Production of Activated Carbon in a Multi-Functional Platform Pilot Plant*

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Abstract

Activated carbons were produced from coconut shells as raw materials in a Multi-Functional Platform (MFP) pilot plant using the chemical activation method.. Derived activated carbon named, C1, C2, C3, C4, C5, and C6, based on the activation agent used and the heating rate employed, were compared to a commercial activated carbon based on acceptable parameters. C1, C2, C3, C4, C5 and C6 had relative hardness of 95.67%, 92.67%, 89.67, 91.6%, 90.67% and 93.33% respectively, averaging 92.26%, as compared to 95.6% relative hardness for the commercial activated carbon used. C6 the best performing activated carbon produced (using KOH and heating rate of 7.67 ºC/min) , had an adsorption rate of 188.68 mg Au/ h g, which was higher than the adsorption rate of the commercial activated carbon, which is 185.19 mg Au/ h g. This validates the possibility of activated carbon production commercially in Ghana.

Keywords: Adsorption, Activated Carbon, Coconut Shells, Relative Hardness, Pilot

1 Introduction

Multifunctional Platform (MFP) is an innovative program developed by the United Nations Development Programme (UNDP), a UN sub-committee that provides expert advice, training, and financial support to developing countries such as Ghana, Mali, and Burkina Faso towards sustainable human development (Inkoom, 2008). This Programme was introduced in Ghana in 2005, to facilitate modern energy services to rural folks and also, increasing the generation of income and social service activities through enterprise services. These Programmes bring about the construction of pilot plants for various productions. And these pilot plants have successively less expensive compared to full-scale plants. The construction of the pilot plants depends on the following factors; reaction kinetics, material types, chemical equilibrium, thermodynamics, equipment selection, and agitation. These pilot plants come in various designs but normally follow these steps: pre-engineering, modeling and optimization, fabrication and assembling, and installation and startup (Inkoom, 2008). Currently, there are a lot of MFP pilot plants in rural areas all over Ghana. The MFP pilot plant, a rotary kiln, used in this research is located in Boankra in the Ashanti Region of Ghana. This pilot plant can be used in the production of biochar, syngas, bio-oil, and activated carbon. In this research, this pilot plant was used to produce activated carbon. Activated carbon (AC) is a specialized produced material with high adsorptive properties. It is produced from organic carbonaceous materials such as coconut shells, palm nut shells, wood, and coal. The process used in the production of activated carbon is either a chemical process, a physical process, or both (Ukanwa *et al.*, 2019; Bergna *et al.*,2018; Marsh and Reinoso, 2006). Activated carbon is widely used in gold mining operations, edible oil treatment operations, pharmaceutical operations, and wastewater treatment operations (Toledo et al., 2020; Meteku, 2013). These industries depend on the supply of activated carbon from outside the country and this increases the cost of production. Ghana is ranked 16th in production of coconut in the world, with the production of 366,183 tonnes of coconuts annually in 2016 (Ofori- Agyeman, 2016). With this high tonnage of coconut, Ghana has a high generation of coconut shells waste. Coconut shells are widely used in the production of activated carbon, which proves that Ghana has enough raw material for activated carbon production.

Most research conducted on activated carbon using either chemical activation or physical activation method are centered on laboratory-scale research. With the need of creating industries, employment, and cutting down the cost of production, these laboratories scale production of activated carbon needs to be developed upon into the pilot-scale production then the commercial-scale production to kickstart the production of ACs in Ghana. This research is to produce activated carbon using chemical activation methods in a Multifunctional pilot plant using coconut shells as raw materials.

2 Resources and Methods Used

2.1 Multi-Functional Pilot Plant

The MFP pilot plant is divided into two sections, the pyrolysis unit shown I Fig. 1 and the activation unit shown in Fig. 2, a rotary kiln.

