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A comparative study of the effects of various locally-prepared agro-based carbons on the *In vitro* adsorption of cyanide using different activating agents

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Abstract

Aim: The study investigated the effect of using different activating agents- Nitric acid, Zinc chloride and Phosphoric acid on the adsorption of potassium cyanide by activated charcoal. We also investigated the *In vitro* effect of various doses of activated charcoal from different agro-based sources (Coconut shell, Rice husk, Castor oil seed shell, Edible clay and Plantain peels) on the adsorption of cyanide.

Materials and methods: 50ml of 1M potassium cyanide was pipetted into two beakers for each sample of the prepared activated charcoal ranging from 0.5 g to 2.0 g. The resultant mixtures were agitated for 1 hour and allowed to stand for 24 hours at room temperature. The mixtures were filtered using whatman filter paper. The filtrates were analyzed for cyanide concentration using ninhydrin method and the absorbance read with ultraviolet-spectrophotometry. The absorbance was then converted to cyanide concentrations in mg/dl using standard cyanide concentrations graphs.

Results: The results showed some statistically significant differences in the potassium cyanide adsorption by the different samples of prepared carbon materials activated with different activating agents except for coconut shell activated with zinc chloride. The cyanide adsorption was found to be in the same range for the activated charcoal samples prepared from different sources irrespective of the doses used in the experiment.

Conclusion: The adsorptive capacities of different samples of activated charcoal varied significantly with the type of chemical activating agents used in the activation of the carbon. Coconut shell activated with HNO₃ and H₃PO₄ showed the best adsorption capacity. The cyanide adsorption by the locally-prepared activated charcoal was found to be better than that of the standard commercial activated charcoal.

Keywords: Activated charcoal; Carbon; Absorbance; Ultra violet-spectrophotometry; Cyanide

1. Introduction

Charcoal for medicinal use is created by the controlled pyrolytic decomposition of carbon-based compounds, such as coconut shells or peat [1]. Thereafter, 'activation' with gases at high temperature removes previously adsorbed

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substances and further reduces particle size, resulting in an exceptionally porous final product [2]. In reality, some 'superactivated' charcoal preparations have a surface area of up to $3500 \text{ m}^2 \text{ g}^{-1}$, or about $175\,000 \text{ m}^2$ per 50 g bottle. (For perspective, the area of a large football pitch is about 10000 m^2 .) This allows the adsorption of drugs and toxins through weak intermolecular forces, with non-ionized, organic compounds binding more avidly than dissociated, inorganic ones [1].

Adsorption capacity of AC depends on the nature of the adsorbent (pore structure, functional groups, ash content) as well as the nature of the adsorbate (functional groups, polarity, molecular size and weight). The type of precursor and the process of activation determine basic properties of AC such as surface area and pore size distribution [2, 4]. The ACs has strong heterogeneous surfaces, in both geometrical and chemical character. The geometrical heterogeneity is the result of differences in the size and shape of pores as well as pits, and vacancies. Chemical heterogeneity is associated to different functional groups at a surface (mainly oxygen) and to various surface contaminants. Both heterogeneities contribute to unique adsorption properties of activated carbons [3, 4, 5, 6, 7, 8].

Cyanides in the form of hydrogen cyanide (HCN) and its alkali metal salts (such as Sodium cyanide [NaCN] and Potassium cyanide [KCN] etc.) are extremely toxic chemicals which widely exist in nature and industrial processes [9,10]. Deliberate or accidental cyanide poisoning is rare but may be fatal to the affected humans or animals [11]. The most common accidental cyanide exposure is the inhalation of smoke from residential and industrial fires that produce cyanides through nitrogen-containing combustibles [12]. The natural sources of cyanides are grossly due to the nitrogen metabolic products of bacteria, fungi and algae and to the degradation of cyanogenic glycosides in some plants including bitter almonds, cassava roots, and whole sorghum [13]. Tobacco smoking also produces cyanide thus increasing cyanide levels in blood and urine of smokers but rarely intoxicating. On the other hand, cyanides have important industrial applications including pesticides, electroplating, mineral refinery, and polymer synthesis [9,14]. These processes may possibly cause cyanide release into the environments.

Cyanide poisoning, whether it is accidental or intentional, remains a major threat to civilians and military personnel worldwide. Unfortunately, cyanide is readily available, highly lethal, and easily weaponized. Oral cyanide in particular poses the largest threat compared to other routes of exposure, with potassium cyanide (KCN) and sodium cyanide (NaCN) being the most frequently ingested cyanide salts [15,16].

