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Preparation and characterization of activated carbon from plantain peel and coconut shell using biological activators

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Abstract

A concern over the toxicity of chemicals used during the activation stage in the preparation of activated carbon is beginning to gain attention. The study therefore looked into the possibility of using bio-activators (lemon juice and potash leached from the peel of unripe plantain) as activating chemicals, for environmentally friendly activated carbon. Coconut shell and the peel from unripe plantain were used as feedstock and pyrolyzed at 400 and 450 ^{0}c. An impregnation ratio of 0.25:1 was used while laboratory grade potassium hydroxide was used as a base activating agent as a control setup. Characterization of the activated carbon was carried out using parameters like bulk density and yield which were obtained using standard procedures. Results showed that activating carbon using bio-activators as activating agents had very good characteristics when compared with the control. Bio-activators are therefore recommended for the production of bio based activated carbon especially in the fields of medicine, food and pharmaceuticals. The effect of carbonization temperature on adsorption efficiency and pore structure were investigated using methylene blue as adsorbate and SEM respectively

Key words: Cocoa beans, marketing risk factor, plant layout

1.   Introduction

Over the years, activated carbon has found immense use in the purification of water and gas, in medicine – as an efficient poison remover from the blood, metal extraction and many other applications [1]. Activated carbon is derivable from organic sources or materials such as wood sawdust, bagasse, rice husks, coconut shell and other agricultural wastes [2-7] They usually will have very high percentage content (greater than 85%) of carbon [8, 9] but with low inorganic matter and cannot be transformed to crystalline graphite when pyrolyzed at temperatures, even up to 3000^0C [10, 11]. Activated carbon is frequently used as adsorbent in the removal of dyes because of its high adsorption capacity.

The method of preparing activated carbon is in two steps, viz: Carbonization and activation using CO_2 or steam (physical activation) and acids, bases or other chemicals (chemical activation). The carbonization stage (pyrolysis) expels the volatile materials from the organic materials, leaving behind only the carbon while the activation stage makes the available pores 'active' enough for adsorption.

Using chemical activators such as $ZnCl_2$ and H_3PO_4 has also been effective. It was used to activate carbon obtained from the waste from Africa star apple and they both gave good results [12]. Traces of chemicals remaining on the surfaces of the activated carbon after series of washing/rinsing steps renders it improper in areas where toxicity is of utmost consideration, like the food processing, pharmaceutical use and medicine, etc. in the washing step during the preparation, the effluent is toxic to both plant and animal life and this aspect has not gained attention yet [13]

Coal-based carbon [14] has been in high demand as precursors in the preparation of activated carbon. Unfortunately, this feedstock is non-renewable. It is therefore imperative to use readily available and renewable feedstock for the preparation of activated carbon of commercial quantity and value. This is the reason agricultural wastes, capable of producing non-graphitizing carbon, is now being considered.

This study is aimed at using agricultural wastes (coconut shells and peel from unripe plantain) as feedstock while using bio-activators (lemon juice and leached potash from peel from unripe plantain) as acid and base activation chemicals respectively. The efficiency of the resultant activated carbon would then be determined and compared with effectiveness of using inorganic potassium hydroxide KOH as chemical activator [6].

The large surface area and numerous pores [9] present in activated carbon make it a good choice for use as catalysts and adsorbents. As adsorbents, the carbon can absorb both colours and odours because the contaminants and/or pollutants are bound to the active and free sites on the carbon surface until it is filled up. There are two major ways of producing activated carbon, namely the physico-chemical method that involves carbonization of the raw material followed by thermal activation [15] and chemical activation which precedes carbonization in a furnace [16]. The carbonization stage rids the feedstock of its non-carbon contents like oxygen, hydrogen, sulphur compouds, other volatiles and a carbonized material that is essentially carbon or char. This char usually lacks the ability for high adsorption since its pore structure has not fully been developed. Activation stage, on the other hand, improves on char's pore structure by an oxidative treatment.

The feedstock used, carbonization method and condition determine the porosity and micro size and distribution [17-19]. Activated carbon can exist in two forms, viz: powder and granular [20]. The choice of the form to be used is largely dictated by the expected performance and material to be adsorbed. While the granular activated carbon are presented in size ranges from 0.2mm to 5mm, are harder and endures longer than the powder counterpart, the powdered form on the other hand, are presented in size ranges of 10 to 160 Å and have lower operational cost and are more flexible.

Processes of producing activated carbon that will maximize yield and also give good pore characteristics are most desirable [20]. Yield is mostly affected by the activation temperature [21], activation time [22] and impregnation ratio [23, 24]. The yield for physical method of producing activated carbon has been found to be less than 17 % [25]

During impregnation, the soaking and subsequent evaporation of the water from the carbon ensures complete absorption of the activation chemical by the carbon.
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2. Methodology

2.1 Materials:

Unripe plantain peel, coconut shell, lemon fruits, indigo dye, Distilled water, potassium hydroxide pellets, 0.05M sodium thiosulphate, iodine and starch solutions,.

