many cases due to unavoidable mishaps in most of the oil-producing nations, including Nigeria. This is despite advances in petroleum exploration and production techniques over the past few decades (Wokoma, 2014). In the Niger Delta region of Nigeria, hydrocarbon pollution frequently results in the death of both flora and fauna. Dambo (1993) reported that hydrocarbon pollution hinders the growth and development of marine organisms, and causes fish, crustaceans, and molluscs to acquire objectionable odours or flavours that lower their market value and acceptability (Wokoma, 2014). Once these substances have contaminated water, it is necessary to adopt approaches to reduce environmental harm.

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To treat and remediate contaminated areas and to lower the costs of the standard technologies—which, in general, use biological treatments, membrane filtration, and adsorption processes for the removal of organic compounds—a variety of strategies (traditional and advanced innovations) have been employed (Cavalcante et al., 2007; Netto et al., 2000).

Adsorption occurs because of the attraction of the atoms or molecules on the surface of solids. The molecules on the surface of solids or liquids have a pulling force inward, which is due to the absence of other forces that compensate. The existence of these forces, which result in solids and liquids, has an adsorption force (Aprianti et al., 2018; Sukarjo, 1990). It is advised to utilise powdered activated carbon (AC) because of its large surface area and higher adsorption rate.

Organic compounds can also be adsorbed by powdered activated carbon. Silica (SiO2), a hard and water-soluble substance, is a component of the AC (Aprianti et al., 2018). Activated charcoal, or carbon, is widely used across the globe as an effective adsorbent for treating wastewater of various contaminants. AC produced from coconut shells (CS) is considered one of the best grades of ACs (Mohammad-Khah and Ansari, 2009). The superior property of these activated carbon materials is due to their ability to provide high adsorption capacity resulting from their large surface area and high pore volumes.

Despite these benefits, the process of producing high-quality activated charcoal, which involves controlled pyrolysis at very high temperatures, makes it an expensive and unsafe process to produce for small-scale use in developing countries. Several researchers have, therefore, attempted to produce relatively inexpensive chemically AC alternatives, using a variety of agricultural waste materials (Díaz-Díez et al., 2004). Although the quality of AC produced is largely influenced by the source of the carbon material used, recent works involving the use of agricultural waste, in successfully removing pollutants from water, suggest the practicality of this approach. There is a need to critically analyze and optimize adsorption processes to obtain better and peak performance of the processes which involves getting the maximum variable settings of the processes.

One of the suitable techniques for analyzing and optimizing processes is the response surface methodology (RSM). RSM can be used to evaluate the relative significance of several factors in the presence of complex interactions. RSM answers the question of how to select the levels for the applied factors to obtain the desirable, smallest or largest, the value of the response function in a reduced number of experiments (Hanrahan and Lu, 2006).

This study aims to investigate the adsorption capacity of coconut shell activated carbon for the removal of total hydrocarbon from oilfield-produced water.

MATERIALS AND METHODS

Waste coconut shells were sourced locally, prepared and pretreated before further use. The effluent waste (produced water) was obtained from Jones Creek Flow Station (OML 42), Delta State, Nigeria. All chemicals/reagents were purchased from local vendors.

Analysis of produced water: The produced water was analyzed for total hydrocarbon content to determine the extent of total hydrocarbon contamination of the water. Other physicochemical properties were determined using standard procedures described by Ademoroti (1996). The properties analyzed include total hydrocarbon, total organic carbon, pH, total dissolved solids, chemical oxygen demand, and total suspended solids.

Total hydrocarbon content (THC): The THC of the produced water was determined using the method described by (Wokoma, 2014). The total hydrocarbons in the water sample were extracted using 30.0 ml of toluene three times in a row before being diluted to 100 ml. Using the spectrophotometer 41D, the absorbances of the filtrates were determined spectrophotometrically at 420 nm. On a dry weight basis, the concentration was determined using the calibration graph. The treatment was carried out with the requisite blanks.

Preparation of coconut shells activated carbon (CSAC): The coconut shell char was prepared using the method described by Mohammed et al. (2018). The coconut shells were thoroughly washed in hot water at 80°C to remove adhering dirt and soluble components. The washed shells were air-dried for one week and then dried at 95°C in an oven until the weight became constant. The coconut shells were then carbonized at 500°C in a muffle furnace for 5 hours after which it was allowed to cool overnight to room temperature. The char was ground and sieved to the desired size of 180 μm. The carbonized coconut shells were activated following the method by Ademiluyi and David-West (2012). The char was subjected to chemical treatment with nitric acid (HNO₃) solution in the ratio of 2 g of carbonized coconut shell to 1 g of nitric acid. The setup was covered and placed in an air...
oven drier at 105°C for 50 minutes after which the temperature was increased to 200°C for one (1) hour to give room for a complete activation. The activated coconut shells sample was allowed to cool in a desiccator and then washed with distilled water.

