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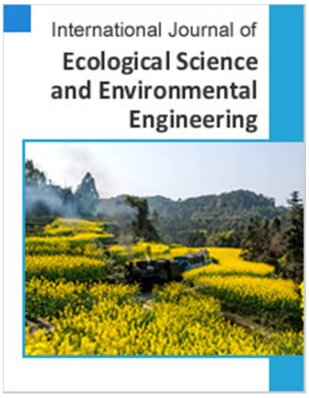

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Preparation and Characterization of Activated Carbon from Avocado Pear (*Persea Americana*) Seed Using H_2SO_4, HNO_3, and H_3PO_4 Activating Agent

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Abstract
Activated carbon is a porous carbonaceous material with high adsorption capacity which can be used as adsorbent material for purification of liquids and gasses. In this research work, production of activated carbon from avocado pear *(Persea americana)* seed using different activation agents was investigated. The study was aimed at comparing various chemical activation agents in order to determine the most suitable chemical activation agents for the preparation of activated carbon from an avocado pear seed. The activating agents used were H_2SO_4, HNO_3 and H_3PO_4. In the course of this study, waste avocado seeds were carbonized at 255°C and activated with different activating agents at 600°C to produce activated carbon. The effect of various activating agents on percentage yield, ash content, percentage burn-off, bulk density and adsorption efficiency in terms of iodine number was characterized. From the analysis, it was observed that using H_2SO_4 as an activating agent at activation temperature of 600°C gave the highest value of bulk density increasing according to the impregnation ratios of 0.05, 0.1, 0.2, 0.3, and 0.4, with the values ranging from 0.29 to 0.34g/cm^3 followed by activating agent of HNO_3 ranging from 0.27 to 0.33 and H_3PO_4 having 0.28 to 0.32g/cm^3. The values obtained for percentage yield ranged from 63% to 77% and that of H_2SO_4 had the lowest percentage yield ranging from 65% to 69% which shows that the activation temperature at 600°C does not favour the impregnation ratios of H_2SO_4 but rather favours HNO_3 and H_3PO_4. With H_2SO_4, low percentage yield resulted in high percentage burn off. It was observed that the ash content of each of the impregnation ratios of different activating agents was high because it ranged from 5% to 17.3%. The iodine number determined showed that iodine adsorption of activated carbon prepared with H_2SO_4 with activation temperature at 600°C is the best because the micropores content on the surface of activated carbon is higher especially at the impregnation ratio of 0.2M which had 1713.28g/kg and that of HNO_3 had the lowest value though chemical activation values were better than thermal valves. From the result, H_2SO_4 as activating agent was found to be the best for the preparation of activated carbon from waste avocado seeds.

1. Introduction

Activated carbon (AC) is a porous carbonaceous material which has a high adsorption capacity to be used as adsorbent in industries for various purposes such as extraction of organic and inorganic pollutants from aqueous or gas phase, removal of highly odorous dissolved organic compounds from industrial wastewater, removal of heavy metals from groundwater, sugarcane juice decolourization, as well as pharmaceutical and agro based industrial wastewater treatment (Lynch, 2001; Marsh and Rodriguez-Reinoso, 2006; Van der Hoek, 1999; Wrench, 2007; Adie et. al., 2012; Ademiluyi et. al., 2009). Recently activated Carbon is applied in the effective filtering out of tar from smoke among recreational users of Cannabis, and other smoking herbs. In the medical field activated carbon is used to treat poisonings and overdoses following oral ingestion. It is thought to bind to poison and prevent its absorption by the gastrointestinal tract. In cases of suspected poisoning, medical personnel administer activated charcoal on the scene or at a hospital's emergency department. Dosing is usually empirical at 1 gram/kg of body mass (for adolescents or adults, give 50–100 g), usually given only once, but depending on the drug taken, it may be given more than once. In rare situations activated charcoal is used in Intensive Care to filter out harmful drugs from the blood stream of poisoned patients. Activated charcoal has become the treatment of choice for many poisonings, and other decontamination methods such as ipecac-induced emesis or stomach pumping are now used rarely.