2.2 Equipment:

A Muffle furnace (Carbolite HTF 1700), pH meter (Jenway 3570), A mass balance (Radwag AS/310/C/2), An oven (Vision Scientific, Japan), juice squeezer, , hot plate magnetic stirrer, 250 and 500-ml measuring cylinders, beakers, Erlenmeyer flasks, glass funnel (Pyrex glassware was used all through), mercury-in-glass thermometer, filter paper (Whatman No 1, 18 cm dia.) and porcelain crucibles

2.3 Procedures

The lemon fruit juice was first extracted before its pH was measured. The acidic juice obtained was stored in glass jars.

2.3.1 Preparation of bio-activators

The unripe plantain peel was washed, sun dried for two weeks and finally transferred to the oven for drying to a constant weight at 105^0C. The dried mass was then ashed in the Muffle furnace for 2 hrs at 600 ^{0}C. A total of 100 g of the ash was extracted at 50 ^{0}C for 1 hr using cumulative 400 ml volume of distilled water in a 500 ml glass beaker. The mixture in the glass beaker was then stirred continuously using the hot plate magnetic stirrer. The mixture was filtered hot after 1 hr to obtain a caustic solution which was then evaporated to obtain the solid potassium hydroxide. The ash residue was again extracted with 200 ml of distilled water in order to completely extract its potash content. This was again evaporated to dryness to obtain KOH. The KOH recovered from the two processes was then weighed and kept in a well secured plastic container.

2.3.2 Carbonization and activation process

The unripe plantain peel and coconut shell were each washed and sundried for two weeks. The drying was then completed in the oven at 105 ^{0}C for 2 hours until a constant weight was achieved. The dried peel and coconut shells then underwent a size reduction to particle size of 500 – 800 microns (feedstock). 150g each of the feedstock per batch was subjected to carbonization at 400 ^{0}C for 2 hrs in the Muffle furnace using porcelain crucibles as holders. A total of 500 g each of the carbonized samples was obtained from the two feedstock materials. The process of carbonization was repeated at 450^0C for both feedstock until 500 g each of carbonized samples was obtained.

2.3.3 Activation of Carbonized Samples using Commercial and Extracted KOH.

The method reported by [26] was used. Here an impregnation ratio of the KOH: carbon ratio of 0.25:1 was used. This meant that per batch, 41.25 g of the KOH was used with 165g carbonised feedstock (from coconut shell). The solid KOH was first dissolved in a 250 ml distilled water and added to the carbonized feedstock. The mixture was stirred until a uniform consistency was achieved. This was left to stand for 24 hrs at ambient temperature and filtered. It was first air-dried and then transferred to an oven for final drying at 110 ^{0}C for 24 hrs. The sample was then further heated in the furnace at 400 ºC for one hr and then cooled. The activated carbon obtained was then subjected to several washings using distilled water in order remove the activating agents used from it. The washing is was topped when the wash-water became neutral. The final sample was dried to a constant weight at 110 ºC.

The carbonized plantain peel was not activated further because it was already by nature an alkaline or basic material. This was labelled self-activating

2.3.4 Activation of Carbonized Samples using Lemon Juice Extract

The extracted lemon juice (250 ml) was added and mixed separately with the carbonized coconut shell and plantain peel samples. They were agitated to form a consistent mixture which was then allowed to stand for 24 hrs at the ambient temperature. This was then filtered oven-dried at 110 ºC for a 24 hrs period. The samples were then transferred to a Muffle furnace for heat treatment at 400 ºC for one hr, cooled and washed several times until it became neutral. The final sample was again dried to a constant weight in an oven at 110 ºC.
The yield of the activated carbon samples was calculated using equation 1.

$$\% \, yield = \frac{\text{mass (g) of activated carbon}}{\text{mass (g) of carbon sample}} \times 100 \qquad (1)$$

2.3.5 Bulk density

A measuring cylinder of 100 ml volume was first zeroed on the weighing balance. The free-flowing and lump-free activated carbon was then taken and poured into the measuring cylinder to the 100 ml mark tapping the cylinder to ensure no void was created. The weight was then noted and recorded. The bulk density was then calculated using equation 2.

$$Bulk \, density = \frac{\text{mass (g) of carbon}}{\text{volume (ml) of carbon}} \qquad (2)$$

2.3.6 Morphology of the Activated Carbon Samples.

The Scanning Electron Microscope (SEM) was used to examine the surface morphology of the produced activated carbon (AC) samples.

2.3.7 Adsorption Study of Dye Solution using the Activated Carbon Samples

0.3 g activated carbon (AC) was first weighed into a conical flask which already contained 100 ppm of 30 ml dye solution. The mixture was agitated intermittently at varied contact times of 1 to 2.5 hours using a magnetic stirrer at a constant rpm. After the regulated contact time, the

mixture was then filtered off. A UV- visible spectrophotometer [27] was then used to measure the absorbance of the filtrate at a wavelength of 665 nm. The corresponding filtrate concentration was determined using the prepared calibration curve [28]. The adsorption capacity of the activated carbon is then calculated from equation 3.

