Characterization of Activated Carbons Produced from Some Agricultural Residues

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Abstract Activated Carbon (AC) is used for absorbing substances of crystalline form, having a large internal pore structures that make the carbon more suitable absorbent. This study entails the experimental studies of the characterization of activated carbon produced from local materials which includes: activated carbon from coconut shell, coconut husk, maize husk and palm kernel shell.

Pyrolysis process was carried out using the pyrolysis setup on the agricultural residues sourced from local farm to produce activated carbon. The carbons were activated through chemical activation process using potassium hydroxide (KOH) and phosphoric acid (H_3PO_4) as activating agents at room temperature. Iodine value and surface area of the activated carbons produced and imported activated carbon were investigated.

The mass of activated carbons produced from coconut shell, coconut husk, maize husk and palm kernel shell are 688.21 g, 539.89 g, 482.53 g and 707.37 g, respectively. The percentage composition of Iodine value obtained from imported activated carbon (12.143 %), coconut husk (60.1 %), maize husk (38.1 %), palm kernel shell (24.286 %) and coconut shell (63.571 %) and the percentage surface area of imported activated carbon (42 %), coconut shell (21 %), coconut husk (24.1 %), maize husk (33.1 %) and palm kernel shell (37 %).

The results revealed that all the activated carbons produced has higher adsorption capacity than the imported carbon, that has the lowest adsorption activity level. Hence, the higher the iodine number the higher the rate of adsorption of activated carbon and locally produced carbon from agricultural residue can be used to replace the imported carbon. It will enhance the economy of the country by reducing the dependence on the foreign product and economy controlled by dollar index.

Keywords Activated carbons, agricultural residues, chemical and physical activations

1. Introduction

Activated carbon is used in gas purification, gold purification, metal extraction, water purification, medicine, sewage treatment, air filters in gas masks and respirators, filters in compressed air and many other applications. Activated Carbon filters have gained popularity among recreational users of Cannabis, and other smoking herbs for their use in effectively filtering out "Tar" from the smoke. They are becoming quick competition for vaporizers as they are only a fraction of the cost and achieve nearly the same thing [1].

Problems associated with the disposal of agricultural waste, especially the volume they occupy (about 96,000 tonnes of palm kernel shell and 7,000,000 tonnes of wet cocoa pod husk in 2011 alone) makes it imperative to find a way of reducing the size (volume) before disposal or finding an alternate use for them [2].. Public conflict arising from site selection for landfills and non-availability of land makes it important to find alternate ways of re-using waste [3]. Environmental issues arising from leachates from cocoa pod husk left on farm lands or dump sites to rot, makes it vital to find alternate use of cocoa pod husk.

The industrial demand, disposal problems and related environmental issues arising from the disposal of these agricultural wastes makes it imperative to turn such waste products into useful products (resources). In addition, agricultural wastes and by-products, hulls of almonds, hazelnuts, and coconuts, and stones of olives, apricots and peaches are also used in activated carbon production [4]. Davis (1880) [5] patented a process for gold recovery from chlorinated solution on wood charcoal.

Activated carbon can be produced from carbonaceous materials such as coconut shell, saw dust, agricultural residues [6-10]. It has been noted that effectiveness in impurity removal is a function of the material and the method of production [11-12]. In oil palm processing, palm kernel shell (PKS) is usually regarded as waste even though it is commonly used as fuel for cooking and in boiler firing during palm oil processing. Most times when individuals produce more PKS than they can immediately use, they heap up the remainder to decay over time in an open storage. By this the heating value is usually reduced thereby devaluing the PKS. An alternative use of PKS which is economically viable is the manufacture of good quality activated carbon due to its high density, high carbon and low ash contents [13].

Many Activated Carbons (ACs) from carbonaceous biomass have been produced and evaluated using different methods. Adewumni and Ogedemgbe (2005) [12] analysed factors which have prominent effect on the quality of Activated Carbon produced from raw or charred palm kernel shell (PKS) carbonized at 400 or 600 °C, for 20 or 40 minutes. It was suggested that optimum quality will be obtained by carbonizing the charred shells at 500 °C for 20 minutes after cessation of fumes and quenching the shells in alkaline water medium. In addition, drying the shells and pulverizing them to the desired grain sizes and activating the pulverized shells at 800 °C for 15 minutes in alkaline steam medium would enhance the quality.

