

RESEARCH PAPER

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBONS FROM *ALBIZIA SAMAN* POD

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ABSTRACT

Activated carbon was prepared from the pods of Albizia saman for the purpose of converting the waste to wealth. The pods were thoroughly washed with water to remove any dirt, air-dried and cut into sizes of 2-4 cm. The prepared pods were then carbonised in a muffle furnace at temperatures of 400°C, 500°C, 600°C, 700°C and 800°C for 30 minutes. The same procedure was repeated for 60, 90, 120 and 150 minutes respectively. Activation was done using impregnation ratios of 1:12, 1:6, 1:4, 1:3, and 1:2 respectively of ZnCl₂ to carbonised Albizia saman pods by weight. The activated carbon was then dried in an oven at 105°C before crushing for sieve analysis. The following properties of the produced Albizia saman pod activated carbon (ASPAC) were determined: bulk density, carbon yield, surface area and ash, volatile matter and moisture contents. The highest surface area of 1479.29 m²/g was obtained at the optimum impregnation ratio, carbonization time and temperature of 1:6, 60 minutes and 500°C respectively. It was recommended that activated carbon should be prepared from Albizia saman pod with high potential for adsorption of pollutants given the high surface area obtained.

Keywords: *Albizia saman pod, activated carbon, carbonization, temperature, surface area*

INTRODUCTION

Heavy metal contamination of water bodies is of public health concern because of their toxic effects on the environment and human health. Some metals such as silver, mercury, cadmium and copper are toxic even at very low levels. Among these toxic heavy metals, cadmium, mercury and lead have been called "the big three" and are in the lime light due to their major impact on the environment (Volesky, 1994;

Volesky and Holan, 1995).

Adsorption is a cost-effective method for the removal of metals (Balkose and Baltacioglu, 1992; Shah *et al.*, 2009; Rahmani *et al.*, 2009) from aqueous solutions. The major advantage of an adsorption system for water pollution control is that it involves less investment in terms of both initial equipment and land cost, simple design and easy operation and no ef-

fect of toxic substances compared to other treatment processes (Markovska et al., 2006). Activated carbon was used as an adsorbent for removal of heavy metal pollutants from wastewater and has proved to be effective.

The conventional activated carbon used in adsorption is usually expensive (Mckay, 1981; 1982). This has stimulated interest in examining the feasibility of using cheaper raw materials. Agricultural adsorbents such as coffee husk (Kumar, 2006), peanut shells (Wafwoyo et al., 1999), mango seed shells (Akpen et al., 2011, 2015), bagasse (Azhar et al., 2005), rice husk (Kumar and Bandyopadhyay, 2006), wheat bran (Dupond and Guillon, 2000), cassava waste (Abia et al., 2003) and sawdust (Argun et al., 2007) have been developed. The use of *Albizia saman* (alternatively called *Samanea saman*) pod as an adsorbent for the removal of cadmium in adsorption columns has not been reported. *Albizia saman* pods are renewable agricultural wastes available abundantly at little or no cost. The elemental composition of *Albizia saman* pod presented in Table 1 shows a high carbon content of 43.61% indicating that it is a good precursor for production of activated carbon.

The use of *Albizia saman* pods as an adsorbent will reduce solid wastes in the environment by converting the agricultural waste to wealth, boost *Albizia saman* plantation, provide employment and hence, improve standard of living of the people. Similarly, cadmium (II) is of

great public health concern because of its toxic effects such as itai-itai disease, cancer, ulceration of the skin, liver and kidney damage in animals and humans. The effective removal of cadmium from industrial wastewaters of industries such as phosphate fertilizer and nickel-cadmium batteries manufacturing industries will improve public health.

MATERIALS AND METHODS

Albizia saman pods sampling and preparation of adsorbent

The *Albizia saman* pods were collected from Makurdi Local Government Area of Benue State, Nigeria. The pods were air-dried and broken to free the seeds. The pods were then thoroughly washed with water to remove any dirt, air-dried and cut into sizes of 2 – 4 cm before carbonization.

Carbonization

Carbonization was performed in a muffle furnace (model; ME 45/12 sob) manufactured by Arnold Schröder (industrieöfen GmbH), Germany. Nine hundred grams (900 g) of the air-dried sample (*Albizia Saman* Pod) was weighed into clean stainless steel containers and placed in the furnace and maintained at a pre-set temperature of 500°C for 30 minutes. This was repeated for 60, 90, 120 and 150 minutes. The carbonization was repeated at different temperatures of 400, 600, 700 and 800°C and at various times of 30, 60, 90, 120 and 150 minutes. This was done to determine the optimum carbonization temperature and time.

