J. Chem. Soc. Nigeria, Vol. 48, No. 5, pp 928 - 935 [2023]

ACTIVATION AND CHARACTERIZATION OF CARBON OBTAINED FROM COCONUT SHELLS.

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ABSTRACT

The objective of this project was to conduct physical and chemical characterization of the carbon derived from carbonized coconut shells using a charcoal kiln at a temperature of 656°C. The carbon of particle sizes of 0.5mm and 2mm where activated using three chemical activating agents H_3PO_4, KOH, and NaCl. The physico-chemical characterization conducted includes particle size analysis, Bulk density, pH, moisture content and iodine number determination using standard procedures. The analysis of the three activated carbons indicated that under conditions of preparation, the activated carbon possess high efficiencies of activation with iodine number ranging from 662.88mg/g - 854.37mg/g (H_3PO_4), 216.09mg/g -850.37mg/g (KOH) and 262.78mg/g -846.02mg/g (NaCl) for 0.5mm activated carbon and for 2mm activated carbon, iodine number values were 318.48mg/g -836.02mg/g (H_3PO_4), 150.90mg/g-818.43mg/g(KOH) and 206.78mg/g-712.75mg/g (NaCl).

KEYWORD: iodine number, activated carbon, coconut shells, carbonization.

INTRODUCTION

In Nigeria many people depend on biomass for their household energy. Charcoal has been the most preferred fuel in the country because gas is costly, kerosene is scarce and power supply is erratic. Charcoal production is a common technique for converting biomass into useful energy source and as raw materials for food, cosmetics, pharmaceuticals, plastic and textile industries. Charcoal can be produced from some NIFOR biomass such as palm kernel shells, coconut shells, Raphia tree stalk, Shea nut shells and date palm tree stalk. This project seeks to design and implement a rural adaptable process for Charcoal or Carbon production with utilization potential of food, non-food products in order to enhance or deepen the downstream section of the value chains of NIFOR mandate crops. The importance of activated carbon cannot be over emphasized due to its applicability in all of almost all of life's endeavours. The production of activated carbon from Agricultural by-products has potential economic and environmental impacts. It converts unwanted Agricultural wastes to high value adsorbents [1]. Activated carbons are increasingly used in water to remove organic chemicals and metals of environment and also it helps to reduce importation of activated carbon into the country. Carbon is a porous solid material that is the result of combustion of precursor materials containing carbon elements while activated carbon is carbon that is activated by immersion in chemicals or by flowing hot steam into the material so that the

ISSN 0795 - 22066

pore size of the material becomes more open with a surface area ranging between 1000 - 1400m2/g. The adsorption properties are selective, depending on the size or volume of the pores and surface area. Carbon activation can be done in two ways, either by physical activation or chemical activation. The physical activation results from heat (steam) treatments and it consists of carbonization and pyrolysis., which is a process that involves the conversion of the precursor materials to carbon through high temperatures of heating The results of pyrolysis are solids (charcoal/carbon), gas (fuel gas), and liquid (bio-fuel) and other products are gases such as carbon dioxide (CO_2), methane (CH_4) etc. In chemical activation different chemicals such as NaOH, KOH, ZnCl, HCl, H_2SO_4, H_3PO_4 etc. are used as activation agents [2].

Precursor Material.

Fig 1*: *Coconut Shells

Fig 2: Activated Charcoal/Carbon

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MATERIALS AND METHODS

Sample collection and preparation

Coconut shells were collected from ADBEEL FOODS GRA, in Benin City.

The coconut shells were washed with clean water to remove sand and dirt and it was sun dried for two days.

All chemical used were of analytical grade and standard.

Carbonization

Carbonization was done using the constructed metal drum kiln. The precursor material (Coconut shells) were measured and put into the kiln, and covered and it was heated at high temperature of above 656^{o}C. An infra-red thermometer (8000series KMAT1600) was used to read the temperature. The process of carbonization was repeated for one more batch and the carbon was allowed to cool for some hours.

Chemical Activation

Chemical activation was done on the carbon using three chemicals which are orthophosphoric acid (H_3PO_4), Potassium hydroxide (KOH) and sodium chloride (NaCl). For the Charcoal/Carbon, to obtain Particle sizes of 0.5mm and 2mm, Particle Size determination was done using AFNOR particle size sieves following standard procedures.