Activated carbon is the most used adsorbents, not only because of its high adsorptive capacity and its intrinsic physicochemical properties such as porosity, specific surface area and the nature of its surfaces, but also because of its availability and affordability which are functions of the abundance of the raw material from which it is manufactured (Kadirvelu et al., 2003; Marsh and Rodriguez- Reinoso, 2006; Sircar et al., 1996). It can be prepared from virtually all carbonaceous materials especially from amorphous carbonaceous materials including nutshells, coconut husk, peat, wood, coir, lignite (brown coal), and petroleum pitch (Hu *et al.,* 2003; Hu and Srinivasan, 2001; Tseng, 2007; Wu and Tseng, 2006). Most commercial activated carbons (CAC) are made from coal, peat or petroleum pitch and have proven to be good adsorbents but are relatively expensive (Netzer and Hughes, 1984; Reed and Arunachalam, 1994; Han et al., 2000; Daorattanachai et al., 2005; Ayotamuno et al., 2007). This fact has led to on-going researches into the feasibility of alternatives to CAC. The most available and affordable carbonaceous materials are agricultural wastes hence; they can be used for the manufacturing of activated carbon (Ademiluyi et. al., 2009). The use of agricultural waste as precursors of activated carbon also meets the 11^{th} sustainable development goal (SDG) of the United Nations agenda 2030 which makes it even more viable economically and environmentally. Thus, a diversity of agricultural raw materials such as woods, palm kernel and coconut husk has been tested for their suitability as adsorbent in the elimination of contaminants (Ademiluyi et. al., 2009; Ayotamuno et al., 2007).

The general process involved in the production of activated carbon is similar despite the precursor. The process involves carbonization and activation. There are two types of activation processes; physical and chemical activation processes. Chemical activation is preferred over physical activation due to higher yields, greater surface area and better development of micro porous structures in carbon (Lua and Gua 2001). Also chemical activation requires lower temperatures for production of activated carbon (Wikipedia, Madu and Lajide 2013), hence it is environment friendly. On the other hand physiochemical characteristics of activated carbon produced are dependent on the precursor and the type of chemical used for the activation. Xiao et al., 2012, observed that using Lignin as the precursor and KOH and K_2CO_3 as activation agents; activated carbon produced using K_2CO_3 gave higher iodine number, greater surface area, and higher methylene blue number than those activated with KOH. Ademuluyi and David-West (2012) reported that with bamboo, coconut shell and palm kernel shell as precursors, and H_2SO_4, HCl, $ZnCl_2$, H_3PO_4, NaOH, and HNO_3 as activation chemicals, bamboo activated with HNO_3 gave the highest iodine number while palm kernel activated with ZnCl gave the least iodine number. Aki et. al., (2013) used HNO_3 and $(NH_4)_2S_2O_8$ as activation chemicals on olive stones. According to report the activated carbon produced with HNO_3 was a better adsorbent than activated carbon produced with ammonium persulphate. Zamora et al. (2000) studied the adsorption capacities of activated carbon from petroleum coke with $ZnCl_2$, NaOH, and H_3PO_4 as activating agents. The physicochemical characteristics determined for these activated carbons as well as scanning electron microscopy showed that H_3PO_4 was the best activation agent. Recent works by Wang et. al., (2010), Anisuzzaman et. al., (2015) and Yakut and El Deen (2016) also reported the use of H_3PO_4 with various precursors. From the above literatures, it can be concluded that the best activation agent is dependent on the precursor and the manufacturing parameters such as time, temperature etc. This research work was therefore focused on the production of activated carbon using avocado pear *(Persea americana)* seed and different activation agents. Therefore the objectives of this research work are; to prepare activated carbon from avocado pear seed as precursor using a laboratory pyrolysis system, to characterize avocado pear seed activated carbon with different activating agents so that it can be used in several industrial applications and to set up high adsorbent materials at lower cost from agricultural wastes in this case avocado pear seed.

2. Materials and Method

2.1. Preparation of Activated Carbon

Avocado pear of no particular specie gotten from the local market in Ikot Akpaden, Akwa Ibom State, Nigeria was used as the precursor material. Nitric acid (HNO_3), sulphuric acid (H_2SO_4) and phosphoric acid (H_3PO_4) all of analytical grade were used as activating agents. Other equipments used include a locally fabricated pyrolytic reactor with thermocouple, laboratory electrical oven, laboratory electrical muffle furnace (F6010-TS) and a spectrophotometer. Two electronic weighing balances were used. One was used to weigh the avocado seed before pyrolysis, while a more sensitive (+0.001) balance was used for other analysis. A step-wise process of chemical activation was employed in the production of the activated carbon.