Adewumi (2009) [10] carried out a survey of the sources and users of Activated Carbon in South-West Nigeria. He found that all the Activated Carbons were imported from Europe and Asia. There was no evidence of local production of this high-demand engineering material. Secondly, there was no standardization of the imported Activated Carbons either. Solution to these is local manufacture and characterization of Activated Carbons.

Methods implored in the production of Activated Carbons affect their adsorption capacity. Chemical and physical methods are available as production methods with chemical method widely used because of lower temperature activation and higher carbon yield than the physical method. Phosphoric acid (H_3PO_4) and Zinc chloride ($ZnCl_2$) are used more predominantly on a larger scale than other chemicals [14].

Since most ACs sold in Nigeria and Ghana [15] and probably West Africa are imported, the need to produce and characterize Activated Carbons locally becomes imperative. This will not only improve the water treatment technology of these countries but also reduce the foreign exchange spending on importation of this industrial raw material. It is therefore the aim of this work to produce and characterize Activated Carbon at laboratory scale) using different agricultural residues.

2. Materials and Methods

The leachate from agricultural residues was used as a chemical activating agent for the production of activated carbon. The chemical method of producing activated carbon was employed using coconut shell, coconut husk, maize husk and palm kernel shell as the raw material.

Five different impregnation ratios were employed at room temperature and at 85 °C and the samples carbonised at between 600 °C and 800 °C. The effect of these varying operating parameters on yield and quality of activated carbon were analysed. The quality parameters of the characterised activated carbon were compared to quality parameters of activated carbon used in the industry. Pyrolysis of coconut shell, coconut husk, maize husk and palm kernel shell were investigated using the pyrolysis setup as shown in Figure 1.

2.1. Components of the Pyrolysis Equipment

The components of the pyrolysis equipment are as follow: electrically operated furnace, retort, condensate receiver, pressure gauge, gas cylinder, pyrometer, contactor, gas collection unit, ice bath, galvanized iron hose, rubber gasket, clips, thermostat, heating element, bolts and nuts, lagging components, weigh balance, stopwatch and moisture content extraction oven.

2.2. Description of Major Pyrolysis Components

The pyrolyzer is the equipment used for converting biomass into useful fuels in the absence of oxygen. The major components are discussed as follow:

2.2.1. Metal Furnace: The furnace was constructed using a metal sheet made of mild steel of high heat resistance; the complete metal furnace. The furnace consists of the following;

Galvanized sheet metal: It serves as a casing or housing for the furnace.

The furnace: Has four tyres that make it to be mobile.

Pyrometer: It is fixed to the furnace to indicate the temperature at which pyrolysis is taking place.

Contactor: This is used to maintain the specific temperature at which pyrolysis is taking place.

Clay: The clay serves as a heat retainer to the generated heat by the heater. It is used because of its low thermal conductivity.

Glass Fiber: It prevents or it minimizes heat transfer from the inner part to the outer. It is lighter in weight, thereby reducing the total weight of the whole furnace.

Heater Band: The function of this is to supply the necessary heat needed for the pyrolysis process.

2.2.2. Retort

The retort is a cylinder container which contains the residues to be pyrolyzed and is fabricated using mild steel. Mild steel is used because of its high melting point (1400-1600 °C), tensile strength, good machinability and good resistance to corrosion.

Gas Collection Unit: The gas collection unit is the unit which receives the non-condensable gases from the condensable receiver structure. It is fabricated with galvanized sheet metal because of its good welderability, bursting, good machinability, heat resistance and corrosion resistance and it is readily available in the market.

Condensate Receiver: The gas coming from the heated material in the furnace is condensed in the condensate receiver which on it is a pressure gauge to know when the pressure in the condensate receiver is increasing.

Gas Cylinder: The gas cylinder serves as storage for gas it receives from the gas collection unit. It is connected to the gas collection unit in order to collect the filtered gas from the gas collection unit.