Adsorption capacity (%) = $\frac{C_1 - C_0}{C_i} 100$ (3)

Where C_1 is the Initial dye concentration (mg/l) and C_0 is the final dye concentration (mg/l)

3. RESULTS AND DISCUSSION OF RESULTS

The yield of the various activated carbon samples are shown in Table 1. These include lemon activated coconut shell (LACS) carbon, lemon activated plantain peels (LAPP) carbon, extracted potash activated coconut shell (EPACS) carbon, self-activated plantain peels (SAPP) carbon, commercial potash activated coconut shell (CPACS) and commercial potash activated plantain peels (CPAPP) carbon.

Table 1: Bulk Density and Percentage Yield of Activated Carbon at Different temperatures

SAMPLES	Bulk density (g/cm^3)		% Yield	
	400 ^{0}C	450 ^{0}C	450 ^{0}C	400 ^{0}C
(LACS)	0.53	0.43	63.04	64.40
(LAPP)	0.46	0.45	46.50	47.40
(EPACS)	0.57	0.45	55.20	55.80
(SAPP)	0.45	0.40	45.58	46.30
(CPACS)	0.56	0.45	62.60	63.70
(CPAPP)	0.45	0.44	41.25	43.56

From Table 1, it was observed that although the carbonization temperature was increased from 400^{0}C to 450^{0}C, it made no appreciable difference in the % yield of the activated carbon produced from the same feedstock. The difference in the percentage yield between the same activated carbon produced at different temperature was only marginal. But the differences in the yield between the activated carbon obtained from coconut shell were generally higher than those obtained from plantain peel. While the highest and least percentage yield of activated carbon from coconut shell was 64.4 and 55.2 % respectively, the highest and least percentage yields of activated carbon from plantain peels gave 47.4 and 41.25 % respectively. This shows that the carbon content per kg of the coconut shell as a feedstock was much more than that obtained from plantain peels. This was actually expected because the moisture content of coconut shell (8.78 %) was much lower than that obtained from plantain peels, 12.78, [29]

Also from Table 1, it was observed that higher bulk densities were obtained for the activated carbon obtained from coconut shell than those from the plantain peel at 400 ^{0}C, whereas, the bulk densities of the activated carbon obtained from both feedstock were almost the same at a higher temperature of 450 ^{0}C. The implication of this is that, activated carbon from coconut shell (at 400^{0}C) will filter better than those from plantain peel at the same carbonization temperature. For the activated carbon produced at 450 ^{0}C lower bulk densities were obtained and implies lower filtering capability of the activated carbon samples from both coconut shell and plantain peels carbon.

The coconut shell carbon activated with both the bio-potash (extracted) and commercial KOH gave better bulk densities than the lemon extract activated counterpart.

3.1 The Effect of Time of Contact on the Adsorption Capacities of Various Activated Carbon Samples
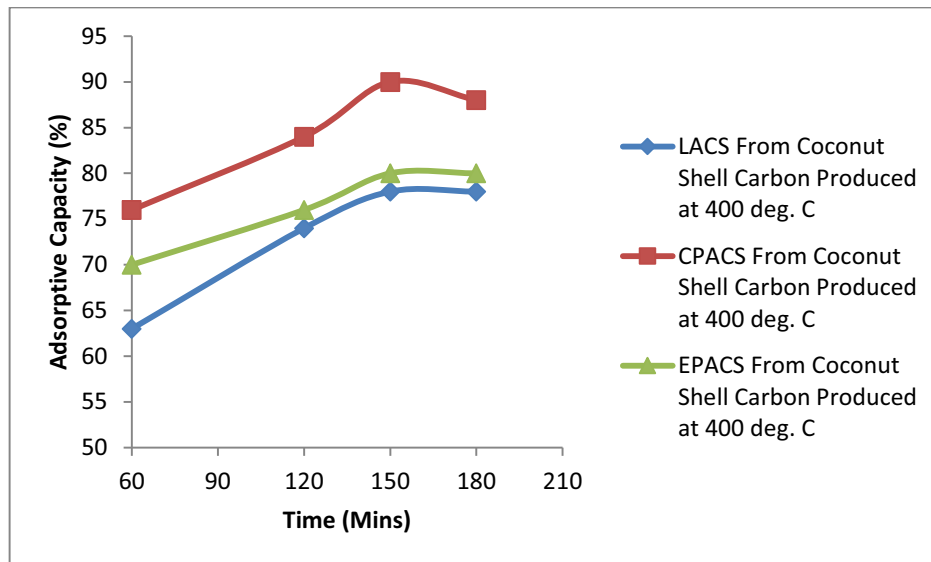

