

Production and Characterization of Activated Carbon from Nigerian Bamboo Using the Two Methods of Activation

Udeh, N.U

Department of Civil and Environmental Engineering University of Port Harcourt, Rivers State, Nigeria

Abstract: The study investigated the production activation carbon with bamboo sticks at different carbonization temperatures (400°C and 500°C), impregnation ratios (1:2; 1:3 and 1:4) and different activation methods (chemical and physic-chemical activation methods). The effects of carbonization temperature, impregnation ratios and activation methods on the properties of bamboo activated carbon were studied by characterizing them in terms of surface area, porosity, bulk density, carbon yield and ash content. The results showed that the unwashed chemically activated carbon (UCABC) had higher surface areas (4839m²/g and 5415 m²/g) at 400°C and 500°C at ratio 1:2 respectively while the washed chemically activated carbon (CABCW) had higher surface areas of 1287m²/g and 1255m²/g (at ratios 1:2 and 1:3 respectively) at 500°C. However, the physic-chemical activated carbon (PABC) had higher surface area of 359m²/g with ratio 1:3 at 500°C. Furthermore, high porosity values ranging from 0.77 to 0.95% was observed for all the bamboo activated carbon indicating highly porous materials and their bulk density were in the range of 0.425g/cm³ to 0.687 g/cm³. Also, the ash content ranged from 8.7 to 15.4 % while the carbon yield at 400°C had the same values of 31.9% for UCABC and CABCW at ratio 1:2. Thus, UCABC at 500°C (ratio 1:2) has the highest surface areas and the activating chemical need not to be washed away to avoid generating more wastewater during production

Keywords: Activation, Bamboo, Characterization, Carbonization, surface area

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I INTRODUCTION

Activated carbon is an adsorbent produced from a wide range of organic based materials such as coal, coconut shell, wood chips, palm-kernel shell, corn cobs, saw dust and many others [1]. The raw material is carbonized to obtain the char or carbonaceous material, which is then activated to yield the highly porous product which has high degree of porosity and an extended internal surface area. Thus, two main parameters are relevant to the performance of the activated carbon namely the surface area and the pore volume or structure. The pore volume limits the size of the molecules that can be adsorbed whilst the surface area limits the amount of material which can be adsorbed. Activated carbon particle has two types of pores existing in it by which adsorption take place. They are the macro pores (>10⁻¹ um) and the micro pores (10⁻³ - 10⁻¹ um). The macro pores provide a passageway to the particle's interior and to the micro pores while the micro pores are responsible for the large surface area of activated carbon particles and adsorption largely takes place in the micro pores. The macro pores and the micro pores are created during the activation process.

Basically, there are two different processes for preparation of activated carbon namely, chemical activation and physical activation[2], [3]. Physical activation involves carbonization of a carbonaceous materials followed by activation of the resulting char in the presence of activating agents such as CO₂, steam or other mixtures [4] while in Chemical activation, the precursor is impregnated with a chemical agent and then pyrolyzed [5], [6]. Chemical activation enjoys the benefit of development of better porous structure in a single process route at low carbonization temperatures as compared to physical activation. The excellent features of activated carbon has made it to be widely used in many fields including, water and wastewater treatment, gas filters, deodorization, decolorization, purification and separation processes [2]. They can be also used as catalysts and catalyst supports in the catalytic processes. However, due to the high cost and non-renewable source of commercially Activated Carbon, researchers have studied production of activated carbons from cheap and renewable precursors. Bamboo appears to be economically attractive because of its abundance, low cost, high carbon content, rapid growth and multiplication speed.

Bamboo is a perennial giant woody grass belonging to the sub family Bambusoideae of the family Graminae and takes only several months to grow and it matures within 3 to 4 years. The dominant bamboo in Nigeria is commonly referred to as the Indian bamboo and is particularly adapted to the rain forest belt of Nigeria where it is found in abundance along river banks and other relatively marshy areas [7]. Bamboo is an abundant natural renewable resource that can be converted into useful materials. In its natural form, bamboo sticks are used as structural materials for steps, scaffolds and as reinforcements in construction sites because of

its inherent mechanical strength [8]. It is a strong, tough, cheap, versatile, and highly renewable material, one that people and communities have known and utilized for thousands of years.

The focus of this research is to produce high surface area activated carbon from bamboo sticks by comparing the two activation methods: chemical activation and physical activation. Conversion of bamboo to activated carbon with high surface area would help to solve part of wastewater treatment in Nigeria.