Fig. 1 Pyrolysis Unit

Fig. 2 Activation Unit

The activation has two different heating inlets, heating inlet 1 and heating inlet 2, as shown in Fig. 3. Heating inlet 3 is denoted when both heating inlets are used simultaneously.
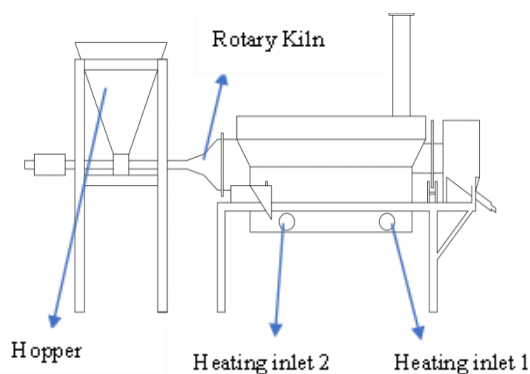

Fig. 3 Skeletal View of the Activation Unit

2.2   Preparation of the Activated carbons

Samples of coconut shells were collected. The coconut shells were washed to remove dirt, sun-dried, crushed, and sieved to obtain the +5 mm fractions, which were pyrolyzed at 500 ºC in Fig. 1. After carbonization, the char derived were crushed to obtain -4mm +2.8mm. The resulting chars obtained were chemically activated using sodium hydroxide and potassium hydroxide as activation agents in the activation unit using different heating rates. The activation unit has two heating inlets producing two different heating rates when used separately and a different heating rate when both are used simultaneously, resulting in three different heating rates. Six samples of ACs were derived and named as follows;

i. C_1 is the activated carbon generated from 5 kg char soaked in potassium hydroxide for 2 hours and heated in the activation reactor (MFP rotary kiln (Fig. 2)) using heating inlet 1.
ii. C_2 is the activated carbon generated from char soaked in potassium hydroxide for 2 hours and heated in the activation reactor (MFP rotary kiln (Fig. 2)) using heating inlet 2.
iii. C_3 is the activated carbon generated from char soaked in sodium hydroxide for 2 hours and heated in the activation reactor (MFP rotary kiln (Fig. 2)) using heating inlet 2.
iv. C_4 is the activated carbon generated from char soaked in sodium hydroxide for 2 hours and heated in the activation reactor (MFP rotary kiln (Fig. 2)) using heating inlet 1.
v. C_5 is the activated carbon generated from char soaked in sodium hydroxide for 2 hours and heated in the activation reactor (MFP rotary kiln (Fig. 2)) using both heating inlets 1 and 2.
vi. C_6 is the activated carbon generated from char soaked in potassium hydroxide for 2 hours and heated in the activation reactor (MFP rotary kiln) using both heating inlets 1 and 2.

After the 2 hours of heating in the activation unit (rotary kiln), that is activation, the ACs produced were water quenched. The ACs were washed first with 10% sulphuric acid to lower down the pH. Then the ACs derived were washed in a lot of tap water to achieve a pH of 7. After washing, the activated carbons derived were placed in an oven at $110\,^{\circ}$C overnight to eliminate moisture. In contrast, the two-step conventioal chemical activation process was used. After that, the yield was calculated from the relation below;

$$Yield\% = \left(\frac{m}{M}\right) * 100\% \qquad (1)$$

where,
m = mass of activated carbon after drying, g
M = mass of coconut shells used, g

2.3 Iodine Adsorption Test

Iodine number is a widely used parameter for activated carbon testing for its simplicity and a quick evaluation of adsorbent quality. It gives an approximation of the activated carbon's surface area and porosity. The iodine number is defined as the milligrams of iodine adsorbed by one gram of material when the iodine residual concentration of the filtrate is 0.02N (0.01 M) (Benzekri *et al.* 2018). A 0.3 g of activated carbon was weighed into a 100 ml conical flask and 25 ml of the 0.02 M iodine solution was added. The conical flask and its content were vigorously shook for 10 minutes and filtered by filter paper. Initial 5 ml of the filtrate was discarded and the remaining filtrate was titrated against the 0.1 sodium thiosulphate solution until a pale-yellow colour appeared. Two drops of the starch indicator were added and titration resumed slowly until a colourless solution appeared. The procedure was repeated two more times for each activated carbon produced. A titration was also repeated on a 20 ml 0.02 M iodine solution (not treated with activated carbon) to serve as a blank. Iodine adsorption number (IAN) was calculated from the relation below;

$$IAN\left(\frac{mmol}{g}\right) = \left(Mt\frac{Va-Vs}{2Ma}\right) \tag{2}$$

where,

Mt = the molarity of sodium thiosulphate, M
Va = volume of thiosulphate used for blank titration, ml
Vs = volume of thiosulphate used for titration, ml
Ma = mass of activated carbon used, g
This was done also for a commercial activated carbon (CC).