Table 1: Elemental composition of *Albizia saman* (*Samanea saman*)

Element	Composition (%)
carbon	43.61
Hydrogen	5.45
Nitrogen	6.28
Oxygen	44.66

Source: (Mythili et al., 2013)

Activation

Carbonised samples weighing 400g each were mixed in standard solutions (0.25M, 0.5M, 0.75 M, 1M, and 1.25M) of zinc chloride (ZnCl₂) and allowed to soak for 24 hours at room temperature. Thereafter, the liquid was discarded and the activated carbon dried in an oven at 105⁰C over night before crushing for sieve analysis. The standard solutions of zinc chloride were prepared by dissolving 34.07 g, 68.145 g, 102.22 g, 136.29 g, and 170.36 g of anhydrous zinc chloride in 1000 ml of distilled water respectively. This resulted in impregnation ratios of 1:12, 1:6, 1:4, 1:3, and 1:2 respectively of ZnCl₂ to carbonised *Albizia saman* pods by weight.

Characterisation of activated carbon produced

Sieve analysis

The activated carbon produced was crushed and sieved through sieve sizes of 850, 600, 425, 300, 150 and 75 µm and those retained on sieves 75, 150, 300, 425 and 600 µm, were used for the adsorption studies.

Carbon yield

As in Fapetu, (2000), the weight, W₀, of each air dried *Albizia saman* pod sample was measured. The carbon yield, Y_{ch} was calculated according to equation 1.

$$Y_{ch} = 100W_{ch} / W_0 \dots\dots\dots (1)$$

Where, W_{ch} is the weight of carbon retrieved from the furnace and W₀ is the weight of air dried *Albizia saman* pod sample.

Bulk density (g/cm³)

Bulk density was estimated using the method of Ahmedna *et al.* (1997), which consisted of placing a known weight of ASPAC of sieve size of 0.03 cm in a 25 ml cylinder to a specified volume and tapping the cylinder for at least 1-2 min and measuring the volume of carbon after settling. The bulk density was calculated as:

$$B_d = W_s / V_s \dots\dots\dots (2)$$

Where B_d = bulk density (g/cm³), W_s = weight of dry ASPAC sample (g), and V_s = volume (cm³) of packed sample.

Surface area

The surface area was determined by the method described by Rouquerol *et al.* (1999) thus: A_s (m²/g) = 6/ (B_dx D_s)

Where A_s = surface area, B_d= bulk density (g/m³) and D_s = particle diameter (m).

Ash content determination

As in Dara, (1991), two grams of the dried ASPAC sample was placed into a porcelain crucible of known weight and transferred into a pre-heated muffle furnace set at a temperature of 900⁰C. The furnace was left on for one hour after which the crucible and its content were transferred to a desiccator and allowed to cool. The crucible and content were reweighed and the weight noted. During this test, the crucible was left open. Ash content (dry basis) was calculated as equation 4:

$$\text{Ash (\%)} = \frac{W_i - W_f}{W_i} \times 100 \dots\dots (4)$$

Where; W_i is the initial weight of crucible and ASPAC sample and W_f is the final weight of crucible and ASPAC sample.

Volatile matter

As in Hesse, (1997), two grammes of oven dried ASPAC was placed in a closed crucible. It was then heated up to 900⁰C and maintained for 10 minutes in the furnace. The crucible was then cooled in a desiccator and reweighed. The difference in weight (W_p) was noted and the volatile content determined using equation 5.

$$V_c = \frac{2 - W_p}{W_d} \times 100 \dots\dots (5)$$

Where V_c = volatile content (%), W_p = difference in weight and W_d = oven dry weight of 2g

of ASPAC.

Moisture content

A sample of activated carbon was weighed into a petri-dish. It was well spread on the dish. It was then heated at 105°C for 3 hrs. The petri-dish was left open during the heating process. After heating, the petri-dish was removed, cooled in a desiccator and then weighed and the moisture content calculated thus:

$$M_{ch}(\%) = [(W_0 - W_1) \times 100] / W_0 \quad \dots\dots (6)$$

Where M_{ch} =moisture content, W_0 = original weight of sample before heating (g) and W_1 = final weight of sample after heating for 3 hr (g).

