

Original Article

Production, Activation and Characterisation of PKS-Biochar from *Elaeis Guineensis* Biomass Activated with HCl for Optimum Produced Water Treatment

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Abstract - The treatment of oilfield produced water for reuse using activated carbons (AC) derived from palm kernel shells (PKS) biomass was studied. The biomass (PKS) was prepared and converted to biochar by pyrolysis and then activated with 0.2M HCl at a temperature of 450°C. The AC (adsorbent) produced was characterized for pH, bulk density, particle size, iodine number, ash and moisture contents. The characteristics of the adsorbent indicate that the iodine numbers ranged from 525.10-918.93mg/g for particle sizes 150-1180 µm); ash content ranged from 2.00-2.80% PKS. Moisture content could get as low as 1.6% for PKS when warmed in an oven at 105°C for 1 hour. The acid concentration and adsorbent particle size were optimized, and based on iodine number, the 300µm particle size (with pH = 7.4, iodine number=918.93, moisture=6.2% and %ash=2.00) showed a good prospect for removing contaminants from produced water. It is recommended that the conversion of *Elaeis Guineensis* biomasses, especially palm kernel shells (PKS), to activated carbon should be encouraged for oilfield produced water treatment in the production phase of the petroleum industry because of its high adsorptive capacity. To improve the adsorption capacity of the PKS, it is also recommended that any other stronger activation reagents should be used to activate PKS because of its bulk density.

Keywords - Activation, Activated carbon, Adsorption, *Elaeis guineensis*, Pyrolysis.

I. Introduction

Activated carbons (ACs) have been prepared from agricultural by-products (also known as agrowastes) such as sawdust, corn cob, coconut husk, waste bamboo, rice husks, nutshells, soya bean oil cake, *Elaeis Guineensis* biomass. "*Elaeis guineensis* is a species of oil palm, sometimes also known as African oil palm or macaw-fat" [1]. It is among the few tropical tree crops (like bananas and citrus) with high productivity in actual growing conditions, i.e. outside of test plots [2]. "The palm oil industry in Africa generates, by the processing of palm oil, a large number of biomass wastes such as palm kernel shell (PKS), empty palm fruit bunch (EPB), oil palm fibre, etc., whose disposal is a challenging task [3]. In fact, for every metric ton of palm oil produced from fresh fruit bunches, a farmer harvests around 6 tons of waste palm fronds, 1 ton of palm trunks, 5 tons of empty fruit bunches, 1 ton of press fibre (from the mesocarp of the fruit), half a ton of palm kernel endocarp, 250 kg of palm kernel press cake, and 100 tons of palm oil mill effluent" [4].

This is forcing an investigation into how these wastes can be turned into wealth by converting them into activated carbons. "It is evident from literature survey that various low-cost adsorbents produced from agrowastes have shown good potential for the removal of various

aquatic pollutants" [5]-[8]. Thus, the conversion of waste materials into ACs would add considerable economic value, help reduce the cost of waste disposal and, most importantly, provide a potentially inexpensive alternative to the existing commercial ACs [7]. These waste materials have proved to be promising raw materials for the production of AC with a high adsorption capacity, considerable mechanical strength, and low ash content" [7]. There are two basic steps for the preparation and production of ACs: (1) Carbonization/pyrolysis of the raw precursor material under anaerobic conditions below 1000 °C, and (2) Activation of the carbonized product (Biochar), which may be either physical or chemical. Mo *et al.* [9] noted particularly that "most agrowastes are not currently used in their raw, original state, but modified in a variety of ways to reinforce the porosity and adsorption surface area of the materials – nanostructuring, carbonization, activation, and grafting are some common modification technologies of agricultural waste adsorbents." They then summarized "the characteristic, preparation and application of adsorbents from various industrial wastes, including natural materials and biosorbents."

Preparation and Production of Biochar. "The carbonization process includes drying and heating to separate by-products, including tars and other



hydrocarbons, from the raw material, as well as to drive off any gases generated. The carbonization process is completed by heating the material at 400–600°C in an oxygen-deficient condition that cannot support combustion [10].

