Attenuation of Heavy Metals from Waste Oil-Based Drilling Mud using Locally Produced Coconut Shell-Based Activated Carbon*

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Abstract

Most toxic Heavy Metals (HM) persist in oil-based mud and ecosystem for many decades after the application of mud for drilling oil and gas wells. This study assessed the attenuation of such heavy metals from waste oil-based mud using activated carbon produced locally. The oil-based mud analysed presented variable levels of Iron (Fe), Copper (Cu), Nickel (Ni), Potassium (K), Arsenic (As), Chromium (Cr), Manganese (Mn) and Zinc (Zn). Adsorption is a highly effective means of separation to remove a wide range of pollutants in waste streams. Coconut shell activated carbon (CS-AC) was locally produced to remove heavy metals from the waste oil-based mud to encourage value addition to waste. The adsorption data was fitted to Langmuir and Freundlich isotherm model using linear regression analysis. The data was more fitted to the Langmuir isotherm as indicated by the high goodness of fit values (R^2). To reduce the heavy metal concentrations below threshold levels optimum CS-AC dosage required for Fe, Cu, Ni, K, As, Cr, Mn and Zn is 5 g/l of CS-AC for a contact time of 30 minutes. The characterization of the produced carbons shows good results comparable to other commercial activated carbons. CS-AC is a viable and economical product for the removal of toxic heavy metals from waste oil-based mud before disposal.

Keywords: Attenuation, Heavy Metals, Waste, Oil-Based Mud, Activated Carbon

1 Introduction

A heavy metal refers to any metallic element that has a relatively high density (>5 g/cm^3) and is toxic or poisonous even at low concentration. These heavy metals are known to have health effects on tissues of living organisms hence the essentiality to remove them before disposal (Vhahangwele and Khathutshelo, 2018). Conventional methods of heavy metal removal include precipitation, ion-exchange, coagulation, and flocculation. However, adsorption is a highly effective means of removing or reducing the amount of a wide range of pollutants both organic and inorganic (Virgen *et al*., 2018; Vo *et al*., 2020).

In the use of oil-based muds (OBMs) in subsurface oil and gas operations, heavy metals (HMs) get into the mud through sources such as: additives used in mud formulation and downhole rock formations. HMs are parameters to be considered among other parameters such as pH, salt, hydrocarbons, and cuttings during disposal of mud. Great emphasis is therefore laid on disposal of spent OBMs into offshore environments. Therefore, treatment before disposal of these muds should be highly encouraged (Amenyah Kove *et al*., 2020).

Adsorbents including activated carbon, clay, silica, alumina, and zeolite are highly effective for removing heavy metals from waste streams (Emam, 2013; Worch, 2012). Because of its highly developed internal porosity and high surface area, activated carbon (AC) is an excellent adsorbent (Buah *et al*., 2015; Yahya *et al*., 2016). Activated carbon is mainly produced from carbon-containing materials using a thermal decomposition process or pyrolysis, followed by activation (Ukanwa *et al*., 2019; Beguin and Frackowiak, 2010). Agricultural wastes such as palm kernel shells, peanut husk, rice husk, wheat bran and coconut shells are a rich-source precursor for production of low-cost AC. Despite the varied use of activated carbon in several sectors including health, manufacturing, waste treatment etc., commercial activated carbon is still considered an expensive material due to the scarcity of conventional raw materials and high preparation costs (Deng *et al*., 2010; Crini *et al*., 2018).

This research produced local and low-cost activated carbon from coconut shells which are available as agriculture waste and evaluated their use in adsorbing heavy metals from spent oil base muds in Ghana. This will not only reduce coconut shell wastes but will help fulfil the local content and participation policy of Ghana.

