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Thermal Degradation and Particle Size Distribution Studies of Activated Carbon obtained from Coconut Shells

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Abstract

Potassium hydroxide as a chemical activator successfully produced activated carbon based coconut shells. The coconut shells were collected from a market in Enugu State, Nigeria, sun-dried and ground into powder. The powdered sample was carbonized for 1 hour at 300°C and then activated with KOH. The thermal stability and particle size distributions of all the samples were then determined using thermogravimetric analysis and dynamic light scattering techniques. The TGA results show that the trend of activated carbon 1:0 < activated carbon 1:1 < activated carbon 1:2 corresponds to the increase in the proportion of KOH. The particle size distribution results of all the samples reveal polydispersity due to their PDI > 0.1 and as well show the presence of additional peaks at small sizes which may be due to additives or buffer components from the activating agents. Thus, both TGA and DLS findings reveal that the prepared activated carbon-based coconut shells are of good thermal stability and particle size distribution

1. Introduction

Activated carbon is an amorphous form of carbon, microcrystalline, non-graphitic in nature, a product of carbonization and activation of carbonaceous material which has been specially treated so that it possesses a very high internal porosity due to its large surface area [1]. A vast number of materials can be used to produce activated carbon; almost any organic matter with a large percentage of carbon could theoretically be activated to enhance its adsorptive characteristics. Two distinct types of activated carbon recognized commercially are (i) Liquid-phase carbon and (ii) Gas-phase carbon [2]. The three major processes of producing activated carbon are Carbonization, purification and activation [1]. The effectiveness of activated carbon as an adsorbent is attributed to its unique properties, including large surface area, a high degree of surface reactivity, universal adsorption effect, and favourable pore size [3]. Activated carbons are used for the following: Sugar Decolourization, Solvent and solution reclamation, refining of oil and fat, removal of impurities, water purification, metal ions removal, decolorizing, drying and degumming of petroleum fractions, removal of industrial odour, removal of small quantities of radioactive contaminant [4]. Owing to this universal usefulness and large applications, research on the use of activated carbon has attracted the interest of different scientists [5]. Hence, effort is made in this study to prepare activated carbon from coconut shell wastes to reduce its environmental pollution due to improper disposal and the same time harness its economic value. Environmental contamination resulting from improper disposal of agricultural wastes has been a great challenge to all the stakeholders in the field [6]. As a matter of fact, different techniques and materials have been applied for remediation of the contaminants from the environment but are not fully efficient [7]. So, researchers are working day in and day out to make sure that environmental pollution is minimized using cheap, biodegradable and eco-friendly materials like coconut shells. These brought about this study. The importance of activated carbon cannot be overemphasized, thus leading to the search for means for its products other than the known traditional methods, due to its applicability in all life's endeavours. Based on this, research is continuously embarked upon to engage the use of different agricultural wastes under various conditions to produce activated carbon. The production of activated carbon using coconut shells should help in reducing the environmental pollution constituted by improper disposal of large quantities of the shells produced annually as wastes. At the same time, activation of carbon using KOH should enhance its thermal stability and average particle sizes. Thus, this study is to prepare activated carbon from coconut shells using different concentrations of KOH as an activation agent and characterise the produced activated carbon for its thermal stabilities and particle size distributions.

2. Methodology

2.1 Sourcing and preparation of

The main chemical (KOH) used in this study was of analytical grade (Sigma-Aldrich, Germany). Coconut shells used for the activated carbon production were obtained from a market in Enugu State, Nigeria and taken to the chemistry laboratory at Prince Abubakar Audu University Anyigba, Kogi State for use.

2.2 Experiments

The coconut shells were thoroughly washed with tap water, sun dried for a week and ground. Sieve size 1.0 – 1.5 mm was used to sieve the ground shells to obtain a uniform size. 200 g of the ground shells was weighed, placed in a crucible and subsequently carbonised in a muffle furnace at 300°C for 1 hour. The hot carbonised coconut shells were allowed to cool and then sieved through of 1.0 – 1.5 mm sieve-size to get uniform particles. These processes were repeated twice after which the pyrolysed coconut shells of 50 g were weighed into three different beakers and treated using KOH at different ratios (1:0, 1:1, 1:2) for 90 minutes to obtained the activated carbons in form of pastes. The pastes were then washed with distilled water until neutral pH to remove excess KOH. The washed pastes were oven-dried at 120°C for 5 hours to obtain the activated carbon samples and the samples were stored in an airtight labelled sample bottle for characterisations.