Figure 1 Effect of time of contact on the adsorption capacity of activated carbon from coconut shell at 400 ºC
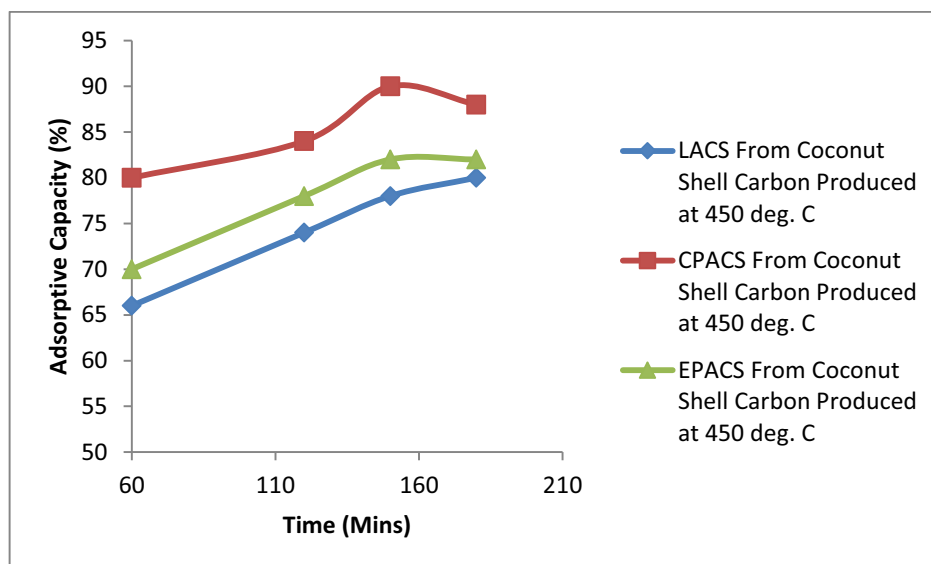

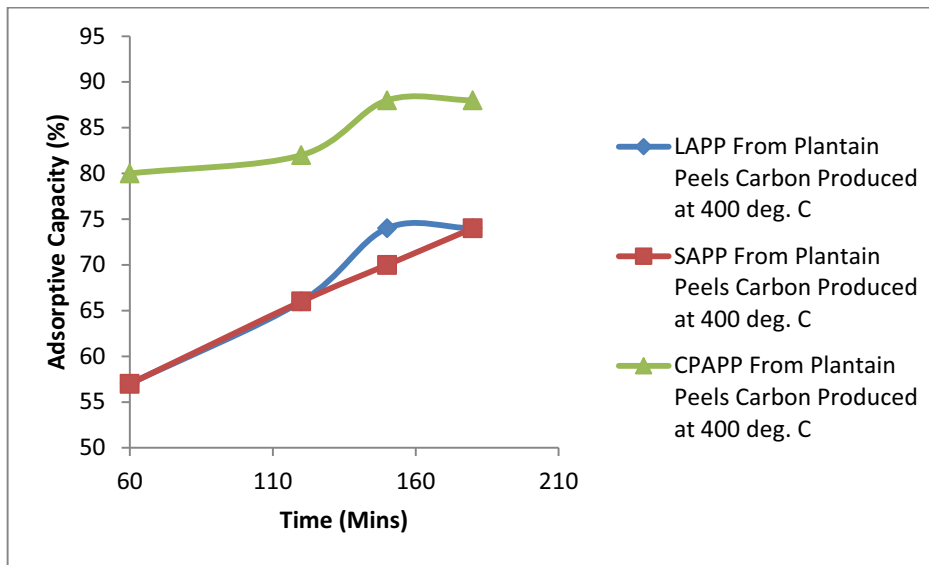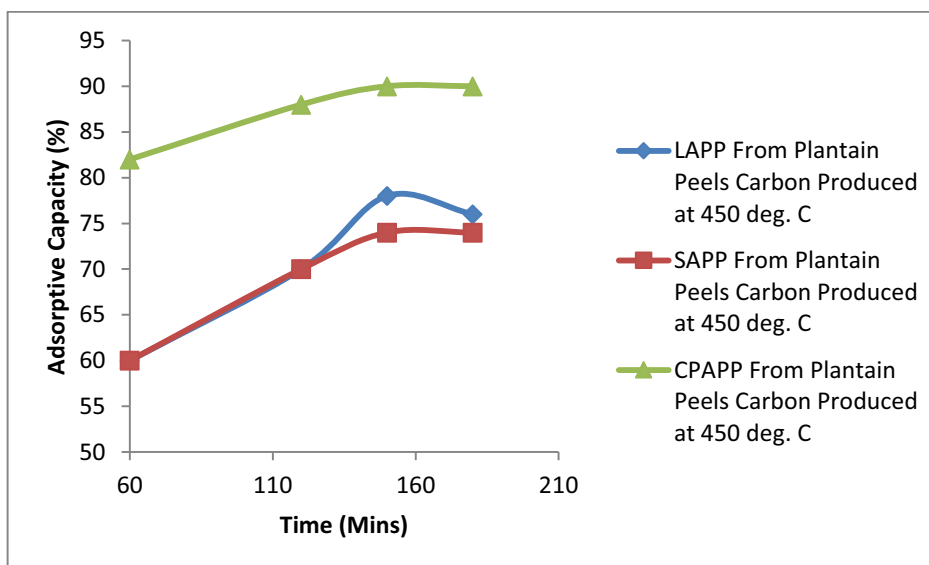Figure 2: Effect of time of contact on the adsorption capacity of activated carbon from coconut shell at 450 ºC

