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Investigating Crude Potash as Activating Agent for the Production of Activated Carbon for Gold Adsorption Operations – A Preliminary Study

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ABSTRACT

Activated carbon was prepared from palm kernel shell using leachates from cocoa husk ash as activating agent. At an impregnation of 1.0, the produced carbons had the best fresh carbon activity at 800°C activation temperature. Crude potash compared favourably with pure potash as activating agent in terms of selected quality indices for gold mining operations as the Independent Sample t-test employed for the analysis recorded p-values which were almost always higher than the α - value for fresh carbon activity, hardness, ash content and apparent density at 95% confidence interval.

Keywords: *activated carbon, impregnation ratio ,chemical activation, crude potassium carbonate, cocoa pod ash leachates, t-test,*

1. INTRODUCTION

Adsorption, the tendency of a molecule from an ambient fluid phase (adsorbate) to adhere to the surface of a solid (adsorbent) is an important unit operation employed extensively in purification and separation processes in the chemical processing industry. Commercially available adsorbents include activated carbon, activated alumina, silica gel, and molecular sieve zeolites of which activated carbon is the most widely used [1]. Activated carbon, a carbonaceous material having high porosity and internal surface area, can be prepared from a number of raw materials ranging from agricultural by-products to petroleum based residues, by either the physical activation or the chemical activation methods [1],[2],[3],[4].

The preferred and most researched method of activation has been the physical method due to the potential effect of residual reagents in chemical activation and their relatively high cost. A number of researchers have reported the use of potassium carbonate to produce activated carbon [5],[6]. In this paper, the use of crude potash – an organic, impure, less expensive form of potassium carbonate obtainable from cocoa husk ash leachates – as activating agent is investigated. Cocoa pod husk is readily available and largely considered as waste and potential source of environmental pollution.

The choice of raw material depends on the price, purity, potential extent of activation and the availability and stability of the supply [7]. Palm kernel shell was chosen as starting material for the production of activated carbon because of the availability of substantial amount of shell all year round, the cheap cost of the raw material, the relatively high fixed carbon content (18% w/w) and low ash content [3].

In the present work, granular activated carbon was prepared from palm kernel shells using leachates from cocoa husk ash (crude potash) as activating agent. Selected quality indices of the produced activated carbon as required in gold mine operations – fresh carbon activity, hardness, ash content and apparent density using crude potash and pure potash as activating agents were ascertained and the statistical tool of t-test was used to determine the statistical difference, if any, between using pure potash and crude potash as activating agents.

2. MATERIALS AND METHODS

2.1 Raw Materials and Reagents

Palm kernel shells (PKS) and cocoa husk were obtained from palm kernel shell dump site at Obuasi, Ghana and a cocoa farm at Akrokerri in the Ashanti Region of Ghana respectively. Pure potassium carbonate (K_2CO_3, 99.0% purity) was obtained from Thomas Baker, U.K.

2.2 Raw Materials Preparation and Analysis

The palm kernel shells were washed thoroughly with water to remove all dirt and sun dried on a clean platform for three weeks. The dried PKS were crushed in a nut cracker and sieved to particle sizes between 2380µm and 1190µm.

The cocoa pod husk was washed with water to remove dirt, sun dried for three weeks and combusted in the open to produce ash. The incompletely combusted dark particles were sieved from the ash. The procedure of Adewuyi *et al.* (2008) for alkali extraction as reported by Ogundiran *et al.* (2011) was adapted [8]. A known mass of the ash was placed in a 1.5L capacity polyethylene terepthalate bottle and an appropriate volume of distilled water was added. The bottle and its content were shaken vigorously and allowed to settle for about 12 hours, to ensure maximum dissolution of soluble mineral component of the ash in the distilled water. For each batch of ash, three different extraction set ups were made using different ratios of ash to water. Four pin holes were bored at the bottom of the bottle, the screw cap was removed and the extract leaked into the collecting beaker beneath it.

The pH of the clear extract solution was measured, and a double indicator titration method was used to determine the K_2CO_3 and KOH content of the extract. [8],[9].

2.3 Chemical Activation

The precursor was impregnated at room temperature for 12 hours with pure K_2CO_3 (Purity 99.0%) and the crude K_2CO_3 leached from cocoa husk ash at an impregnation ratio (I.R). of 1.0. The samples were then dried in an oven at 110°C for 8 hours. Impregnation ratio is defined as:

$$I.R = \frac{Mass\ of\ K_2CO_3\ in\ solution}{Mass\ of\ PKS}$$ [5],[10]

The impregnated samples were carbonised and activated simultaneously in a Nabertherm muffle furnace heated at a rate 8°C/min. until the final activation temperatures (600°C, 700°C and 800°C) were attained. The samples were maintained at those temperatures for a further 2 hours and then allowed to cool to room temperature. The prepared carbon was washed thoroughly with distilled water to remove residual chemicals and dried at 110°C for 24 hours.