2.1.1. Carbonization

Avocado seeds were washed, cut and dried in laboratory electrical oven for 2-3 hours during which gases were emitted and a black oily liquid was released from the seeds and collected in a measuring cylinder. The carbonization process was completed when no gaseous emission was observed and at which point the volume of oil collected in the measuring cylinder was constant for 30mins. Avocado seeds were carbonized at a temperature of 250 ± 5°C. Charred products formed during the carbonization process were allowed to cool to room temperature. The charred material was crushed using mortar and pistle and sieved with a manual standard mesh size of 2.80mm. Yield of pyrolysis (carbonization) was calculated from the weight before carbonization (wb) and after carbonization (wa). The % yield is thus calculated as stated in accordance with (Yoshiyuki and Yutaka, 2003). Yield (%) $=\frac{Wa}{Wb} \times 100$

2.1.2. Activation Processes

Carbonized avocado seed samples were activated chemically in various concentrations of 0.05, 0.1, 0.2, 0.3, and 0.4M of nitric acid, sulphuric acid, and phosphoric acid. Known grams of carbonized samples were weighed and mixed in separate crucibles containing the different concentrations of activating agents (nitric acid, sulphuric acid, and phosphoric acid) in the ratio of 1:2 (g/vol). Samples were impregnation for 4 hours after which samples transferred into a Petri dish and oven dried at 120°C for three hours before being placed into the furnace at 600°C for one hour. The activated carbon generated was then removed from the furnace and allowed to cool at room temperature and washed with distilled water to remove residual acid. Washing was completed when pH value of wash wastewater was 7. The activated carbon generated was then dried in an oven at 120°C for 4 hours and allowed to cool. The final product was kept in an air tight polyethylene bag for characterization.

2.2. Characterization of Adsorbent

2.2.1. Percentage Burn off

10g of each carbonized char were perfectly weighed and noted to be Wa and also allowed to pass through activating process step at 600°C for one hour. Thereafter the samples were weighed and noted as Wi.

$$\% \text{ burnoff} = \frac{Wa-Wi}{Wa} \times 100 \text{ (Itodo et al., 2010).}$$

Wa = weight of char after pyrolysis, Wi = weight of carbon after activation.

2.2.2. Determination of Ash Content

Ash content was determined using the standard test method-ASTM D2866-94. 10g activated carbon of different concentration was recorded as D_i and taken into the muffle furnace at the temperature of 600℃ for one hour. It was removed from the muffle furnace and allowed to cool to room temperature and was reweighed and recorded as D_f. The percentage ash content was determined as$=\frac{D_f}{D_i} \times 100$. Where D_i = initial weight of sample and D_f = final weight of sample.

2.2.3. Determination of Bulk Density

Bulk density was determined as stated in the work of Yoshiyuki and Yukata, (2003). An empty measuring cylinder was weighed and the weight was recorded. Activated carbon of different samples were placed into a plastic measuring cylinder of known weight and compacted by tapping on the bench top until the volume of the sample stopped decreasing. The mass and volume were recorded and bulk density was calculated as:

$$\rho = \frac{Mass}{Vol.Occupied}$$

2.2.4. Determination of Iodine Number

0.2g of activated carbon of different concentration of activation agent was properly mixed with 30ml of 0.1M of standard iodine solution in a beaker and left for 24 hours and then filtered using filter paper. 10ml of the filtered solution of different activated carbon samples was measured into a beaker and 2drops of starch solution was introduced into each solution as an indicator. Titration of the formed solution was done against 0.1M sodium thiosulphate till it became colourless and initial and final readings were taken for each samples. Calculation for iodine number is as follows

$$B - \frac{S}{B} \times \frac{V}{W} \times M \times 126.91$$

Where M= molarity, B= blank reading (ml), S= volume of iodine solution used (ml), W = weight of sample used (g). v = volume of the iodine used (ml)

2.2.5. Determinatioin of Percentage Yield

According to Anisuzzaman et al., 2015; the percentage yield of activated carbon to that of the original precursor with both weight on a dry basis can be %yield $= \frac{W_f}{W_i} \times 100$. Where; W_i=initial mass of dry impregnated sample, W_f = final mass of sample at the end of the activation process.