Figure 3: Effect of time on the adsorption capacity of activated carbon from plantain peel at 400 ℃

Figure 4: Effect of time on the adsorption capacity of activated carbon from plantain peel at 450 ℃

From Figures 1- 4, the adsorption of dye from its solution using the obtained activated carbon samples as adsorbents was observed to be initially fast at the start of experimental runs while the temperature was kept constant at 400 and 450 ^{0}C respectively. The adsorption rate however, became slower as the contact time was increased. This was expected and it may be attributed to the long time exposure of active pore sites of the activated carbon samples to the adsorption of the dye molecules such that as the contact time increased more of the available active sites become covered by the indigo dye molecules thereby making the active site area smaller for adsorption. Initially, owing to the availability of the unused active large surface, adsorption

became very fast. The obtained results indicate that the activated carbons produced from both the peel of plantain and coconut shell are capable (removing) or adsorbing the dye from its solution. The optimum contact time of 150 mins was obtained using the various activated carbon samples to achieve equilibrium.

The Effect of Bio and Inorganic Activating Agents on the Adsorption Capacity of Dye from its Solution
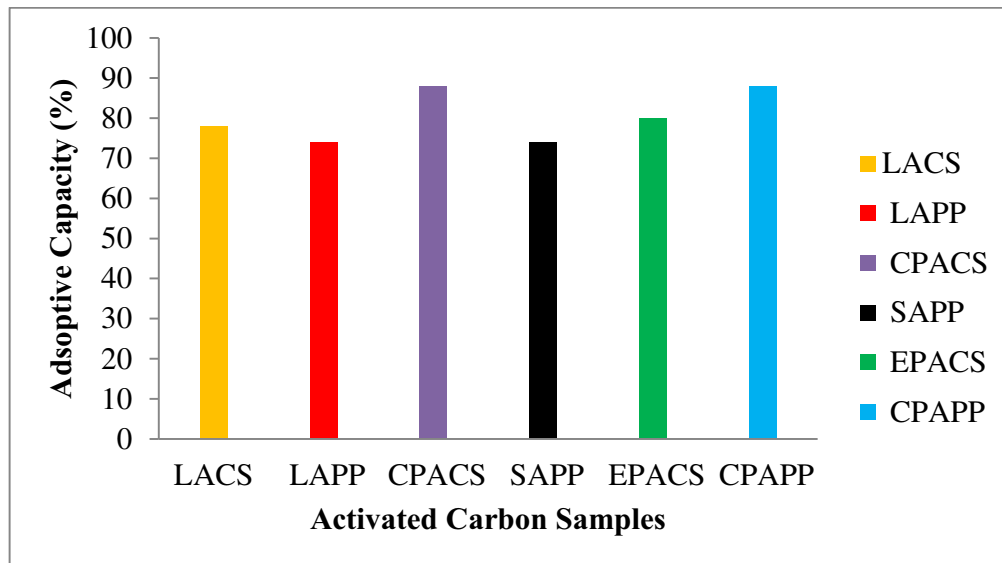

Figure 5: Effect of Bio and Inorganic activating agents on the adsorptive capacity of activated carbon samples of indigo dye at 400 °c
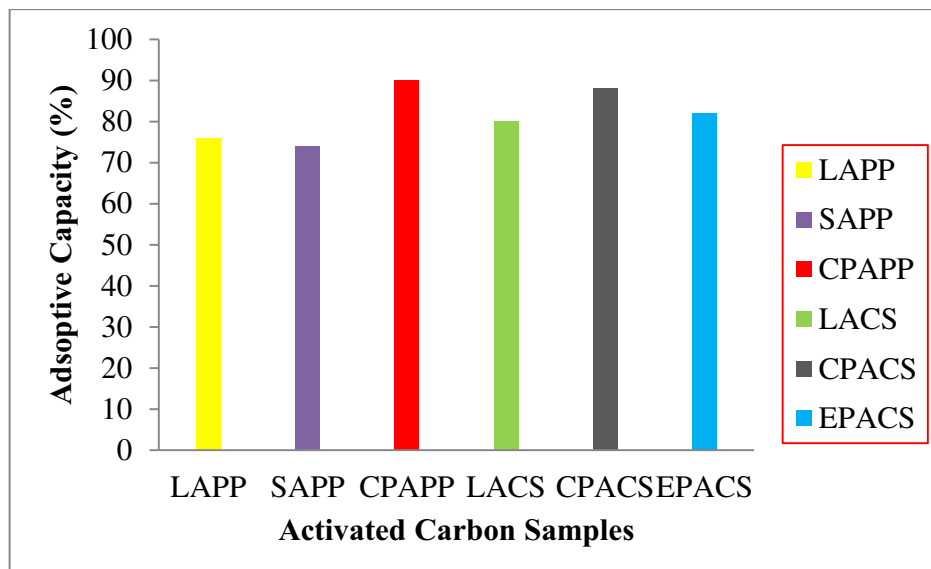
Figure 6: Effect of Bio and Inorganic activating agents on the adsorptive capacity of activated carbon samples of indigo dye at 450 °c