2.3. Raw Materials Preparation and Analysis

2.3.1. Production of activated carbon

The palm kernel shell samples were sourced from Sabo market in Ogbomoso Oyo state, Nigeria. The following steps are taken for the carbonization process; The palm kernel shell was cleaned from other soil material. The palm kernel shell was crushed into smaller sizes. It was then sun dried. The palm kernel shell was weighed using a sensitive electronic weighing balance and recorded. The palm kernel shell was then loaded into the retort. The furnace was switched on and allowed to reach the temperature of 600 °C. The retort was then placed into the furnace and closed. The PKS was allowed to burn for 55 minutes. The retort was then brought out of the furnace after 55 minutes and allowed to cool for about 3-5 minutes before unloading the retort. The charcoal formed was weighed using an electronic weighing balance and sealed in a nylon to prevent contamination and inlet of oxygen (O_2). The pyrolysis set up is shown in Figure 1.

Figure 1: Complete Pyrolysis Experimental Setup

The coconut shell samples were obtained from Sabo market in Ogbomoso Oyo State, Nigeria. Carbonization of precursor was carried out at a temperature of 600 °C, for coconut shell, the carbonization was achieved in a furnace for 30 minutes. The coconut shell was cleaned from other coconut fibres or soil. The coconut shell was crushed into smaller sizes. It was then sun dried. The coconut shell was weighed using a sensitive electronic weighing balance and recorded. The coconut shell was then loaded into the retort. The furnace was switched on and allowed to reach the temperature of 600 °C. The retort was then placed into the furnace and closed. The coconut shell was allowed to burn for 30 minutes. The retort was brought out of the furnace after 30 minutes and allows to cool for about 3-5 minutes before unloading the retort. The charcoal formed was weighed using an electronic weighing balance and sealed in a nylon to prevent contamination and inlet of oxygen (O_2) [7, 16]. The pyrolysis set up is shown in Figure 1 and the experiments were carried out at the department of Mechanical Engineering, LAUTECH. Ogbomoso, Oyo state, Nigeria. The same procedures were repeated for coconut husk and maize husk.

2.4. Carbonization and Chemical Activation

The sample materials were carbonized in the absence of air in a muffle furnace at a temperature of 550- 700 °C for 30 minutes and the 600 g of carbonized sample was mixed with an aqueous solution of phosphoric acid (activating agent). The mixture was then subjected to heat at a temperature of 120 °C for 3 hours to vaporize the water.

2.5. Activation process for coconut shell and Palm kernel shell

In the activation process of both coconut shell and palm kernel shell, two chemical are used are Phosphoric acid (H_3PO_4) and Potassium hydroxide (KOH). The volume of the Phosphoric acid diluted was 6.8 cm^3 or 0.0068 dm^3 in distil water, using equation (2.1)

$$C_1V_1 = C_2V_2 \qquad (2.1)$$

For the Phosphoric Acid (H_3PO_4) calculation, Concentration of the acid C_1= 14.7 M, Volume of acid V_1=?, Final concentration of the acid C_2 = 0.1 M, Final volume of acid V_2= 1 dm^3, $14.7 \times V_1 = 0.1 \times 1$.

V_1= 0.0068 dm^3 = 6.8 cm^3 Since, 1 dm^3= 1 litre

For the amount of Potassium Hydroxide (KOH) required:

The mass of the KOH to be dissolve in 1 litre of distil water was 5.87 g. Therefore, for the acid 6.8 cm^3 of H_3PO_4 was diluted with 1 litre of water and for the base (KOH) 5.87 g was dissolved in 1 litre of water. After the carbonization of the two materials (Coconut and Palm kernel shell). The Materials are activated using Phosphoric acid (H_3PO_4) and Potassium Hydroxide (KOH) which are diluted with water to reduce the concentration of the Acid (H_3PO_4) and Base (KOH). About 6.8 cm^3 of (H_3PO_4) was diluted in 1 dm^3 of water and about 5.87 g of KOH was diluted in 1 dm^3 of water.