"Biochar preparation methods are mainly pyrolysis, hydrothermal carbonization, gasification, and other methods. Pyrolysis is a decomposition reaction under high temperatures and anoxic conditions. Pyrolysis is the thermo-chemical decomposition of organic material at elevated temperature in the absence of oxygen" [11]. "Pyrolysis is a thermochemical treatment that can be applied to any organic product. Based on pyrolysis time, temperature, and heating rate, it can be divided into slow pyrolysis, rapid pyrolysis, and 'flash' pyrolysis (heating rate up to 1000°C/s) [12]-[14]. Slow pyrolysis is "characterized by lengthy solids and gas residence times, low temperatures and slow biomass heating rates. In this mode, the heating temperatures range from 0.1 to 2 °C per second, and the prevailing temperatures are nearly 500°C. The residence time of gas may be over five seconds, and that of biomass may range from minutes to days. During slow pyrolysis, chars are released as main products as the biomass is slowly devolatilized [15].

Characteristics of Biochar. "Biochar, with high carbon content and void structures, has abundant aromaticity oxygen-containing functional groups. The Physico-chemical properties of biochar vary with the types of raw material, the particle size of the feedstock, the means of pyrolysis, the temperature (including the rate of temperature rise), the time of pyrolysis, and the modification conditions. Although the structure of biochar is affected by many factors, in general, biochar has abundant surface functional groups (hydroxyl, carboxyl, carbonyl, and methyl), a developed pore structure, a high specific surface area, and the stable molecular structure with good adsorption performance, which is favourable to adsorb pollutants" [16], [17].

Activation. "The carbonized product is 'activated' by exposing it to an activating agent, such as steam, acid, etc., at high temperature. The size of the pores developed during activation is a function of the time they are exposed to the activating agent. Longer exposure times result in larger pore sizes. The most popular aqueous phase carbons are bituminous based because of their hardness, abrasion resistance, pore size distribution, and low cost, but their effectiveness needs to be tested in each application to determine the optimal product" [10]. "Because the product will not be porous, it needs additional treatment or activation to generate a system of fine pores. The yield and the characteristics of the prepared activated carbon, however, depend on the process of preparation used. Adsorption capacity is enhanced by activating the surfaces" [18], [19]. Activation of the carbonized product (char) may be either physical or chemical.

Physical activation is "a process in which the precursor is developed into AC using gases and is generally carried out in a two-step process. It involves contacting the char with an oxidizing gas, such as carbon dioxide, steam, air or their mixtures, in the temperature range between 600 and 900 °C, which results in the removal of the more disorganized carbon and the formation of a well-developed micropore structure" [18].

Chemical activation. "In this process, the two steps are carried out simultaneously, with the precursor being mixed with chemical activation agents (such as ZnCl₂, KOH, NaOH or K₂CO₃) at very low temperatures. This agent will increase the surface area and reduce the ash content of final carbonized products: the chemical incorporated into the interior of the precursor particles reacts with the products resulting from the thermal decomposition of the precursor, reducing the evolution of volatile matter and inhibiting the shrinking of the particles; in this way, the conversion of the precursor to carbon is high, and once the chemical is eliminated after the heat treatment, a large amount of porosity is formed" [20].

"After activation, the char may then be processed into two finished products: a powdered form called PAC (powdered activated carbon) and a granular form called GAC (granular activated carbon). PAC is normally less than 200 mesh, and GAC greater than 0.1 mm in diameter" [18].

Characterization. The characterization of activated carbon involves determining such properties as particle size distribution, surface area, apparent and bulk densities, "pore volume, percentage burnt off, moisture and ash contents, methylene blue adsorption, and iodine number" [21], [22]. Ademiluyi & Nze [23] characterized "activated carbon from waste Bamboo used for the batch adsorption of Ni, Cu, Zn, Pb, Cd, and Cr ions in aqueous solution from refinery wastewater compared with reference activated carbon."

This study aims to characterize the PKS biochar produced from *Elaeis guineensis* biomass and activated with HCl for optimum produced water treatment.

2. Materials and Methods

2.1. Materials

The basic materials used in this work were: agro-waste [*Elaeis Guineensis* Biomass – PKS] and reagents (such as thiosulfate solution, Potassium Iodide (KI), and starch).

2.2. Methods

2.2.1. Production of Biochar from PKS by Pyrolysis

Carbonization of the PKS (2 kilograms of washed, cut, and dried oil palm biomass) was carried out in a local pyrolysis reactor. The agro-waste material was carbonized at about 300-350°C for two or more hours, after which the biochar was allowed to cool to room temperature. The charred material was crushed using a mortar and a pestle.