KOH extract from the ash of plantain peel and lemon juice extract from lemon fruits were used as the bio-activators or bio-activating agents. The aim was to ascertain their ability to serve as activating agents in activated carbon production. From Figures 5 and 6, six each of the activated carbon samples produced at 400 and 450 ºc were respectively produced. They include LACS - lemon activated coconut shell carbon, LAPP - lemon activated plantain peels carbon, SAPP - self activated plantain peels carbon, EPACS - extracted potash activated coconut shell carbon, CPACS - commercial potash (inorganic) activated coconut shell carbon and CPAPP - commercial potash activated plantain peel carbon. The four bio-activators showed good applicability for producing bio-activated carbons. Their bio-activated carbons (EPACS, LACS, LAPP and SAPP) at the two temperatures (400 and 450ºC) performed well compared to the conventional chemically activated carbons, (CPACS and CPAPP). From the bio-activated carbons at 450 ºc (Figure 6), EPACS gave the highest adsorptive capacity of 82 % followed by LACS (80 %), LAPP (78 %) and SAPP (74 %). The same trend was observed with the bio-activated carbons produced at 400 ºC (Figure 5), EPACS gave the highest adsorptive capacity (80 %) followed by LACS (78 %), LAPP (74 %) and SAPP (74 %). Although, the commercial or chemical KOH activated carbons (CPACS and CPAPP) produced the overall highest adsorptive capacities compared to the bio-activated carbons. At 400ºC, the adsorptive capacity was 88 % for both CPACS and CPAPP), while at 450 ºC, the adsorptive capacities were 90 % and 88 % for CPAPP and CPACS respectively. The difference between the adsorptive capacities of the chemically activated and bio activated carbons is encouraging to warrant further research and improvement of bio-activators. The fundamental difference between them is due to the development of better porous structure and larger pores in the chemically activated carbons at 400 ^{0}C and 450 ^{0}C. Generally, the activated carbon produced at 450 ^{0}C performed better than those at 400 ^{0}C. As temperature is increased, the unconverted biomass residue in the carbonized sample is fully converted to carbon and hence increasing the quantity of carbon available for adsorption.

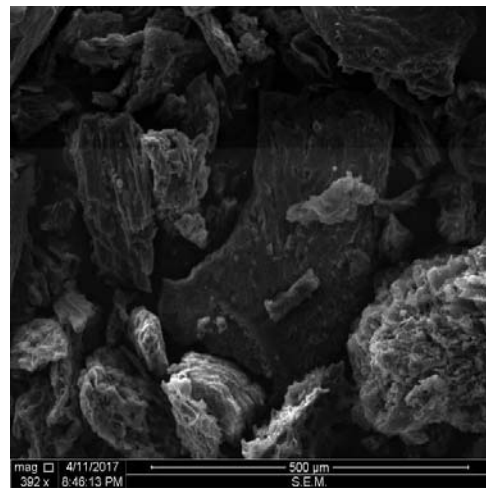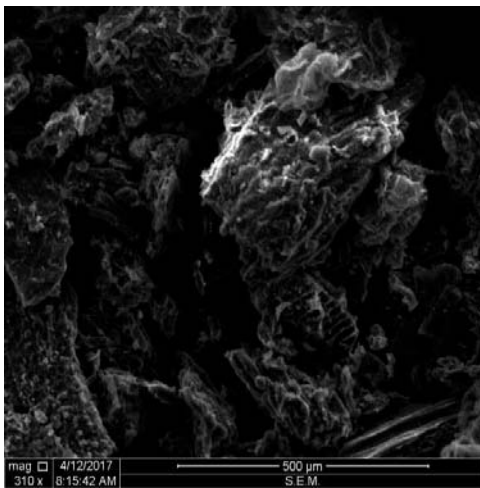**Images from of Scanning Electron microscope Electron (SEM) and their Implications**