Cyanide remains on the list of potential terrorist threats according to the various United States governmental agencies. One of the most renowned incidents of a large-scale oral cyanide poisoning was the Jonestown massacre, which resulted in more than 900 deaths after drinking cyanide-laced Flavor-Aid [17]. In 1982, seven people in the USA died after ingesting an over-the counter medication laced with cyanide [18]. In 2015, the Office of the Director of National Intelligence released documents revealing that some terrorists planned to contaminate food supplies with cyanide [19].

Once absorbed into the blood stream, cyanide equilibrates between the cyanide anion (CN^-) and un-dissociated hydrogen cyanide (HCN) [20]. In this form (HCN), cyanide can easily cross the cell membranes and inhibit multiple enzymes including succinic dehydrogenase, superoxide dismutase, and cytochrome oxidase [20, 21]. The latter enzyme is part of complex IV of the mitochondrial electron transport chain. The anion of cyanide (CN^-) has a high affinity for the ferric iron (Fe^{3+}) on cytochrome c oxidase, forming a complex that leads to inhibition of the electron transport chain, and thus aerobic respiration [22, 23]. The development of anaerobic metabolism leads to acidemia with hyperlactatemia, a hallmark of cyanide poisoning [22].

On exposure to acidic gastric conditions, ingested cyanide ion is rapidly converted to hydrogen cyanide gas due to its pKa of 9.3 and boiling point of 26.3°C . Thus, cyanide is likely absorbed as hydrogen cyanide gas, rather than as cyanide ion [24].

2. Material and methods

2.1. Collection and Preparation of the local Adsorbents.

All the agricultural biomass (Plantain peels, Rice husks, Edible clay, Coconut shell and Castor oil seed shell) were collected from from Ogbete main market, a local market in Enugu metropolis, Enugu state, Nigeria. The samples were properly washed and dried in the sun and 150g of each sample was packaged and properly labelled for oven-drying. The raw materials were dried in an oven at the temperature of 110°C for 24 hours. All the oven-dried samples were pulverized manually using electric blender into fine particles. This is to increase the surface area of the samples.

A known quantity (150g) of each of the agricultural biomass was used. The adsorbents were prepared as activated carbon by chemical-physical methods as described by [25] with slight modification. The dried samples were pulverized using electric blender, to increase the surface area that will enable more efficient chemical activation of the raw materials. Samples were sieved after pulverization to obtain particle sizes smaller than 600 μm .

2.2. Chemical Activation and Carbonization of samples

The fine oven-dried pulverized powder of our raw materials were each divided into three and each were separately treated with different activation agents; Phosphoric acid (H_3PO_4), Zinc chloride (ZnCl_2) and Nitric acid (HNO_3). The activated carbons were produced with a slight modification using standard methods of Ekpete et al., 2011 and Mopoung & Dejang, 2021 [26, 27]. Three beakers were used in the activation process for each sample. In the first beaker, 50 g of the fine oven-dried pulverized powder was mixed with 100 ml 10% w/v of ZnCl_2 . In the second beaker, another 50g was mixed with 500 ml 0.3 mol dm^{-3} H_3PO_4 . In the third beaker, another 50g was mixed with 500 ml 0.5 mol dm^{-3} HNO_3

To ensure the entry of the activating agent into the interior of the fine oven-dried pulverized powder samples, these mixtures were left for 24 hours at room temperature with constant stirring using a magnetic stirrer. To form a paste, the beakers were heated using oven at a constant temperature of 80 $^\circ\text{C}$ until a fine oven-dried powder were obtained. The fine oven-dried activated powders were then each transferred into a labelled ceramic crucible. The fine oven-dried pulverized activated powder of our raw materials were carbonized using the box-type resistance muffle furnace at a temperature of 800 $^\circ\text{C}$ for 1 hr. The ceramic crucibles were placed in the muffle furnace where they were heated at a temperature of 800 $^\circ\text{C}$ for 1 hr.

The activated carbon products formed were transferred into separately labelled desiccators to cool to room temperature. After completion of the process, the produced activated carbon products were soaked with 0.5 N HCl (hydrochloric acid) solution for 24 hr. The soaked activated carbonized materials were washed several times with hot double distilled water and finally with cold double distilled water until the pH of the wash water was neutral. The wet activated carbonized materials were dried in an oven at 110 $^\circ\text{C}$ for 24 hours. They were then kept in air-tight containers for further analysis and were properly labelled.

