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OPTIMUM CONDITIONS FOR CARBONISATION OF COCONUT SHELL

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ABSTRACT

The optimum conditions that are useful in the carbonization of coconut shell have been examined. The carbonization was effected using particle sizes (150 – 2000µm) at carbonization temperatures between 200 and 900^{0}C in a laboratory muffle furnace. The study involved determination of yield, rate of weight loss, optimum temperature, as well as determination of ash and moisture contents of the carbonized carbon and suitable resident time for carbonization. The result showed a maximum yield of 27% of carbonized product. It had 1.03 and 5.50% ash and moisture contents respectively. The characteristic particle size of 500µm, carbonization temperatures of 500 – 600^{0}C at resident time of 5 minutes were the optimum production conditions.

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INTRODUCTION

Carbonization is the production of charred carbon from a source material. The process is generally accomplished by heating the source material usually in the absence or limited amount of air to a temperature sufficiently high to dry and volatilise substances in the carbonaceous material (Hassler, 1963). Coconut shells are cheap and readily available in high quantity. Coconut shell contains about 65 – 75% volatile matter and moisture which are removed largely during the carbonization process (Gimba, 2001). The cellulosic structure of the coconut shell determines the end product.

Coconut-shell-based activated carbon has unique properties as a superior adsorbent, making it the carbon of choice for a wide range of liquid and gas/vapour phase applications. It has been recognized that for an effective activated carbon the preliminary carbonization process is very essential (Gimba, 2001). Therefore, parameters such as temperature, particle size and resident time for carbonization will affect the overall texture, quality and quantity of the carbonized product with the attendant effects on the ash, moisture and possibly metal contents. Available information on carbonization of source materials such as coconut shell is still scanty. For instance, the recommended particle size of shell for carbonization reactions is between 150 – 850µm (Gimba, 2001). This wide range could be reduced through a properly monitored carbonization process to obtain the characteristic particle size. It is hoped that the results obtained in this study would give more specific optimum conditions for carbonization of coconut shell. Carbonised products with more definite characteristic data can therefore be produced in large quantity from the relatively cheap coconut shell for subsequent production of activated carbon.

EXPERIMENTAL

Sample preparation

The coconut shells were collected from market places, homes and dumps around Zaria, Nigeria. They were washed with water, dried in an oven at 105^0C, crushed and sieved into different particle sizes of 150, 250, 300, 355, 425, 500, 710, 850, 1180, 1400, 1700 and 2000μm, respectively.

Carbonisation of the samples

Carbonization was achieved using a muffle furnace (Model GLMB + PD/IND) manufactured by Carbolite Barnford, Sheffield, England. Two grammes of a specific particle size sample was weighed into clean and previously weighed crucible and placed in the furnace at already set temperature for various resident times. After each resident time, the crucible was removed from the furnace, allowed to cool and the weight determined. The heating (resident) times were 0.25, 0.5, 0.75 ………. 20 minutes each at carbonization temperatures of 200, 250, 300, 400, 500, 600, 700, 800 and 900^0C, respectively.

Determination of Time of Commencement of Carbonization

This is the time when a 'pop' sound is accompanied by dense brown fumes from the furnace and inflammation of the sample for each particle size and/or carbonization temperature. It was repeated three times. The average time of four determinations were computed.

Determination of Percentage Carbon Brunt-Off (CBO)

Ground coconut shell materials (2.0g) was heated in a pre-weighed crucible at each carbonizing temperature for 5 or 10 minutes. The crucible was allowed to cool and the weight of the charred carbon residue (carbonized product) was determined using Mettler Analytical Balance. This was repeated thrice and the average of three determinations obtained.

