Activated Carbon Prepared in a Novel Gas Fired Static Bed Pyrolysis-Gasification Reactor for Gold Di-Cyanide Adsorption*

W. K. Buah, J. S. Y. Kuma, T. Williams and S. A. Ndur

Abstract

A novel gas fired static-bed pyrolysis-gasification/activation reactor has been designed. A special feature of the reactor is its rectangular cross-section, having a square groove positioned symmetrically at the bottom part of the reactor, which allows efficient heat transfer into the bed of material being pyrolysed. The reactor is designed also to allow easy feeding of precursors as well as easy discharging of carbonised products. The operating parameters of the reactor such as temperature, which is dependent on the fuel gas flow rate; and the rate of flow of steam into the reactor for gasification, can be monitored. Palm kernel shells based activated carbon was prepared by carbonisation of the shells at 900 ℃ pyrolysis temperature, followed by steam activation of the derived char also at 900 ℃ for 6 hours. Activation was done at steam addition rate of 0.2 mol/h/g in the reactor. The derived activated carbon was characterised using the hard groove test method and the carbons were found to possess relative hardness comparable to the commercial one in use in some gold processing companies. The gold di-cyanide adsorption characteristics of the derived activated carbon compared very well with that of the commercial activated carbon, Norit RO 3515 used in most mines in Ghana.

Keywords: Pyrolysis, Gasification, Activated carbon, Gold processing, Adsorption

1 Introduction

Activated carbons are carbonaceous materials having highly developed porous structure and a large internal surface area. Generally, activated carbons with higher surface areas exhibit higher adsorption capacities and their applications also depend on their pore characteristics.

Activated carbons have worldwide applications including removal of colour. For example, for removal of colour from sugar syrup during commercial production of sugar and also for removal of odour and tastes from edible oils, potable water, alcoholic beverages and fats (Mudoga *et al.,* 2008; Satyawall and Balakrishnan, 2007; Tennant and Mazyck, 2003).

More recently, they have found use in pollution abatement systems due to increasing concern regarding emissions from industrial processes and the introduction of stricter legislation designed to reduce the release of hazardous chemicals into the environment. For example, mercury emissions from coal-fired utility boilers vary in total amount and speciation, depending on the coal types, boiler operating conditions, and configurations of Air Pollution Control Devices (APCDs) (Yang *et al.,* 2007). The APCDs, such as Fabric Filter (FF) bag house, Electrostatic Precipitator (ESP), and wet Flue Gas Desulfurization (FGD), can remove some particulate-bound and oxidised forms of mercury. Elemental mercury often escapes from these devices. Activated carbon injection upstream of a particulate control device has been shown to have the best potential to remove both elemental and oxidised mercury from the flue gas (Yang *et al.,* 2007).

In the gold industry, activated carbon is used mainly to recover dissolved gold complexes from solutions (Soleimani and Kaghazchi, 2008a; Navarro *et al.,* 2006; Yalcin and Arol, 2002). Activated carbons have been widely employed for the adsorption of gold from potassium or sodium aurocyanide solutions as well as from gold chloride solutions. The problem of gold recovery from their ores by adsorption onto activated carbon is complex and a number of adsorption mechanisms of gold adsorption from potassium or sodium aurocyanide solutions and gold chloride solutions onto activated carbons have been proposed over the years in the literature (Adams *et al.,* 1987a, b; Lagerge *et al.,* 1997).

Activated carbon can be produced from almost all carbon containing materials. In practice, materials such as coal, bones, sawdust, palm kernel shells and coconut shells, wood, peat, lignite, nut shells and fruit stones have been used for activated carbon manufacture (Daud and Ali, 2004; Su *et al.,* 2003; Laine and Yunes, 1992; Guo *et al.,* 2007; Guo and Lua, 2000).

Activation of carbon is achieved by removal of hydrogen rich fractions and other volatile constituents from the carbonaceous raw material to produce a porous residue with a large internal surface area.