2.3. Data Analysis

The data collected were analyzed using SPSS version 26.0. Data were expressed in mean \pm SD. Groups were analyzed using one way anova (analysis of variance) followed by Post-hoc (Duncan) test. A difference in the mean with $p < 0.05$ was considered to be statistically significant.

3. Results and discussion

The table below shows the effect of different quantities (doses) of the prepared carbon samples activated with different activating agents on their cyanide adsorption capacities compared with the standard commercial activated charcoal. The cyanide concentrations recorded in this experiment is a measure of the cyanide adsorption capacity of the respective samples. The amount of cyanide that was present in the filtrate after passing the cyanide solution treated with the adsorbents through the whartman filter paper. So the amount of cyanide in the filtrate is the quantity that is not adsorbed by the adsorbent. This shows that the higher the concentration of measured cyanide in the solution, the lower the adsorption capacity of the adsorbent. The good adsorbents showed low cyanide concentrations in the experiment.

At 0.5g dose of the samples, coconut shell/ H_3PO_4 recorded the lowest cyanide concentration of $42.78 \pm 1.21 \text{ mg/dl}$ while the standard activated charcoal sample recorded the highest cyanide concentration value of $185.27 \pm 1.85 \text{ mg/dl}$. This cyanide concentration value for the standard activated charcoal is significantly higher than all the other values obtained for the other samples in the experiment ($p < 0.05$).

Across the four different doses of 0.5g to 2.0g, plantain peels/ ZnCl_2 recorded $46.66 \pm 1.22 \text{ mg/dl}$, $45.61 \pm 1.33 \text{ mg/dl}$, $44.56 \pm 0.70 \text{ mg/dl}$ and $43.51 \pm 1.27 \text{ mg/dl}$; castor oil seed shell/ HNO_3 recorded $44.31 \pm 1.74 \text{ mg/dl}$, $43.46 \pm 0.81 \text{ mg/dl}$, $43.82 \pm 1.73 \text{ mg/dl}$ and $43.78 \pm 1.80 \text{ mg/dl}$; coconut shell/ H_3PO_4 showed $42.78 \pm 1.21 \text{ mg/dl}$, $41.03 \pm 0.58 \text{ mg/dl}$, $40.73 \pm 0.19 \text{ mg/dl}$ and $39.33 \pm 2.33 \text{ mg/dl}$ respectively.

The cyanide concentrations for the standard activated charcoal across the quantities of 0.5g to 2.0g were $185.27 \pm 1.85 \text{ mg/dl}$, $175.52 \pm 0.64 \text{ mg/dl}$, $125.75 \pm 1.21 \text{ mg/dl}$ and $118.36 \pm 0.58 \text{ mg/dl}$ respectively. These values of obtained for the standard activated charcoal are significantly higher ($p < 0.05$) than the values obtained for the other activated charcoal samples in the experiment

Table 1 The cyanide adsorption capacities for the different adsorbents at various doses with different chemical activating agents

Sample	Treatment	0.5 g	1.0 g	1.5 g	2.0 g
Rice Husk	HNO ₃	54.27±2.30 ^{h*}	52.69±1.21 ^{b*}	53.93±1.73 ^{f*}	51.45±0.69 ^{ab*}
	H ₃ PO ₄	53.33±1.34 ^{gh*}	52.84±1.17 ^{b*}	52.04±1.15 ^{ef*}	52.23±1.00 ^{ab*}
	ZnCl ₂	53.95±0.72 ^{gh*}	52.69±1.33 ^{b*}	51.75±0.66 ^{ef*}	51.34±0.75 ^{ab*}
Edible Clay	HNO ₃	50.04±1.17 ^{defg*}	50.25± 1.28 ^{b*}	50.19±0.64 ^{de*}	49.85±1.73 ^{ab*}
	H ₃ PO ₄	51.04±0.60 ^{efgh*}	50.40±1.96 ^{b*}	49.85±1.15 ^{de*}	47.60±1.21 ^{ab+}
	ZnCl ₂	51.69±0.75 ^{fgh*}	50.17±1.37 ^{b*}	49.51±0.81 ^{de*}	48.00±1.73 ^{ab+}
Plantain Peels	HNO ₃	50.49±1.39 ^{defgh*}	50.00±2.31 ^{b*}	48.46±0.81 ^{cd+}	47.55±0.69 ^{ab+}
	H ₃ PO ₄	48.91±1.74 ^{cdef+}	47.91±1.16 ^{b+}	48.36±1.33 ^{cd+}	47.49±1.79 ^{ab+}
	ZnCl ₂	46.66±1.22 ^{abcd+}	45.61±1.33 ^{b+}	44.56±0.70 ^{b+}	43.51±1.27 ^{a+}
Castor oil seed shell	HNO ₃	44.31±1.74 ^{ab+}	43.46±0.81 ^{ab+}	43.82±1.73 ^{b+}	43.78±1.80 ^{a+}
	H ₃ PO ₄	47.36±0.17 ^{bcde+}	46.72±1.79 ^{b+}	45.78±0.40 ^{bc+}	46.12±1.21 ^{ab+}
	ZnCl ₂	46.87±0.59 ^{abcd+}	31.67±15.85 ^{af}	45.59±0.89 ^{bc+}	47.12±0.58 ^{ab+}
Coconut shell	HNO ₃	45.12±1.16 ^{abc+}	43.33±1.17 ^{ab+}	45.18±0.64 ^{b+}	43.43±1.29 ^{a+}
	H ₃ PO ₄	42.78±1.21 ^{a+}	41.03±0.58 ^{ab+}	40.73±0.19 ^{a+}	39.33±2.33 ^{a\$}
	ZnCl ₂	45.22±0.59 ^{abc+}	44.87±1.22 ^{ab+}	43.84±0.65 ^b	42.78±1.28 ^{a+}
STD activated Charcoal	-----	185.27±1.85 ^{id}	175.52±0.64 ^{cw}	125.75±1.21 ^{gβ}	118.36 ±0.58 ^{ba}