3. Results and Discussion

3.1. Effect of Activating Agents on Percentage Yield (%)

A high percentage yield is required for a feasible economic production of activated carbon. Activated carbon generated from Avacado pear seeds showed high yield (65-78%) similar to commercial activated carbon produced from soft coal (65-80%). As shown in Figure 1, the percentage yield is strongly influenced by the acid type and the impregnation concentration. Activation of the char with mineral acids decomposed the polymeric structure of the carbonized precursor. The acids served as shield and acidic catalysts in promoting several reactions including aromatic condensation reactions, aromatic substitution reactions, bond cleavage reactions and formation of cross links. Percentage yields of activated carbon from nitric and phosphoric acids progressively decreased with increased acid concentration. This can be explained by the effect of aromatic condensation reaction which is accompanied by dehydration and decarboxylation. It is such that as concentrations of nitric and phosphoric acids increased, dehydration and decarboxylation increased which led to the loss of small molecules and ultimately a lower % yield. The inverse relation of burn off and percentage yield with acid concentration supports this explanation (See Figure 2 and 3). On the other hand, as concentration of sulphuric acid increased the loss of small molecules was reduced and carbon was retained. Again this explanation is supported by the inverse relation of burn off and percentage yield with acid concentration (see Figure 4). However percentage yield values of activated carbon from nitric and phosphoric acid activation were higher than percentage yield values from sulphuric acid especially between 0.05M and 0.2M (see Figure 1). This was attributed to cross link formation along adjacent aromatic molecules and the shielding effect of amine and phosphate over the internal carbon structure to prevent excessive loss of carbon. The sharp drop in % yield from 76% to 68% between 0.2M and 0.3M as against the progressive decrease from 78% to 76% for 0.05M to 0.2M for phosphoric acid suggests that at concentrations greater than 0.2M that bond cleavage and polyphosphate substitution reactions occurred leading to the loss of carbon.

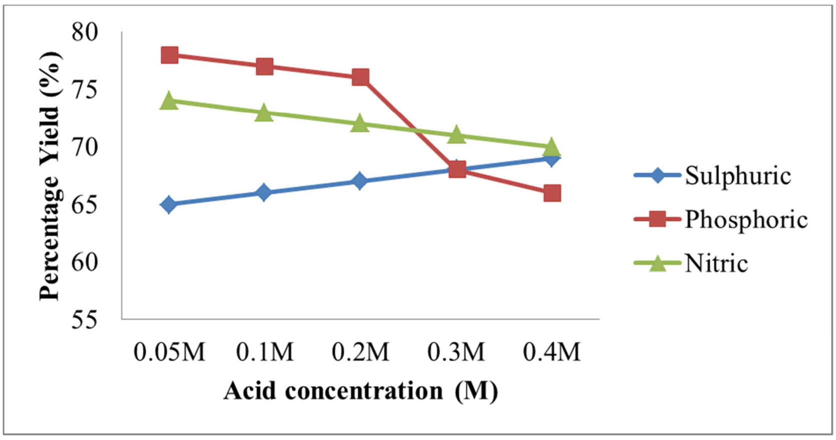

Figure 1. *Effect of activating agent on percentage yield (%) of activated carbon from avocado seed.*