2.2.2. Activation of Biochar

The procedure for activating the biochar started with the dilution of the concentrated acid: the reagent (hydrochloric acid, HCl, of a particular concentration) was prepared from the concentrated acid. The volume of concentrated acid added to 1 litre (1000 ml) of distilled water was calculated. For the purpose of characterization and to determine the optimum concentration of HCl to be used to activate the biochar, 100g of the carbonized sample was carefully weighed and put in a beaker containing about 300 ml of dilute HCl acid. The five concentrations of the dilute acid considered were 0.05, 0.1, 0.2, 0.3 and 0.4M. The content of the beaker was thoroughly mixed until it formed a paste. The paste was then transferred to a crucible, which was placed in an electric furnace (Carbolite AAF1100) and heated to 450°C for 1 hour. The activated sample was then allowed to cool to room temperature before washing with distilled water to a pH of 7.0 – 7.5 and dried in the electrical oven (Memmert UN75) at 105°C for 1 hour. The pH tester used was the Erma Instruments pH tester calibrated using a 7.0 buffer solution prepared at 25°C.

Characteristics of the Activated Carbon

Characterization of the PKS-activated carbon involved the determination of properties such as particle size, bulk density, moisture content, ash content, benzene adsorption, methylene blue adsorption, iodine number, chemical composition and surface morphology, specific surface area, pore volume and pore size. The results of the determination of the chemical composition and surface morphology, specific surface area, pore volume and pore size of the adsorbent using X-ray fluorescence (XRF), scanning electron microscopy (SEM), etc., are not included in the paper.

Particle Size Distribution

Sieves were used to determine the following particle sizes: 75, 150, 300, 600, 1180, and 2360 µm. The sieves were arranged with the sieve with the highest mesh size (2360µm) on the top and the 75-µm mesh size at the bottom. Sieving was done by pouring the grind AC sample onto the topmost sieve, then with gentle shaking, the various particle sizes: 75, 150, 300, 600, 1180, and 2360µm were obtained.

Bulk Density

An empty test tube was weighed using the portable digital electronic weighing balance (Type: Professional Digital Table Topscale), and the tare mass in grams (g) was recorded. The tube was then filled to the brim with the sample, tapped 10 times gently on the table and weighed. The mass of the tube plus the sample in grams was also recorded. The calculation of the bulk density then followed.

Ash content: ASTM (D2866 Standard Method Test)

The mass of an empty crucible was determined with the aid of the "portable electronic weighing balance and

recorded as W_1 ." Five (5) grams of the sample of AC were measured into the crucibles, and the mass of the crucibles plus the samples were recorded as W_2 . The crucibles and samples were then put into the electric furnace (Carbolite AAF1100), set at 600°C for 6 hours. After 6 hours, the crucible plus dried or ashed samples were left in the furnace to cool to room temperature before they were removed from the furnace, weighed, and the masses recorded as W_3 . Calculation of the ash content was done using Equation 1:

$$\% \text{ Ash} = \frac{W_3 - W_1}{W_2 - W_1} \times 100 \quad (1)$$

Adsorption Capacity - Methylene Blue Test

This test was done using the AAS (atomic absorption spectrophotometer) to analyze the filtrate from the samples to determine the adsorption capacity of the adsorbents. One (1) gram of methylene blue dye was measured/weighed into 1000 ml of distilled water and stirred to mix. Then 30 millilitres of it was measured out into a new bottle, and 0.3 grams of the activated carbon was added to it and stirred to mix. The electric shaker (Stuart Orbital Shaker/ SSL1) was used at an appropriate rotation speed of 200 rotations per minute (200rpm) for 30 minutes to shake and mix samples properly, but the mixed samples were allowed for 2 hours before decanting. Using filter paper, filtration of each of the mixtures of methylene blue was done. The AAS was then used to analyze the filtrate, to measure the absorbance using a wavelength of 600 metres, "to determine the adsorption capacity of the adsorbents." The absorbance (result) for each sample was recorded, and then the determination of the methylene blue (MB) absorbance [as the difference between the blank and Absorbance (Abs) values; i.e. MB = Blank – Abs] followed.