Characterization of CSAC: Five basic techniques were used for the characterization of the prepared CSAC: pH at point of zero charges (pHPZC) using the “eleven point PZC measurement” adapted from methods described by Regalbuto and Robles (2004) and Cechinel et al. (2014), surface morphology and chemical composition was determined using a scanning electron microscope (SEM) coupled with an energy dispersive X-ray spectroscopy (XRD and XRF) and the pore size distribution and specific surface area of CSAC was determined by N2 adsorption method using Brunauer–Emmett–Teller (BET) method.

Batch adsorption studies: The experiments were carried out using the method of Argun et al. (2007) with modification to include central composite design. Exactly 200 ml of produced water was measured into polythene bottles and adsorbent was transferred into these bottles. The mixtures in polythene bottles were stirred simultaneously with a basic reciprocating shaker at 200 rpm for the contact time and then left undisturbed on the desk for 24 hours to allow the system to equilibrate before filtering with a Whatman filter paper into another 100 cm³ polyethylene bottle (Okieimen and Okieimen, 2001). The factors and range of values considered are CSAC dose of 0.4 to 1.0 g, contact time of 20 to 60 mins and temperature of 30 to 60°C. The concentrations of the residual or remaining THC in the filtrate after the adsorption process were determined. The amounts of the THC adsorbed will be calculated using Equation (1).

\[ q_e = \frac{\nu(C_0-C_e)}{m} \]  

(1)

Where \( q_e \) is the amount of THC adsorbed in mg/g of the CSAC; \( C_0 \) is the initial concentration of the THC before the adsorption process in mg/L; \( C_e \) is the equilibrium concentration of the THC in the filtrate after the adsorption process in mg/L; \( m \) is the mass of the adsorbent (g) and \( V \) = volume of the solution in L.

Data Analysis: The amount of THC adsorbed was analysed to fit a second order regression model and then optimized using numerical optimization technique. Analysis of variance (ANOVA) was used to determine the significant factors/terms at 5% significant level using Design Expert software (Version 11.0).

RESULTS AND DISCUSSIONS

Characterization of coconut shell activated carbon (CSAC): The micrograph of the CSAC as produced by the scanning electron microscope (SEM) at 750 resolutions is shown in Figure 1. The micrograph demonstrates the highly developed irregular pore structure of CSAC, which showed significant pore gaps between the CSAC particles which may be due to impurities from the carbonation process.

![SEM micrograph of CSAC](image)

The primary indicator for the surface characteristics of activated carbon/catalyst is the BET surface area (Chandra et al., 2009; Kalderis et al., 2008). From this study, CSAC's surface area, pore volume, and pore size were found to be 689.406 m².g⁻¹, 0.266 cm³.g⁻¹, and 2.153 nm, respectively.

In the report of the International Union of Pure and Applied Chemistry (IUPAC), CSAC is classified to have mesoporous pore size distribution with a pore size of 2.153 nm (Pandolfo and Hollenkamp, 2006). Due to the reaction between activating chemicals and carbon, activation causes the considerable production of micropores and/or mesopores, which increases the specific surface area and total pore volume of adsorbents.

The elemental compositions of CSAC as analyzed using XRF spectroscopy are given in Table 1. It can be seen that CaO is the predominant metal oxide in CSAC with a composition of 73.22%, the other metal oxide with high composition is P₂O₅ of 20.59% while the remaining metal oxides are very low in composition. The ability of CSAC to be employed in adsorption/catalytic processes is indicated by the presence of metal and transition metal oxides. And this is due to their ability to alter the oxidation state or, in
the case of metals, to adsorb other compounds on their surface.

| Component | Composition (%) |
|-----------|-----------------|
| CaO       | 73.22           |
| FeO       | 20.59           |
| AlO       | 1.76            |
| SO        | 1.51            |
| Cl        | 1.26            |
| CaO       | 0.72            |
| MgO       | 0.28            |
| SiO       | 0.16            |
| FeO       | 0.13            |