II MATERIALS AND METHODS

2.1 Collection and Preparation of materials

The Bamboo sticks used for this study could be referred to as Indian bamboo which is the dominant bamboo in Nigeria found in the rain forest belt. Fully matured Bamboo sticks were cut into smaller sizes (2- 4cm), washed, air dried and then taken to the laboratory for further processing. Preliminary analyses were carried out on the dried Bamboo materials to determine the Fixed Carbon and Volatile matter content. Zinc Chloride ($ZnCl_2$) of about 80% purity was used as the activating agent.

2.2 Production of Bamboo Activated Carbon

The Bamboo Activated carbons were produced by two methods: Physico-chemical activation method (which involved two- step pyrolysis) and Chemical activation method (which involved the single step pyrolysis).

2.2.1 Chemical Activation Method (Single Step Pyrolysis)

The bamboo samples were chemically activated using Zinc Chloride ($ZnCl_2$) as the activating agent. 500g of dried bamboo was mixed with $ZnCl_2$ solution at different impregnation ratios (1:2, 1:3 and 1:4) $ZnCl_2$ /Bamboo. The mixture was then heated on a hot plate to almost dryness. The activated bamboo samples were air- dried and then carbonized at two different temperatures 400°C to 500°C for 3 hours using a muffle furnace and then grinded into powder. The carbonization temperature and time ranges were in accordance with previous studies on Nigeria Bamboo [9], [10], [11]. Part of the grinded activated bamboo carbon were washed with distilled water to a pH of 6 (to remove the activating chemical) while the other part was left unwashed. The washed samples were dried in an oven at a temperature of 110°C for a period of 6 hours. The washed bamboo samples were named Chemical Activated Bamboo Carbon washed (CABCW) while the unwashed samples were named Unwashed Chemical Activated Bamboo Carbon (UCABC). This nomenclature was used to identify the two variations of bamboo activated carbons produced by chemical activation.

2.2.2 Physico -Chemical Method of Activation

500g of dried Bamboo samples were carbonized in a muffle furnace at two different temperatures (400 and 500°C) for 3 hours. The carbonized materials were grinded into smaller sizes and impregnated with Zinc Chloride ($ZnCl_2$) at different weight ratios of 1:2, 1:3 and 1:4 ($ZnCl_2$ /bamboo). The resultant mixtures were heated on a hot plate until a paste was formed. The carbon samples were allowed to cool and then washed with distilled water to a pH of 6 and then dried in an oven between temperature ranges of 100°C to 110°C for 6 hours. The samples were named Physical Activated Bamboo Carbon (PABC).

2.3 Characterization of Produced Bamboo Activated Carbon

The bamboo activated carbon samples produced (PABC, UCABC and CABCW) were characterized and the following parameters were determined: surface area, porosity, bulk density, carbon yield and ash content. Surface area and porosity in particular were adopted as criteria because it has been reported that carbons with high surface area and porosity are the most suitable for adsorption of adsorbates in aqueous solution [12], [13], [14], [15].

The Surface area was calculated using Sear's method as reported by [16]. Carbon yield was measured using the method of [17] in which the dried weight of each bamboo sample was determined before and after carbonization and the carbon yield, Y_{ch} calculated from Equation 1:

$$Y_{ch} = \frac{W_o}{W_{ch}} \times 100\% \quad (1)$$

Where: W_o = mass of bamboo carbon retrieved from the furnace. W_{ch} = mass of air-dried bamboo samples before carbonization.

The Porosity which is the ratio of the volume of voids to the total volume of the activated carbon particles and was calculated from Equation 2:

$$n = 1 - \frac{\rho}{\rho_w G_s (1+w)} \quad (2)$$

Where: n = Porosity, ρ = Bulk density of activated carbon particles, ρ_w = Density of water, G_s = Specific gravity of activated carbon particles and w = Moisture content.

Thus, to calculate the porosity, first calculate the Bulk density, specific gravity and moisture content of the activated carbons. The Bulk density was measured using the method of [18]. The moisture content was

calculated as the ratio of the change in weight to the original weight expressed as a percentage. It was calculated from Equation 3;

$$w = \frac{W_o - W_d}{W_o} \times 100\% \quad (3)$$

Where: W_o = original weight (g), W_d = weight after drying (g), w = moisture content (%)

A pycnometer was used in the determination of the specific gravity of the activated carbon particles. The Specific gravity G_s was calculated from Equation 4:

$$G_s = \frac{M_2 - M_1}{(M_2 + M_4) - (M_1 + M_3)} \quad (4)$$

Where M_1 = mass of the empty pycnometer, M_2 = mass of oven dried activated carbon inside the pycnometer, M_3 = mass of oven dried activated carbon inside the pycnometer filled with water, M_4 = mass of pycnometer filled with water.