2 Resources and Methods Used

2.1 Materials Collection

The agro-waste product employed in this research is coconut shells. Fig. 1 shows an image of a typical coconut fruit outlining the different parts of the fruit. In this research, the endocarp was used as the precursor in the production of the CS-AC. The coconut shells were sourced from vendors in Tarkwa in the Western Region. The sources of the samples were chosen based on the aim of the research to convert waste agro-products into valuable activated

carbon to remove wastes from contaminated drilling mud while reducing pollution caused from these agro-waste products. Waste oil-based mud was collected from Halliburton in Takoradi.
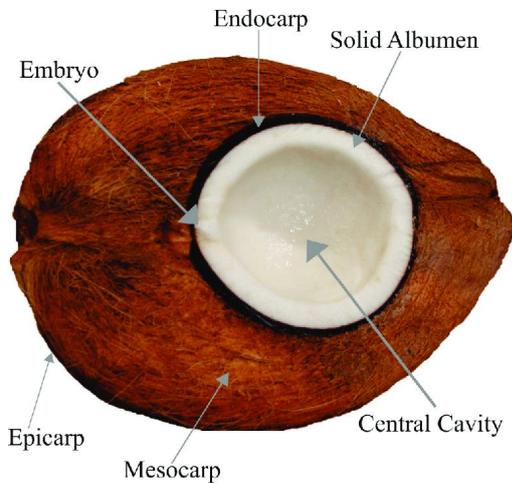

Fig. 1 Different Parts of the Coconut Fruit, *Cocos nucifera* **(Passos** *et al.***, 2018)**

2.2 Carbonisation

In the manufacturing of activated carbons, two steps are involved namely, carbonisation of the raw materials (sometimes called charring) and activation of the carbonised product. The carbonisation process is necessary to create an imprint effect on the final product. The carbonisation operation is used to increase the carbon content and create preliminary porosity in the carbon. The activation expands the porosity and orders the framework to produce a high porosity solid as the finished product. Two main activation methods/processes exist for manufacturing of activated carbon: physical (using steam, CO_2 etc.) and chemical activation (using H_3PO_4, $ZnCl_2$ and K_2CO_3 etc.) methods. In this research physical activation using steam was employed because it has been found to be inexpensive and more environmentally friendly (Maniscalo *et al.*, 2020; Tan *et al.*, 2021).

Precursor of 833.46 g of particle size range of -5.6 mm +4.0 mm was fed into a reactor. The temperature was increased from room temperature to a final carbonisation temperature of 900˚C. Researchers have reported several temperature ranges required for pyrolysis in the range of 500 ˚C to 900 ˚C (Okoroigwe *et al.*, 2013; Buah *et al.*, 2015; Hidayu and Muda, 2016; de Souza Souza *et al.*, 2021). The resulting char was then removed and allowed to cool in a desiccator, weighed and recorded.

2.3 Activation Process

Activation is essential in building additional porosity and organising of the char structure, resulting in a much more porous solid of activated carbon. These include opening of pores that were not accessible, development of new pores due to selective activation and widening of already existing pores. The char was sieved to obtain a particle size of +2.80. The char was fed into the reactor and covered. De-ionised water was then injected into the reactor through a pipe found at the bottom at a flowrate of 4.5 ml per minute. The activation was carried out at a temperature of 900 ˚C for 3 hours. After completing the activation process, the reactor was allowed to cool down, the samples were taken out from the reactor and washed using distilled water. The resultant activated carbon (CS-AC) from coconut shells were weighed and recorded and then subsequently characterised and used for adsorption studies. Fig. 2 shows the locally produced activated carbons.