Coconut and palm kernel shells as precursors for activated carbon production are found in significant quantities in developing countries including Ghana but up till now, commercial production of activated carbons in Ghana has only been a dream. The design of an activated carbon production reactor, easy to fabricate from locally available materials in Ghana can go a long way to promote production of activated carbon in the country.

This paper presents a novel Gas Fired Static Bed Pyrolysis-Gasification/Activation Reactor and demonstrates the suitability of activated carbon produced from this reactor for gold di-cyanide adsorption. The Reactor is fabricated mainly from materials available in Ghana. The gold adsorption characteristics of the derived activated carbon from gold di-cyanide solution have been compared with that of a commercial activated carbon used for gold di-cyanide adsorption in the gold industry in Ghana.

2 Materials and Methods Used

2.1 Materials

The palm kernel shells were obtained from a palm kernel oil processing mill in Tarkwa in the Western Region of Ghana. They were crushed and sieved to obtain a particle size range of 3.5 – 10 mm, which was washed with water and dried at 110 °C in an oven. Some of the shells were pulverised and used for their proximate and ultimate analyses. The proximate analysis, determined as moisture content 4.65 wt%, ash content 4.42 wt%, volatile matter 62.05 wt%, and fixed carbon 28.90 wt% was performed using thermogravimetric analysis (TGA) in a Shimadzu TGA-50H. The carbon, hydrogen and nitrogen contents of the shells were also determined using a FlashEA 1112 analyser and gave the carbon content at 53.38wt%, hydrogen at 6.00wt%, nitrogen at 0.87wt%, and oxygen by difference at 39.75wt%.

De-ionised water obtained from the Minerals Engineering Laboratory of the University of Mines and Technology was used as the activation agent.
A commercial activated carbon, Norit RO 3515 was used as a reference standard to assess the gold di-cyanide adsorption ability of the activated carbons produced in this work.

2.2 Design of the Reactor

The reactor is fabricated using stainless steel plates of 4 mm thickness. The reactor is made up of a stainless steel sample chamber in the shape of a rectangular truncated prism with a cover to enable pyrolysis of up to 4 kg of palm kernel shells. The reactor has a rectangular cross-sectional groove, positioned symmetrically at the bottom part of the reactor, which allows efficient heat transfer into the bed of material in it. The furnace is a rectangular stainless steel chamber fired by combustion of Liquefied Petroleum Gas (LPG). There is a 40 mm fibre glass jacketed heat insulation layer round the furnace. The reactor is designed also to allow easy feeding of precursors as well as easy discharging of carbonised products. The reactor also houses a perforated stainless steel pipe through which water is introduced into the reaction chamber. The simplicity of the design and the ease of feeding and unloading the reactor are some of the unique advantages compared to other static bed pyrolysis-gasification reactors.

2.3 Carbonisation of Raw Palm Kernel Shells

The palm kernel shells were obtained from Tarkwa in the Western Region of Ghana. A 4 kg sample of the shells, pre-dried at 104 °C was fed into the reactor and heated to a final pyrolysis temperature of 900 °C and held at the final temperature for a period of 1 h. The carbonisation and activation temperature of 900 °C was chosen because Guo and Lua (2000) investigated the effect of heating temperature on the properties of chars and activated carbons prepared from oil palm stones and found that as the activation temperature increased from 750 up to 900 °C, the Brunauer Emmett and Teller (BET) and micropore surface areas increased progressively since the activation process not only enlarged the pores created during the pyrolysis but also generated some new pores. However, at 950 °C, the surface area, especially the micropore surface area dropped dramatically due to the over-reaction of carbon with the activating agent. A number of researchers (Lua and Guo, 2001; Teng and co-workers, 1996) have also successfully produced quality activated carbons at 900 °C.