% carbon-burnt – off = Wt of carbon residue x 100 (for a particular particle size)
2g of ground coconut- shell

Determination of the Amount of Iodine Adsorbed

Into a $100cm^3$ volumetric flask, 0.5g of the carbonized carbon and $10cm^3$ of 5% $^v/v$ HCl were introduced and then swirled. $100cm^3$ of the previously standardized iodine solution was added and agitated at a test speed using electric shaker for a period of 1 hour. This was followed by filtration of the whole solution using sintered glass crucible. Aliquots of the filtrate was titrated against 0.1M $Na_2S_2O_3$ using starch indicator. By difference, the amount of iodine adsorbed (in mg) by carbonized carbons of each particle size was calculated. The amount of iodine adsorbed was calculated as percentages of carbon.

Determination of the Amount of Indigo Blue Adsorbed.

The method described by Mykayula and Matumbo (1994) was adopted. The absorbances of standard solutions of indigo blue at 0.05, 0.1, 0.2, 0.5, 1.0g concentrations in chloroform were used to prepare a calibration curve.

1.0g of carbonized samples of the different particle sizes were dispensed into a $250cm^3$ conical flask followed by $50cm^3$ of standard indigo blue solution in 5% v/v chloroform and stoppered. The mixture was agitated using an electric shaker for 1 hour at room temperature and then filtered by suction through a sintered glass crucible. The concentration of the filtrate was determined at λ_{max} 602nm against a reference solution of 5%

chloroform using a digital CORNING colorimeter.

Determination of Metal Contents of the Activated Carbons

The method used here was adopted from the standard methods for the examination of water and wastewater (1992) and the method described by Mkayula and Matumbo, (1994). The amount and types of metals present in a sample can be satisfactorily determined using the Atomic Absorption Spectrophotometry (AAS).

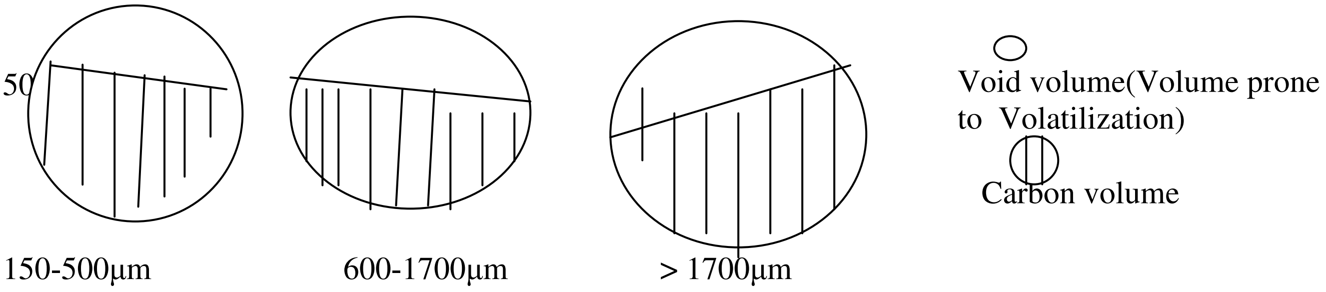
In the determination of metal contents of the carbons prepared, 0.5g of the ash obtained was added to 25cm^3 of 2M HCl and the mixture boiled gently for 5 minutes. The mixture was filtered through a glass crucible (porosity 4) and washed three times with distilled water. The filtrate and washings were put into 250cm^3 volumetric flask and diluted to the mark with distilled water. The solution obtained was then analysed for metal content for each carbon activated using the Atomic Absorption Spectrophometer.

For the determination of the amount of different metals that can be adsorbed by each of the activated carbons prepared, standard solutions of each of the metals containing 2000 ppm were agitated with each of the carbons for a period of 1 hour. It was then filtered through a glass crucible with porosity 4. The amount of each metal adsorbed by the different activated carbons was then determined by using AAS.