Fig. 2 Locally Prepared Activated Carbon(CS-AC)

2.4 Characterisation of Activated Carbons

The moisture content (C_{moist}) of the produced carbons was found by the oven-drying test method. The ASTM D2867 – 09 standard test method was employed. A sample of carbon is put into a dry, closed capsule (of known weight) and weighed accurately. The capsule is opened and placed with the lid in a preheated oven (145 – 155 ºC). The sample is dried to constant weight them removed from the oven and with the capsule closed, cooled to room temperature. The closed capsule is weighed again accurately. The percentage difference (Equation (1)) of weight is expressed as the moisture content of the sample.

$$C_{moist} \ (\%) = \frac{Loss\ in\ weight\ on\ drying\ (g)}{Initial\ weight\ (g)} \times 100\% \qquad (1)$$

To determine the volatile matter (C_{vol}) as expressed in Equation (2), approximately 1.0 g of the sample was taken in a crucible with cover (of known weight). The covered crucible was placed in a muffle furnace regulated at 950 °C for 7 min. Then the covered crucible was cooled to room temperature in a desiccator and weight read and recorded. The percentage weight loss was regarded as the percentage of volatile matter.

$$C_{vol}(\%) = \frac{weight\ of\ volatile\ component\ (g)}{oven\ dry\ weight\ (g)} \times 100\% \qquad (2)$$

To determine the ash content (C_{ash}) (Equation (3)), a dried sample of activated carbon weighed to the nearest 0.1 mg was taken into a crucible (of known weight). The crucible was placed in the muffle furnace at 650 °C and ashing is completed when constant weight is achieved. The crucible is cooled to room temperature in a desiccator and the percentage weight of the sample that remained was considered as ash content. This was done following the ASTM D2866 – 94 standards.

$$C_{ash}(\%) = \frac{Ash\ weight\ (g)}{Oven\ dry\ weight\ (g)} \times 100\% \qquad (3)$$

The surface area of the activated carbons was determined by adsorption of Nitrogen at 77 K onto the activated carbons using the Brunauer-Emmett-Teller (BET) method.

2.5 Drilling Mud Characterisation

The drilling mud was separated into the solid and liquid phase to determine the heavy metal distributions. To do this, the spent mud samples were placed in a rack on the Centurion CR200 Centrifuge. The centrifuge was then set to run for 2 000 rpm for 30 minutes. After 30 minutes the test tubes were removed from the centrifuge and the liquid separated. The liquid on the top was poured into a funnel fitted with a filter paper. Fig. 3 shows the separated solid and liquid fractions of the drilling mud. The concentration of heavy metals were then determined by AAS analysis.

Fig. 3 Drilling Mud Separated into the Solid and Liquid Phases

2.5 Adsorption Studies

The initial HM concentration of the emulsion (waste OBM) determined by the AAS recorded Iron (Fe), Copper (Cu), Nickel (Ni), Zinc (Zn), Chromium (Cr) and Manganese (Mn) at varying levels of 851.11, 51.21, 20.62, 2.31, 0.74, 0.92 and 5.21 mg/l. 2 g of the prepared activated carbon (CS-AC) was weighed into a beaker. A 40 ml volume of the liquid phase of the mud was added to the CS-AC. The mixture was placed on an electronic shaker and rotated at 150 rpm. The samples were rotated for 5, 10, 15, 30 and 60 mins. It was all carried out at a constant room temperature. The remaining HM concentration in the emulsion was determined by atomic absorption spectroscopy (AAS).

2.5 Isotherm Modelling

Linear regression has been one of the most viable tools defining the best-fitting relationship quantifying the distribution of adsorbates, mathematically analysing the adsorption systems, and verifying the consistency and theoretical assumptions of a specific model. Hence, the accuracy of the fit of an adsorption model to experimental data is typically assessed based on the magnitude of the linear coefficient of determination, with R^2 values closest to unity being deemed to provide the best fit. Accordingly, linear adsorption isotherm models (shown in equations 4 and 5) in this research were solved in Origin 8.5 and the constants determined.