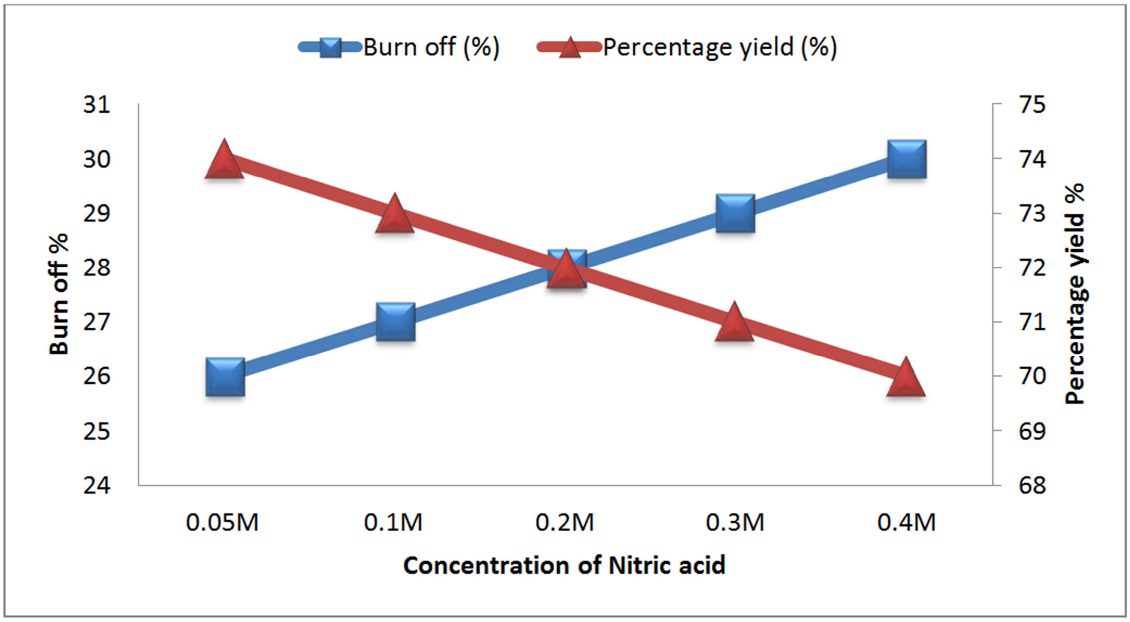

Figure 2. *Relation between burn off and percentage yield of activated carbon with HNO_3 concentration.*

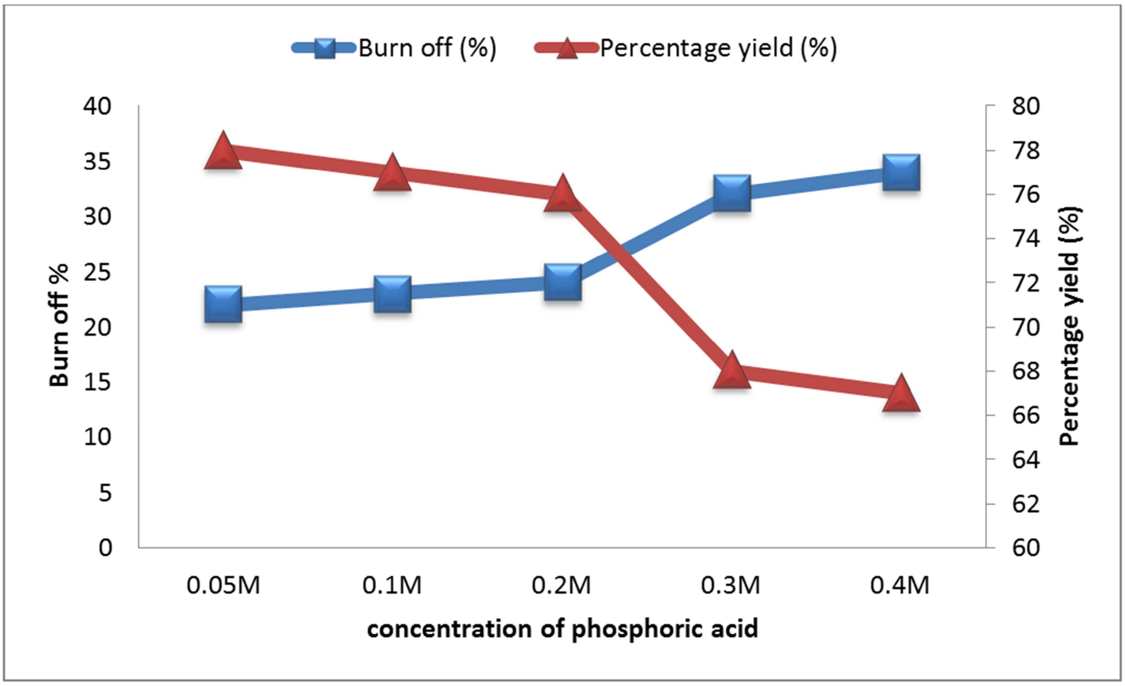

Figure 3. *Relation between burn off and percentage yield of activated carbon with H_3PO_4 concentration.*