RESULTS AND DISCUSSIONS

Carbonization temperature and time for formation of *Albizia Saman* pod activated carbon (ASPAC)

For ASPAC produced after 30 min and 60 min of processing, the surface area of ASPAC increased from 675.904 m²/g at 400°C to 1194.743 m²/g at 500°C and decreased to 458.926 m²/g at 800°C (Fig. 1). Among the activation temperatures investigated, that of

500°C gave the highest surface area in the activated carbon (Fig. 1). At higher temperatures, such as 800°C, more ash was produced because the burn-off was higher causing the formation of tars that filled the pores of the samples. Increasing temperature should ordinarily lead to increase in porosity by the release of tars from the cross-linked framework formed by chemical impregnation. However, studies done by Ahmadpour and Do (1996, 1997) suggested that temperature above 500°C resulted in the loss of weight, shrinkage in carbon structure and porosity reduction. This trend is similar with the findings of Teng *et al.* (1998) and Girgis and El-Hendawy (2002). Anisuzzaman *et al.* (2015) also reported a similar trend on the removal of 2, 4-dichlorophenol by modified commercial activated carbon. Teng *et al.* (1998) stated that extensive contraction during thermal treatment collapses the porous structure.

As can be observed on Fig. 1, the surface area of ASPAC increased from 1064.396 m²/g for material processed for 30 minutes to 1194.743 m²/g after 60 minutes of processing at the temperature of 500°C for instance. However, the

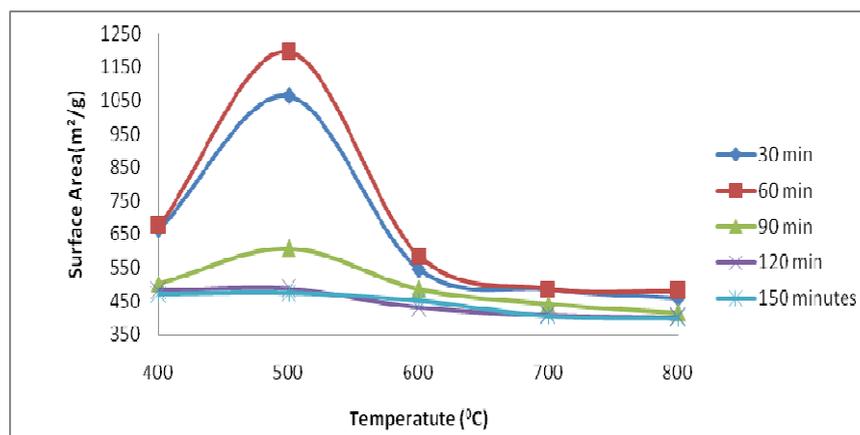


Fig. 1: Surface Area (m²/g) of ASPAC at various carbonization temperatures and times (Impregnation ratio = 1:3, Particle size = 0.03 cm)

surface area decreased to 471.809 m²/g for ASPAC processed for 150 minutes at the same temperature of 500°C. The carbonization time of 60 minutes gave the highest surface area in the activated carbon. At longer times, such as 120 minutes and above, the surface area was reduced because more ash was produced because the burn-off period was longer.

Effect of impregnation ratio on surface area of ASPAC

The surface area of ASPAC was 1267.427 m²/g at impregnation ratio (IR) of 1:12 and it increased to 1479.29 m²/g at impregnation ratio (IR) of 1:6 for carbonization time of 60 minutes (Fig. 2). However, the surface area decreased to 1184.133 m²/g when the IR was further increased to 1:2. The trend was similar for other contact times investigated as the surface area increased to a maximum at IR of 1:6 and decreased with further increase in the IR. This finding is similar to the study done by Girgis and El-Hendawy (2002). Introduction of ZnCl₂ beyond the optimum degree of impregnation might form an insulating layer covering the particles that will reduce the activation process and the contact with the surrounding atmos-

phere (Girgis and El-Hendawy, 2002), leading to a lower surface area available for adsorption. Owabor and Iyaomolere (2013) explained that the decrease in surface area observed with increasing concentration of activating agent maybe due to a breakdown in the physical structure of the precursor caused by excessive pore pathways there by leading to shrinkage in particle size and hence surface area of the AC.

