PRODUCTION AND CHARACTERIZATION OF ACTIVATED CARBON FROM LEATHER WASTE, SAWDUST, AND LIGNITE

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ABSTRACT

Powdered activated carbon (PAC) was prepared from leather buffing waste, sawdust and lignite by carbonization at temperatures between 500 – 800°C followed by steam activation. Experimental results reveal a general decrease in yield of carbon residue with increase in temperature of carbonization. Samples of lignite recorded the highest yield (49.80 – 67.70%) followed by leather buffing dust (30.70 – 39.70%) and sawdust (25.10 – 37.20%). Activated carbon from these precursors, were also evaluated for percentage ash, fixed carbon, pH and bulk density. Adsorption studies carried out with methylene blue indicate that low temperature carbonization of precursors such as leather buffing waste favour production of carbon with better adsorption efficiency while high temperature carbonization produced carbon with better efficiency from sawdust and lignite. Activated carbon from sawdust and leather buffing waste show result which compare favourably with the reference carbon used. These carbons are recommended for use in the adsorption of dyes or decolourization of organic compounds and other substances in aqueous solutions.

Key words: Activated carbon, carbonization, Steam activation, Adsorption efficiency, Leather buffing waste

INTRODUCTION

Activated carbon has been found to be the common product used in arresting or controlling pollutants in soil, air or water. (Baksi et al., 2006; Pollard et al., 1992) It is a substance known to possess extensively developed porous structure and large surface area (Ahmedna et al., 2000).

Almost any carbonaceous material can serve as precursor for the production of activated carbon (Rodriquez – Reinoso, 1997). However, the common feedstocks for its commercial production include anthracite and bituminous coal, lignite, lignocellulosic materials, wood and coconut shell (Yenkie et al., 1993). Research has also been undertaken on agricultural by-products such as grain hulls, corn cobs (Young, 1996; Ahmedna, et al., 2000), almond shell, bamboo (Baksi et al., 2006) and sewage sludge (Rozada et al., 2003).

Activated carbon can be produced by a two-staged process of carbonization followed by activation at temperature of 500 -800°C (Christopher et al., 2000) The raw material may be impregnated with common activating agents such as zinc chloride or phosphoric acid and calcined at the desired temperature. The efficacy of activated carbon in removing pollutants or contaminants depends on the type of feedstock, operating conditions during carbonization and method of activation (Heskel and Close 1995; Christopher et al., 2000).

The objective of this study is to produce, characterize and determine the adsorptive efficiency of activated carbons from leather buffing waste from the tanning industry and from conventional precursors such as sawdust and lignite using similar methods of preparation. The efficiency of these carbons would further be compared with commercially, available activated carbon.

EXPERIMENTAL

Sample Collection and Preparation

Samples of leather buffing dust were collected from tanneries located in Challawa industrial estate in Kano – Nigeria. Sawdust samples were collected from a saw mill at Katako "timber shed" in Jos, while lignite was obtained from National Metallurgical Centre NMDC also in Jos. The samples were dried in an oven at 100°Cfor a period of 6hrs pulverized and passed through a mesh size of 0.50 mm.

Carbonization of Samples

The various samples were carbonized at temperatures between 500 – 800°C, using a muffle furnace (Phoenix Furnace-Alpha Model).

Twenty grams each of leather buffing dust, sawdust and lignite were placed in three clean pre-weighed crucibles. They were then heated for 30 mins on attaining the temperature of 500°C. The samples were then removed and quenched in three separate beakers, one for each sample. The samples were subsequently dried at 100°C in an oven for 2hrs, cooled in desiccators and stored in sample bottles. The same procedure was adopted for carbonization of the samples at 600°C, 700°C and 800°C.

The percentage yield of carbon residue of each sample after carbonization was calculated.
The samples were coded as follows:
BD_5-BD_8: for buffing dust carbonized at 500– 800°C.
SD_5-SD_8: for sawdust carbonized at 500– 800°C.
LG_5- LG_8: for lignite carbonized at 500– 800°C.

Activation of Carbonized Samples

The technique of steam activation was employed in the activation of the carbonized sample.
Twenty grammes (20g) of each carbonized sample was impregnated with 40% solution of zinc chloride and kept for 24 hrs with intermittent stirring at ambient temperature.

An improvised steam activator was developed to carry out the activation process. It consisted of a steam generator (steam can) connected to a tubular pyrex glass reactor which is heated from beneath by a finken burner.