The FTIR analysis was conducted to confirm the presence of functional groups in the CSAC that are capable to adsorb the THC in produced water. The FTIR spectrum is shown in Figure 3. The results showed a broad absorption peak at 3336.0 cm⁻¹ indicating the presence of the O-H group, which was derived from the alcohol compounds. Absorption peaks at 1558.0 cm⁻¹ indicate the presence of aromatic C=C groups. The presence of alkanes and alkyl bonds was seen by the C-H strong absorption broadband at a wavelength of 1416.4 cm⁻¹, whereas the presence of alkyl halide groups with very strong absorption bands was observed at 1021.3 and 764.1 cm⁻¹. A strong absorption band with a C-H bend belonging to aromatic compounds was observed at 708.2 wavelengths. The p\(\text{H}_{\text{PZC}}\) is approximately 4.9, as shown by the curve in Figure 4. The surface of CSAC is mostly negatively charged at pH values more than 4.9 and positively charged at pH values lower than this. Activated carbon typically has a pH at the point of zero charges that is close to 7.0. (Babić et al., 1999). However, the CSAC employed in this investigation did not exhibit this tendency. Since the H⁺ ions existing on the surface of the activated carbon after treatment will be released into the solution and lower the pH, the value obtained (4.9) may be explained by the acid treatment that the activated carbon underwent previous to usage (Cechinel et al., 2014). Due to electrostatic repulsion, the adsorption of cations, such as metal ions, is preferred at pH > p\(\text{H}_{\text{PZC}}\), while the adsorption of anions is favoured at pH < p\(\text{H}_{\text{PZC}}\). The THC adsorption process was therefore carried out at a pH of 4.9 or higher.

The pH of the produced water was found to be slightly alkaline with a value of 7.78 and within the standard for pH of water. According to Neff et al. (2011), TOC of produced water range from < 0.1 to > 11,000 mg.l⁻¹. The TOC of the produced water (this study) of 80.80 mg.l⁻¹ was found to be within the range given. The TDS of 837.69 mg.l⁻¹ was below the DPR standard of 5000 mg/l for discharged water and the TSS of the produced water (2.40 mg.l⁻¹) was found to be below the standard of 50 mg.l⁻¹ (Nwosi-Anele and Iledare, 2016). Electrical conductivity (EC) is a measure of the saltiness of the water and is measured on a scale from 0 to 50,000 µS.cm⁻¹. Freshwater is usually between 0 and 1,500 µS.cm⁻¹ and typical seawater have a conductivity value of about 50,000 µS.cm⁻¹. Natural waters contain small amounts of salt, which are essential for the growth of both plants and animals. High salt concentrations in freshwater may have negative effects on aquatic ecosystems and impede...

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human usage (Mary River Catchment Coordinating Committee, 2013). The EC value of 1.55 µS.cm\(^{-1}\) showed that the produced water is safe for discharge into water bodies. The THC of the produced water was observed to be very high with a value of 13990.00 mg.l\(^{-1}\). This is because produced water is an effluent of petroleum exploration/production as petroleum is mainly made up of hydrocarbons. THC at low concentrations is known to be detrimental to aquatic life and may end up in the system of humans directly or indirectly through the consumption of aquatic animals.

Analysis of THC adsorption: Table 3 gives the experimental design matrix of THC adsorption (showing the combination of conditions) from produced water by CSAC and the actual and predicted amounts of THC adsorbed.

| Run | Coded factors | Actual factors | Amount THC adsorbed (mg.g\(^{-1}\)× 10\(^3\)) |
|-----|---------------|----------------|-----------------------------------------------|
|     | A  | B  | C    | CSAC dosage (g) | Contact time (mins) | Temperature (°C) | Actual | Predicted |
| 1   | 1  | -1 | 1    | 1.0           | 20               | 50            | 6.25   | 6.30       |
| 2   | 0  | 0  | -1.68| 0.7           | 40               | 23.18         | 7.43   | 6.55       |
| 3   | 0  | -1.68| 0    | 0.7           | 6.36             | 40            | 9.21   | 8.60       |
| 4   | 0  | 0  | 0    | 0.7           | 40               | 40            | 6.80   | 5.69       |
| 5   | 0  | 0  | 0    | 0.7           | 40               | 40            | 5.71   | 5.69       |
| 6   | -1 | -1 | 0    | 0.4           | 20               | 30            | 1.00   | 1.04       |
| 7   | 0  | 0  | 0    | 0.7           | 40               | 40            | 5.36   | 5.69       |
| 8   | 0  | 0  | 0    | 0.7           | 40               | 40            | 5.43   | 5.69       |
| 9   | 0  | 1.68| 0    | 0.7           | 73.64            | 40            | 7.14   | 6.72       |
| 10  | 0  | 0  | 0    | 0.7           | 40               | 40            | 5.21   | 5.69       |
| 11  | -1.68| 0  | 0    | 0.2           | 40               | 40            | 12.8   | 12.20      |
| 12  | 0  | 0  | 1.68| 0.7           | 40               | 56.82         | 6.14   | 5.98       |
| 13  | 1  | 1  | -1   | 1.0           | 60               | 30            | 4.25   | 4.52       |
| 14  | 1  | -1 | -1   | 1.0           | 20               | 30            | 3.75   | 4.45       |
| 15  | 1.68| 0  | 0    | 1.2           | 40               | 40            | 3.94   | 3.51       |
| 16  | -1 | 1  | 1    | 0.4           | 60               | 50            | 8.13   | 8.16       |
| 17  | -1 | 1  | -1   | 0.4           | 60               | 30            | 10.00  | 10.7       |
| 18  | -1 | -1 | 1    | 0.4           | 20               | 50            | 10.00  | 10.5       |
| 19  | 1  | 1  | 1    | 1.0           | 60               | 50            | 3.50   | 3.81       |
| 20  | 0  | 0  | 0    | 0.7           | 40               | 40            | 5.43   | 5.69       |