$$logC_e = \frac{1}{n}logC_e + logK_F \qquad (4)$$

where Q_e is the amount of iron ions adsorbed per unit mass of adsorbent (mg/g) at equilibrium, C_e is the equilibrium concentration of the adsorbate (mg/l), n is a constant which describes the intensity of adsorption and K_F is a Freundlich constant used to describe the capacity of the carbons $((mg/g)/(mg/l)^{1/n})$. The constant n is a measure of adsorption intensity, which varies with the heterogeneity of the adsorbent surface. The surface becomes more heterogeneous as the value of 1/n gets closer to zero. The adsorption process is favourable when 1/n value lies between 0.1 and 1.

$$\frac{C_e}{Q_e} = \frac{1}{K_L Q_m C_e} + \frac{1}{Q_m} \qquad (5)$$

where Q_e is the amount of iron ions adsorbed per unit mass of adsorbent (mg/g) at equilibrium, C_e is the equilibrium concentration of the adsorbate (mg/l), K_L is a constant related to the affinity between the adsorbent and the adsorbate, and Q_m is the Langmuir constant related to the energy of adsorption.

The vital aspect of the Langmuir model is the dimensionless constant separation factor, sometimes called the equilibrium parameter, R_L. The expression to derive R_L is shown in Equation 6.

$$R_L = \frac{1}{1+K_L C_e} \qquad (6)$$

3 Results and Discussion

As the ash content decreases in value, the prepared AC's efficiency and adsorptive capacity increases. Ash content affects adsorption properties by creating inactive sites. While working on this factor, Abdullah *et al*. (2001) concluded that ash values that make a carbon suitable for absorption should be in the range of 1 – 20 %, making 3.67 % value obtained suitable.

It is recommended that for best results the volatile matter content of activated carbons should be ≤ 15 %. This criteria indicates that the C_{vol} value is higher than the required value. The moisture content of the AC was found to be within the quality specification limits. According to Madhavakrishnan *et al*. (2008), there was no correlation between moisture content and adsorption capability of activated carbon. For fixed carbon content the higher the value the better. The most commonly reported recommendation for fixed carbon content is that it should be ≥ 65 % which makes the value obtained for CS-AC suitable.

The BET surface area achieved for the locally produced activated carbons was significantly higher than expected when compared to the acceptable range for commercial activated carbon (500 – 1500 m^2/g). The BET surface area of 787.88 is good.

Table 1 Characteristics of the Locally Produced Activated Carbons

Parameter	Value	Unit
Hardness	99	-
Density	1.98	g/cm^3
Volatile Matter	23.54	%
Ash Content	3.67	%
Moisture Content	4.30	%
Fixed Carbon	68.49	%
BET Surface Area	787.88	m^2/g
Total Pore Volume	0.415	cm^3/g
Micropore Volume	0.381	cm^3/g
Mesopore Volume	0.031	cm^3/g
pH	6	-

Adsorption percentage of heavy metals (Fe, Cu, Ni, K, As, Cr, Mn, and Zn) from the OBM by CS-AC with respect to time is presented in Fig. 4. From the results, adsorption increases within the first 15 minutes to maximum of 75.26 %, 73.05 %, 34.05 %, 81.77 %, 74.88 %, 81.07 %, 62.72 %, 83.63 % for Fe, Cu, Ni, K, As, Cr, Mn and Zn respectively. This

pattern is followed with a gradual fall in adsorption efficiency after 30 minutes of contact time. This can be associated with exhaustion of CS-AC active sites due to metal filling capacity as adsorption is an equilibrium process (Saleem *et al*., 2019). The results show Nickel adsorption using CS-AC presents the least adsorption efficiency while Zinc shows the highest. The results also show that the optimum contact time is 30 minutes.
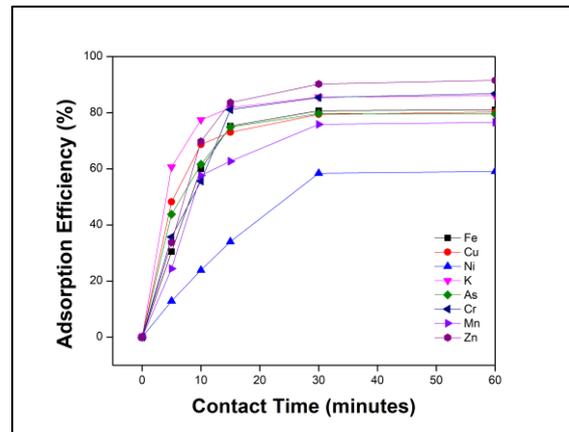