2.4 Activation of the Carbonised Palm Kernel Shells

The carbonised shells obtained after pyrolysis of the shells at 900 °C in the reactor were crushed and sieved to obtain (-2.8 +1.7) mm size fraction, which was subjected to steam activation at 900 °C in the same reactor. During the activation process, the char weighing 500 g was loaded into the reactor and covered by the reactor cover. De-ionized water was introduced through the water pipes at the bottom of the reactor. The perforations on the sides of the pipes allow vapourised water into the reactor to cause activation. The flow rate of water in the pipes was maintained at 0.2 mol/h/g.

2.5 Characterisation of the Derived Activated Carbon

The yield of activated carbon, resulting after activation of a given quantity of the carbonised palm kernel shells is calculated as follows:

$$Yield, \quad Y = \frac{W_2}{W_1} \times 100\% \qquad (1)$$

Where: W_1 = Initial dry mass of char (g) and W_2 = Dry mass of carbon after activation (g).

Other properties of the derived activated carbon determined in this research are the relative hardness, the gold di-cyanide adsorption rate and capacity. These parameters are necessary for assessing the suitability of activated carbon for gold di-cyanide adsorption from solution. The hardness was determined relative to the hardness of a typical commercial activated carbon, which is used for gold di-cyanide adsorption in some gold processing companies. In this determination 2 g of the activated carbon was subjected to abrasion in a vibrated hard groove machine in the presence of a steel ball for 1 minute. The commercial activated carbon was also subjected to the same treatment in the machine. The hardness of the derived activated carbon relative to that of the commercial activated carbon, Norit RO 3515 was determined according to Eq. (2) on the basis of the amount of abraded test material retained on a 25 mesh (0.707 mm) screen:

$$Relative \ Hardness = \frac{W}{W_r} \times 100\% \qquad (2)$$

Where, W and W_r represent the weight of the test activated carbon and the reference activated carbon retained on a 25 mesh screen, respectively. A similar procedure was used by Yalcin and Arol (2002) for assessing the relative hardness of activated carbons produced from steam activation of hazelnut shell, apricot and peach stones using a coconut shell activated carbon as the reference carbon.

Tests on adsorption of gold di-cyanide by the derived activated carbon was carried out by equilibrating 1.0 g of the (-2.8 +1.7) mm size fraction of the activated carbon with 500.0 mL of a 3.624 mg L^{-1} gold di-cyanide solution in a glass bottle. The bottle was rolled on a set of rollers at a low speed to prevent attrition of the activated carbons, but high enough to ensure good solid/liquid contact. A similar test was run using the commercial activated carbon for the purpose of comparison. All experiments were conducted at room temperature and the speed of rotation of the activated carbon/gold solution system was kept constant for all the experiments to offset the effect of stirring rate on the adsorption characteristics of the activated carbons. The adsorption of gold with respect to time was determined by removing 10 ml aliquots of the solution at 1 h time intervals for 12 h and analyzing the gold concentration with a Varian Fast Sequential Atomic Adsorption Spectrophotometer (Varian AA240FS).

3 Results and Discussion

3.1 The Reactor Design

Fig. 1 shows the photographs of the newly designed Gas Fired Static Bed Pyrolysis-Gasification/Activation Reactor system. The various stages of loading the reactor and discharging carbonised materials from the reactor are shown in Figs.1a to 1c: (a) Reactor in opened position to receive precursor; (b) Reactor in closed position for pyrolsis/gasification; (c) Reactor lifted for discharging of carbonaceous products. Detailed engineering description of the newly designed Gas Fired Static Bed Pyrolysis-Gasification/Activation reactor system is beyond the scope of this work.

3.2 Production of Activated Carbon

The palm kernel shells, carbonised in the reactor at a pyrolysis temperature of 900 ℃ is allowed to cool to less than 200 ℃ and then discharged from the reactor. The yield of the char obtained was 28.89% as shown in Fig. 2. The yield of char obtained from the reactor in this research matches well with yields obtained by other researchers (Hu, et al., 2001; Tsamba et al., 2006). It has been established that the percentage yield of char products from the pyrolysis of various carbonaceous materials to various final temperatures depended strongly on temperature. For example, Buah et al., (2007) established during pyrolysis of Refuse Derived Fuel (RDF) samples in a fixed bed reactor at 10 K min^{-1} to various final temperatures that as the temperature was raised from 400 to 700 ℃ the yield of char fell from 49.8 to 32.3 wt%. Demirbas (2004) also observed a decrease in the yield of bio-char resulting from the slow pyrolysis of agricultural residues such as olive husk, corncob and tea waste when the pyrolysis temperature increased from 177 to 977 ℃.