RESULTS AND DISCUSSION

Figure 1 shows that carbonization of coconut material commenced earlier in the smaller particle sizes than in the larger particle sizes at each carbonization temperature. The higher the temperature, the earlier was the commencement of carbonization. Carbonization involves loss of volatile materials which are presumably fewer in smaller particles than in the larger ones and escape of the volatiles increases with increasing temperature. The weight fraction of the carbon residue generally increased to a maximum at about 500µm particle size for each carbonizing temperature and decreased at higher particle sizes up to 1700µm (Fig. 2). The maximum values decreased with increasing temperature and practically no maximum at the highest carbonization temperature in this study (900^{0}C). The minimum weight fractions occurred at 1700µm for samples carbonized at 200^0 – 400^{0}C**,** 1000µm for the 500^{0}C to 800^{0}C. The declining weight fractions between 600 – 1700µm at the lower temperatures suggest that the carbonized products were either thermally unstable at these particle sizes or the latter contained smaller amounts of carbonaceous material, that is, they contained more of volatile substances. The smaller weight fraction of carbonized product at bigger particle sizes than 500µm is indicative of this particle size being the optimum (characteristic) particle size for effective carbonization of coconut shell. It would have been expected that increase in particle size would lead to greater weight fraction of carbonized product but these results have proved otherwise. Scheme 1 proposes a particle size model during carbonization.

In relation to Fig. 2 the first set of particles between 150–500µm have an increasing weight fraction of carbon volume greater than void volume, from 600 – 1700µm, void volume > carbon volume with decreasing weight fraction and the third set of particles weight fraction becomes higher as carbon volume is greater than the void volume with less ratio than the first set. It, therefore, means that, at least, there are three categories of particle sizes between 150 – 2000µm available in coconut shell. With this model, weight fractions will not increase linearly with particle size but have the pattern shown in Fig. 2.



Scheme 1: Schematic particle size model during carbonization

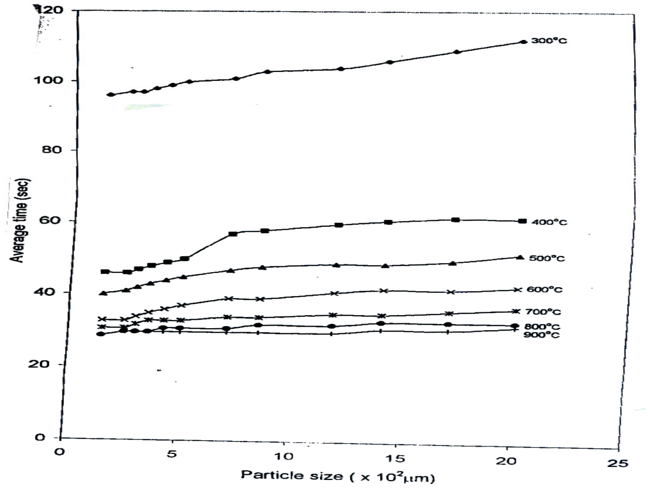


Fig. 1: Effect of Particle size on average time of commencement of carbonization temperatures (30 –0 900 °C).

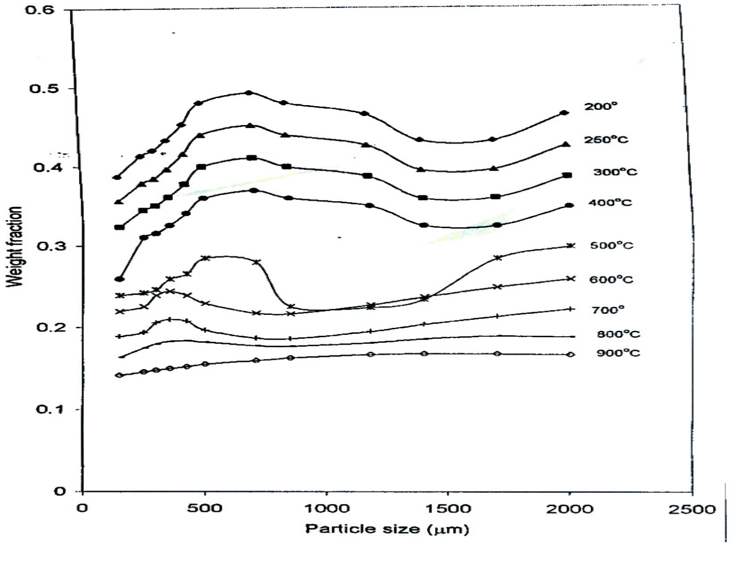


Fig. 2: Effect of Particle size on weight fraction of carbon for carbonization temp.(200–900 °C).