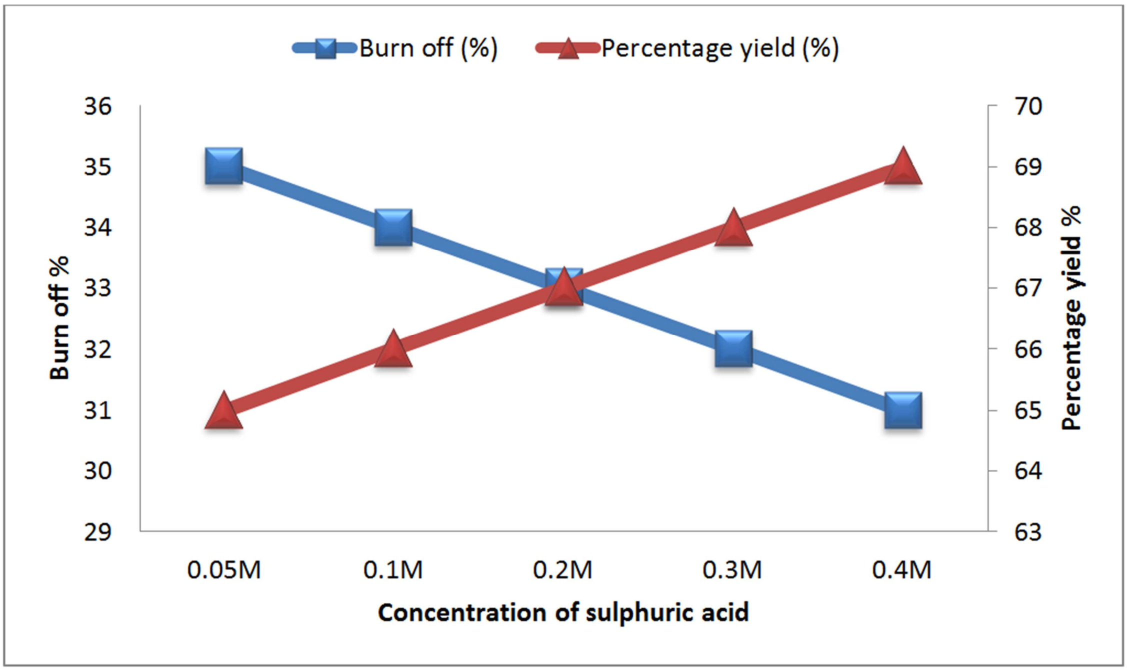

Figure 4. *Relation between burn off and percentage yield of activated carbon with H_2SO_4 concentration.*

3.2. Effect of Different Activating Agents on Ash Content of Activated Carbon

Ash content is one physiochemical property that influences the adsorptive capacity of activated carbons as it is directly related to porosity. When ash content of activated carbon is low, the adsorptive capacity is high due to increased surface area and high porosity. Ash content of activated carbon is dependent on the ash content of the precursor, a measure of activating agents (inorganic impurities) entrapped in the carbon structure (Bansode et al., 2003) and the time of activation. In this work, the ash content of activated carbon was notably high compared to previously recorded value of ash content for avocado pear seed of 2.4% (Arukwe et. al., 2012). The high ash content in this work is an indication that the one hour duration was not sufficient for complete vapourization of the inorganic minerals. However, the high ash content (5%-17.3%) was found to be in the range of most ash contents of activated carbon from agricultural waste.

3.3. Effect of Different Activating Agents on Bulk Density (g/cm^3)

Bulk density is an important characteristic of activated carbon as it is a measure of the amount of adsorbate the carbon can hold per unit volume. A high bulk density is an indication of better quality activated carbon because there is more available volume for adsorption. In this work it was observed that the bulk density of the generated activated carbon for all concentration of the different activating agents agrees well with that of commercial activated carbon. As shown in Figure 4, the bulk density of the chemically activated carbon was in the range of 0.27-0.34g/cm^3. The bulk density increased with concentrations of all activating agents. The highest value of bulk density was recorded for H_2S0_4 at 0.4M.