The pH was determined by suspending one gram of carbon in 100ml distilled water of pH 7.0 and heating at 90°C for 20 minutes. The solution was then cooled and pH measured using a digital pH meter [12].

III RESULTS AND DISCUSSION

3.1 Preliminary Analysis of Raw Bamboo

The results of the preliminary analysis of raw bamboo indicated a relatively high volatile matter content (86.4%) and fixed carbon content of 12.12% and may be considered as a good material for activated carbon. This confirms with the study of [19] reported that bamboo is rich in volatiles (70%) and has a fixed carbon of 11.32%. Also, [20] reported a high carbon content of 43.8% for raw bamboo sticks. Note that fixed carbon content of bamboo changes when exposed to high temperature during carbonization and activation. The fixed carbon content would increase steadily while volatile matter would decline with temperature [19].

3.2 Results of Characterization of Bamboo Activated Carbon

The effect of the optimum conditions (carbonization temperature and impregnation ratios) in the production of Bamboo activated carbon using Physico-Chemical and Chemical method of activation were analyzed in terms of surface area, porosity, ash content and carbon yield.

3.2.1 Effects of Carbonization Temperature on properties of Bamboo carbon

The results of characterization of the different samples of Bamboo activated carbon produced using the two activation methods at two different carbonization temperatures (400°C and 500°C) were shown in Table 1.

Table 1: Characterization of Bamboo Activated Carbon at 400°C and 500°C

Parameters	400°C	500°C	400°C			500°C		
	Bamboo Charcoal		PABC	UCABC	CABCW	PABC	UCABC	CABCW
Impregnation ratio	No impregnation		1:2	1:2	1:2	1:2	1:2	1:2
Surface area (m ² /g)	151	156	167	4839	1159	247	5415	1287
Bulk density (g/cm ³)	0.476	0.454	0.473	0.524	0.543	0.53	0.687	0.653
Specific gravity	1.11	0.91	1.27	2.23	1.78	1.98	2.066	1.403
Moisture content (%)	2.7	1.95	1.60	0.98	1.20	1.48	1.30	8.2
Porosity	0.88	0.83	0.86	0.88	0.86	0.89	0.86	0.95
Ash content (%)	11.3	8.7	8.6	11.1	14.1	10.4	9.4	9.9
Carbon yield (%)	24.5	27.69	24.5	31.9	31.9	27.69	25.6	25.6
pH	10.10	10.10	6.0	6.4	6.2	6.4	6.5	6.3

From Table 1, it was observed that the bamboo carbons had higher surface areas at higher temperature. High surface areas of 5415m²/g, 1287m²/g and 247m²/g were obtained for UCABC, CABCW and PABC at 500°C respectively while the surface area of Bamboo charcoal (no activation) was 156m²/g at 500°C. It was also observed that the chemically activated carbons had higher surface area than the physically activated carbon. However, the high surface area in UCABC may be as a result of the unwashed chemical used for activation which increased the titre volume used in calculation of surface area. The same trend was observed by [21], [22]. Note that surface area is a measure of the adsorptive capacity of the adsorbent and the larger the surface area, the higher the number of adsorptive site available and thus, the higher the adsorption capacity of a particular carbon [23]. The effects of different carbonization temperature on the surface area of bamboo activated carbons are presented in Figure 1.