Iodine Number Determination

The materials and reagents for determining the iodine number were thiosulfate solution, Potassium Iodide (KI), and starch. The thiosulfate solution was prepared by dissolving 17.6 grams of the thiosulfate crystals into 1 litre of distilled water and leaving it for 24 hours. A solution of Potassium Iodide (KI) was also prepared by dissolving 24g of KI in 1 litre of distilled water. Both solutions were kept for the same 24 hours. The indicator, the starch solution, was then prepared by dissolving 0.3 grams of it in 30ml of distilled water, with 70ml boiled distilled water added to it and stirred until properly mixed. At this point, 10ml of the filtrate was measured into a beaker, and 2-3 drops of the starch solution were added to the filtrate as an indicator. Titration of the thiosulfate solution against the filtrate of the KI solution was done until the filtrate turned colourless. The initial and the final volumes were recorded. The titre value, S , was obtained by subtracting the initial value from the final value and recording it. The procedure was repeated for the blank (which is the control without the adsorbent).

Moisture Content

The moisture cup was used for the moisture content determination. The procedure was simply to measure the mass of the empty moisture cup and record it as M_1 . Then, measure 5 grams of the sample of AC into the moisture cup, weigh the cup plus the sample and record it as M_2 . Put the moisture cup plus the sample into the electric oven (Memmert UN75) and heat it at 105°C for 3 hours until the moisture content is minimal; the moisture cup plus the sample was removed from the oven and weighed every 1 hour until the mass was constant. The moisture cups containing the dry samples were removed from the oven, weighed, and the masses were recorded as M_3 . Calculation of the moisture content (MC) was performed using Equation 2.

$$\% \text{ MC} = \frac{M_3 - M_1}{M_2 - M_1} \times 100 \quad (2)$$

3. Results and Discussion

3.1. Production of the Adsorbents from *Elaeis Guineensis* Biomass

The experimental results of the pyrolysis process performed on the *Elaeis Guineensis* agro-waste material, namely PKS, at the Petroleum Laboratory, Rivers State University (RSU), Port Harcourt, Nigeria, are presented in Figures 1 and 2.

From Figure 1, the pyrolysis time is a function of several factors, such as the pyrolysis temperature, the strength of the material, heat intensity, and draught (breeze/wind speed).

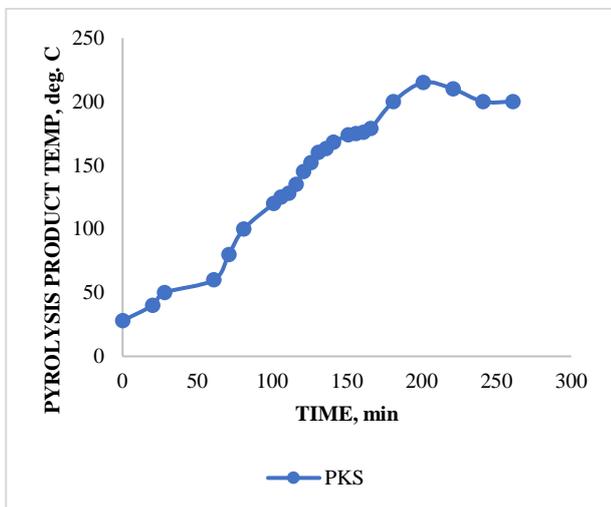


Fig. 1 A plot of pyrolysis product temperature versus time for the biomass of *elaeis guineensis* (PKS)

The temperature depends on the intensity of the heat energy used. The higher the temperature, the shorter the time taken to carbonize the biomaterial samples. The heat was applied to the pyrolysis reactor until the volume of bio-liquid recovered was constant, as can be seen in Figure 2. The palm kernel shell (PKS) is carbonized at more than 250 minutes because of its relative strength; the PKS is a strong material.

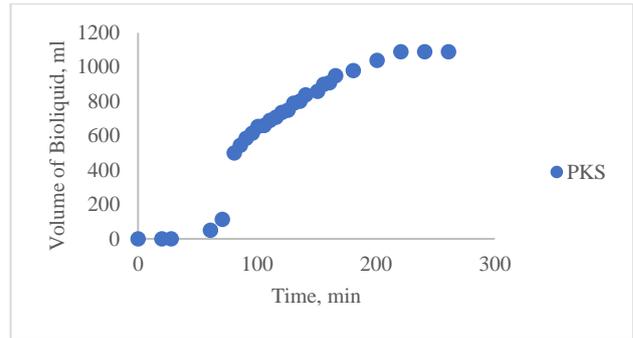


Fig. 2 A plot of the volume of bio-liquid recovered from pks biomass at various times during pyrolysis of samples.