Activation in three different chemical media were done using the procedure of Kra *et al*, 2019. with slight modification.

For chemical Activation with orthophosphoric acid [H_3PO_4], 75g of 2 mm and 0.5 mm carbon of coconut shell was weighed into two different beakers and 1000ml of 10% H_3PO_4 acid was added and the mixture was stirred and it was allowed to stand for 24 hours.

For KOH Activation of the carbon, 75g of 0.5mm and 2mm carbon was weighed in 2 beakers, 1000ml of 1M KOH solution was added and the mixture was stirred and it was allowed to stand for 24hours.

Also for the NaCl activation of the carbon, 75g of 0.5mm and 2 mm carbon were weighed and put into 2 different beakers and 1000ml of 1M solution of NaCl was added, the mixture was stirred and allowed to stand for 24 hours.

Finally, the slurry obtained was filtered and the activated carbon was dried in an oven at 105^{o}C for 24 hours. It was allowed to cool in a desiccator and then kept in air tight pack for further analysis.

pH Determination

The activated carbon samples of the three different chemicals used was washed with distilled water until a neutral pH was determined using a pH meter (Hanna pHep model HI98107).

Bulk Density Determinations

The determination of bulk density for the 0.5 mm and 2 mm particle sizes of the carbon was determined by using a 100 ml glass cylinder. The measuring glass cylinder of 100 ml volume was first zeroed on the weighing scale and each particle size carbon of either 0.5 mm or 2 mm was loaded into the 100ml glass cylinder. The cylinder was tapped for 1 -2 minutes to compact the carbon and weighed and the bulk density was calculated and presented as g/ml following this formular:

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$$Bulk\,density = \frac{mass\,of\ sample}{volume\,of\ measuring\ cylinder} \times 100$$

Moisture content Determination

1.0g of the carbonized samples of 0.5mm and 2 mm weighed in triplicate and placed in clean dried and weighed petri dishes. The petri-dishes with the samples were placed in an oven (Jenlab Drying oven Model 0-18L) at 105^{o}C for 4 hours to obtain a constant weight. The difference between the initial and final mass of the carbon represents the moisture content. It was calculated as

$$Moisture\,\% = \frac{Loss\,in\,weight\,on\,drying(g)}{Initial\,weight(g)} \times 100$$

Iodine Number Determinations

Iodine number is determined according to the ASTM D4607.94 2006 method. The iodine number is defined as the milligrams of iodine adsorbed by 1.0g of carbon where the iodine concentration of the filtrate is 0.02N [3]

A standard iodine solution[12.700g iodine and 19.100 of KI in 1L distilled H_2O] is treated with the three different weights (dosage) 0.3g, 0.5g and 0.7g of activated carbon under specified conditions.

Each sample of the carbon (0.3g, 0.5g and 0.7g) was put into 250ml conical flask and 10ml of 5% HCl was added and stirred until the carbon sample was well wetted. The mixture was boiled for 30s and then cooled. Then, 100ml of 0.1N iodine solution was added to the mixture and stirred for 30s. The resulting solution is filtered using a filter paper, an aliquot portion of 50ml of the filtrate is titrated with 0.1N solution of sodium thiosulphate using starch as an indicator. This procedure was done in triplicates,

Iodine number is calculated using this formular:

$$\frac{X}{M} = \left[\left(N_I \times 126.93 \times V_I \right) - \left(\frac{V_I + V_{HCl}}{V_F} \right) \right] \times \left(N_{Na\,2503} \times 126.93 \right) \times \left(\frac{V_{Na_2S_2O_3}}{MC} \right)$$

Where

$C = \left(N_{Na_2S_2O_3} \times V_{Na_2S_2O_3} \right)$

N_1 = Conc. I_2

V_1 = added volume of Iodine solution (100ml)

V_{HCl} = Added volume of 5% HCl = (10ml)

V_F = is the filtrate volume used in titration (50 ml)