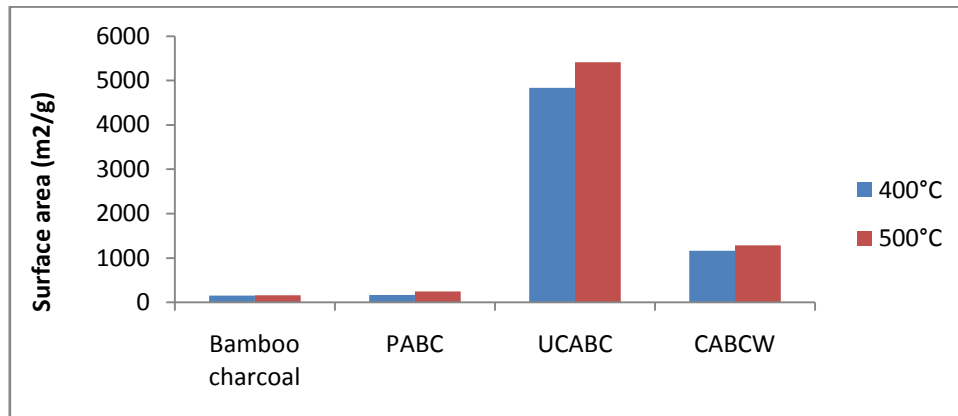


Figure 1: Surface Area as a function of Carbonization Temperature ($ZnCl_2$ /Bamboo ratio 1:2)

The effect of carbonization temperature on the porosity of bamboo activated carbon was shown in Figure 2. The porosity of all the bamboo activated carbon was between the ranges of 0.83% to 0.95% at the two temperatures tested. CABCW and PABC had porosity of 0.95% and 0.89% at 500°C indicating higher porosity at higher temperature. However, UCABC had porosity of 0.86 at 500°C indicating that the unwashed chemical might have blocked some of the pores. Note that porosity enhances adsorption capacity of the adsorbent and activated carbon with high surface area will be highly porous.

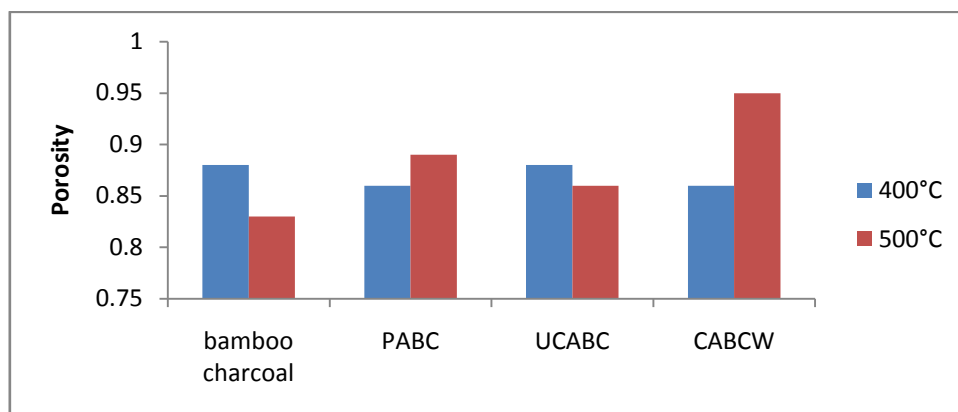


Figure 2: Porosity as a function of carbonization Temperature ($ZnCl_2$ /Bamboo ratio 1:2)

The effects of different carbonization temperature on ash content of bamboo carbons were shown in Figure 3. The ash content of bamboo carbons were in the range of 8.7% to 14.1%. Higher ash content values were observed at 400°C carbonization temperature with CABCW having the highest ash content of 14.1%. Note that the inorganic materials contained in activated carbon are measured as the ash content and it reflects the purity of the carbon and the organic residue left after the carbon source has been heated. The ash content values for the bamboo carbon at 500°C were within the range of 2 - 10% which were the range recommended by [24]. Low ash content value indicates that the inherent carbon, in the starting material is high [15].

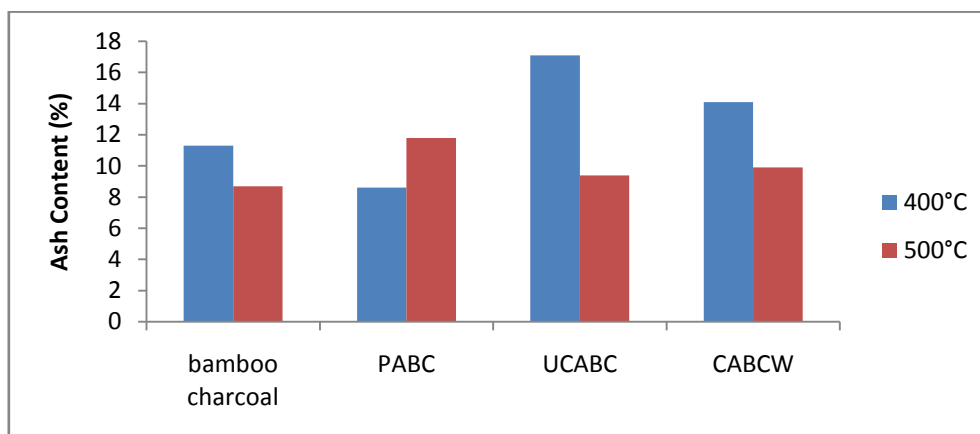


Figure 3: Ash content as a function of carbonization Temperature ($ZnCl_2$ /Bamboo ratio 1:2)