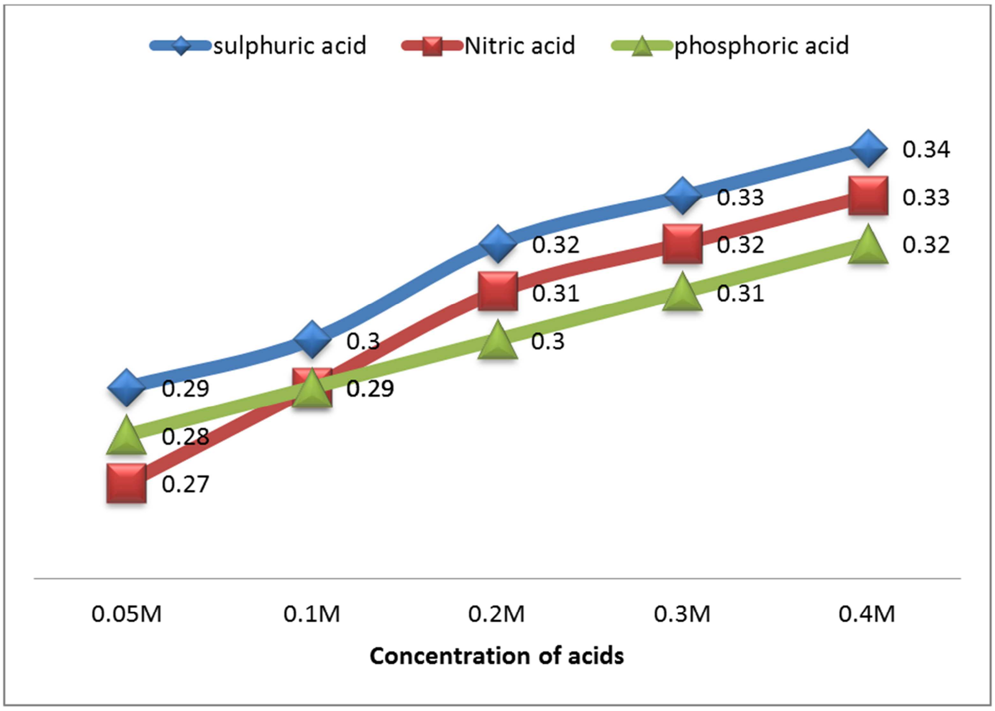

Figure 5. *Effect of activating agent on bulk density of activated carbon.*

3.4. The Effect of H_2SO_4, HNO_3, and H_3PO_4 Activation on the Iodine Number of Activated Carbon from Avocado Seed

Determining the iodine number is one of the methods to determine the adsorption capacity of activated carbons. It is a measure of the micropore content of the activated carbon by adsorption of iodine from solution. Iodine number for commercial adsorbent range from 300 to 1200g/kg and above is good. On the other hand the result of adsorptive property indicates that avocado seed is a very good source of raw material for the production of activated carbon of high efficiency. It can be seen from Figure 7; that iodine adsorption of activated carbon prepared with sulphuric acid with activation temperature at 600°C is the best because the micropore content on surface of activated carbon is higher especially at the concentration of 0.2 than that of nitric acid and phosphoric acid. This is due to more extensive volatile matter degradation. From experimental result it is observed that iodine number for sulphuric acid was remarked to attain the highest iodine number while that of nitric acid was recorded the least iodine number of all. It means that activation favours sulphuric acid because sorbents with high iodine number performs better in removing small sized contaminants. It is the most fundamental parameter used to characterize the performance of activated carbon. High value indicates high degree of activation (Aziza et al., 2008; Itodo et al., 2010).

Iodine Number of activated carbon from avocado seed of Different reagent after Chemical and Thermal Activation

Figure 7; shows the difference between Chemical and Thermal Activation. It was observed that chemical activation is better than thermal activation. The smaller the volume of iodine adsorbed the bigger the iodine number adsorption. Consequently, the bigger the surface area and the more effective it is. Iodine number is used to characterize activated carbon performance. The highest iodine adsorption number obtained for sulphuric acid was 1713.28g/kg at chemical activated temperature of 600°C. It is known that activated carbon do absorb iodine very well and hence the iodine adsorption number is an indication of the total surface area of the activated carbon. It is a measure of activity level, the higher the number the higher the degree of activation.