$V_{Na_2S_2O_3} = Conc._{Na_2S_2O_3}$

MC = Mass of Activated carbon dosage

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RESULTS AND DISCUSSION

Table 1 Percentage Yield of carbon

Raw materials	Weight before carbonization (Kg)	Yield	Average percentage (%)	Time of carbonization (mins)
Coconut shells	10	1.768	17.68	60
Coconut shells	10	1.820	18.20	60

Table 2: Physical Characterization of the activated carbon from Coconut shells

Property	**0.5mm carbon sample**	**2 mm carbon sample**
Ph	6.58±0.05	7.24±0.01
Bulk Density (g/ml)	74.4±1.8	67.9±0.1
Moisture content %	10±0.00	10±0.00

Results are given as mean ± standard deviation (triplicate values)

Table 3a: Iodine number determination of Activated Carbon of 2 mm particle size

Dosage (grams)	**Activating Chemicals**		
	H_3PO_4 (mg/g)	**KOH (mg/g)**	**NaCl (mg/g)**
0.3	318.48	150.93	206.78
0.5	492.54	690.81	650.04
0.7	836.02	818.43	712.75

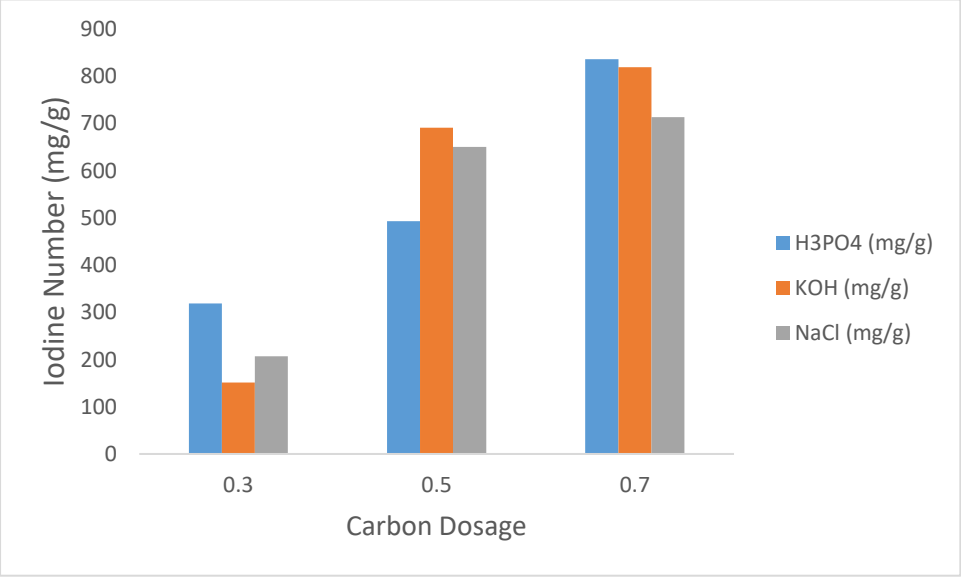

Figure 3a. Iodine adsorption chart of 2 mm activated carbon

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Table 3b: Iodine number determination of Activated Carbon of 0.5 mm particle size

Dosage (grams)	Activating Chemicals		
	H_3PO_4 (mg/g)	KOH (mg/g)	NaCl (mg/g)
0.3	662.88	216.09	262.63
0.5	690.81	693.60	685.22
0.7	854.37	850.38	846.39