The following steps are taken for the charcoal activation; The charcoal of both Coconut shell and Palm kernel shell were soaked in H_3PO_4 for 24 hours. The activated Carbon was dried in an ovum for about 1 hour for each material (Coconut shell and Palm kernel shell) to obtain the initial mass of the material before activation. Initial mass of coconut shell, coconut husk, maize husk and palm kernel shell are 688.21 g, 539.89 g, 482.53 g and 707.37 g, respectively. The activated charcoal was then washed with water and KOH, to neutralize the material been activated. The activated charcoal was place outside for about 3 hours to evaporate the moisture in it. The activated charcoal dried in an ovum for 1-2 hours with a temperature of about 170 °C. The activated charcoal was grounded to a smaller size and then, was packed in a dry container.

2.6. Chemical Activation

The precursor was impregnated at room temperature for 24 hours with diluted K_3PO_4 and K_3PO_4 leached from coconut shell ash at an impregnation ratio (I.R). of 1.0. The samples were then dried in an oven at 150 °C for 2 hours. The impregnated samples were carbonised and activated simultaneously in a Nabertherm muffle furnace heated at a rate 8 °C/min. until the final activation temperatures (600 °C, 700 °C and 800 °C) were attained. The samples were maintained at those temperatures for a further 2 hours and then allowed to cool to room temperature. The prepared carbon was washed thoroughly with distilled water to remove residual chemicals and dried at 110 °C for 24 hours.

2.7. Characterisation of the Activated Carbon
2.7.1. Proximate Analysis – Moisture Content Determination
The aluminium dish was first weighed, with the first one weighing 14.57 g, second dish weighing 14.46 g and third dish weighing 15.27 g. A mass of 3 g of the coconut shell was added to each of the three dishes and they were reweighed again, before drying for a time limit of 30 minutes. After 30 minutes the dishes are weighed again until the weight of the dishes contain the coconut shell are stable.

Also, a mass of 5 g of palm kernel shell was added to the dishes, before drying for 30 minutes and it was reweighed again. After the final weight has been determined, to obtain the actual mass of the material (coconut shell and palm kernel shell) weight of the aluminium dish is subtracted from the final weight.

Actual mass = weight of dish – final weight after dry
Therefore, the moisture content is calculated using, using equation (2.2);

$$Moisture Content = \frac{A - B}{A} \times 100\% \quad (2.2)$$

Where, A – Mass of the sample before drying, (g) and B – Mass of the sample after drying, (g)

2.7.2. Procedure for iodine solution test on activated carbon
Iodine indicates the micro pore (0 - 20 A) content in the activated carbon. Reagents used in the iodine value test are as follow:

Step 1:
10 cc of 0.1 N Iodine solution was put into a conical flask and drops of starch solution was added to it. The pale yellow colour of iodine solution turns blue. Titration of the formed solution was done with 0.05 N Sodium Thiosulphate ($NaS_2CO_3.5H_2O$) till it becomes colourless. Burette reading corresponds to blank reading (B) was taken

Step 2:
0.1 gm of Activated carbon was weighed accurately. It was poured into a dry flask. 10 cc of 0.1 N Iodine solution was added into the flask containing the activate carbon. The flask was shaken properly for 4 minutes and then filtered. The filtrate was the titrated against standard Sodium Thiosulphate ($NaS_2CO_3.5H_2O$) using starch as indicator. Burette reading corresponds to A was taken. In calculating the Iodine valve (ID) using equation (2.3)

$$ID = C \times Conversion\ factor\ (mg/gm)$$

Conversion factor =

$$\frac{Molar\ weight\ of\ Iodine(127) * Normality\ of\ Iodine}{Weight\ of\ carbon * Blank\ reading} \quad (2.3)$$

Where Blank reading = B and Burette reading = A

$$C = B - A \quad (2.4)$$

Iodine adsorption number
This is the most fundamental parameter used to characterize activated carbon performance. It is a measure of activity level (Higher degree indicates higher activation), often reported in mg/g (with typical range of 500 – 1200 mg/g). It is a measure of the microspore content of the activated carbon (values > 0 to 20 AO, or up to 2 nm) by adsorption of iodine from solution. It is equivalent to surface area of activated carbon between 900 m^2/g and 1100 m^2/g [17].