The yield of activated carbon was determined by weighing 200 g of the coconut shells into three burners. These were carbonized separately and the carbonized products were cooled to room temperature and weighed to calculate the yields.

2.3 Product characterisation

The characterisations of the prepared activated carbon were carried out using thermogravimetric analysis and dynamic light scattering techniques for the determination of their thermal stabilities and particle size distribution respectively. To determine the thermal stabilities of the prepared activated carbons, 5.00 mg of each of the samples were heated from ambient temperature 30 to 600°C, at a heating rate of 10°C/min under a nitrogen atmosphere using a TGA-STA 449F3 NETZSCH5 analyser.

Particle size distributions of the prepared activated carbons were determined in a water medium by a laser particle size analyser (BT-9300Z, Better size Instruments Co., Ltd., Dandong, China).

3. Results and Discussion

3.1 Percentage Yield of Carbonized Product and Thermograms of the prepared activated carbons

The thermal stabilities of the prepared samples were determined using thermogravimetric analysis (TGA) and the results are shown as TGA and DTG curves in Figures 1a and b respectively. Information obtained from the curves is presented in Table 1.

The percentage yields of carbonized products were given in Table 1**.** Three different activated carbons were prepared from coconut shells using KOH as an activating agent and they were identified as follows; activated carbon 1:0 activated carbon 1:1, and activated carbon 1:2. The result of the yield of carbonized products is stated in Table 1. The coconut shells were found to have a higher yield and the values obtained are in the range of those reported by [8] and [9].