On the other hand, the lower surface area development recorded at lower IR like 1:12 was because less ZnCl₂ was available for the activation reaction per given quantity of *Albizia saman* pod. While the increase in surface area with increase in impregnation ratio was due to increase in pore formation because of the enhanced reaction between the readily available ZnCl₂ and the *Albizia saman* pod. A similar trend was reported by Uner *et al.* (2015) on the preparation of AC from waste watermelon rind using ZnCl₂ as the activating agent.

Characteristics of *Albizia Saman* pod activated carbon (ASPAC)

The characterizing parameters (surface area, bulk density, ash content, volatile content,

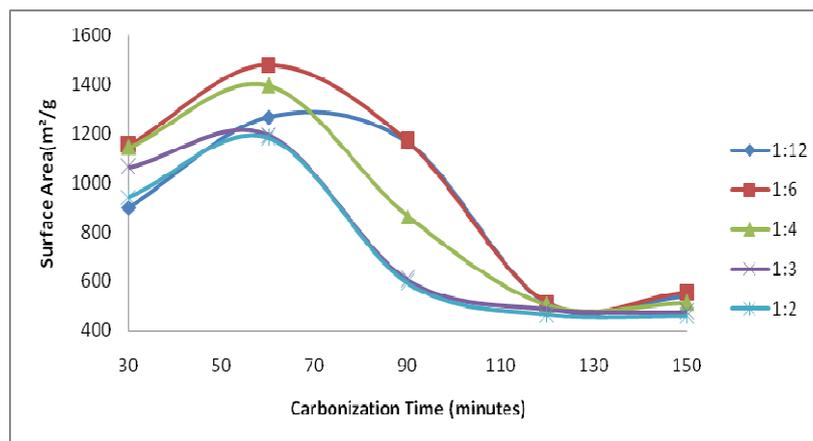


Fig. 2: Surface area of ASPAC at various impregnation ratios and times (Temperature = 500°C, Particle size = 0.03 cm)

carbon yield, and moisture content) were investigated and the results are shown in Figs 3-5. The optimum characteristics are presented in Table 2. The bulk density was lowest at the carbonization temperature of 500°C for all the carbonization periods investigated (Fig. 3). The least value of 0.167 g/cm³ was obtained at carbonization time and temperature of 60 minutes and 500°C respectively. Fig. 4 revealed a least

bulk density value of 0.135 g/cm³ corresponding to impregnation ratio of 1:6, carbonization time and temperature of 60 minutes and 500°C respectively. These values gave the highest surface area of the activated carbon (Fig. 2) and were chosen as the optimum values because the higher the surface area and porosity of a carbon, the higher the adsorptive capacity of the carbon (Karthikeyan *et al.*, 2008, Aloko and

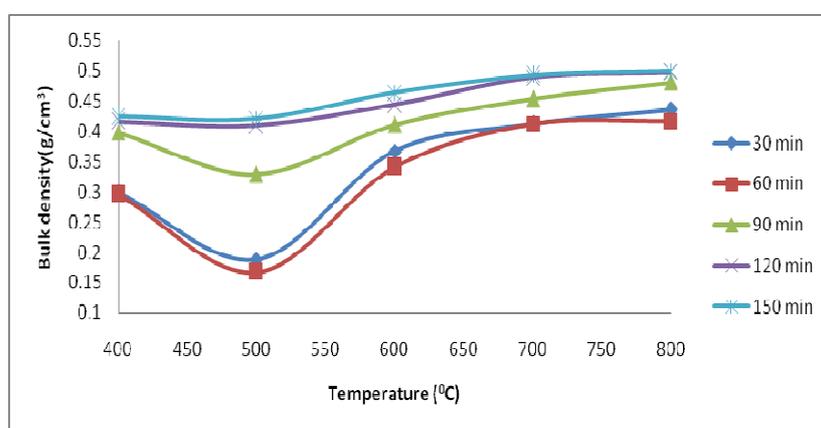


Fig. 3: Bulk Density of ASPAC at various carbonization temperatures and times (Impregnation ratio = 1:3, Particle size = 0.03 cm)