2.4 Adsorption Test

A 0.5 g sample of activated carbons produced from each activating agent and heating rates of the size range of -2.8 mm + 1.4 mm were placed into bottles containing 200 ml of gold solution. The bottles were placed on rollers for 24 hours. A 0.5 g of commercial activated carbon was also subjected to the same procedure. Samples of the solution were taken at various times and analysed for gold using a Varian Fast Sequential Atomic Absorption Spectrophotometer (Varian AA240FS).

2.5 Hardness Test

A 50 g of -3.36 um + 1.18 um activated carbons produced was placed on a 1.18 um sieve of 200 mm diameter. 20.7 mm diameter steel ball was added then shook for 10 minutes using a screen shaker. 30 g of the oversize after 10 minutes of shaking was left on the 1.18 um sieve together with the 20.7 mm diameter steel ball and shook for 60 minutes using a shaker. After 60 minutes the oversize was weighed and the hardness was calculated using;

$$Hardness\ \% = \left(\frac{mass\ of\ oversize\ after\ 60\ minutes}{mass\ of\ ACs\ used\ for\ the\ 60\ minutes}\right) * 100\% \tag{3}$$

3 Results and Discussion

3.1 Proximate Analysis of Raw Materials

The coconut shell used in this project have a very low ash content of 0.72% as shown in Table 1. This makes the coconut shells used a good raw material for activated carbon production. According to Bansal et al, (1998) and reported by Meteku, (2013), a low ash content grade of a raw material generates an activate carbon with low inorganic matter which increases its adsorption ability.

Table 1 Proximate Analysis of Coconut Shells used

Parameter	Value (%)
Moisture	6.80
Volatile Matter	80.05
Ash content	0.72
Fixed Carbon	12.43

3.2 Heating Rate

In Fig. 4, the heating rate for pyrolysis was 6.79° C/min, indicating slow pyrolysis, which is the best pyrolysis method for this research due to its high yield in biochar as compared to the fast and intermediate pyrolysis as indicated by Jonsson (2016) and Yoder et al. (2011). During the pyrolysis, it took the vapour two hours to stop flowing out of the pyrolysis reactor which is also an indication of slow pyrolysis.
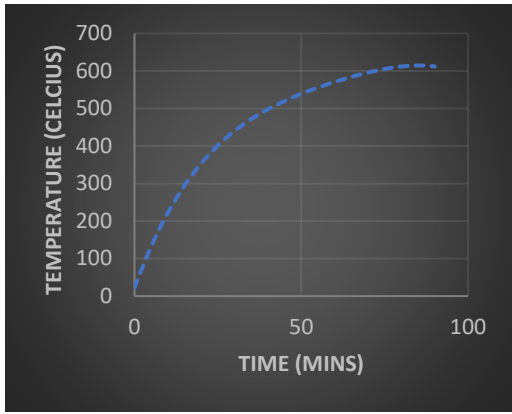
Fig. 4 Rate of Temperature Rise for Pyrolysis.

From Fig. 5, heating inlet 2 produced the highest heating rate with heating inlet 3 producing the least heating rate. Also, the average maximum temperature obtained in the MFP activation unit was approximately 900 ºC.
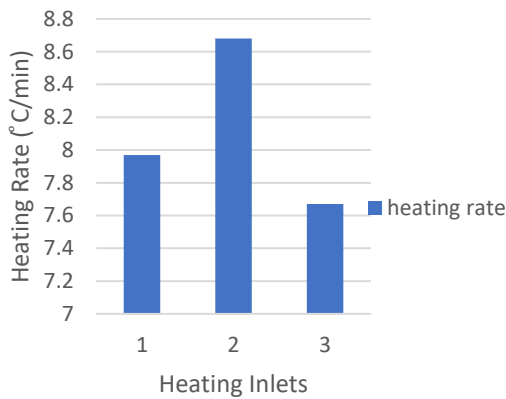