The reactor was manually rocked to ensure uniform distribution of steam and heat as they came in contact with the carbon. For each sample, a slurry was made and fed into the Pyrex glass reactor. The activation of each sample was carried out for a resident period of 30 mins before the process was discontinued.

After activation each sample was washed with 30% phosphoric acid followed by repeated reflux in a soxhlet with distilled water. All samples were further washed with distilled water and filtered until aliquots of the last $100cm^3$ were neutral to blue litmus paper. The samples were then dried, pulverized and sieved through a mesh size of 0.25mm.

Determination of Ash Content and fixed carbon
Ash constituents of activated carbon represent the amount of inorganic residue in it. The ash content of

$$B.D = \frac{W_2 - W_1}{1}$$

Where:

W_2 = Weight of micropipette + sample
W_1 = Weight of micropipette
1 = Volume (cm^3) of micropipette occupied by sample

Determination of pH of Activated Carbons
The pH of the various activated carbons produced was determined using a portable pH meter by taking the pH of $50cm^3$ aliquot of the final $200cm^3$ of the filtrate which was obtained after thorough washing of the carbons after activation with distilled water.

Determination of Amount of Methylene Blue Adsorbed
This test was carried out by contacting standard solution of methylene blue with known amounts of the activated carbons produced and measuring the absorbance of the solutions at pre-determined wavelength of maximum absorbance (520nm) for the solution. The amount of the dye (methylene blue) adsorbed from the solution can then be determined.

each activated carbon produced was determined by adopting the method described in ASTMD2667-75[8] For each sample 2g was put in a clean pre-weighed crucible and placed in a muffle furnace at 600^oC for 15 minutes after which each sample was cooled in a desiccator and weighed. This procedure was repeated with the resident period extended to 1 hr until a mass constant within 0.2mg was obtained.

From the weight of the residue in the crucible, the percentage ash content of each sample was calculated.

Determination of Apparent Bulk Density
The bulk density of all samples of activated carbon produced was calculated using weight of a unit volume ($1cm^3$) of each sample. This was done by weighing a micropipette and carefully filling it with each sample of the activated carbon to the $1cm^3$ mark after tapping several times. The micropipette with each sample was then weighed. The bulk density of each sample was calculated using the formula:

In this study, 0.5g of each activated carbon was weighed and added to separate $50cm^3$ of methlene blue solution (containing 100mg/L methylene blue) in a $250cm^3$ cornical flask. The mixtures were shaken vigorously with a mechanical shaker for a period of 1hr after which they were filtered using filter paper (Whatman No.42). The absorbance of each filtrate was measured at the pre-determined wavelength of maximum absorbance of methylene blue (λ_{max} 630nm) using a spectrophotometer.

From the data obtained, the amount of methylene blue adsorbed by each activated carbon was calculated and expressed in percentage using the following formula:

$$\% \text{ MB adsorbed} = \frac{AB_b - Ab_a}{AB_b} \times 100$$

Where:
AB_b = Absorbance of MB before carbon dosage
AB_a = Absorbance of Mb after carbon dosage.

Effect of Activated Carbon Dosage on Decolourization of Methylenene Blue
Different doses of each activated carbon, in the range of 8 – 18 g were contacted with $50cm^3$ solution containing 100mg methylene blue to determine the optimum dose to effect the best adsorption. The result obtained was recorded.

RESULTS AND DISCUSSION
Table 1 represents percentage carbon residue after carbonization of the samples at temperature range of 500 – 800^oC. The yield of carbon residue in sample of lignite was greater than the other two precursors.

This can be understood from the fact that lignite being a coal-based compound, is likely to volatilize to a much lesser extent, thus less weight loss is experienced especially during low temperature pyrolysis (Gergora et al., (1994). It can also be observed that there was a general downward trend in yield of carbon residue with increase in temperature of carbonization for all sample used.

The apparent bulk density, pH, percentage ash and fixed carbon of each sample of activated carbon produced are presented in table 2. Lower apparent bulk densities (0.32 – 0.34) were recorded for carbon from sawdust with slight variations in values obtained.

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The value obtained for the reference carbon (0.37) came closer to the carbon from sawdust than the apparent densities of carbon from leather buffing waste (0.43 – 0.53 and lignite (0.71 – 0.81) which increased with increase in temperature of carbonization. This can be attributed to the fact that high bulk density in activated carbon is usually associated with feedstock having high carbon content (http//www.answers.com). The apparent bulk densities of activated carbon from sawdust fell far below the ASTM recommended values of 0.42 – 0.52g/cm while that from leather waste was within this range but for sample BD8. Lignite based activated carbon however recorded higher values for reasons previously stated.