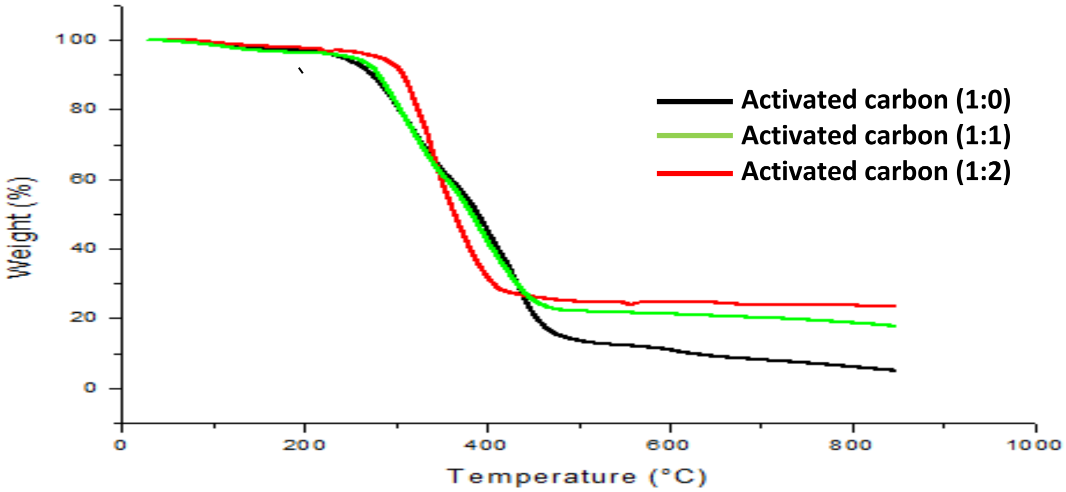

Figure 1a: Thermograms of the prepared activated carbons

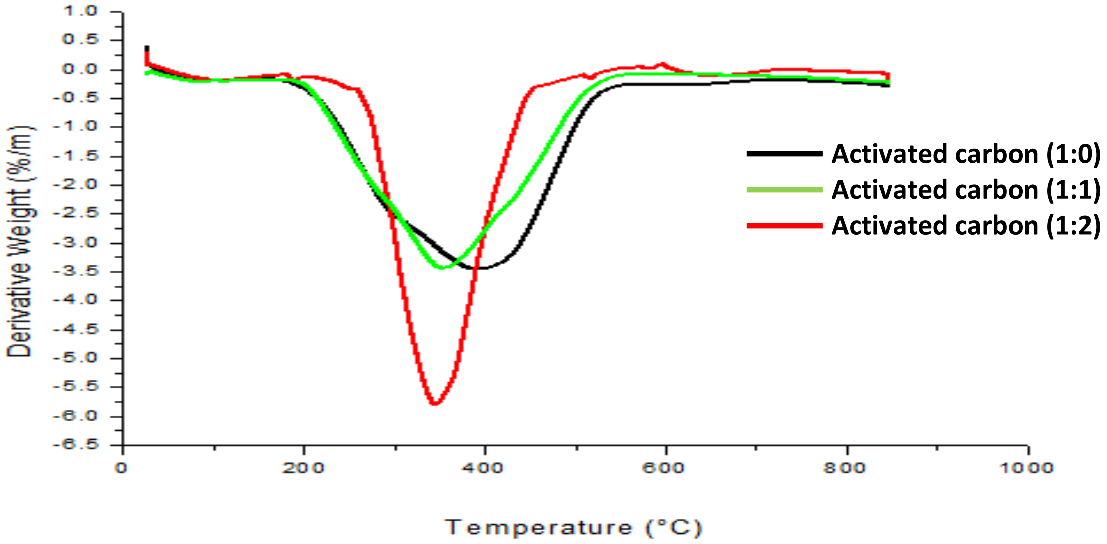

Figure b: Thermograms of the prepared activated carbons

IDT (or onset): Initial decomposition temperature is the point where a material starts disintegrating and is the measure of the thermal stability of that material. It can be seen that the TG curves are smooth, with only two weight loss steps which shows that the thermal degradation of activated carbons (Activated carbon 1:0, Activated carbon 1:1 and Activated carbon 1:2) in a nitrogen atmosphere is simple and is a one-step reaction apart from the loss of moisture. The first weight loss at temperatures around 100^{0}C for activated carbons respectively is associated with the loss of low molecular weight volatile matter and moisture. Moisture content is the amount of moisture in a sample given as a percentage of the sample's original (wet) weight [10].

From Table 2, 4.76 and 5.56% moisture contents for Activated carbon 1:1 and Activated carbon 1:2 respectively were observed at higher temperatures than that for activated 1:0 (4.56). It implies that they contained more bonded moisture which can be attributed to the interaction of KOH that are hydrophilic materials [11]. The variation in the moisture content could be connected to the different proportions of KOH present in the samples. Meanwhile, it has been reported that the activation by KOH usually results in higher moisture content due to the formation of –OH on the activated carbon by KOH [12]. Also, it is believed that, due to the distinct hydrophilic nature of KOH, activated carbon with more amount of KOH is expected to be more hydrophilic [13, 14]. Hence, this claim was observed for the prepared activated carbons. The ash content of activated carbon is the residue which remains when the carbonaceous portion is burnt off and ash determination is important only for organic adsorbents like activated carbon [15, 16].

Table 1: Percentage yield of carbonized shells

Raw material	Weight before carbonized (g)	Weight after carbonization (g)	Percentage yield (%)	Average yield (%)
Coconut shells	200	62.10	31.05	31.55
	200	64.00	32.00	
	200	63.20	31.60	

Table 2: Thermal behaviours of the prepared activated carbon

Sample	Moisture content (%)	Ash content (%)	Fixed content (%)	Onset degradation temperature (^{0}C)	Peak degradation temperature (^{0}C)
Activated carbon (1:0)	4.56	27.84	67.60	225.45	350.20
Activated carbon (1:1)	4.72	23.10	72.18	246.11	400.18
Activated carbon (1:2)	5.56	18.83	75.61	295.99	339.61

Obviously from the thermal information in Table 2, a higher amount of ash contents of 27.84 and 23.10% obtained for activated carbon (1:0) and activated carbon (1:1) compared to activated carbon (1:0) at temperatures above 500°C was attributed to the low proportion of KOH as an activating agent. From the results, activated carbon 1:0 has the lowest fixed carbon. It was expected that samples with the lowest fixed carbon will have the lowest adsorption capacity and this finding agreed with that of [12]. Activated carbon 1:2 has the highest percentage of fixed carbon and high adsorption capacity in the adsorption test.

The second weight-loss stage which commences at onset degradation temperatures of 225.45, 246.11 and 295.99°C, illustrates the rapid weight loss of activated carbon 1:0, activated carbon 1:1, and activated carbon 1:2 at temperatures of 200 to 600°C respectively. This rapid degradation may be attributed to the formation of ash as a result of devolatilization [17]. More so, the higher values of the onset degradation temperatures of the activated carbon 1:1 and activated carbon 1:2 show that they were more thermally stable compared to activated carbon 1:0. The trend in the increase in thermal stability of the samples is activated carbon 1:0 < activated carbon 1:1 < activated carbon 1:2 which corresponds to the increase in the proportion of KOH used for activation [18]. Thus, the activated carbon 1:2 is the most thermally stable of the samples.

Particle size distributions of the prepared activated carbon

The particle size distributions of the prepared samples were determined using dynamic light scattering (DLS) and the results are shown as charts in Figures 2 a, b and c respectively. Information obtained from the charts is presented in Table 3.