The total volume of the bio-liquid recovered from the PKS biomass depends on the quantity that entered the pyrolysis reactor; the bio-liquid comprising mainly of oil and water was 1104.00ml. The distillation process will confirm the actual proportion of the liquids (volume of oil and water) recovered during the pyrolysis process. The components that could not be condensed were basically vapours (gases) produced when the agro-waste was subjected to heat in the absence of air. These bio-liquids can be refined into biofuels, but this work did not consider this aspect.

3.2. Particle Size Distribution

Sieves were used to determine the following particle sizes: 75, 150, 300, 600 μm , 1180 μm (1.18 mm), 2360 μm (2.36 mm), and remnant particle sizes ($> 2.36\text{mm}$, granular AC). The total mass of the biomass before the pyrolysis was 3.6 kg of PKS, pyrolyzed at a maximum temperature of 350°C . The total mass of biochar recovered after the process was 1.747 kg PKS, translating to 485.27g biochar/kg of PKS pyrolyzed.

3.3. Bulk Density of the Activated Carbon

The results of the weighing and computation of the bulk density of the various sample (sieved) sizes were also plotted in Figure 3. The average bulk density was determined as 1.1894 g/cm^3 . From the work of Ademiluyi and Nze [Ref. 22], activated carbon from Nigerian waste bamboo had an average bulk density of 0.458 g/cm^3 (reference range: $0.2\text{-}0.6 \text{ g/cm}^3$). Therefore, PKS having a bulk density of 1.1894 g/cm^3 , is a heavier material than the AC from waste bamboo. "Higher apparent or actual density provides greater volume activity and normally indicates better-quality activated carbon" [10].

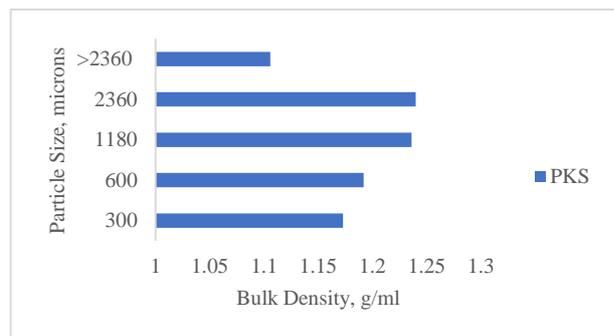


Fig. 3 Bulk Density of PKS AC samples according to their Particle Sizes

3.4. Results of the Activation of Samples

3.4.1. Optimizing Acid Concentration

The results (of the pH of activated samples for the 600-micron particle size after washing with distilled water) are plotted in Figure 4. The activated and washed samples were dried in an oven at 105°C for 1 hour. For economic reasons (that is, because of cost), one particle size (600 µm) was chosen and used – the intent was to find an optimum concentration of the HCl acid with which to activate all the particle sizes.

The observed trend of the pH versus acid concentration in Figure 4 is interesting. There was an initial gradual increase in pH with increasing acid concentration from 0.05M, stabilized from 2.0 to 3.0M, then dropped from 3.0M to 4.0M. This is in agreement with the work of Rabia *et al.* [Ref. 24]. What could be responsible for the fall? The gradual increase in pH with increasing acid concentration is correct, but acid concentration increasing beyond 3.0M may have caused starch to hydrolyze to glucose, thereby causing the drop in pH. From Ref. [24], the removal of nutrients/pollutants from a waste stream increases with increasing pH.

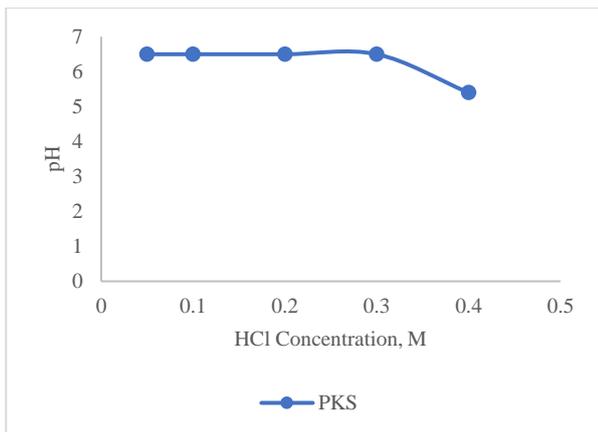


Fig. 4 pH of the washed 600-micron biochars activated at various acid concentrations

Following the above argument, the optimum acid concentration is 0.2M, as can be observed from Figure 4; the 0.2M concentration of the HCl acid is optimum and was thus used for the activation of all the sample sizes for the purpose of the treatment. The final activated products were used for the characterization.