In a study of the influence of process variables on char yield and characteristics during carbonisation of bagasse in a fixed bed reactor, Katyal et al. (2003) also observed a decrease in the yield of char, with a corresponding increase in the volatiles (gas and liquid) yield as the pyrolysis temperature increased. Onay (2007) investigated the influence of pyrolysis temperature and heating rate on the production of bio-oil and char from safflower seed by pyrolysis, using a well-swept fixed-bed reactor. His results also showed that, as the final pyrolysis temperature was raised from 400 to 700 °C for each heating rate investigated, the char yield

significantly decreased. The decrease in the yield of char with increasing temperature was attributed to either greater primary decomposition of the safflower seed at higher temperatures or to secondary decomposition of the char residue. Tippayawong *et al.* (2008) also showed that the yield of solid product decreased from 55 to 49 wt% as the pyrolysis temperature of RDF was increased from 400 – 700 °C.

To obtain a size fraction of (-2.8 +1.7) mm activated carbon, suitable for gold adsorption, the char was subjected to crushing and sizing. The (-2.8 +1.7) mm fraction of the char constitutes 39.42% of the char obtained immediately after the pyrolysis. It was this sized char that was subjected to the activation process in order to obtain activated carbons in the size range of (-2.8 +1.7) mm, suitable for gold adsorption as recommended by Yalcin and Arol (2002).

After reasonable cooling of the reactor was allowed it was lifted from the furnace to hinge at the discharge end of the furnace to allow easy discharge of the product. After discharging of the product the reactor was allowed to fall back into the furnace and the reactor was ready to receive new load of precursors. The yield of the activated carbon obtained was 93.80% in relation to the quantity of the char that was activated. Fig. 3 shows how well the derived activated carbon visually compares with the commercial activated carbon.

In a previous investigation (Buah and Williams, 2013), carried out in a comparatively smaller reactor on the production of granular activated carbons from palm nut shells for gold di-cyanide adsorption, it was observed that the yields of the activated carbons decrease from 100 wt% to 75.3 wt% after 3-h activation and further to 46.9 wt% after 6-h activation. The yield of activated carbon obtained in the current research is far above what has been obtained previously. This could be due to inadequate burn-off of the carbon in reaction with the steam, which subsequently would affect the degree of activation. It is intended that the operating variables of the new rig would be optimized to obtain the best product.

3.3 Characterization of the Derived Activated Carbon

Adsorption of gold from the gold di-cyanide solution by the derived activated carbon with respect to time is presented in Fig. 4, from which the rate of gold adsorption is determined. The rate of gold adsorption is measured by the R-value which is the reciprocal of the intercept at zero time of plotting time divided by loading of gold on carbon versus time when contacting the activated carbon with gold solution. Yalcin and Arol (2002) determined the R-value of activated carbons by the Calgon Test Method 1983 (Anon., 1983; Urbanic *et al.*, 1985). In their experiment, a 5 mg L^{-1} gold cyanide solution was contacted with a closely sized (-2.83 + 1.41) mm fraction of granular activated carbon. The data obtained in such adsorption experiments yield a straight line that can be expressed by Eq. (3) (Urbanic *et al.*, 1985).