2.4 Characterisation of the Produced Activated Carbon

The produced activated carbon using both pure K_2CO_3 (PPAC) and crude K_2CO_3 (CPAC) as activating agents were characterised based on the usual quality indices used in gold mine operations.

2.4.1 Carbon Activity

Fresh Carbon Activity was determined by adding about 9g of prepared carbon to 0.1% NaCN in a Winchester bottle. A standard gold solution (to make 10 ppm of the final solution) was added and the bottle was whirled for about an hour and allowed to settle. The clear solution was analysed with the Atomic Absorption Spectrometer (AAS) to determine the percentage of gold left in the solution.

2.4.2 Hardness

The hardness was determined by the wet attrition method of Toles *et al*, (2000) [11],[12]. One gram (1.0 g) of the activated carbon was placed in 100 mL of 0.1M acetate buffer in a 150 mL beaker. The solution was stirred at 500 rpm for 2 hours at ambient temperature. The sample mixtures were then poured on a 0.30mm screen and washed sequentially with 250 mL of distilled water. The retained samples on the screen were transferred onto an aluminium pan and dried in the oven at 110°C for 2 hours. The samples were finally cooled in a desiccator. The percent attrition or hardness is calculated using the relation:

$$Attrition\ (\%) = \frac{Initial\ mass - Final\ mass}{Initial\ mass} x100$$

The same procedure was used to determine the attrition of a sample granular activated carbon used in commercial gold adsorption process which was coded S-GAC. The attrition value for the S-GAC served as the benchmark with which to compare the attrition values of the produced AC.

2.4.3 Ash Content

The ash content of the AC was determined using ASTM D 2866 – 94 procedure. 1.0 g of the prepared AC that has been dried at 150°C for 3hrs was weighed into a crucible. The sample was ashed in the furnace for 3 hours at 630°C and then cooled. The loss in weight expressed as percentage is the ash content.

2.4.4 Apparent Density

The apparent density of the prepared AC was determined using the method of Norazatul (2005) [13]. The prepared AC of known mass was wrapped in a plastic bag and dropped gently into 70mls of water in a 100ml capacity measuring cylinder. The difference in the level of water was measured and the density calculated as the mass of the AC divided by the volume of water displaced.

2.5 Statistical Analysis of Results

The Independent Samples t-test analysis was used to determine whether there was any significant difference in the quality indices of the produced activated carbon from pure potash (PPAC) and crude potash(CPAC) as activating agents at 95% confidence level. When the significance level (p-value) is higher than the α – value (in this case 0.05), then no difference exist in the use of the pure potash and the leachates from cocoa husk ash, crude potash as activating agents.

3. RESULTS AND DISCUSSION

3.1 Quality Indices

The fresh carbon activity using the two activating agents is shown in figure 1. The activity generally increases as the activation temperature increases. At higher temperatures, substantial devolatilisation is expected to occur in the precursor as a result of the high reaction rates leading to the creation of numerous pores and hence the high surface area which translates into high activity [5][14]. It appears the use of the crude potash produces a slightly higher activity than when the pure potash was used. In gold mine operations, an activity of 55% or more is considered acceptable [15]. This requirement is met by activation at 700°C and 800°C using both CPAC and PPAC.

The percent attrition, a measure of carbon hardness, increased with increasing carbonisation/activation temperature for both PPAC and CPAC as shown in figure 2. Biomass generally tends to disintegrate at high temperatures. The rate of dehydration and calcination increases and the granules become weaker [5].

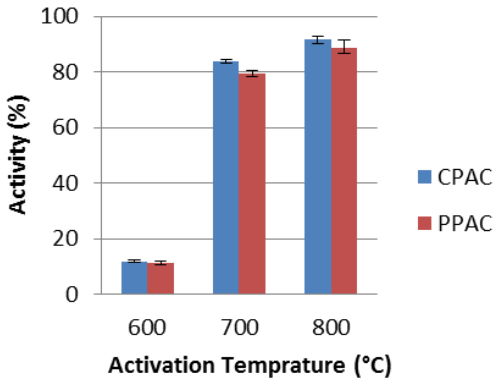

Fig. 1: Effect of Activation temperature on activity

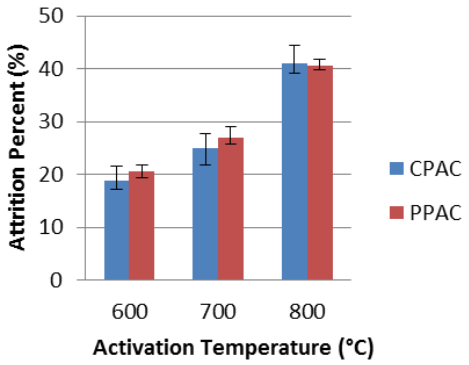

Fig. 2: Effect of activation temperature on attrition

From figure 3, it can be observed that the ash content of the produced AC increased steadily when the activation temperature was increased. As the activation temperature increased, more volatile components were driven off and the inorganic constituents present with the carbon such as SiO_2 residues are left over as ash [16],[17]. Similar trend was observed by Yagsi (2004) in chemical activation of apricot stones [18].