Fig. 4 Removal Percentage of Ca, Fe, Cu, Ni, Zn, Cr, Mn, K and As using 2 g/L of CS-AC at varying Contact Times

The results of the linear fit CS-AC adsorption to the Freundlich isotherm model is shown in Fig. 5. Using the goodness of fit (R^2) shown in Table 2, it is seen that for Fe, Cu an Zn, adsorption using CS-AC shows is more fitted to the Freundlich isotherm. However, comparatively, Langmuir isotherm model is best at explaining the adsorption of all the metals under consideration in this study.
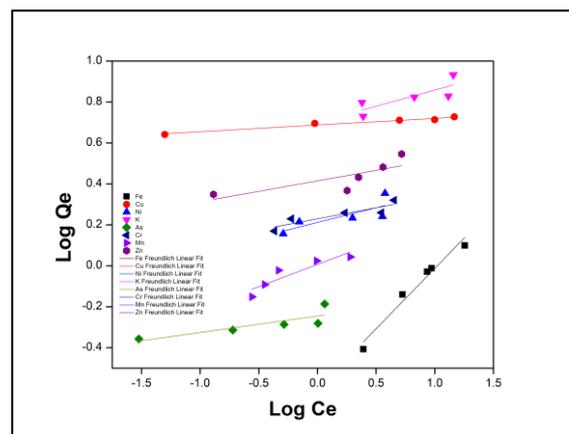

Fig. 5 Linear Freundlich Isotherm Fit of Fe, Cu, Ni, K, As, Cr, Zn and Mn Adsorption using Locally Produced (CSAC)

The results of the goodness of fit (R^2) to the Langmuir isotherm model for CS-AC are shown in Fig. 6. The results indicate CS-AC adsorption of Cu, Ni, Fe, K, Cr, As, Mn, Ni and Zn shows a good fit

to the Langmuir isotherm model. The R^2 values were between 0.926 and 0.999 respectively. This shows that 92.6 % and 99.9 % of the adsorption results using CS-AC is described by the Langmuir isotherm model.
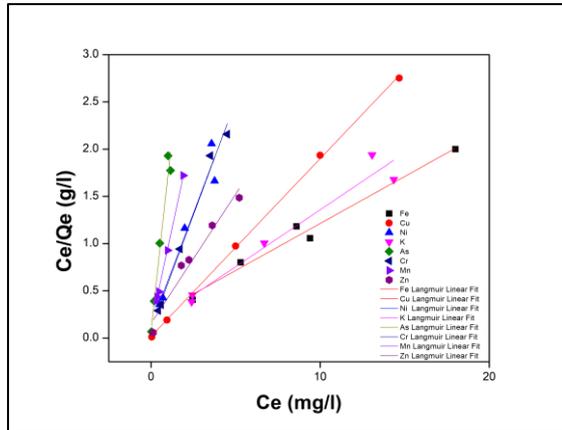

Fig. 6 Linear Langmuir Isotherm Fit of Fe, Cu, Ni, K, As, Cr, Mn and Zn Adsorption using Locally Produced (CS-AC)

Table 2 shows the Langmuir and Freundlich isotherm model constants, R^2, K_L, R_L, Q_m, K_F, $1/n$, obtained by linearly fitting the adsorption of Fe, Cu, Ni, K, As, Cr, Mn and Zn onto CS-AC. For a favourable adsorption, $0 < 1/n < 1$. From the results shown in Table 2, all values of $1/n$ for Fe, Cu, Ni, K, Cr, Mn, As and Zn satisfy the condition and therefore show a favourable adsorption condition. The value of K_F signifies the adsorption intensity and the higher K_F the higher the intensity of adsorption on the adsorbent. The results of the maximum adsorption capacities Q_m follows the trend Cu > As > Cr > Mn > Ni > K > Zn > Fe, showing CS-AC shows a higher capability of adsorbing Cu ions.

