Preparation of Activated Carbon from Cassava Root for Cadmium Removal in Aqueous Solution Sample

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Abstract

The purpose of this research was to study the properties of activated carbon produced from cassava root using phosphoric acid as catalyst at various ratios between charcoal and phosphoric acid and activated at different temperature levels. The chemical properties such as moisture, ash, volatile matter content and the amount of fixed carbon were determined. The surface morphology was observed using scanning electron microscopy (SEM); and elemental analysis were done using energy-dispersive X-ray spectroscopy (EDX). The study analyzed the effectiveness of the activated carbon in removing cadmium in water. The cadmium adsorption was determined using an atomic adsorption spectrometer (AAS). The results indicated that activated carbon could remove from 99 - 100 % of cadmium in an aqueous solution. The cassava roots are agricultural residues that can be used to produce activated carbon, which can adsorb heavy metals from industrial wastewaters. As such, it can add value to cassava roots, providing an effective and profitable way for disposal of agricultural waste.

Keywords:
Activated Carbon, Cassava Root, Phosphoric Acid, Heavy Metal

1. Introduction

Water is for direct consumption to sustain life. It is an important input in all economic activities, in agriculture, fishery and industry. Water is especially important in industrial processes, both as raw material and for cleaning and washing operations. For cleaning and washing operation, water is released back to the environment as wastewater. The wastewater or drain water from the industries might be contaminated with toxics materials and other pollutants which may result to environmental damages that negatively affects the living things in the surrounding environment [1]. As such, the wastewater must be treated before it is returned to the environment, into its source body of water (such as river, lake, or the sea).

In a primary treatment system, larger size solids floating in the wastewater are removed using screens. Smaller size solids and other suspended particles are removed in the second phase, using chemicals to precipitate them out and then again screened or filtered. Colorization and bad smell are generally removed through use of charcoal filters or activated carbon. The harmful pathogens are then wiped out in the third phase by biochemical reactions using beneficial microbes [2].

Activated carbon has proven to be significantly effective for eradicating smell, color, as well as heavy materials. Contaminated water can be purified and made less toxic to the environment using activated carbon. Activated carbon is thus used in many industries for wastewater treatment, such as in the plating and food industries. In addition, it is also used in production processes like in the production of bottled water and other highly purified drinks. These all result to higher demand for activated carbon [3].

The use of biomass from agriculture as the raw material to make activated carbon has been previously widely explored. Agricultural wastes can be used to produce cheaper activated carbon due to those materials being readily available and inexpensive. They are also environmental-friendly [3-4]. Agricultural wastes such as apricot shell, corncobs, coconut husk, jackfruit shell, olive trunk, cassava rhizome and root, etc. has already been extensively used.

Activated carbon is generated via chemical and physical simulations [5-9]. Wooden materials are mostly used. Carbonization is done at temperature ranging from 150 - 900 °C. Chemicals consisting of zinc chloride ($ZnCl_2$), phosphoric acid (H_3PO_4), and potassium chloride (KCl) are commonly utilized in activation. Phosphoric acid is more popular because the carbon activation process requires not too high temperature at about 400 – 500 °C, and the acid is reusable [10].

2. Objective of the Study

This study aimed to develop a process to produce activated carbon from cassava roots, an agricultural waste, by phosphoric acid stimulation. In harvesting cassava, cassava roots are cut off and then disposed of by burning. The burning process produces smoke and other air pollutants that can cause poisoning from smoke inhalation. It can severely impact the respiratory systems of people inhabiting in risk areas [11-12]. Using cassava roots to produce activated carbon will avoid the waste disposal and burning of cassava roots and thus also, the smoke and air pollution

Previous studies regarding the appropriate conditions to produce charcoal and activated carbon from several agricultural materials i.e. root, rhizome and peel of cassava have been widely documented. Treatment with phosphoric acid at 700 °C for 1 hour resulted to a product of 6.23% moisture content and 609.6 mg/g carbon content, while at 800 °C, resulted to 1.17% moisture content and 602.9 mg/g carbon content.

After the stimulation process, the activated carbon has become more porous with a lower iodine number at 800 °C. The higher temperature made the porous structure smaller and the iodine number lower. It was possible that smell, color, and heavy material adsorption have slightly decreased. Moreover, the XRD examination showed that the activated carbon produced was amorphous and that phosphates compound was also included. Additionally, a few nonrelated inorganics remained. These imply that activated carbon produced was good for adsorption [13].

As mentioned earlier, this study aimed to produce activated carbon from waste cassava root by phosphoric acid stimulation at lower temperature levels of 400, 500, 600, and 700 °C. This was to create wider porous surface, which raised the level of adsorption. This study analyzed the chemical structure, the structure and porousness of the surfaces, and the number of substances in the activated carbon through the Energy Dispersive X-ray Spectrometer (EDX) by a scanning electron microscope (SEM). The study then investigated the adsorption of heavy materials (i.e., cadmium) using the activated carbon produced from waste cassava roots to clean and purify water.