3.5. Characteristics of the Biochar produced from *Elaeis Guineensis* Biomass – PKS

3.5.1. Iodine Number

From the results of the characterization of the biochar of the biomass (PKS), activated with different HCl concentrations, the plot of the iodine numbers is displayed in Figure 5. The acid concentration was optimized. The iodine number of 918.93 g of Iodine/kg of carbon, corresponding to an acid concentration of 0.2M HCl, is within the acceptable range (500-1200 g of Iodine/kg of carbon) [22], [25]. At 0.2M concentration of HCl acid, the iodine number is highest for the adsorbent. Therefore, the 0.2M HCl concentration (having the highest iodine

number) was chosen as the optimum concentration to be used to activate all the sample sizes for the treatment of the produced water for best adsorption. This is also in agreement with what was obtained in Figure 4.

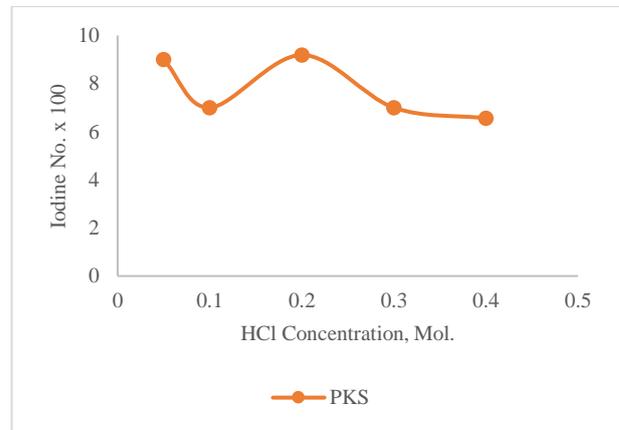


Fig. 5 Iodine number of biochars activated with different HCl Acid concentrations for the 600µm particle size.

3.5.2. Ash Content

The ash content for the PKS adsorbent at 0.2M acid concentration for the 600 µm size was 2.6%. This one particle size of the adsorbent was chosen just for the purpose of optimizing the concentration of the acid so that that particular concentration would be used to activate the adsorbent for the treatment. It is important to remark that ashing. It was performed at a lower temperature of 600°C but for a long time because the first samples done at a high temperature of 800°C cracked all the crucibles used.

3.5.3. Methylene Blue

Results of the Methylene Blue Test for 600 µm size were respectively 0.16, 0.26, 0.24, 0.28 and 0.26 for 0.05, 0.1, 0.2, 0.3, 0.4M HCl concentrations. Although these values are low due to the reagent used, it means that the PKS may have a good micropore structure that can adsorb adsorbate well. Some ACs have a mesopore "(2 to 5 nm) structure, which adsorbs medium-size molecules, such as the methylene blue dye" [10], [27].

3.5.4. Characteristics of Biochar Activated with 0.2M HCl

The characteristics of the particle sizes considered in terms of moisture content, ash content and iodine numbers were compiled from experimental results determined after the activation of all the AC samples with 0.2M HCl. Figures 6-8 show the moisture content, ash content and iodine numbers, respectively, of the Biochar activated with 0.2M HCl for the stated particle sizes.

Figure 6 shows the moisture content of biochar of different sizes activated with 0.2M HCl were plotted. It should be noted that the adsorbent is naturally hygroscopic, meaning it attracts a lot of moisture from the atmosphere. Therefore, the particle size chosen for the treatment of the waste (produced) water has to be dried in the oven at 105°C for at least 1 hour before use to reduce the moisture content.