It can be observed that despite the high yield (69.3%) of carbon residue recorded in samples of lignite (Table 1), the same sample exhibited lower amount of fixed carbon after activation when compared with the other precursors. This is because lignite being of mineral origin is likely to contain an appreciable amount of minerals. Moreover, the carbons from lignite may still contain some hydrocarbon withholding mineral components especially after low temperature pyrolysis.

The percentage ash and fixed carbon from all the precursors exhibit little variation at the various temperatures of carbonization indicating negligible effect of temperature on these parameters. Apart from activated carbon from leather buffing waste with ash content of 4.34 – 4.44%, carbon from other precursors had the higher figures in terms of ash than the ASTM recommended maximum value of 5%. This suggests further modification and thorough washing of the activated carbons to remove soluble mineral matter.

The pH of the activated carbon also presented in table 2 was in the range between pH 6.3–pH 6.9. This can be as a result of washing out of the ortho-phosphoric acid used after activation with distilled water until it was almost neutral to blue litmus paper. After carbonization and activation, it is conventional to wash activated carbons thoroughly with distilled water after an acid wash, thus bringing the pH of most of them within this range.

Generally, the colour adsorption property is important in considering the quality of activated carbons and is measured in terms of percentage methylene blue adsorption. The higher the percentage adsorption, the better the quality of the activated carbon (Kalappan et al., 2000) Results of adsorption studies carried out by determining the quantity of methylene blue adsorbed by the various carbon in aqueous solution shown in figure I and II indicate that

sample of activated carbon from leather buffing waste carbonized at 500^oC and that from sawdust carbonized at 800^oC show very good adsorption capacity comparing favorably with the reference carbon at a carbon dosage of 12 - 14g/l and resident period of 1 hr while activated carbon from lignite do not give encouraging degree of adsorption of the methylene blue especially at low temperature of carbonization. There was no significant improvement in adsorption even at higher carbon dosages. This invariably means that low temperature carbonization produces carbon with better activity from precursors such as leather buffing waste while high temperature carbonization produces carbons with better activity from sawdust and lignite. The recorded low performance of lignite can be attributed to its not being adequately carbonized at these temperature and or period used in this study hence it would not possess adequately developed pores and surface area for adsorption. The good adsorption quality of the activated carbon stated above can be due to their possessing well developed porosity.

The result from these adsorption studies reveal that for some precursors such as leather buffing waste , higher temperature of pyrolysis does not necessarily result in higher adsorption capacity of the resultant activated carbon. The low temperature involved can reduce cost in terms of energy consumption.

CONCLUSION
From the results obtained, it can be concluded that low temperature carbonization of precursors such as leather buffing waste favored the production of carbon with better adsorption activity while high temperature carbonization produced carbons with better activity from sawdust and lignite. The adsorption properties of activated carbon from leather buffing waste compare favorably, with those from conventional precursors such as sawdust and even better than those from lignite under the same experimental condition.

The low temperature of carbonization involved and high yield of carbon residue obtained from sample of leather buffing waste makes the production of activated carbon from it more cost effective produce than the other precursors used in this study.

The performance of the samples of the activated carbon produced is encouraging, thus further modification can also be recommended to increase their adsorption capacity and serve as effective adsorbent.