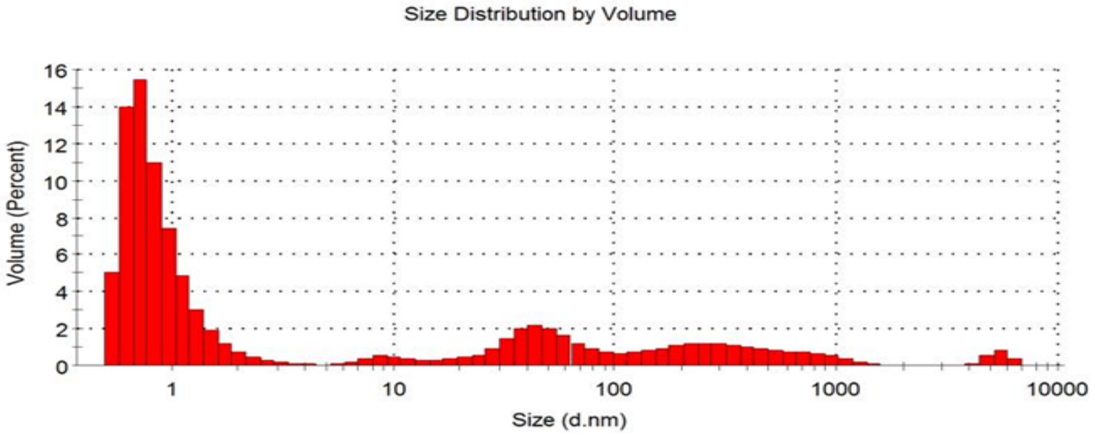

Figure 2a: Particle size distribution chart for the activated carbon 1:0

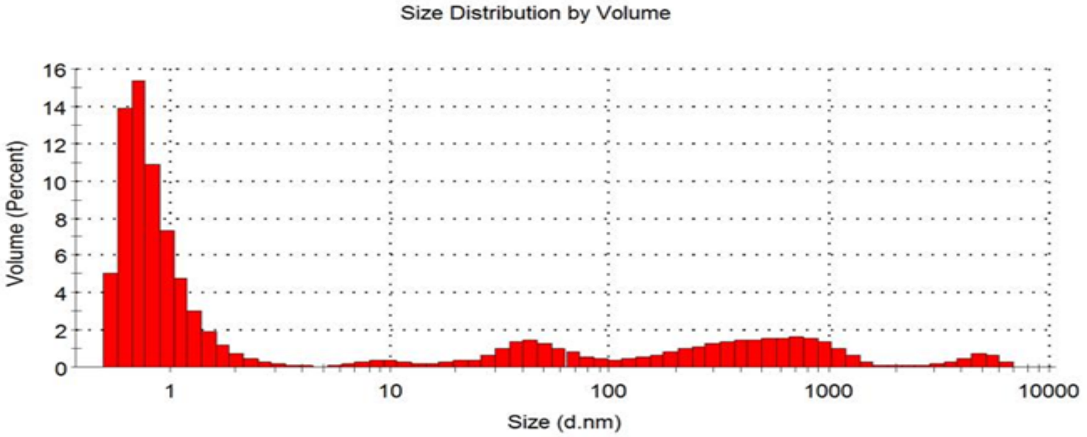

Figure 2b: Particle size distribution chart for the activated carbon 1:1

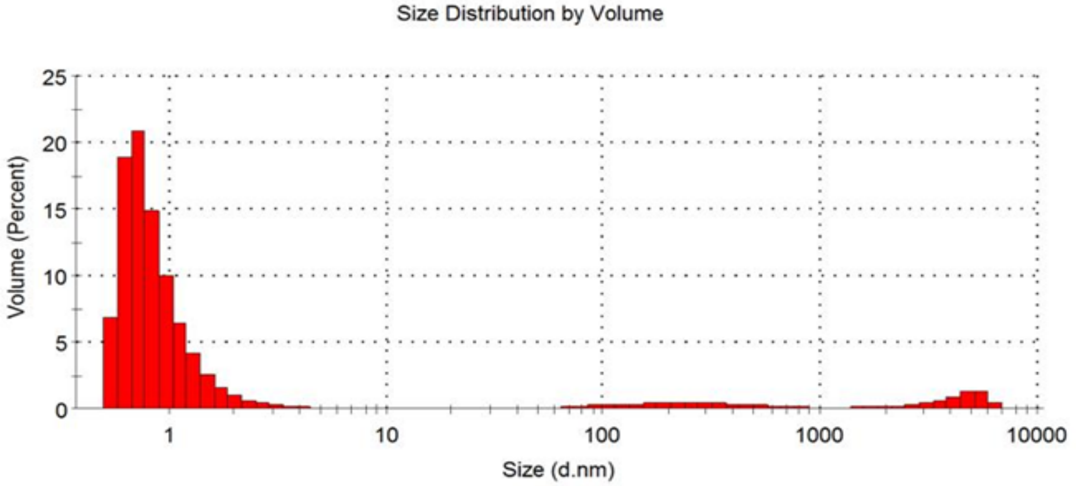

Figure 2c: Particle size distribution charts for the activated carbon 1:2

Table 3: Results of size distribution of the prepared activated carbons

Sample	Particle sizes (nm)	PDI (nm)	Peak 1 (nm)	Peak 2 (nm)	Peak 3 (nm)
Activated carbon (1:0)	102.00	0.694	0.881	10.18	48.87
Activated carbon (1:1)	78.80	0.696	49.96	10.23	0.881
Activated carbon (1:2)	69.29	0.881	282.2	0.885	4392

Dynamic light scattering is a technique for measuring the characteristic particle size distribution and dispersive index of a substance typically in the sub-micron region. Figure 2 a, b, and c show that the particle size distributions by volume of the prepared activated carbons have three peaks and the recorded peak sizes are as shown in Table 3. As a whole, the characteristic particle sizes for the activated carbon 1:0, activated carbon 1:1 and activated carbon 1:2 were recorded as 102, 78.80 and 69.29 nm respectively. This showed that the particle sizes of the samples decreased as the proportion of the activating agent (KOH) increased in the samples. This thereby results in to increase in specific surface area which may be attributed to less agglomeration caused by the presence of KOH **[19]**. Table 3 shows that based on the particle size distribution; activated carbon produced was mostly 50– 150 nm in diameter with more than 80% of particles of size less than 120 nm. According to the Chinese activated carbon classification standard (GB/T-32560-2016), the activated carbon of particles mostly of less than 180 nm is classified as powdered. Thus, the produced activated carbon in this study is therefore powdered in nature. This increase in the specific surface area of the samples is typically linked to an increase in the adsorption potential of the samples [20]. Hence, among the prepared activated carbons samples, activated carbon (1:2) can be the best used as an adsorbent.

Another important parameter measure using DLS is the polydispersity index (PDI) of a material. The PDI is a measure of the variability of sizes of molecules or particles in a material. It is used to describe the degree of non-uniformity of the size distribution of particles [21]. PDI which ranges from 0 to 1 displays the width of particle size distribution, indicating the nature of dispersion [22]. In this study, the values of PDI were 0.694, 0.696 and 0.881 for the activated