Mean± SEM, different letters down the column connotes significantly different ($p < 0.05$); different symbols across the row denotes significant difference ($p < 0.05$)

The study revealed that the coconut shells activated with phosphoric acid (H₃PO₄) demonstrated the highest adsorption capacity as shown in the experiment. Next was the castor oil seed shell activated with phosphoric acid, nitric acid and zinc chloride. This finding is similar to that found in the study by Ates and Ozcan, 2018 [28] in which the adsorption capacity of coconut shells activated with phosphoric acid (H₃PO₄) was found to be higher than carbon produced from other agro-based sources and activated with other chemical activating agents. The enhanced adsorption capacity for coconut shell activated with phosphoric (H₃PO₄) can be attributed to higher BET Surface area (BSA) and higher micropore area (MA) it possessed over the other agro-based sources of carbon [28, 29]. Phosphoric acid (H₃PO₄) has also been found to increase bond cleavage reactions and the formation of crosslinks via processes such as cyclization and condensation and combines with organic species to form phosphate bridges such as phosphate and polyphosphate esters [30, 31].

Plantain peels activated with Zinc chloride (ZnCl₂) also showed good cyanide adsorption with cyanide concentration values ranging from 43.78mg/dl to 44.31mg/dl. Zinc chloride (ZnCl₂) at high concentrations gives Bronsted acidity to the solution. The surface area increased when the temperature is increased from 500°C to 800°C. Zinc chloride has also been found to play an important role during thermal degradation at higher temperatures [28, 32].

We also discovered that there is no significant difference in the potassium cyanide adsorption following different doses of the locally-prepared activated charcoal samples. The cyanide adsorption was found to be in the same range for the activated charcoal prepared from different sources irrespective of the doses used. This contrasts with the findings in the study done by Dada et al in 2012 [33] in which the adsorption capacity of the different samples of activated charcoal increased with higher doses of the samples. The adsorption capacity of the standard commercial activated charcoal however, increased with higher doses in this study [33].

Our study also showed that the potassium cyanide (KCN) adsorption by the locally-prepared activated charcoal samples were all better than that of the standard commercial activated charcoal. This result is in conformity with the findings in other studies that compared the adsorption capacity of locally-prepared activated charcoal and standard commercial activated charcoal [33, 34].

4. Conclusion

The results of our study revealed that the adsorption capacities for cyanide for different samples of prepared activated charcoal varied significantly with the type of chemical activating agents used in the activation of the carbon and the raw materials used for the activated charcoal. Coconut shell activated with HNO₃ and H₃P₀4 showed the best adsorption capacity in the experiment. The cyanide adsorption by the locally-prepared activated charcoal was found to be better than that of the standard activated charcoal. Given the foregoing, locally-prepared agro-based activated charcoal can be a viable alternative to the standard activated charcoal in the treatment of cyanide poisoning in humans to curb the over-reliance on foreign standard commercial activated charcoal that is currently widely used globally.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors strongly declare no conflict of interest in this study.

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