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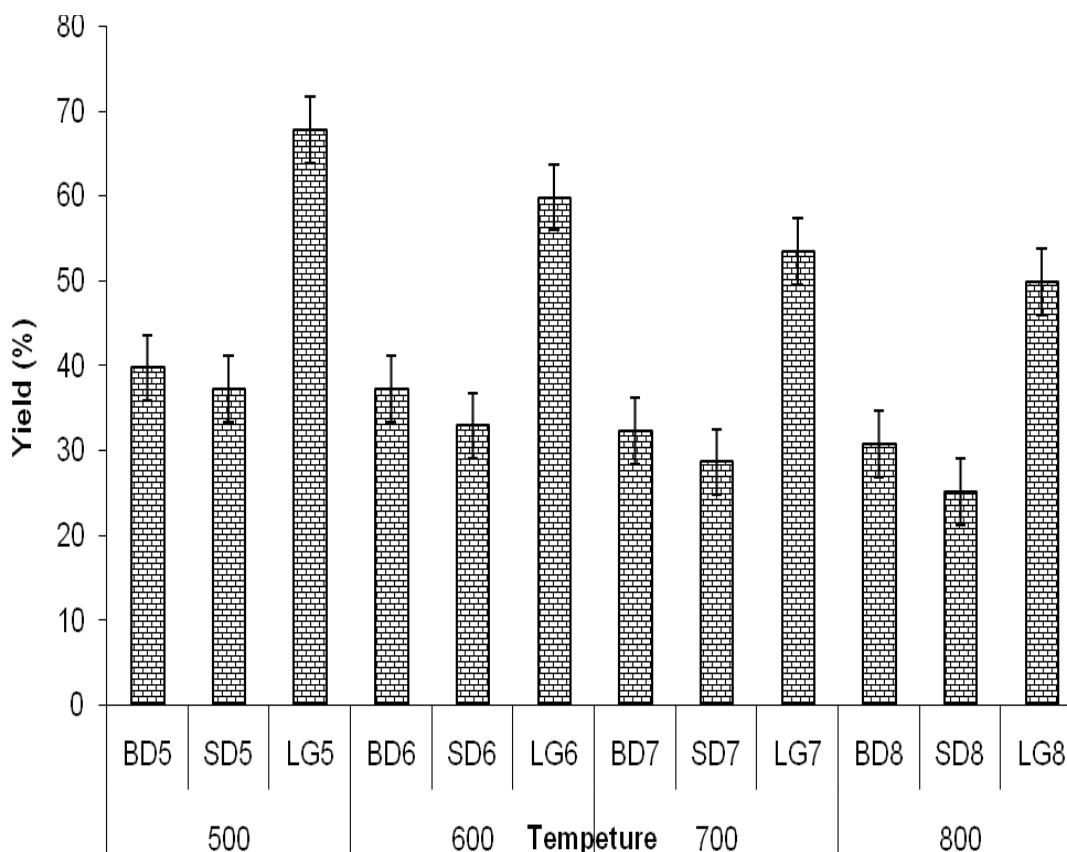

Figure 1: Percentage Yield of Carbon Residue after Carbonization at 500 – 800°C

Table 1: Physical and chemical properties of activated carbons

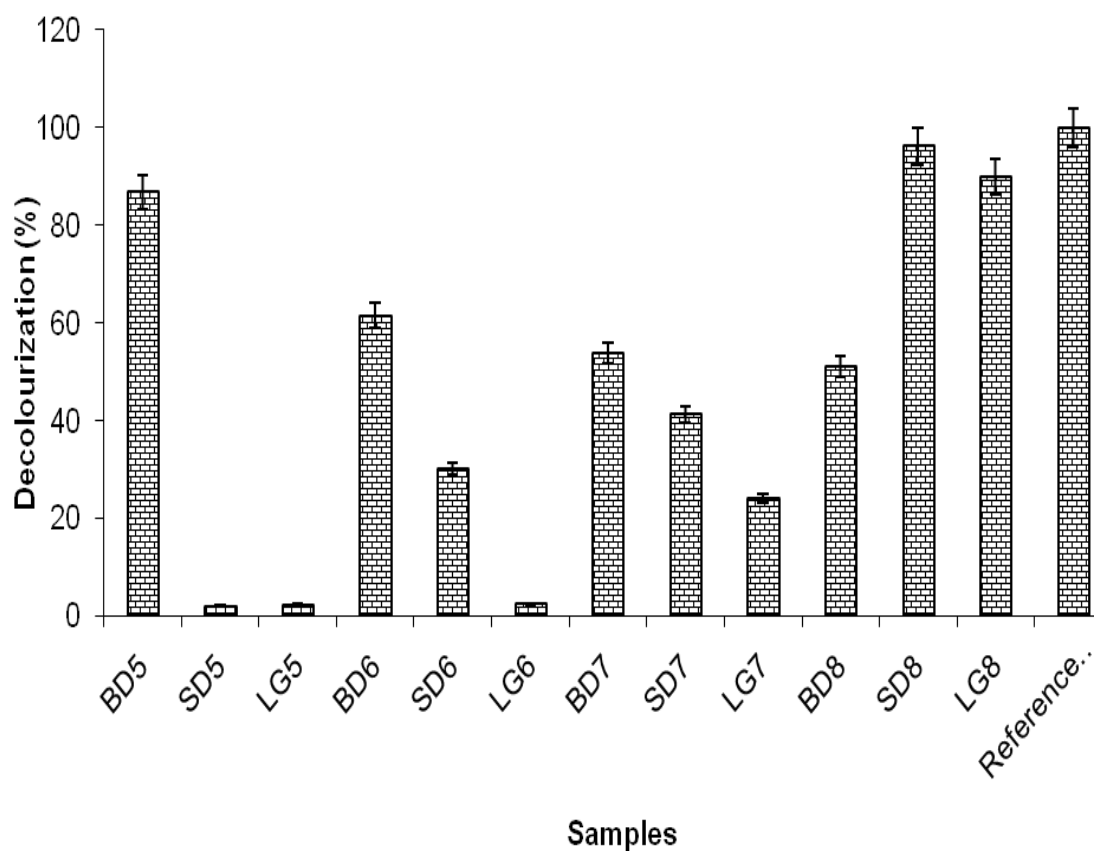| Sample	Apparent Bulk Density (g/cc)	pH	Ash (%)	Fixed Carbon (%)
BD5	0.43	6.7	4.34	95.66
SD5	0.33	6.8	5.23	94.77
LG5	0.71	6.6	9.98	90.02
BD6	0.46	6.9	4.38	95.62
SD6	0.34	6.9	5.35	94.65
LG6	0.74	6.8	11.62	88.38
BD7	0.5	6.7	4.55	94.45
SD7	0.33	6.9	5.54	94.46
LG7	0.76	6.8	11.63	88.37
BD8	0.53	6.3	4.44	95.56
SD8	0.32	6.5	5.78	94.22
LG8	0.81	6.9	11.61	88.39
Reference carbon	0.37	6.7	-	-