Fig.5 Various Heating Rate of the Activation Unit

3.3 Activated Carbon Yield

Yield of activated carbon produced were evaluated using the initial mass of coconut shells used as shown in equation (6). In Fig. 6, the activated carbon generated using potassium hydroxide had a higher yield as compared to the activated carbon produced using sodium hydroxide as a result of the higher reactivity power of sodium hydroxide than potassium hydroxide. The yield decreased gradually from using both heating inlet one and two, then heating inlet one with heating inlet two producing the least yield, regardless of the activation method used.

This is a result of higher volatile content being expelled out of carbonaceous material at a higher heating rate as compared to a lower heating rate as suggested by Masoumi et al. (2021), Maguyon-Detras et al. (2020) Hosney *et al,* (2016), which result to the trend of yields. The yields obtained were with the average yield range for two-way

conventional chemical activation process, that is 30 - 40 % (Ukanwa *et al.*, 2019).
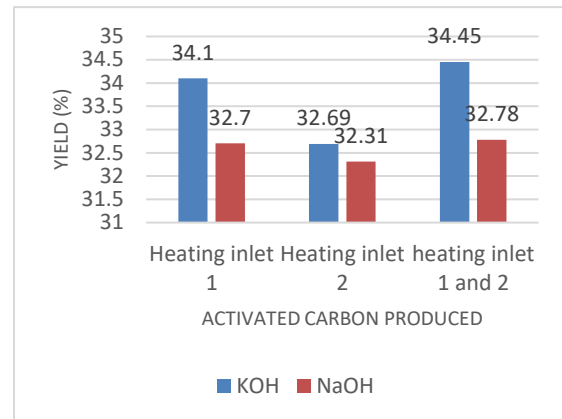

Fig. 6 Activation Real Yield (%)

3.4 Iodine Adsorption Number (IAN)

From Fig. 7, the iodine adsorption number increase from heating inlet 3, 2, and then 1, for chemical activation using sodium hydroxide. This as a result of higher heating rate leads to diverse pore distribution, with micropores widening up into mesopores and macropores (Leng et al., 2021; Liu et al., 2019; Chen et al., 2017; Ma *et al.*, 2014). The Iodine Adsorption Number is for micropore volume determination hence would not quantify the other pores in the activated carbon except micropores. Chemical activation using Potassium Hydroxide had Iodine Adsorption Number increased from the heating inlet 3, 1 with the heating inlet 2, resulting from low reactivity of potassium hydroxide as compared to sodium hydroxide, hence, leading to slow pores development. IAN of ACs derived are lower than the IAN of commercial carbons (CC) used as a reference for this research.
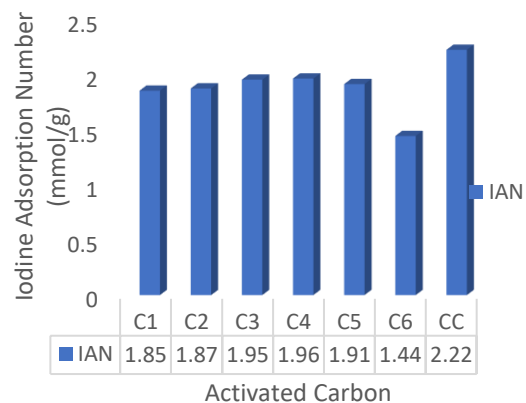

Fig. 7 Iodine Adsorption Number

3.5 Adsorption Test

Adsorption of gold from gold di-cyanide solution with an initial concentration of 14.15 mg/L, by the

commercial activated carbon and the activated carbons derived, is presented in Fig. 7.

In Fig. 7, the reference AC performed approximately equal as ACs produced This is an indication that the derived activated carbons are as active as the commercial activated carbon, although derived ACs had lower IAN than reference activated carbon (CC).

From Fig. 8, the curves show the activated carbon-produced adsorption trends, which shows that all produced activated carbons performed very well, with each adsorbing about 95% of the gold in solution within two hours.