3. Research methodology and process

The following is a step-by-step presentation of the methodology and process used in this study:

3.1. Preparation charcoal from cassava root

(1) Bake the root at 105 °C for 3 hours
(2) Carbonize at 700 °C for 1 hour
(3) Grind the charcoal and winnow by using a 140-micron sieve.

3.2. Preparation of activated carbon from cassava root

(1) Soak the charcoal with 85-percent gradient phosphoric acid. The proportion of charcoal and phosphoric acid is 1:0.5, 1:1, and 1:1.5, for 24 hours, then dry it.

(2) Stimulate the dried charcoal by burning at 400, 500, 600, and 700 °C for 1 hour, let cool in a desiccator.

(3) Wash with 5-normal hydrochloric acid, then with distilled water until its pH value is neutral.

(4) Dry activated carbon at 105 °C and let cool in a desiccator.

3.3. Moisture content analysis

The method of moisture content was employed based on ASTM: D 2867 – 95.

(1) Burn a crucible at 600 °C for 10 minutes, then let cool in a desiccator.
(2) Weigh the crucible with activated carbon to be analyzed for 1 gram, then record the weight.
(3) Dry at 150 °C for 3 hours, then let cool in a desiccator.
(4) Weigh and record the crucible with activated carbon after drying, then calculate with equation (1)

Equation of moisture content calculation:

$$Percentage\ of\ moisture\ content\ by\ weight = \frac{C-D}{C-B} \times 100 \quad (1)$$

When B = weight of a crucible
C = weight of crucible with the sample before drying (g)
D = weight of crucible with the sample after drying (g)

3.4. Ashe content determination

Analysis for ashes in activated carbon from cassava root based on the standard of ASTM: D 2866-94

(1) Burn crucible at 900 °C for 10 minutes, then let cool in a desiccator.
(2) Weigh the crucible with activated carbon for 1 gram, with the cover.
(3) Burn it at 650 °C for 4 hours, then let cool in a desiccator.
(4) Weigh and record the activated carbon with crucible and cover after burning, then calculate with equation (2)

Equation of percentage of ashes calculation:

$$Percentage\ of\ ashes = \frac{B-D}{D-C} \times 100 \quad (2)$$

When B = weight of crucible with cover and the sample after burning (g)
C = weight of crucible with cover (g)
D = weight of crucible with cover and the sample before burning (g)

3.5. volatile matter detection

Analysis of volatile matter of activated carbon from cassava root based on the standard of ASTM: D 5832-95

(1) Burn crucible at 600 °C for 10 minutes, then let cool in a desiccator.
(2) Weigh the crucible with cover
(3) Fill 1 gram of the sample into the crucible, then weigh the crucible with the sample and cover.
(4) Burn at 950 °C for 7 minutes, then let cool in a desiccator.
(5) Weigh and record the crucible with the sample and cover, then calculate for volatile matter with equation (3)

Equation of volatile matter calculation:

$$Percentage\ of\ volatile\ matter = \frac{W_{before} - W_{after}}{W_{before}} \times 100 \quad (3)$$

When W_{before} = weight of the sample before burning (g)
W_{after} = weight of the sample after burning (g)

3.6. Fixed carbon analysis

This method is calculated with equation:

$$Percentage\ of\ fixed\ carbon = 100 - \left(\%\ ashes + \%\ volatile\ matter + \%\ moisture\right) \tag{4}$$

3.7. Physical properties

Surface and porous analysis and substances analysis by Energy Dispersive X-ray Spectrometer (EDS/EDX) using scanning electron microscopy (SEM).

(1) Bake activated carbon from cassava root at 120 °C, then let cool in a desiccator.
(2) Put the dried activated carbon on conductive tape as thin as possible, then stick with 1-cm Stub.
(3) Coat the activated carbon on the conductive tape with mixed gold with palladium in the evaporator that works in vacuum state for 2 minutes.
(4) Examine the coated sample with scanning electron microscopy (SEM).

3.8. Batch adsorption investigation

An experiment was carried out in a flask with 4.80 mg/L of adsorbent concentration of Cd^{2+} solution (pH 7.0). Activated carbon was prepared at different ratios and then mixed with initial solution. The flask was placed in a constant temperature shaker and shacked at 250 rpm for 60 min. After that the mixture was filtrated using a filter paper no. 42 to separate the adsorbent from supernatant. All experiments were replicated thrice for all the adsorbents and results were averaged. After attaining solutions, they were estimated by atomic absorption spectrometry (AAS). The removal percentage (R%) of cadmium was computed for each run by following expression (5):

$$R(\%) = \frac{C_i - C_e}{C_i} \times 100 \tag{5}$$

Where C_i = The initial concentration of cadmium in the solution (mg/L)

C_e = The final concentration of cadmium in the solution (mg/L)

4. Results and Discussions

4.1. Chemical property examination

The chemical properties of activated carbon made from cassava roots stimulated by phosphoric acid at several temperature levels is shown below in (Table 1).