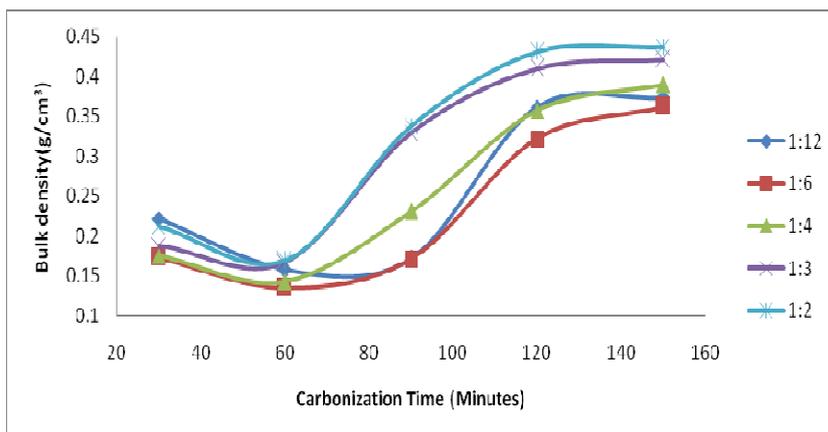


Fig. 4: Bulk density of ASPAC at various impregnation ratios and carbonization times (Temperature = 500°C, Particle size = 0.03 cm)

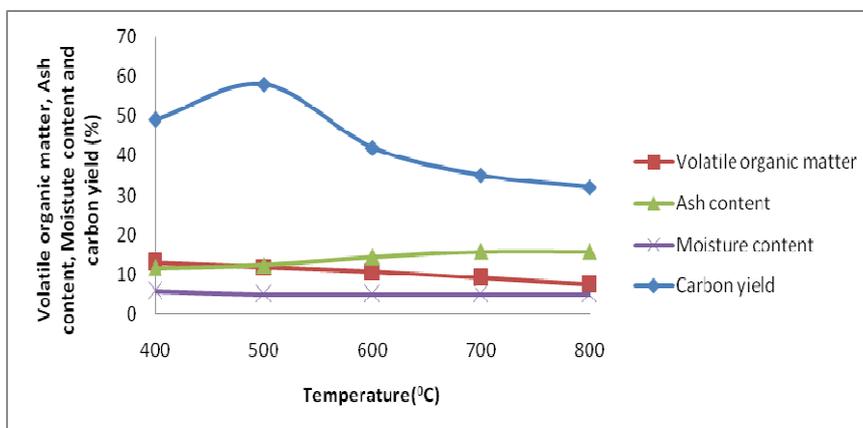


Fig. 5: Other characteristics of *Albizia saman* pod activated carbon (ASPAC) (Impregnation Ratio = 1:6, time = 60 minutes)

Table 2: Optimum conditions for preparation/characteristics of *Albizia Saman* pod activated carbon (ASPAC)

S/N	Parameter	Value
1	Carbonization temperature (°C)	500
2	Carbonization time (min.)	60
3	Impregnation ratio	1:6
4	Bulk density (g/cm ³)	0.135
5	Surface area (m ² /g)	1479.29
6	Moisture content (%)	5
7	Carbon yield (%)	58
8	Volatile organic matter (%)	11.7
9	Ash content (%)	12.35

Adebayo, 2007). The result is in agreement with common knowledge that the lower the bulk density of activated carbon (AC), the higher the surface area of the carbon.

It is observed in Fig. 5 that increase in carbonization temperature resulted in a corresponding increase in ash content, while volatile matter and carbon yield generally decreased with increase in temperature. It follows that the low ash content yielded comparatively more active

carbon sites for adsorption. The same result was reported by Mythili *et al.* (2013) and Abdullah and Gerhauser (2008). The highest carbon yield of 58% obtained at 500°C points to the fact that *Albizia saman* pod is a high yielding precursor for the production of AC which is in line with the results of the elemental analysis reported by Mythili *et al.* (2013) which gave a carbon content of 43.61%.

CONCLUSION AND RECOMMENDATIONS

The optimum conditions for the production of activated carbon from *Albizia saman* pods were investigated in this research. The results obtained revealed optimum carbonization temperature and time of 500°C and 60 minutes respectively. The impregnation ratio of 1:6 gave the highest surface area (1479.29 m²/g) for adsorption. Activated carbons produced at the optimum values of impregnation ratio, carbonization time and carbonization temperature had bulk density and carbon yield of 0.135 g/cm³ and 58% respectively. It is recommended that activated carbons should be prepared from ASPAC with high potential for adsorption of pollutants given the high surface area obtained.

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