2.7.3. Surface Area
The specific surface area of activated carbon was estimated using sear method by agitating 1.5 g of activated carbon samples in 100 ml of dilute hydrochloric acid at pH= 3. Then a 30 g of sodium chloride was added while stirring the suspension. The volume was made up to 150 ml with deionised water. The solution was titrated with 0.1N NaOH to rise the P^H from 4 to 9 and volume V recorded.

The surface area according to this method was calculated as:

$$S = 32V - 25 \qquad (2.5)$$

Where S = surface area of activated carbon, V = volume of sodium hydroxide required to raise the pH sample from 4 to 9. The value 32 in equation (2.5) is a dimensional constant measured in per centimetre (cm^{-1}).

3. Results and Discussions

3.1. Moisture content.

The following results were obtained for the moisture contents present in all the raw materials used (coconut shell and palm kernel shell). Moisture content for coconut shell, coconut husk, maize husk and palm kernel shell are presented in Figure 2.
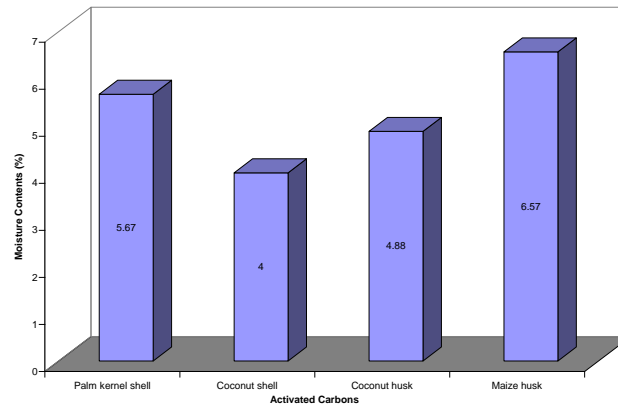
Figure 2: Percentage of moisture contents in the raw materials

The percentage moisture content of the activated carbon produced from coconut shell (5.67 %), coconut husk (4.88 %), palm kernel shell (4 %) and maize husk (6.56 %) were shown in Figure 2. The results revealed that maize husk (6.56 %) has higher moisture content than others. Hence, higher moisture content in the material used lowers the amount of char to be produced.

3.2. Pyrolysis of Coconut shell and Palm kernel shell

The result of the pyrolysis of coconut shell, coconut husk, maize husk and palm kernel shell for the production of activated charcoal, the mass by percentage of raw materials with corresponding activated carbon are presented in Figure 3.
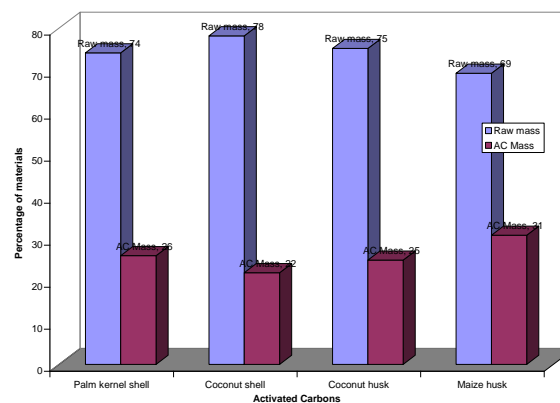

Figure 3: Percentages of raw materials and activated carbon masses produced

The percentages of mass of activated carbons produced from coconut shell, coconut husk, maize husk and palm kernel shell are 26 %, 22 %, 25 % and 31 %, respectively with corresponding percentage of mass of raw materials as 74 %, 78 %, 75 % and 69 %, respectively as presented in Figure 3. The results revealed that maize husk (31 %) has higher activated carbon than others in one run.

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3.3. Characterization of activated carbon produced