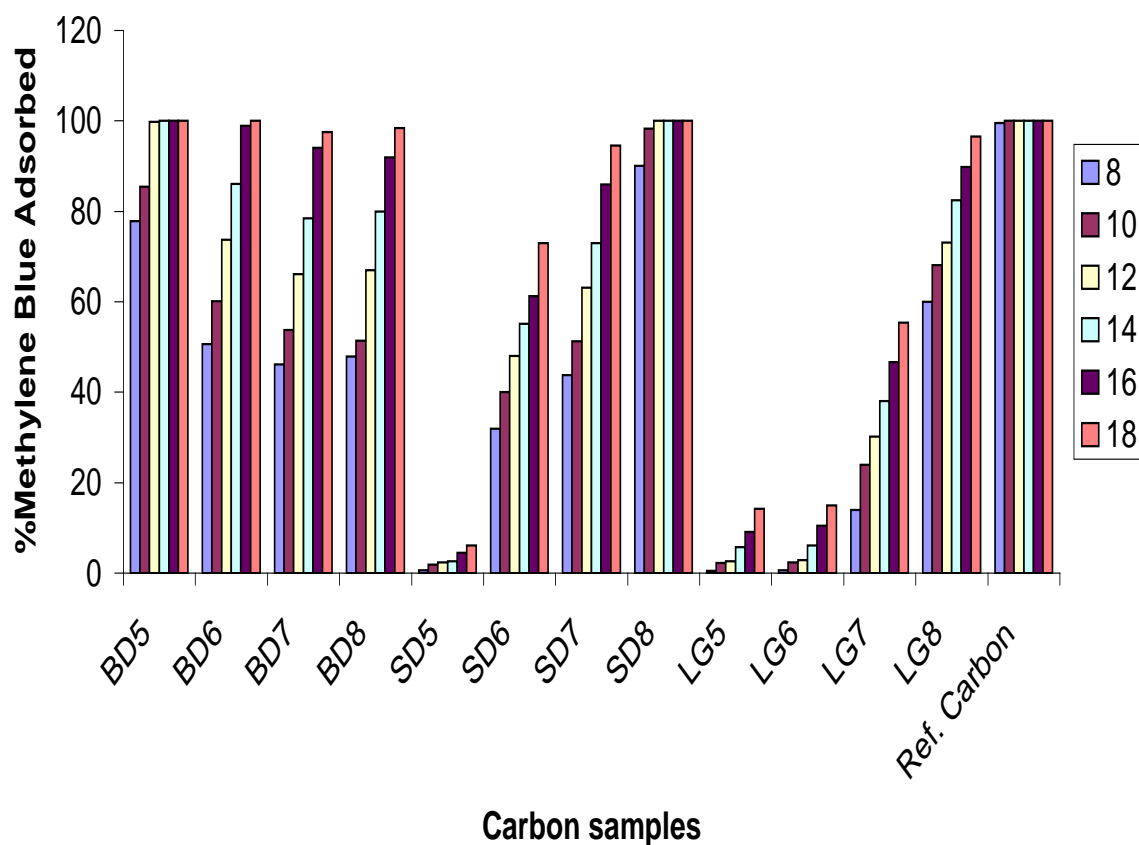**Figure 2: Percentage adsorption of methylene blue by activated carbons**

Figure 3: Effect of Carbon Dosage on adsorption of Methylene Blue