The effects of carbonization temperature on carbon yield shown in Fig. 4 revealed higher carbon yield at lower temperature for UCABC and CABCW while the PABC had higher carbon yield at higher temperature. CABCW and UCABC had carbon yield of 31.9% at 400°C while PABC and bamboo charcoal had carbon yield of 27.69% at 500°C. Carbon yield is the amount of original precursor remaining after carbonization and activation treatment. The carbon yield for all the bamboo carbons were within the range of 20% recommended by [25].

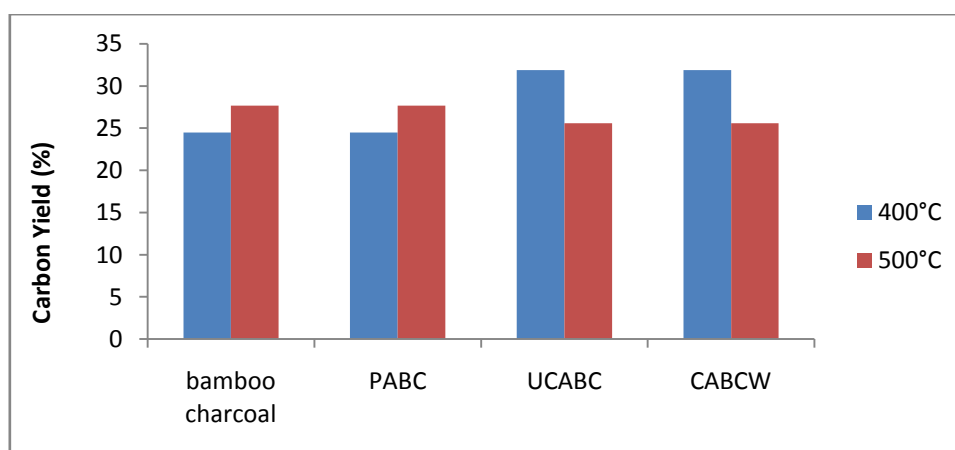


Figure 4: Carbon Yield as a function of carbonization Temperature ($ZnCl_2$ /Bamboo ratio 1:2)

It then follows that the carbonization temperature has significant influence on the properties of activated carbon. This could be seen in very high surface area values observed at 500°C for all the bamboo carbon tested (see Table 1). Note that the higher the porosity and surface area of a carbon, the higher the adsorptive capacity of the carbon [12], [13], [15].

3.2.2 Effects of Impregnation Ratio on Properties Bamboo Carbon

Results on the effect of different impregnation ratios on the properties of the bamboo activated carbons produced at two different carbonization temperatures (400°C and 500°C) were presented in Tables 2 and 3. The results revealed that surface area decreased as the ratios increased for the chemically activated carbon. UCABC had higher surface area of 5415m²/g and 4839 m²/g at ratio 1:2 at 500°C and 400°C respectively while the CABCW had surface area of 1287m²/g and 1159 m²/g at ratio of 1:2 at 500°C and 400°C respectively. However, PABC had lower surface areas (167m²/g, 199m²/g and 135m²/g at 400°C) and (247m²/g, 359m²/g and 225m²/g at 500°C) at ratios of 1:2, 1:3 and 1:4 respectively. These were presented in Figure 5.