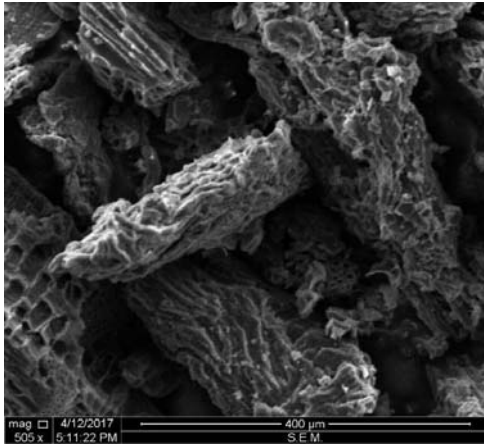Figure 7 SEM of CPACS at 450 ºc   Figure 8 SEM of CPACS at 400 ºc
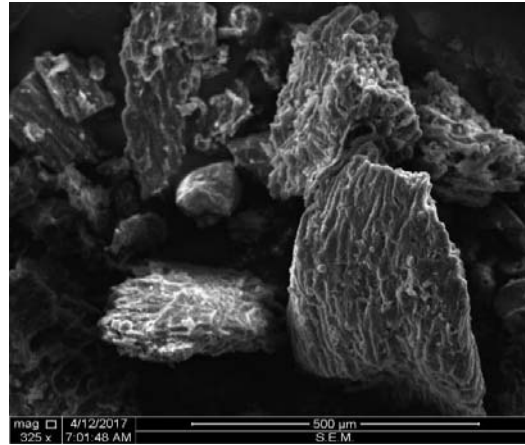
Figure 9 SEM of LACS at 450 ^{0}C
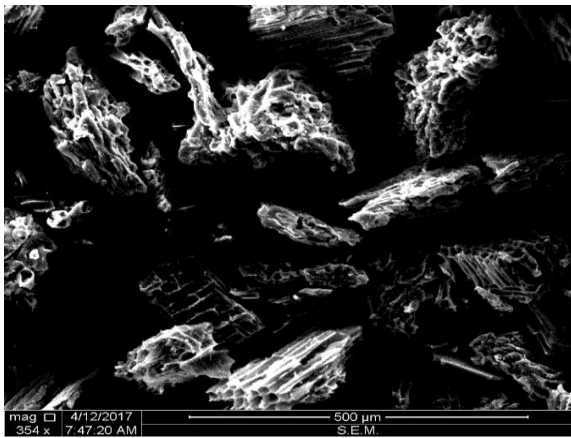
Figure 10 SEM of LACS at 400 ^{0}C

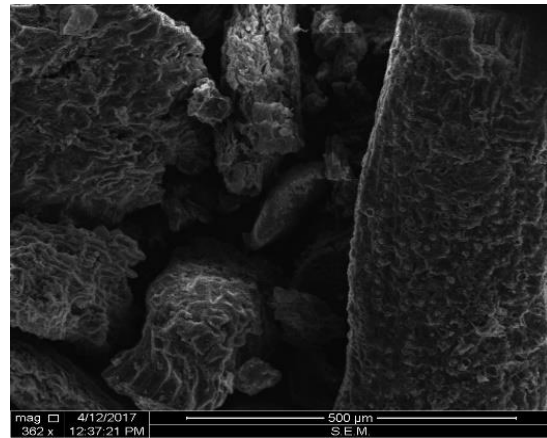Figure 11 SEM of EPACS at 450 ^{0}C

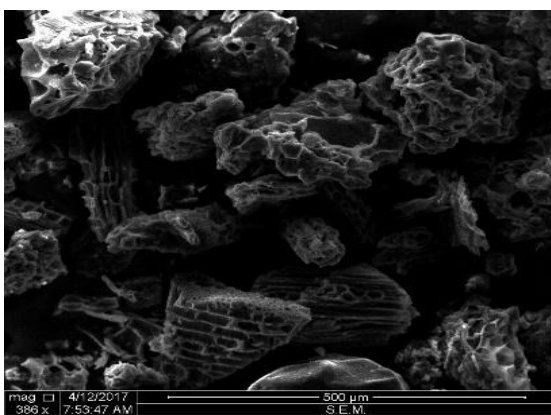Figure 12 SEM of EPACS at 400 ^{0}C
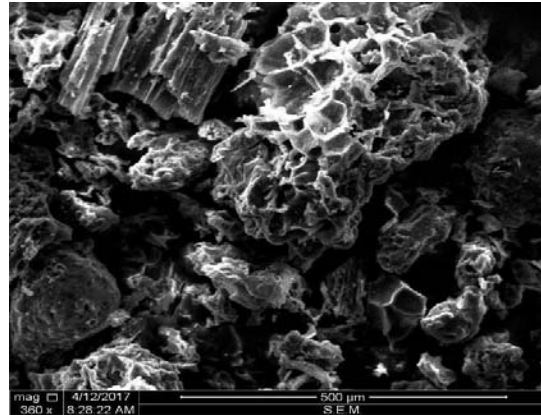

Figure 13 SEM of SAPP at 450 ^{0}C
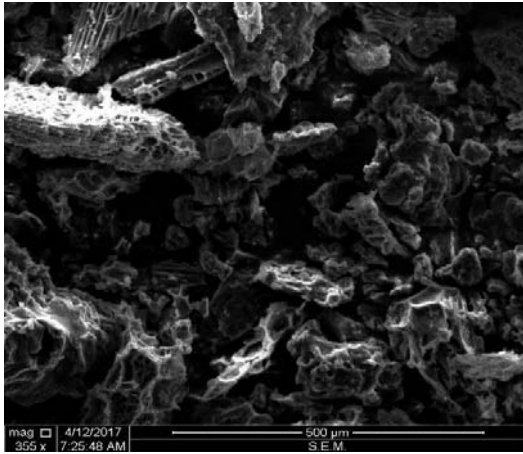
Figure 14 SEM of SAPP at 400 ^{0}C
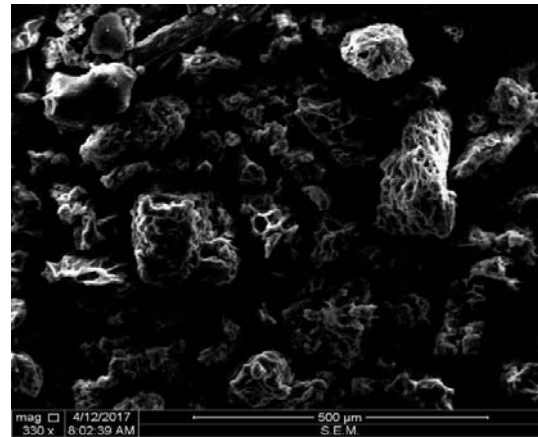
Figure 15 SEM of CPAPP at 450 ^{0}C