3.3.1. Iodine value

The Iodine value for all the produced activated carbon from local materials and imported activated carbon were obtained as shown in Figure 4.3. The percentage composition of Iodine value obtained for imported activated carbon (12.143 %), coconut husk (60.1 %), maize husk (38.1 %), palm kernel shell (24.286 %) and coconut shell (63.571 %) as presented in Figure 4.
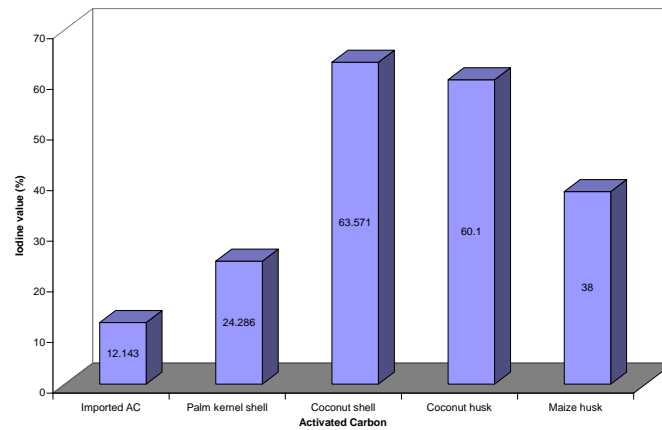

Figure 4: Iodine values of different activated carbon samples

The Iodine value for the imported activated carbon as shown in the result was very low (128.512 mg/g) compared to the produced activated carbon from coconut shell and palm kernel shell which were 672.79 mg/g and 257.02 mg/g, respectively. Since, the material with the highest Iodine value shows highest activation (i.e. the higher the degree of Iodine value indicates higher activation), hence, the imported activated carbon have the lowest activation which was 128.511 less than that of the palm kernel shell and also about 544.278 less than that of the coconut shell.

3.3.2. Surface Area

The surface area of surface area of imported activated carbon, coconut shell and palm kernel shell are determined and the percentage surface area of imported activated carbon (42 %), coconut shell (21 %), coconut husk (24.1 %), maize husk (33.1 %) and palm kernel shell (37 %) are as presented in Figure 5.
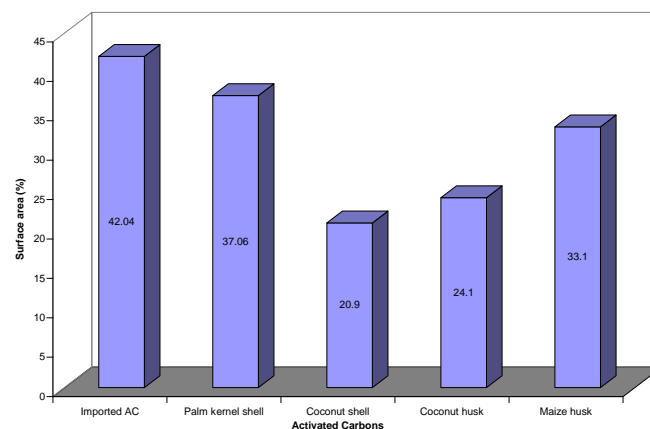

Figure 5: Surface area of different activated carbon

The smaller the surface area of a material, the higher the adsorption capacity within the material, from the result it can be deduce that the imported activated carbon have the highest value of surface area due to the size of the particle compare with the produced activated carbon. It can also be deduce the surface area for coconut shell is lower than that of imported activated carbon and other produced activated carbons.

3.3.3. Electron microscope image of Activated Carbon sample

The images of the micropore structure for the produced activated carbon from coconut shell, palm kernel shell, unactivated carbon and imported activated carbon were studied using SEM are presented in Figure 3.5, 3.6, 3.7 and 3.8, respectively.
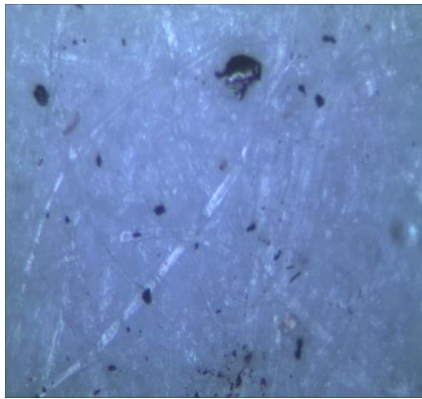

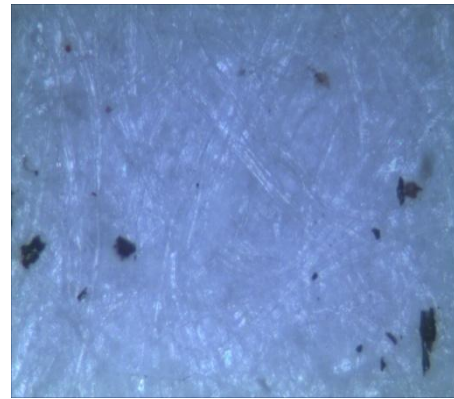*Figure 6: Activated carbon from Coconut shell (mg ×10)*