Table 2: Characterization of Bamboo Activated Carbon at 400°C at different impregnation ratios

Parameters	Bamboo charcoal	PABC			UCABC			CABCW		
	400°C	400°C			400°C			400°C		
	No impregnation	1:2	1:3	1:4	1:2	1:3	1:4	1:2	1:3	1:4
Surface area (m ² /g)	151	167	199	135	4839	4231	2567	1159	999	871
Bulk density (g/cm ³)	0.476	0.473	0.42	0.48	0.524	0.592	0.589	0.543	0.527	0.50
Specific gravity	1.11	1.27	5	9	2.23	2.00	1.79	1.78	1.60	1.59
Moisture content (%)	2.7	1.60	0.98	1.14	0.98	0.75	1.5	1.20	0.56	1.65
Porosity	0.88	0.86	1.73	1.04	0.88	0.83	0.87	0.88	0.79	0.86
Ash content (%)	11.3	9.4	0.82	0.79	12.1	11.7	11.0	14.1	11.4	9.8
Carbon yield (%)	24.5	24.5	8.9	8.6	31.9	30.8	29.6	31.9	30.8	29.6
pH	10.10	6.0	24.5	6.3	6.4	6.5	6.7	6.2	6.4	6.2

Table 3: Characterization of Bamboo Activated Carbon at 500°C at different impregnation ratios

Parameters	Bamboo charcoal	PABC			UCABC			CABCW		
	500°C	500°C			500°C			500°C		
	No impregnation	1:2	1:3	1:4	1:2	1:3	1:4	1:2	1:3	1:4
Surface area (m ² /g)	156	247	359	225	5415	4103	2663	1287	1255	1191
Bulk Density (g/cm ³)	0.454	0.53	0.56	0.58	0.687	0.610	0.602	0.653	0.625	0.592
Specific gravity	0.91	1.98	1.28	1.13	2.066	1.916	2.089	1.403	1.715	1.162
Moisture content (%)	1.95	1.48	2.95	1.05	1.30	1.45	1.21	2.8	1.03	1.14
Porosity	0.83	0.89	0.89	0.75	0.87	0.87	0.86	0.95	0.82	0.77
Ash content (%)	8.7	11.8	6.70	6.70	9.4	7.8	15.4	11.9	9.9	8.20
Carbon yield (%)	27.69	27.69	27.6	27.6	24.6	25.5	26.0	24.6	25.5	26.0
pH	10.10	6.4	9	6.6	6.5	6.5	6.5	6.3	6.5	6.2

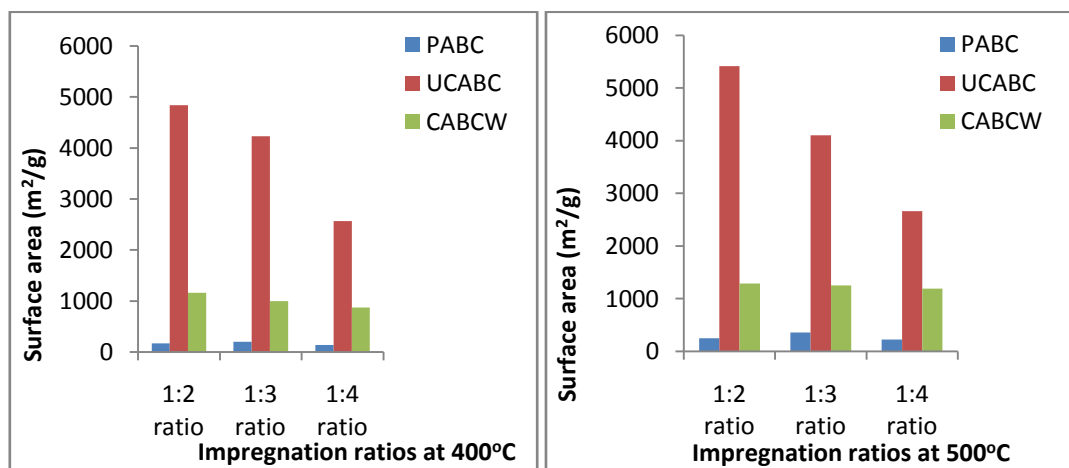


Fig. 5: Surface area as a function Impregnation Ratio at 400°C and 500°C respectively

The effects of different impregnation ratios on the porosity of bamboo activated carbon at 400°C and 500°C carbonization temperatures were shown in Figure 4.7 and 4.8. At 400°C, the porosity values ranged between 0.79% and 0.88% at all the ratios tested. It was observed that the porosity decreased as the ratios increased. High porosity values were observed at ratio 1:2 for PABC (0.86%), UCABC (0.88%) and CABCW (0.88%) at 400°C.