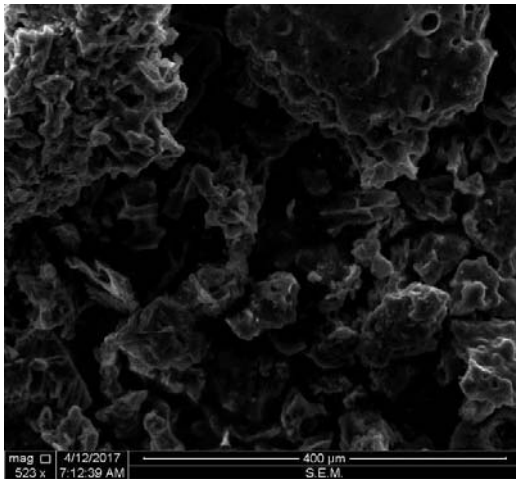Figure 16 SEM of CPAPP at 400 ^{0}C

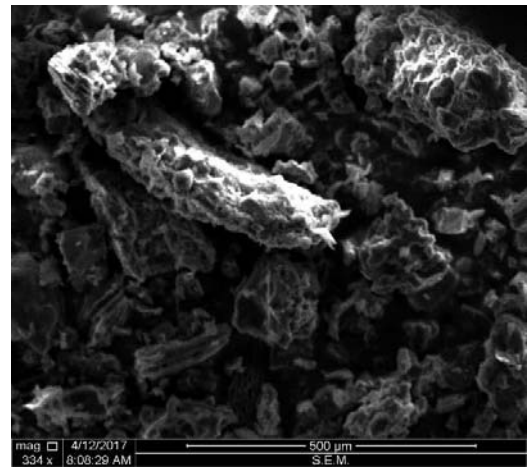Figure 17 SEM of LAPP at 450 ^{0}C

Figure 18 SEM of LAPP at 400 ^{0}C

The SEM images of the various activated carbons are displayed in Figures 7 – 18. The micrographs show surface topography and how fully developed the pore sizes are. The SEM micrographs of the activated coconut carbons at 400 and 450 ^{0}C are shown in Figures 7 – 12, while the activated plantain carbons at 400 and 450 ^{0}C are shown in Figures 13 – 18. Varied pore sizes were observed to have been formed in all the activated carbon samples. This is attributed to the thermal treatment which expelled the volatile matters during the activation and carbonization stages. This resulted in the production of active and fixed carbon mass with and larger pore sizes observed in all the samples. The presence of micro-pores as seen in the activated carbon samples is responsible for the high adsorptive capacity exhibited by both the bio- and chemically activated carbons [30]. The SEM micrographs revealed that the activated carbon samples obtained from the coconut shell (EPACS and LACS) at 400 ^{0}C and 450 ^{0}C (Figures 9 – 12) looked spongy in their structures consisting of many pores but CPACS (Figures 7 and 8) showed that they have better formed or developed surfaces with well-ordered micro cavities and pores.

Same pore structure was observed in all the activated carbon samples obtained from plantain peel. Generally though, CPAPP, LAPP and SAPP produced at 450 ^{0}C showed much more developed structural topography, well-structured area containing many active sites (pores and cavities) than the corresponding activated plantain carbons at 400 ^{0}C (see Figures 13 – 18).

4.   Conclusion

- The adsorption study indicated that the activated carbon samples obtained from the two precursors (coconut shell and plantain peel) were effective for removing or adsorbing dye molecular components from its solution.
- As the contact time increased the adsorptive capacity also increased proportionately until after an optimum point it then began to reduce.
- Lemon juice extract and extracted potassium hydroxide from plantain peel proved very effective and excellent as bio-activating agents.
- The resultant bio-activated carbon samples were found to be good and comparable with the activated carbon produced using common chemical activating agents such as KOH.
- The activated carbon sample produced at 450°C had better adsorptive capacity compared to those produced at 400°C.
- The self-activated plantain peels carbon, SAPP, which did not require any external agent for activation performed well when compared with other bio-activated plantain peels carbons.

3.   Recommendation

Pilot scale investigation of this study and its application to a wide range of other agricultural wastes is encouraged.

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