The amount of THC adsorbed (\(\text{mg.g}^{-1}\)) = 568600 − 258100A − 55713.52B − 16748.40C − 4687.50AB + 45312.50AC − 64062.50BC + 76383.55A² + 69728.09B² + 20483.15C²

Batch experiments were performed for the uptake of THC from produced water by CSAC using RSM-based CCD. Equation (2) in terms of coded units is the empirical model that demonstrates the mathematical correlation between the process factors taken into consideration and the quantity of THC adsorbed from the adsorption process using CSAC.

When an independent variable in a regression equation has a positive sign, it implies that increasing the variable will result in a rise in the response, whereas a negative sign would cause the response to decrease (Lenth, 2009; Ocholi et al., 2018). All the main factors; CSAC dosage (A), contact time (B) and temperature (C) had negative effects on the amount of THC adsorbed from produced water by CSAC. CSAC dosage with a higher coefficient had the highest effect than both contact and temperature on the amount of THC adsorbed from produced water by CSAC. Interaction factor (AC) had positive effects on the amount of THC adsorbed while AB and BC had negative effects. All quadratic factors (A², B² and C²) had positive effects on the amount of THC adsorbed from produced water by CSAC. The model’s fit statistic, which is shown in Table 4, may be used to further evaluate the model.

| Parameter | Value |
|-----------|-------|
| Standard deviation (mg.g\(^{-1}\)) | 71.691.18 |
| Mean (mg.g\(^{-1}\)) | 682.400 |
| Coefficient of variation, C.V (%) | 10.51 |
| R² | 0.9571 |
| Adjusted R² | 0.9185 |
| Predicted R² | 0.7591 |
| Adequate precision | 17.13 |

The coefficient of variation (C.V.) in Table 4 is 10.51%, which is within an acceptable range. Since the CV is a measure of how standard deviation is expressed as a percentage of the mean, smaller values of the CV result in higher reproducibility. A high CV, in general, suggests that the mean value’s variability is

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large and that an acceptable response model has not been adequately developed (Liyana-Pathirana and Shahidi, 2005). To prove the validity of the THC adsorption model equation, the coefficient of regression $R^2$ was utilized. The high $R^2$ value of 0.9571 in Table 5 indicates that the model can account for nearly 95% of the variation in the quantity of THC absorbed by the variables under consideration. Further evidence that the model (Equation 4) accurately predicted the THC adsorption process using a quadratic polynomial is provided by the reasonable agreement between the adjusted $R^2$ (0.9185) and the predicted $R^2$ (0.7591) values (difference between actual and predicted $R^2$ values is less than 20%). The signal-to-noise ratio is measured with adequate precision. More than 4 is preferred as a ratio. A strong signal may be seen from the ratio of 17.13. (Lenth, 2009). This suggests that the model may be used to explore the design environment. The summary of the analysis of variance (ANOVA) for the quadratic model is presented in Table 5. It was noted that the regression model for the removal of THC was significant at a confidence level of 95% ($p < 0.05$) indicating the aptness of the model. The quantity of THC that CSAC adsorbed from produced water was significantly influenced by two primary parameters, CSAC dose and contact duration, both of which had low $p$-values of <0.0001 and 0.0166, respectively. The temperature was shown to have no significant impact on this amount of THC adsorbed. Among the interaction variables, only BC have significant effects ($p$-value = 0.0300 < 0.05) on the amount of THC adsorbed. Quadratic factors ($A^2$ and $B^2$) had significant effects on the amount of THC adsorbed. Lack of fit for the model was not significant ($p > 0.05$) which indicates that the model was predicted for reducing COD and NH3-N and adequate for the prediction within the range of the variables studied. The low residual values are an indication of the good agreement of the experimental data with the mathematical model.