Table 2 Langmuir and Freundlich Isotherm Constants

	R^2	K_L	R_L	Q_m	R^2	$1/n$	K_F
Fe	0.97	2.18	0.01	4.60	0.96	0.59	2.28
Cu	1.00	0.10	0.25	55.52	0.96	0.03	1.88
Ni	0.93	0.29	0.25	7.35	0.52	0.14	1.20
K	0.94	1.27	0.02	6.53	0.55	0.16	2.14
As	0.96	0.05	0.87	12.19	0.60	0.08	0.77
Cr	0.98	0.21	0.31	10.06	0.75	0.11	1.31
Mn	1.00	0.16	0.54	7.45	0.79	0.22	1.40
Zn	0.94	0.57	0.10	6.42	0.51	0.10	1.53

The rates and equilibrium of HM adsorption depends upon the loading of HM already on the carbon i.e., the number of active sites available (Saleem *et al*., 2019). Fig. 7 shows the adsorption of

heavy metals (Fe, Cu, Ni, K, As, Cr, Mn and Zn) from the liquid phase OBM by CS-AC with respect to adsorbent dosage. The percentage of HM adsorbed by the CS-AC increases with increasing dosage with 100 %, 100 %, 97.38 %, 93.52 %, 99.81 %, 76.32 %, and 82.49 % for Fe, Cu, Ni, K, As, Cr, Mn and Zn respectively at CS-AC dose of 7 g/l. This may be attributed to the increase in total surface area for adsorption. The results show that, with increase in CS-AC dose up to 7 g/l, all Fe and Cu ions are removed from the liquid phase of the mud.
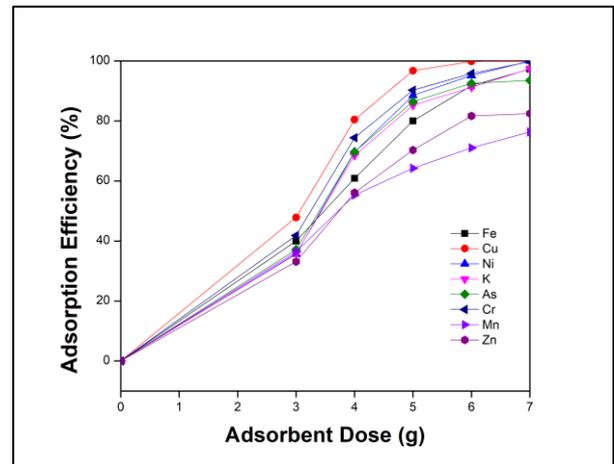

Fig. 7 Percentage Adsorption of Fe, Cu, Ni, K, As, Cr, Mn and Zn by CS-AC of varying Dosages (2, 3, 4, 5, 6, 7 g/l)

4 Conclusions

Heavy metals are present in waste oil-based mud in varying concentration levels and activated carbon produced had good adsorption characteristics for adsorbing these heavy metals from the mud. From the results it can be concluded that to reduce the heavy metal concentrations below threshold levels optimum CS-AC dosage required for Fe, Cu, Ni, K, As, Cr, Mn and Zn is 5 g/l for a contact time of 30 minutes. The characterization of the produced carbons shows good results comparable to other commercial activated carbons. The locally produced activated carbons from waste coconut shells are capable of removing heavy metals from the spent oil-based. The use of waste coconut shells will help to reduce the amount of waste in the environment while reducing the overall concentration of heavy metals in spent oil-based mud.

Acknowledgements

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