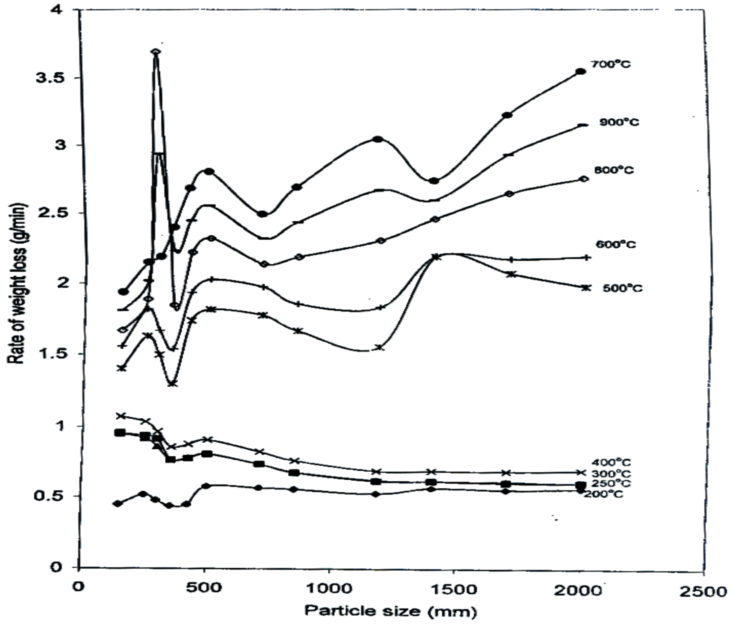


Fig. 3: Effect of Particle size on rate of weight loss at carbonization temp.(200–900^{0}C)

Figure 3 shows that there was a general pattern of rise, fall and then rise in the rate of weight loss for 150 to about 500μm particle size between 500 – 900^{0}C carbonization temperatures and another pattern of fall, rise and fall for <500^{0}C temperatures. These initial behaviours tend to suggest that the shell samples experienced some fluctuating structural changes involving a mixture of predominantly condensable and non-condensable cellulosics, depending on the carbonization temperature and particle sizes. This result also shows that the carbonization temperatures considerably affect the nature and quantity of these cellulosic intermediates. The latter probably exist at the higher rather than at the lower temperatures (200 – 400^{0}C) as particle size increases. Therefore, it is clear that particle size and temperature affect both the carbonization reactions and the carbonized product. The effect of carbonization temperature on the weight fraction of carbonized product using particle size of 500μm. Fig. 4 shows that the linear segments, at 200 – 300^{0}C, 400 – 600^{0}C and 700 – 900^{0}C, had the slopes as indicated on the curve. The decreasing slopes indicate greater level of volatilization with increasing temperature.

Figure 5 shows a general increase in weight loss to a maximum at 600^{0}C and, thereafter, a drop at 700^{0}C, before the final increase up to 900^{0}C. The results indicate that the most appropriate carbonization temperature range for coconut shell was 400 – 600^{0}C at the characteristic particle size of about 500μm. Increasing the temperature beyond this range may increase the rate of internal burning which would affect the type and nature of the pores developed during carbonization and the ultimate properties of the carbonized product. Table 1 shows that carbonization of coconut shell at 500^{0}C gave the highest adsorption capacity for the test adsorbates.