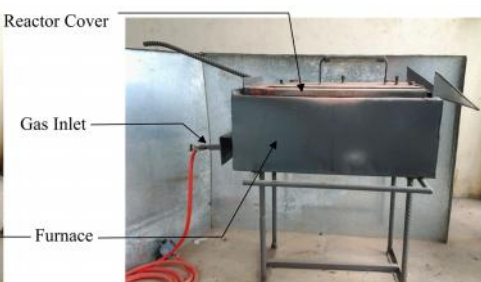Fig. 1(a) Reactor in Opened Position to Receive Precursor

Fig. 1(b) Reactor in Closed Position for Pyrolsis/ Gasification

Fig. 1(c) Reactor Lifted for Discharing of Carbonaceous Products

Fig. 1 A Gas Fired Static Bed Pyrolysis-Gasification/Activation Reactor System
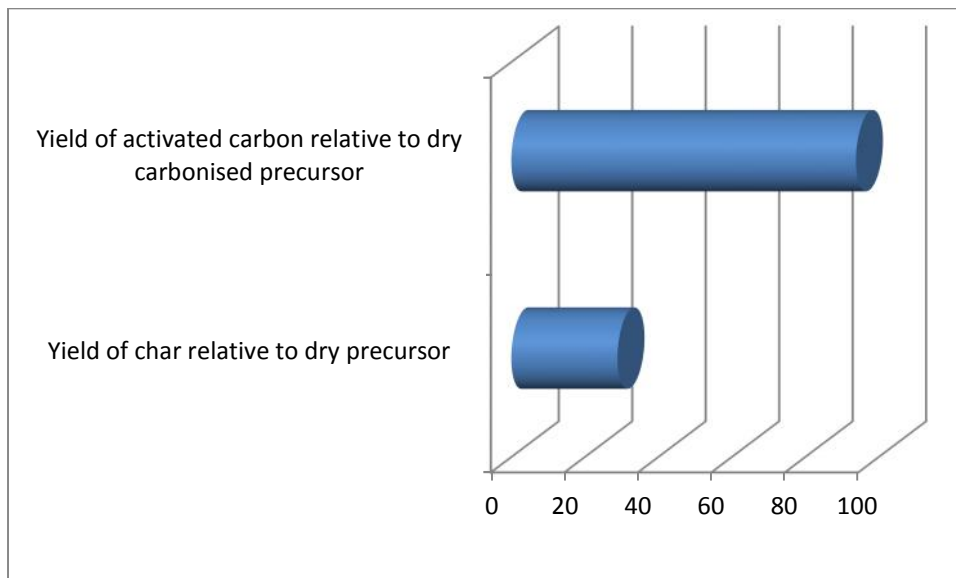
Fig. 2 Yield of Char and Activated Carbon

$$\frac{t}{x/m} = \frac{1}{M}t + \frac{1}{R} \qquad (3)$$

Where, x/m represents the carbon loading in mg Au/g of carbon, t is the time and M and R are reciprocal of the slope and intercept at zero time, respectively.

Fig. 3 Activated Carbon Produced in the New Reactor (a) Compared with a Commercial Activated Carbon (b)