Furthermore, the effects of impregnation ratios on the ash content of bamboo activated carbon revealed that the ash content decreased as the ratios increased. At 400°C, UCABC and CABCW had ash content of (12.1%, 11.7% and 11.0%) and (14.1%, 11.4% and 9.8%) at ratios of 1:2, 1:3 and 1:4 respectively while PABC had lower ash contents of 9.4%, 8.9% and 8.6% at ratios 1:2, 1:3 and 1:4 respectively. However, at 500°C, a high ash content of 15.4% was observed for UCABC at ratio 1:4 while the ash content at ratio 1:2 and 1:3 were 9.4% and 7.8% respectively. Also, the ash content of PABC was in the range of 6.7% to 11.8% and 8.6% to 9.4% for 500°C and 400°C respectively (see Tables 2 and 3).

The effects of different impregnation ratios on carbon yield revealed that at 400°C, the carbon yield decreased as the ratios increased. CABCW and UCABC had higher carbon yield of 31.9%, 30.8% and 29.6% at ratios 1:2, 1:3 and 1:4 respectively while PABC had the same value of carbon yield (24.5%) at the different ratios. However, at 500°C, the carbon yields of CABCW and UCABC increased as the ratios increased (24.6%, 25.5% and 26% at ratios 1:2, 1:3 and 1:4 respectively) while PABC had the value of carbon yield (27.69%) at the different ratios. These were presented in figure 6. Furthermore, the bulk densities of all the bamboo carbon were within the range of 0.425g/cm³ to 0.687g/cm³. Generally, any adsorbent with high bulk density need not to be regenerated frequently because, it can hold more adsorbate per unit weight [26] and a carbon with a bulk density of about 0.5g/cm³ is adequate for adsorption [27].

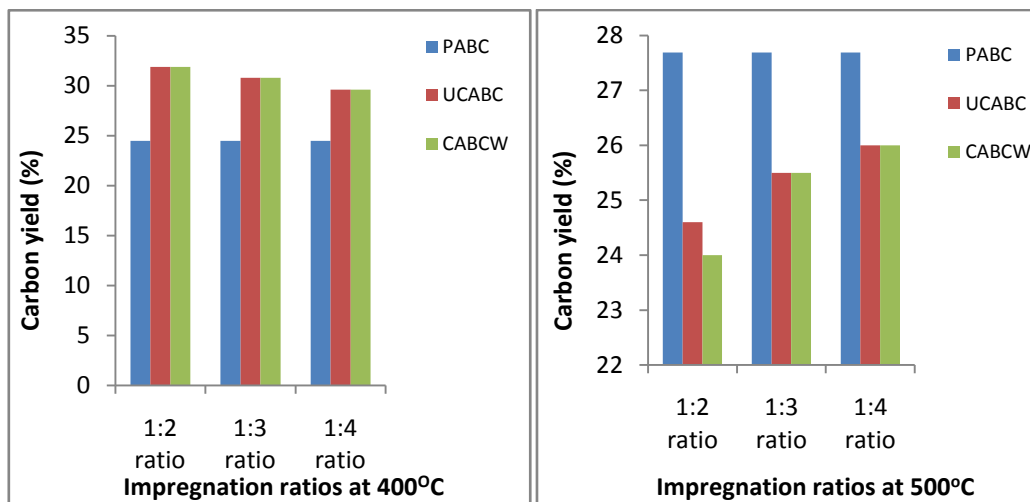


Figure 6: Carbon yield as a function of Impregnation ratios at 400°C and 500°C respectively

3.3 Morphology Structure of Bamboo Carbons

The morphology structure of Bamboo carbons was observed using the scanning electron microscope (SEM). The SEM was taken for bamboo charcoal, PABC, UCABC and CABCW as shown in Figures 7 to 10. The SEM micrographs of the activated carbon particles showed cavities, pores and rough surfaces on the carbon samples. SEM photography of UCABC shows that the surface is more pitted and fragmented due to carbonization and activation with ZnCl₂, thus having a well developed porous structure (see Fig. 9). The external surface shows a rough area having different pore diameters distributed over the surface of activated carbon. Similar observations were reported [28]. However, the pores of bamboo charcoal, PABC and CABCW were not as developed as the UCABC.