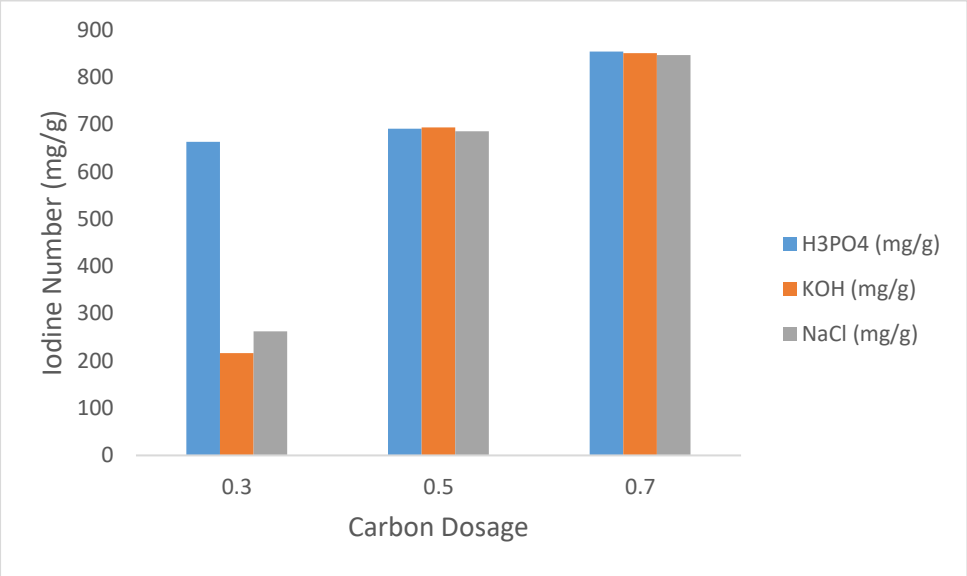

Figure 3b: Iodine adsorption chart of 0.5 mm activated carbon.

Table 1: Shows the percentage yield of the carbonized coconut shell carbon. It was observed that after carbonization at high temperature of 656^{o}C, 10kg of coconut shells done in 2 batches at 60mins gave a percentage yield of 17.68% and 18.20% respectively. The values obtained are in the range as reported by Dada *et al* [4]. Which indicated that activated carbon derived from coconut shells gave an appreciable percentage yield after carbonization.

Table 2: Shows the physical characterizations of the carbon after carbonization and activation. The percentage moisture content for the 0.5mm and 2mm AC was 10%, which indicated a low moisture content but higher than the values obtained by Efeovbokhan *et al* [5] which was 8.78%. The pH values of 6.58± 0.05 and 7.24 ± 0.01 respectively for the 0.5mm and 2mm indicated a neutral pH. While the bulk density obtained was 72.6g ± 3.6 for 0.5 mm and 67.9g ± 0.1 for 2 mm which indicated that activated carbon from coconut shells will serve as good adsorbent with high filtering capabilities.

Tables 3a and 3b with fig. 3a and 3b shows the iodine number values obtained with

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different activating chemicals used on the coconut shells carbon of 0.5 mm particles size and 2 mm particle size.

Chemical activation leads to the creation of new pores and enlargement of available pores, from mesopores to micropores, therefore having a greater iodine adsorption capacity. The determination of porosity and adsorbed capacity with iodine number helps to indicate that the coconut shell activated carbon has a very high number of small pores hence a higher iodine number [6].

The results of iodine adsorbed by the different carbon dosages of the sample at 0.3g, 0.5g and 0.7g are represented in figures 3a and 3b of 2mm and 0.5mm respectively.

The high iodine numbers obtained was due to the presence of micropores structures in the activated carbon of the coconut shells. As observed in table 3a and 3b.the iodine values obtained increased with increase carbon dosage [7].

From the three activating chemicals used H_3PO_4, had the highest iodine values compared to KOH and NaCl in the order of H_3PO_4 > KOH > NaCl for both 0.5 mm and 2mm activated carbons. The values obtained in the iodine number determination for 0.5mm were higher compared to 2mm particle size carbon because of the increase in surface area of the 0.5mm compared to the 2mm activated carbon which is granular. Similar observations were reported by Haimour and Emeish [8].

CONCLUSION

This project has been able to construct a carbonization kiln using a metal drum which was successfully used for the carbonization of the precursor material (coconut shells) to produce charcoal/carbon**.** The studies shows that the activated carbon derived from Coconut shells activated with H_3P0_4, KOH and NaCl gave high iodine numbers indicating that Coconut shells activated carbon has high micropores making it suitable as an adsorbent having high adsorption capacity [9]. Though H_3P0_4 gave the highest iodine number values in both 0.5mm and 2mm activated carbon making it a suitable activating agent in the preparation of activated carbon from coconut shells under appropriate process conditions.

ACKNOWLEDEMENT

The Authors wish to express their profound gratitude to the Director, HOD and staff of Biochemistry Division NIFOR, for making this project a success.

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ISSN 0795 - 22066