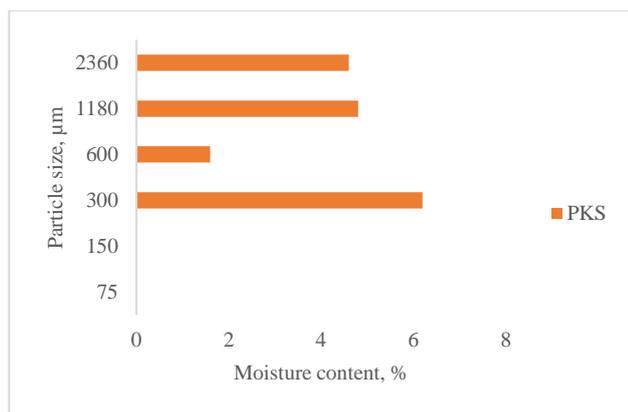


Fig. 6 Moisture content of biochars activated with 0.2M HCl.

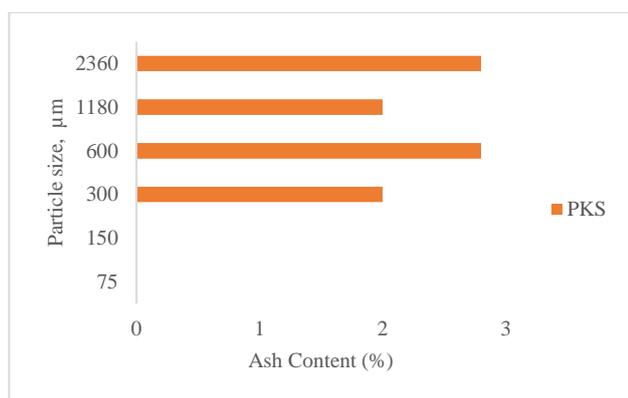


Fig. 7 Ash content of biochars activated with 0.2M HCl.

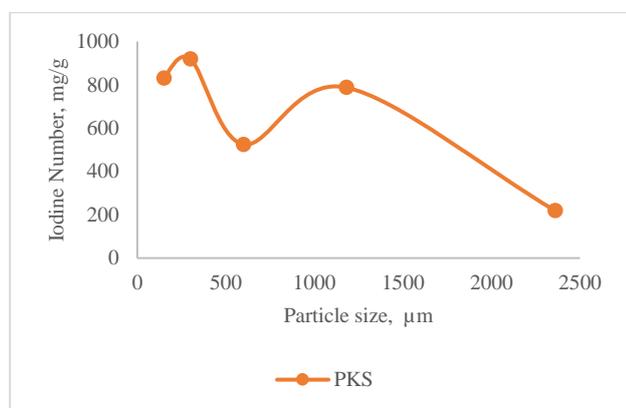


Fig. 8 Iodine number of biochar of different particle sizes activated with 0.2M HCl.

Figure 7 is a plot of the ash content versus particle size. As can be observed from the graph, the ash of the PKS-activated carbon is low. The ash "reduces the overall activity and efficiency of activated carbon reactivation. From the results obtained and presented in Figure 8, iodine numbers are observed to decrease steadily with increasing particle size for the activated carbon; a high iodine number means a larger surface area and greater adsorption capacity for the particle sizes.

The optimum particle size needs to be found since all the particle sizes cannot be used for the treatment for economic reasons. Looking at Figure 8, it was clearly obtained that the 300-micron particle size (with 918.93mg/g) has the highest iodine number for the AC (adsorbent) and was therefore chosen as the optimum particle size to be used for the treatment.

4. Conclusion

Activated carbon (AC) has been produced by pyrolysis from *Elaeis Guineensis* biomass (PKS), which has excellent qualities and can be used for produced water treatment for either disposal or reuse. The adsorbents' characteristics indicate that the iodine numbers ranged from 525.10-918.93 for PKS particle sizes 150-1180 µm); ash content ranged from 2.00-2.80%. Moisture content could get as low as 1.6% when warmed in an oven at 105°C for 1 hour. The acid concentration and adsorbent particle sizes were optimized and based "on iodine number and methylene blue" adsorption capacity. The 300µm particle size for the adsorbent showed good prospects for removing contaminants and can therefore be chosen to treat produced water. Therefore, the conversion of *Elaeis Guineensis* biomasses, especially palm kernel shells (PKS), to activated carbon should be encouraged for oilfield produced water treatment in the production phase of the petroleum industry because of its high adsorptive capacity. Hydrochloric (HCl) acid was used in this research work to activate the biochar in AC production. It is, however, recommended that the PKS, in further research, should be activated with a stronger acid or reagents to improve its adsorptive capacity.

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