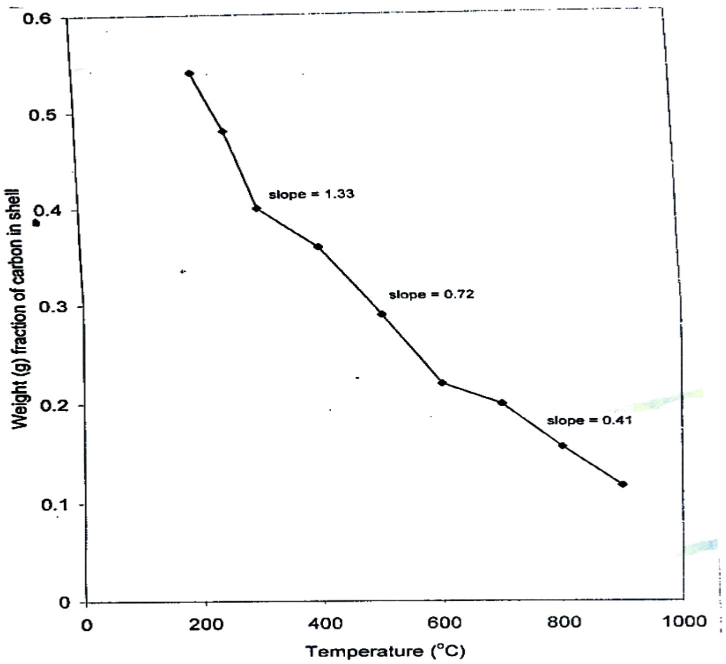


Fig. 4: Effect of carbonization temperature on weight of shell at 500 μm particle size.

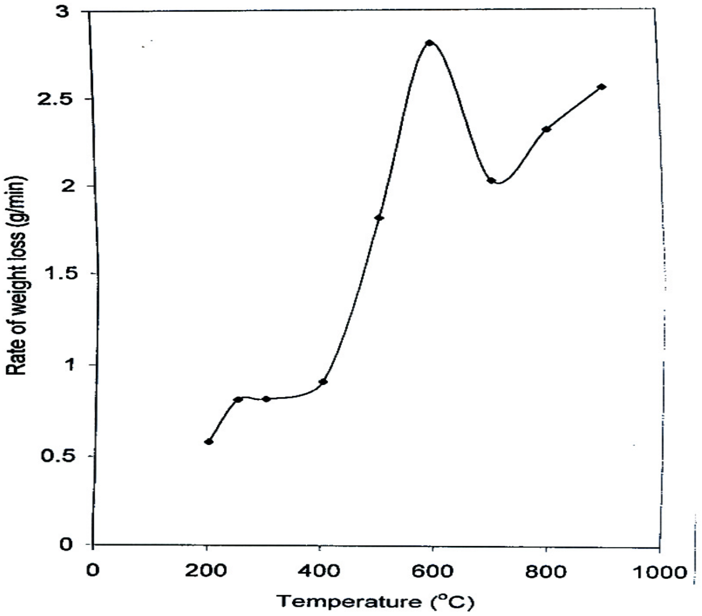


Fig. 5: Effect of carbonization temperature on weight of shell at 500 μ

Table 1: Effect of carbonization temperature on the activity of carbonized product (500µm particle size).

	Carbonization temperatures (^0C)							
Adsorbate	200	300	400	500	600	700	800	900
% Iodine	15	27	33	52	48	35	24	20
% Indigo blue	29	31	32	50	48	41	37	25

The effect of carbonization times on the weights of the coconut shell of 500µm particle size between 200 – 500^{0}C (Fig.6) and 600 – 900^{0}C (Fig.7), shows that longer heating time resulted in lower equilibrium weights of carbon. Almost equal weight of 0.54g, representing 27%, was obtained at 400, 500 and 600^{0}C at full carbonization for 5 minutes. Increasing temperatures produced relatively lower weights at equal resident times when full carbonization was achieved. It can also be seen from this result (Fig. 6) that at the lower temperatures, the weights of shell did not reduce to the constant values that were observed at higher temperatures (Fig.7). Thus, carbonization is not complete at lower temperatures (200 – 300^{0}C) and the leveling off of curves indicates full carbonization. This result shows that the resident time for carbonization of coconut shell with the characteristic particle size of 500µm was about 5 minutes. The percentage carbon burnt off (CBO) in (Table 2) further suggests that shorter time as 5 minutes was more effective for carbonization.