Considering the data in Fig. 4, the rates of gold adsorption by the activated carbons investigated in this research are presented in Fig. 5. The R-value for each carbon is relative, i.e. a higher value indicates faster adsorption. The R-value estimated from Fig. 5 for the commercial activated carbon is higher than that of the activated carbon produced from the newly designed reactor. However, it is observed that gold recovery by both carbons hovers around 87% - 88% after 10 h of contact time.
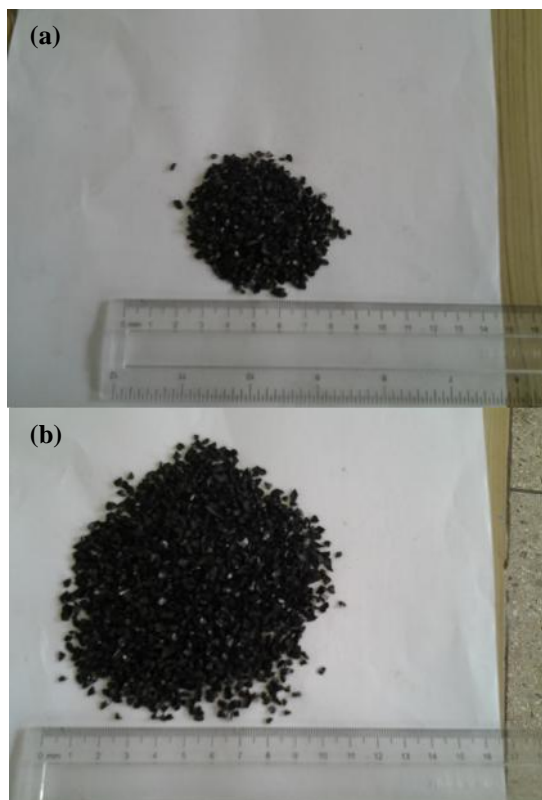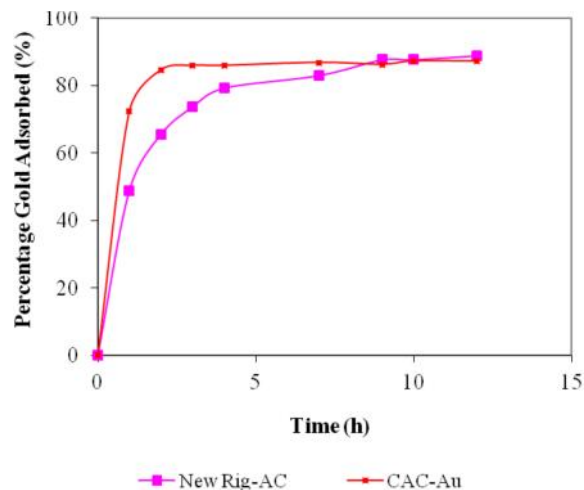

Fig. 4 Percentage Gold Adsorption by the Activated Carbons in Relation to Contact Time with the Gold Solution. *(New Rig-AC represents activated carbon produced in the New Reactor; CAC-Au represents a commercial activated carbon used by a mine in Ghana)*
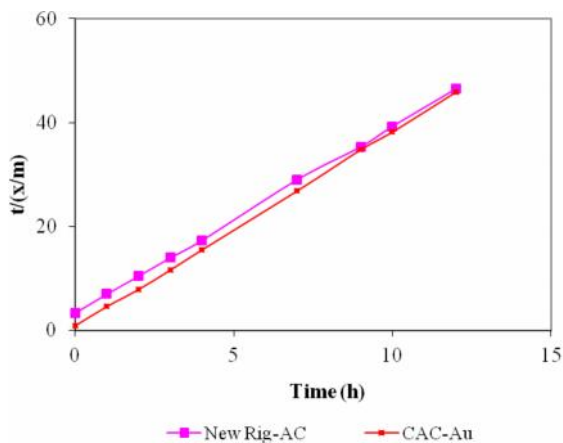
Fig. 5 Rate of Gold Adsorption from the Gold Solution by the Activated Carbons *(New Rig-AC represents activated carbon produced in the New Reactor; CAC-Au represents a commercial activated carbon used by a mine in Ghana)*

The results obtained in this research demonstrate the potential of the derived activated carbon for industrial application.

4 Conclusions

Activated carbon has been prepared successfully from the Novel Gas Fired Static Bed Pyrolysis-Gasification/Activation Reactor. The activated carbon was prepared by carbonisation of palm kernel shells in the reactor at 900 ℃ pyrolysis temperature, followed by steam activation of the (-2.8+1.7) mm fraction of the carbonised palm kernel shells at activation temperature of 900 ℃ for 6 h and water introduction rate of 0.2 mol/h/g. This paper has demonstrated that activated carbon produced from this reactor exhibits gold extraction efficiency comparable to the commercial carbon in use in some gold processing companies. The rate of gold adsorption of the derived activated carbon is however lower compared to that of the commercial one, a situation attributed to low degree of carbon burn off achieved in the Reactor. There is therefore the need for optimisation of the operating variables in the Reactor.

Acknowledgements

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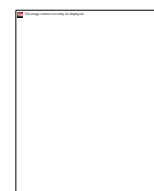
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