carbon 1:0, activated carbon 1:1 and activated carbon 1:2 respectively. The PDI values 0.694 and 0.696 for activated carbon 1:0 and activated carbon 1:1 were due to the non-uniformity of their particle sizes, which show the constricted size distributions due to clumping of particles while the value 0.881 for the activated carbon 1:2 indicate that the sample has a very wide particle size distribution. Meanwhile, all the samples were of polydisperse particle size distributions because their PDI are more than 0.1 [23]. However, the presence of additional peaks (peak 2 and peak 3) at small sizes may be due to additives or buffer components from the activating agents and they are usually referred to as solvent peaks that could cause an average size lower than the expected peak size [24].

Conclusion

From the results of this work, it is concluded that coconut shells can be used as raw materials for the preparation of activated carbon. TGA results show that activated carbons obtained from coconut shells by chemical activation technique have good practical thermal stability. One of the originalities of this work is the use of KOH as the activating agent during carbonization. Results showed that treatment of the coconut shells with KOH solution during carbonization increased the values of particle sizes and surface area of the activated carbon prepared. The activated carbons obtained from KOH treated coconut shells gave better particle size distribution and good polydispersion indices. KOH impregnation improved the particle sizes of the prepared activated carbon.

Activated carbon has numerous industrial applications depending on its nature and adsorption potential. To increase the surface area of the activated carbon produced from coconut shells, it is recommended to carry out experiments with different experimental parameters. Some of the most critical parameters are the impregnation ratio, heating rate, and carbonization temperature and carbonization time. Future efforts in this study should include the optimization of the effect of KOH concentrations. To get more idea about the kinetics of carbonization of the impregnated samples, it is better to examine the gaseous products. Therefore, it is also recommended to carry out the carbonization experiments in a fixed bed reactor. To decrease the ash content value, it is recommended to change the heating rate.

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Conflict of Interest: The authors declare that there are no conflicts of interest.

Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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