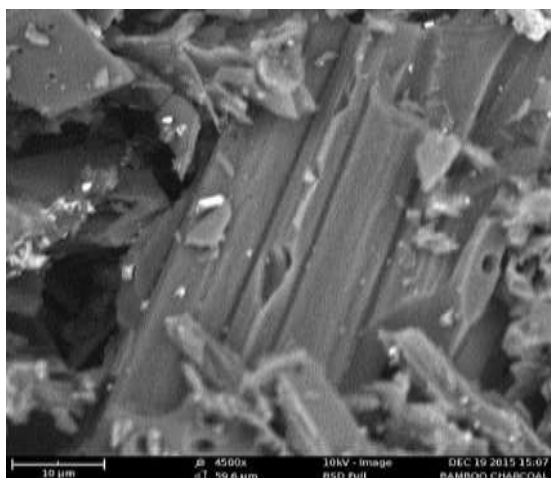


Figure 7: SEM of Bamboo Charcoal

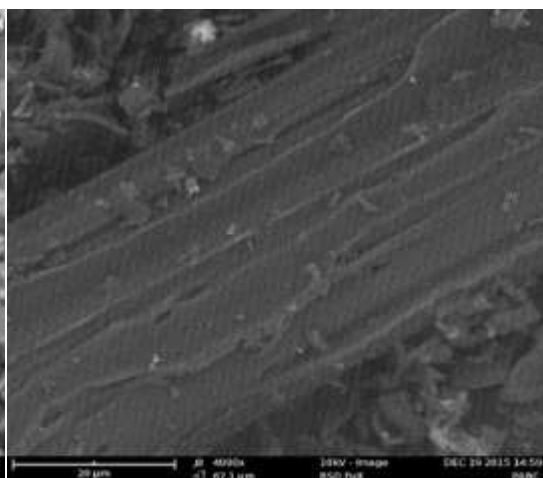


Figure 8: SEM of PABC

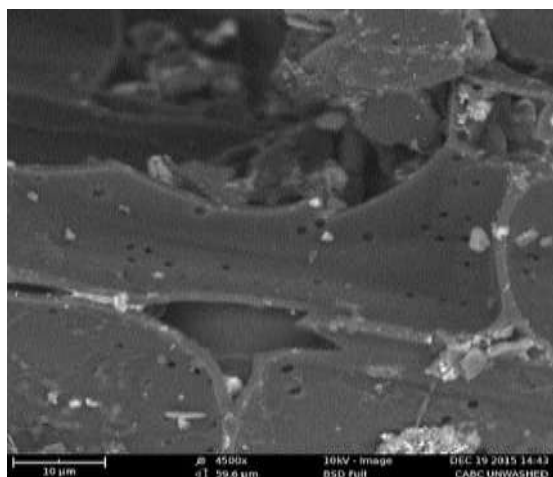


Figure 9: SEM of UCABC

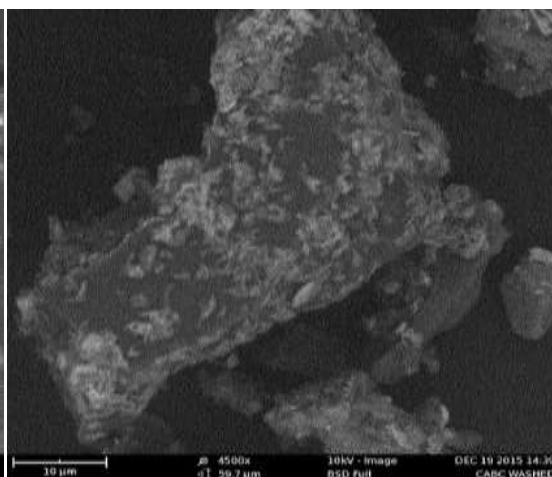


Figure 10: SEM of CABCW

IV CONCLUSION

This study revealed that carbonization temperature and impregnation ratio has significant influence on the properties of activated carbon. This could be seen in very high surface area values observed at 500°C and other impregnation ratios tested. Similar trend was also reported by [11]Evbomwan et al, (2013). Also, production of Bamboo activated carbon using chemical method of activation with Zinc Chloride (ZnCl₂) gave activated carbon with higher surface areas than the physic-chemical activated method. Thus, the unwashed chemical activated carbon (UCABC) gave the highest surface area for all the carbon samples tested. This was confirmed by the SEM morphology structure of UCABC which showed more cavities and porous structure. This indicates that the activating chemical need not to be washed away to avoid generating more wastewater during production. Therefore, the optimum conditions for production of activated carbon from Nigerian Bamboo were presented in Table 4.

Table 4: Optimum conditions for the production of Bamboo Activated Carbon

Type of Activated carbon	Carbonization Temperature (°C)	Impregnation ratios
PABC	500	1:3
CABCW	500	1:2
UCABC	500	1:2

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