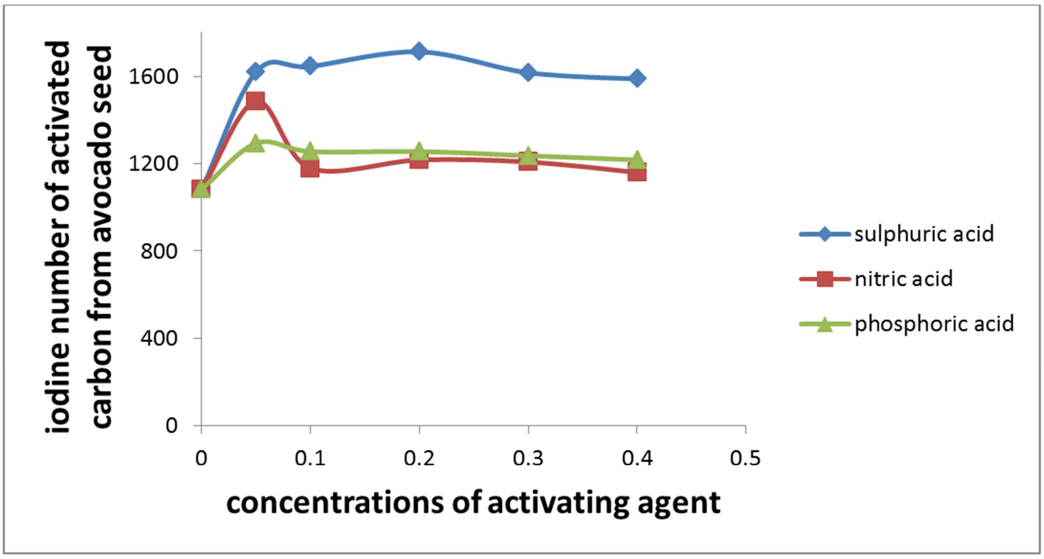

Figure 6. *Effect of H_2SO_4, HNO_3, and H_3PO_4 activation on the Iodine Number of activated carbon from avocado seed.*

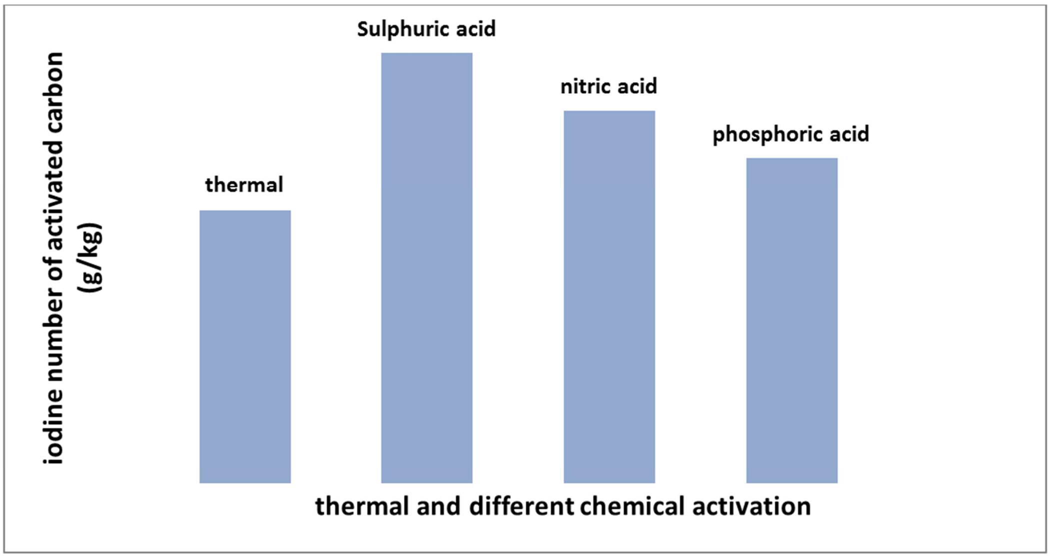

Figure 7. *Showing the Iodine Number of Activated Carbons from avocado seed after Chemical and Thermal Activation.*

4. Conclusion

A low cost and high quality active carbon should be of high surface area, produced at low temperature. The characterization of the adsorbents includes estimation of various parameters such as ash content, iodine number, burn off, bulk density, % yield, where carried out. Precursors of high % burn off gave corresponding low % yield. The sorted precursors could be prescribed as potential substrates for sorbent generation and utilization. It is concluded that Ash content gives an idea of the amount of inorganic constituents associated with the carbon. The iodine number of the activated carbon product of H_2SO_4 was the best with the highest iodine adsorption number of 1713.28g/kg at chemical activation temperature of 600°C. A longer activation time could induce negative effect on the carbon structure and thus decrease the iodine value. It is known that activated carbon do absorb iodine very well and hence the iodine adsorption number is an indication of the total surface area of the activated carbon. It is a measure of activity level, the higher the number the higher the degree of activation.

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