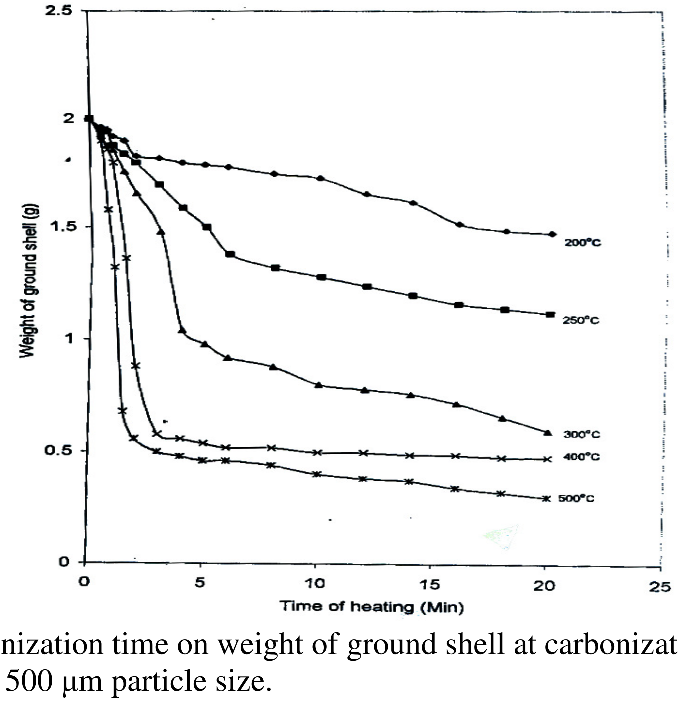


Fig. 6: Effect of carbonization time on weight of ground shell at carbonization temperatures 200-500°C for 500 µm particle size.

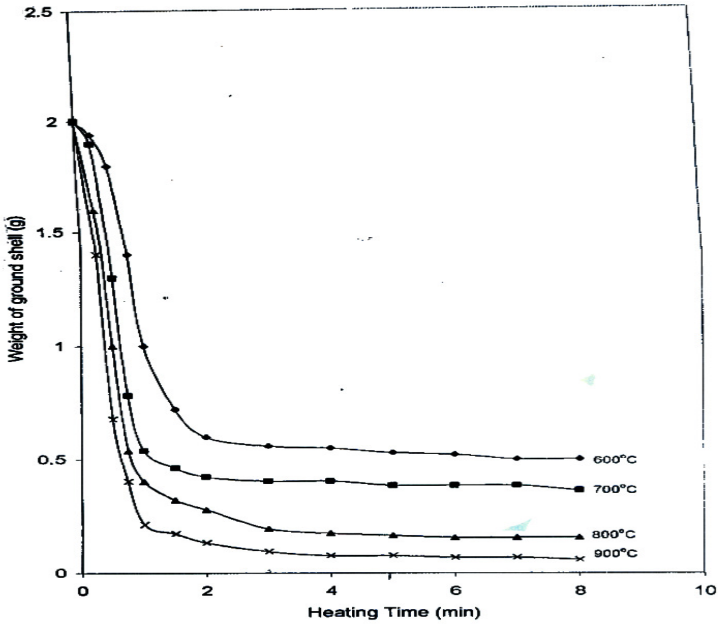


Fig. 7: Effect of carbonization time on weight of ground shell at carbonization temperatures 300-900°c for 500 µm particle size.

Table 2: Effect of resident time on percentage carbon burnt-off (CBO) at carbonization temperatures, (200 - 900^{0}C).