Table 5: ANOVA for the quadratic model of THC adsorption

| Source          | Sum of Squares | df  | Mean Square | F-value | p-value |
|-----------------|----------------|-----|-------------|---------|---------|
| Model           | 1.147E+12      | 9   | 1.274E+11   | 24.79   | < 0.0001* |
| A – CSAC dosage | 9.100E+11      | 1   | 9.100E+11   | 177.06  | < 0.0001* |
| B – Contact time| 4.239E+10      | 1   | 4.239E+10   | 8.25    | 0.0166*  |
| C – Temperature | 3.831E+09      | 1   | 3.831E+09   | 0.7454  | 0.4082   |
| AB              | 1.758E+08      | 1   | 1.758E+08   | 0.0342  | 0.8570   |
| AC              | 1.643E+10      | 1   | 1.643E+10   | 3.20    | 0.1041   |
| BC              | 3.283E+10      | 1   | 3.283E+10   | 6.39    | 0.0300*  |
| $A^2$           | 8.408E+10      | 1   | 8.408E+10   | 16.36   | 0.0023*  |
| $B^2$           | 7.007E+10      | 1   | 7.007E+10   | 13.63   | 0.0042*  |
| $C^2$           | 6.046E+09      | 1   | 6.046E+09   | 1.18    | 0.3035   |
| Residual        | 5.140E+10      | 10  | 5.140E+09   |         |         |
| Lack of Fit     | 3.449E+10      | 5   | 6.898E+09   | 2.04    | 0.2264   |
| Pure Error      | 1.691E+10      | 5   | 3.382E+09   |         |         |
| Cor. Total      | 1.198E+12      | 19  |             |         |         |

* = significant model factors

Fig 5a: Effect of interaction of CSAC dosage and contact time on the amount of THC adsorbed

Fig 5b: Effect of interaction of CSAC dosage and temperature on the amount of THC adsorbed
Fig 5c: Effect of interaction of contact time and temperature on the amount of THC adsorbed

Figures 5 (a) to (c) show how the interaction of factors affected the quantity of THC that CSAC adsorbed from produced water. Figure 5(a) depicts the amount of THC that CSAC adsorbed from produced water as a result of the interaction between adsorbent dose and contact time (AB). From the plot, an increase in the adsorbent dosage at constant contact time lead to a decrease in the amount of THC adsorbed by CSAC while an increase in the contact time at constant adsorbent dosage leads to a decrease in the amount of THC adsorbed. The concurrent increase in the adsorbent dosage and contact time at constant temperature leads to an increase in the amount of THC adsorbed by CSAC. The relationship between adsorbent dose and temperature (AC) on the quantity of THC absorbed by CSAC is shown in Figure 5(b). As shown in the plot, an increase in adsorbent dose at a constant temperature result in a reduction in the amount of THC adsorbed by CSAC, whereas an increase in temperature results in a decrease in the amount of THC adsorbed at a constant adsorbent dosage. The concurrent increase in adsorbent dosage and temperature leads to an increase in the amount of THC adsorbed at constant contact time by CSAC. The interaction between contact duration and temperature (BC) has an impact on how much THC is adsorbed by CSAC, as shown in Figure 5(c). According to the plot, an increase in contact time at a fixed temperature causes a reduction in the amount of THC adsorbed, while an increase in temperature causes a decrease in the amount of THC adsorbed. The quantity of THC that the adsorbent can absorb decreases when the contact duration and temperature are simultaneously increased while maintaining a fixed adsorbent dose (CSAC).

**Optimal conditions of THC adsorption:** A numerical optimization technique using desirability functions employed for the optimization of the response (amount of THC adsorbed) from produced water by the CSAC gave maximum desirability of 0.771 which indicates that there is a greater than 77% chance of achieving the maximum amount of THC adsorbed. The maximum amount of THC adsorbed in the adsorption process predicted was 1,068,451.73 mg·g⁻¹. This maximum was achieved using a CSAC dosage of 0.40 with a contact time of 60 mins at a temperature of 30°C.

**Conclusion:** Information and data was provided from this study on the elemental composition, surface area, surface morphology and functional group of the coconut shell activated carbon prepared which showed that the activated carbon can be used as adsorbent. The physico-chemical properties of the produced water before treatment revealed water was contaminated with hydrocarbon. The maximum amount of total hydrocarbon adsorbed showed that the activated carbon is efficient in the treatment of wastewater streams laden with hydrocarbon.

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