The apparent density, the mass per unit volume (including pores and voids between particles) is a parameter used to estimate the surface area and activity values of AC. Smaller apparent density values therefore implies the presence of large number of pores and for that reason higher surface area. As shown in figure 4, the apparent density decreased with increasing carbonisation and activation temperature as a result of the loss of weight and the development of larger pores as more volatile components evolve. [6],[19].

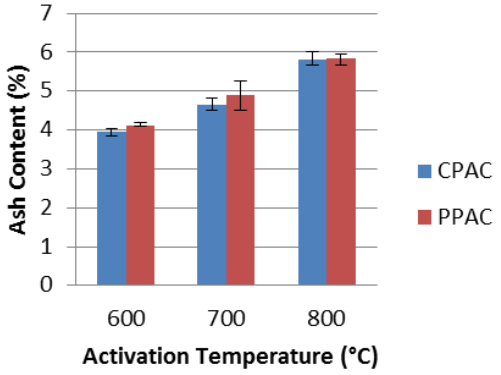

Fig. 3: Effect of Activation temperature on Ash Content

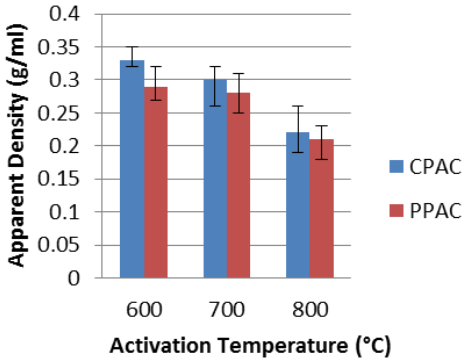

Fig. 4: Effect of Activation temperature on Apparent Density

3.2 Statistical Analysis

In all cases, equal variances were assumed and at a confidence interval of 95%. Generally, for the analysis of fresh carbon activity values and ash content, the p-values were higher than the α-value (0.05), an indication that there is no significant difference between the activity values of PPAC and CPAC (table 1). An exception is observed in the fresh carbon activity at 700°C activating temperature and ash content at 600°C. However, all values are within acceptable limits for gold adsorption operations.

Table 1: Summary of Statistical Analysis of Selected Quality Indices (t – test)

PROPERTY	CARBON TYPE	ACTIVATION TEMPERATURE	MEAN	DF	P- VALUE	DECISION
Activity	CPAC PPAC	600°C	11.600 11.267	4	0.492	No Significant Difference
	CPAC PPAC	700°C	83.900 79.500	4	0.002	**Significant Difference**

	CPAC PPAC	800°C	91.667 88.883	4	0.153	No Significant Difference
Hardness	CPAC PPAC	600°C	18.820 20.620	4	0.300	No Significant Difference
	CPAC PPAC	700°C	24.957 27.027	4	0.324	No Significant Difference
	CPAC PPAC	800°C	41.020 40.560	4	0.802	No Significant Difference
Ash Content	CPAC PPAC	600°C	3.937 4.120	4	0.029	**Significant Difference**
	CPAC PPAC	700°C	4.660 4.810	4	0.635	No Significant Difference
	CPAC PPAC	800°C	5.800 5.853	4	0.725	No Significant Difference
Apparent Density	CPAC PPAC	600°C	0.333 0.293	4	0.145	No Significant Difference
	CPAC PPAC	700°C	0.308 0.277	4	0.476	No Significant Difference
	CPAC PPAC	800°C	0.220 0.210	4	0.731	No Significant Difference

DF = Degree of Freedom

CPAC = Crude Potash Activated Carbon (Carbon prepared by impregnation with crude potash)

PPAC = Pure Potash Activated Carbon (Carbon prepared by impregnation with pure potash)

4. CONCLUSIONS

The preliminary studies of production granular activated carbon from palm kernel shells using crude potash and pure K_2CO_3 as activating agents show that, activated carbon can be effectively prepared from palm kernel shell using crude potash as activating agent with a fresh carbon activity of 91.7% at 800°C activation temperature. No significant difference exists in the quality indices in relation to gold mine operations of the produced activated carbon using pure K_2CO_3 and crude potash as activating agents. Increasing activation/carbonisation temperature also enhanced the fresh carbon activity and apparent density.

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