Temperature ^{0}C	% CBO	
	Resident time(mins) 5	Resident time(mins) 10
200	96.6	92.3
250	86.7	75.0
300	64,6	47.2
400	49.0	30.6
500	39.7	27.3
600	28.0	22.2
700	22.0	16.7
800	10.1	8.3
900	5.3	2.8

The results shows that carbonization of coconut shell involves volatilization resulting in formation of char carbon at full carbonization. Such view has been recognized earlier (Tang and Bacon, 1964) in cellulosic materials.

$$(C_6H_{10}O_5)_{n(s)} + O_{2(g)} \longrightarrow \underset{\text{Char Carbon}}{C_4} + nCO_{(g)} + nCO_{2(g)} + 5nH_2O_{(g)}$$

This chemical process commences early enough that by about 500^{0}C char carbon is formed as earlier reported (Morterra *et al*, 1984). However, at higher carbonization temperatures, after full carbonization has occurred, it is very likely that conversion of the char carbon to CO or CO_2 in the presence of oxygen occurs. This leads to reduction in the equilibrium weight of the carbonized product. The reduction becomes pronounced at higher temperatures, larger particle sizes and at longer carbonization time.

$$C_{(s)} + O_{2(g)} \longrightarrow 2CO_{(g)} + C_{n-1} \quad \text{(reduced mole of char) (reduced wt of char)}$$

$$\text{or} \quad C(s) + O_{2(g)} \longrightarrow CO_{2\,(g)} + C_{n-1} \quad \text{(reduced mole of char)}$$

The latter reactions do not only reduce the quantity of carbonized product (char carbon) the nature, pore size and shape as well as activity would also be attenuated. Such effects are most probably due to excessive burning /oxidation and collapse of pore structures which predominate at higher temperatures and longer resident (carbonization) times.

The percentage ash and moisture (Table 3) of the carbonised product show that both parameters decreased with increasing particle size. The presence of ash plays a vital role in decolourising carbon as earlier reported (Abraham, 1974). The present work shows that the most effective particle size for carbonization of coconut shell is 500 um which had 1.03 and 5.05 percentage ash and moisture contents respectively. It has also been shown in previous sections that particle sizes too far from 500um are generally not effective and as such higher or lower ash and moisture contents than the values obtained at the characteristic particle size may not be good for carbonized product from coconut shell. Table 4 indicates low metal content of K, Na, Ca, Mg, Fe and Zn.

Table 3: Effect of particle size on ash and moisture contents of carbonised carbon from coconut shell.

	Particle Size								
Property	150	250	355	425	500	710	860	1410	2000
% ash	2.49	2.03	2.24	1.13	1.03	0.78	0.53	0.42	0.29
% moisture	10.01	8.03	7.85	6.07	5.50	3.91	3.03	2.82	-

Table 4: Percentage metal content in carbonized coconut shell

Metal	K	Na	Ca	Mg	Al	Fe	Zn	Pb	Cr	Hg
% Metal	3.71	1.17	0.27	0.23	ND	0.11	0.05	ND	ND	ND

While the alkaline and alkaline-earth metals had higher % values than Fe and Zn, Al, Pb, and Cr were not detected in the carbonised product by the AAS method. The relatively low metal content shows that coconut-shell contains more of organic matter than inorganic compounds a situation considered favourable for production of carbonized carbon.

CONCLUSION

The present work has shown that optimum parameters for effective and full carbonization of coconut shell include a particle size of 500 µm, carbonization temperature at 500^0C and carbonization time of 5 minutes. Below these conditions, there is generally incomplete carbonization due to partial or incomplete removal of volatile products resulting in pore blockade and less adsorptive capacity. Beyond these conditions, the carbonized product (charred carbon) are generally reduced in quantity and activity most probably due to excessive or post-carbonization process of oxidation. For an effective carbonization product, the ash and moisture contents of the coconut shell should be approximately 1.03 and 5.50%, respectively, under the optimum conditions.

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