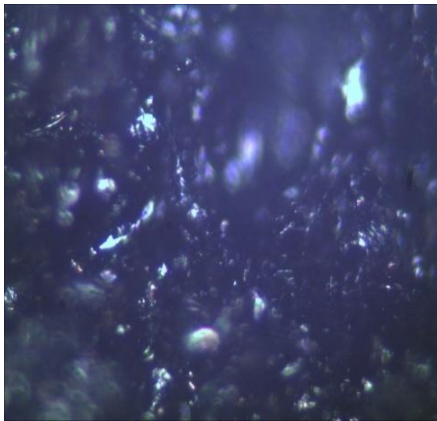*Figure 7: Activated Carbon from Palm Kernel shell (mg ×10)*

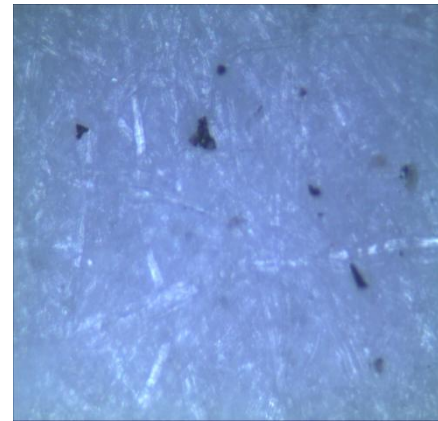*Figure 8: Unactivated Carbon (mg ×10)*

Figure 9: Imported Activated Carbon (mg ×10)

The micro pore space structure on Figure s 3.5 and 3.6 represent activated carbon produced from coconut shell and palm kernel shell respectively and the micro pore space structure on Figures 3.7 and 3.8 represent unactivated carbon and imported activated carbon respectively. Comparing pore space of the produced activated carbon with the unactivated charcoal or coal, it can be seen that the open pore space for the unactivated coal was smaller, but that of the produced activated carbon was larger.

But it can be seen from Figure 3.5 (coconut shell) that the pore space were larger than that of on Figure 3.6 (palm kernel shell), which implies that coconut shell have a larger pore space than that of palm kernel shell. In relation with the surface area of activated carbon from coconut shell and palm kernel shell, it can be deduced that activated carbon from coconut shell is having a surface area of 403.8 cm^2, compare to that of palm kernel shell of 227.8 cm^2.

Comparing the unactivated material (Figure 3.7) and produced activated carbon with the imported activated carbon (Figure 3.8), it can be seen that the pore space is the largest of all, which implies that the surface area is the largest (458.2 cm^2) as calculated and it also have the largest pore space as shown on Figure 3.6.

4. Conclusions and Recommendations

4.1. Conclusions

The following conclusions were drawn from the work carried out. The mass of activated carbons produced from coconut shell, coconut husk, maize husk and palm kernel shell are 688.21 g, 539.89 g, 482.53 g and 707.37 g, respectively. The investigation of the activity level of each of the produced activated carbon through iodine test,

shows that the level of adsorption activity of the produced activated carbon from local material is higher than that of the imported activated carbon, hereby giving the activated carbon produced from coconut shell and palm kernel shell an edge over the imported activated carbon in terms of activity level. The percentage composition of Iodine value obtained from imported activated carbon (12.143 %), coconut husk (60.1 %), maize husk (38.1 %), palm kernel shell (24.286 %) and coconut shell (63.571 %).

Activated Carbon produced from local materials can perform the same function as the imported activated carbon. And locally produced carbon from agricultural residue can be used to replace the imported carbon. It will enhance the economy of the country by reducing the dependence on the foreign product and economy controlled by dollar index.

4.2. Recommendations

The following future work can be carried out later; Investigations can also be carried out on how to improve the adsorption capacity of activated carbon produced locally and further experimental work could be done on the determination and comparison of the thermal properties of the activated carbon in term of adsorption rate.

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