Table 1 The chemical analysis of activated carbon done from cassava root.

Temperature (°C)	Ratio of charcoal: phosphoric acid	Moisture content (%)	Ash content (%)	Volatile matter (%)	Fixed carbon (%)
400	1:0.5	13.3094	5.8372	1.0979	79.7556
	1:1	14.2173	7.1562	1.1298	77.4967
	1:1.5	13.4558	12.8153	0.9439	72.7850
500	1:0.5	13.8376	3.4556	1.1290	81.5778
	1:1	13.2230	8.0558	1.0596	77.6617
	1:1.5	14.1861	6.9175	1.0697	77.8267

Temperature (°C)	Ratio of charcoal: phosphoric acid	Moisture content (%)	Ash content (%)	Volatile matter (%)	Fixed carbon (%)
600	1:0.5	13.0997	10.1685	1.1593	75.5725
	1:1	11.6112	11.0729	1.1512	76.1647
	1:1.5	11.9095	9.6216	1.0572	77.4117
700	1:0.5	12.8552	10.4945	1.2272	75.4232
	1:1	10.5988	8.2251	1.1095	80.0666
	1:1.5	15.9185	6.7057	0.9739	76.4018

Table 1 shows that moisture content decreases as the temperature increases even if the ratio between charcoal and phosphoric acid remains constant. At higher temperatures, there is heat that causes water evaporation that led to decreases in moisture content [14].

The ash content decreased from 5.8372 to 3.4556 % when the temperature increased from 400 to 500°C, and then increased when the temperatures increased to 600 °C and then to 700 °C when the ratio of charcoal and phosphoric acid was 1:05. When this ratio was increased to 1:1 the ash content increased at temperatures from 400 to 500 to 600 °C but decreased at 700 °C. At a higher ratio of 1:1.5, the ash content was fluctuating; with values of 12.8153, 6.9175, 9.6216, 6.7057% corresponding to temperatures 400, 500, 600, and 700 °C respectively. As a result, the amounts of ashes were relatively lower. It might be stated that phosphoric acid had dissolved or destroyed with the heat the inorganic matter within the porous surface.

In terms of the number of volatile matters, when the proportion of charcoal: phosphoric acid was 1:0.5 at 400, 500, 600 and 700 °C, the number of volatile matters were 1.0979, 1.1290, 1.1593, and 1.2272%, respectively. When the proportion of charcoal to phosphoric acid was 1:1 the amounts of volatile matters were 1.1298, 1.0596, 1.1512 and 1.1095, at 400, 500, 600, and 700 °C, respectively. The proportion of charcoal: phosphoric acid was 1:1.5 at 400, 500, 600, and 700 °C, the amounts of volatile matters were 0.9439, 1.0697, 1.0572 and 0.9739 %, respectively. It was suggested that phosphoric acid could clear volatile matter up from activated carbon, resulting in the increase of carbon content [14] because of the structural disintegration of activated carbon, in which the heat breaks the structure so that it evaporates from such a structure [15].

The fixed carbon content in the activated carbon produced using charcoal to phosphoric acid ratio of 1:0.5 and 400, 500, 600, at 700 °C were 79.7556, 81.5778, 75.5725, and 75.4232%, respectively. The fixed carbon content in the activated carbon produced using charcoal to phosphoric acid ratio of 1:1 and 400, 500, 600, at 700 °C were 77.4967, 77.6617, 76.1647 and 80.0666 %, respectively. The fixed carbon content of activated carbon produced using charcoal to phosphoric acid ratio of 1:1.5 at 400, 500, 600, and 700 °C were 72.7850, 77.8267, 77.4117 and 76.4018%, respectively. The analysis showed that the amount of carbon declined with higher values of moisture, ash, and volatile matter content.

The carbon content is the remaining part of the charcoal after moisture, ashes and volatile matter were removed due to the disintegration of structure that is lost as volatile matter or it might be a reaction between stimulant and carbon structure that resulted in the break of its structure caused by heating. Additionally, the stimulant might make the reaction by removing the water (H_2O) from the carbon structure [15]. ASTM defines that well-produced activated carbon should contain more than 80% carbon. The study shows that activated carbon with 70-85% carbon content can be produced at 500 °C and ratio 1:0.5 at 81.5778%; and at 700 °C and ratio of 1:1 at 80.0666%.