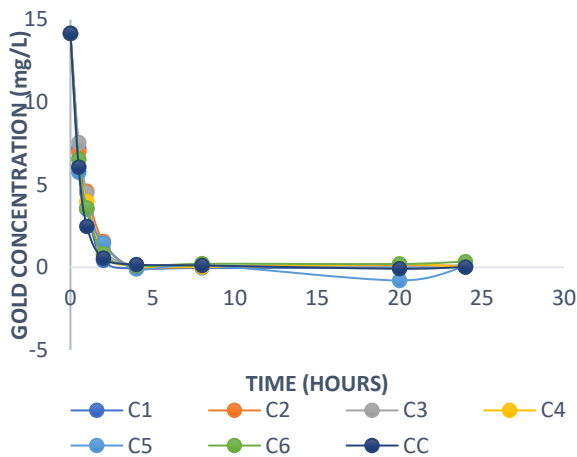From the results in Fig. 8, a graph of t/(x/m) against time was drawn in Fig. 9.

Fig. 8 Recovery of Gold from Solution by the Activated Carbons obtained and the Reference Activated Carbon
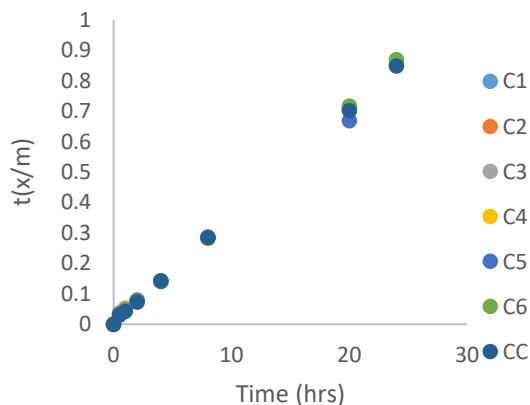

Fig. 9 Adsorption Rate of ACs obtained

The R-values of ACs in Table 2 were determined plots presented in Fig. 9 using Eq. (4)

$$t/(x/m) = (1/M)t + 1/R \qquad (4)$$

where x/m represent the carbon loading in mg Au/g of carbon, t is the time and M and R are reciprocal of the slope and the intercept at t =0

A higher R-value of carbon indicates faster adsorption, implying less gold loss at the plant, as stated by Buah et al. (2019) and Yalcin and Arol, 2002. From Table 2, ACs derived have lower R-value than reference AC except for C6 which has a higher R-value than CC.

Table 2 R-values of ACs Produced

Activated Carbon Sample	R-Value
C1	169.49
C2	172.41
C3	109.89
C4	172.41
C5	93.46
C6	188.68
CC	185.19

3.6 Hardness Test

The hardness of ACs produced is represented by Fig. 10.
From fig. 9, hardness decreased with increasing heating rate. This is because of higher devolatilisation of higher heating rate, hence, a larger number of pores in the processor which leads to weakening the carbon as confirmed by Buah and Kuma (2012)
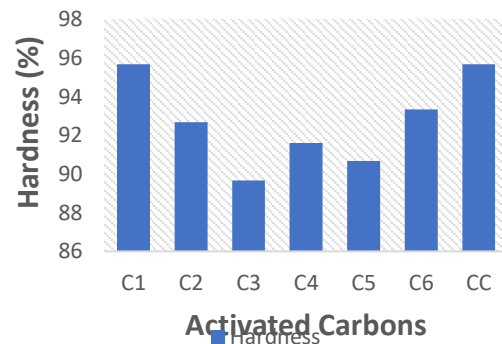

Fig. 10 Relative Hardness of Activated Carbons Obtained

4 Conclusion

The Activated Carbon produced from the MFP pilot plant had equally good iodine number, hardness, and adsorption rate as the commercial carbon. The activated carbon generated from char soaked in potassium hydroxide for 2 hours and heated in the activation reactor (MFP rotary kiln) using both heating inlet 1 and 2, C6, had a better gold adsorption rate than the commercial carbon. This

validates the possibility of commercial production of activated carbon in Ghana.

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