Based on the examination of the chemical properties of the activated carbon, it was revealed that stimulating activated carbon with phosphoric acid with various temperatures had good numerous porous surfaces. Especially, at 700 °C and charcoal to phosphoric ratio of 1:1 could give great properties (see

Table 1). However, the stimulation at lower temperature levels could serve as high activated carbon properties. As a result, it was able to adsorb serval substances such as Methylene Blue, color and heavy materials.

The quality of activated carbon made from cassava roots using phosphoric acid stimulation at 400, 500, 600, and 700 °C for one hour, were examined. The examinations were done on the porous surfaces of the activated carbon using Scanning Electron Microscopy (SEM). They were done for the following proportion of charcoal to phosphoric acid; 1:0.5, 1:1, and 1:1.5, The results of the examination are given below.

The quality of activated carbon produced using different proportions of charcoal to phosphoric acid at 1:0.5, 1:1, and 1:1.5 under 400 °C, are shown on Figure 1. At 500X magnification (see Figures 1-a, 1-c & 1-e) - the outside structures of activated carbon can be seen to be incompletely e porous. There are broken scratches on the edge of the porous surface because of trituration. At 1,500X magnification, the remaining inorganic matters stuck on the outside structure of activated carbon can be observed (see Figures 1-b, 1-d & 1-f). The photos also show the inside structure having broken scratches on the edge of the porous surface and containing remaining inorganic matters, which at 400 °C, could not be eradicated by evaporation. These results were consistent with the results the examination of the porous surface using scanning electron microscopy [13].

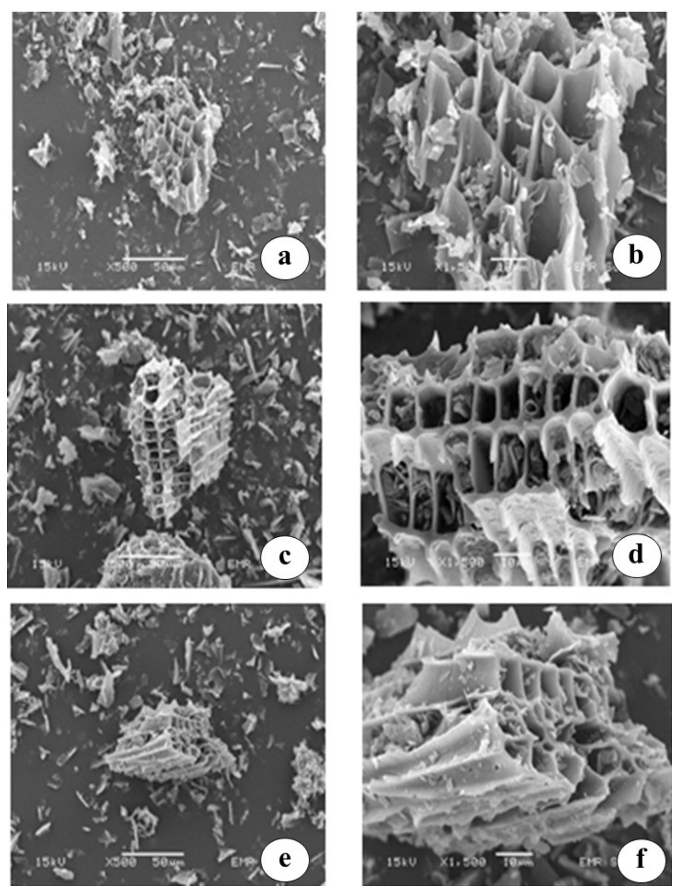

Fig. 1 The porous surface of activated carbon is stimulated at 400 °C with (a) the portion 1:0.5 at 500-time enlargement, (b) the portion 1:0.5 at 1,500-time enlargement, (c) the portion 1:1 at 500-time enlargement, (d) the portion 1:1 at 1,500-time enlargement, (e) the portion 1:1.5 at 500-time enlargement, and (f) the portion 1:1.5 at 1,500-time enlargement.

The quality of activated carbon produced using different proportions of charcoal to phosphoric acid at 1:0.5, 1:1, and 1:1.5 under 500 °C, are shown on Figure 2a to 2f. At 500X magnification (see Figures 2-a, 2-c, and 2-e) the incomplete porous surface of the outside structure, containing broken scratches on the edge of the porous surface can be seen. This is from trituration, which causes broken edges on the porous surface and unarranged porous surface. It means that this higher temperature level produces more porous surface [16]. A 1,500X magnification (see Figure 2-b, 2-d, and 2-f) shows the inside structure containing broken scratches on the edge of the porous surface, and a cleaner surface because the higher temperature destroyed the inorganic matters on the activated carbon surface. [17].

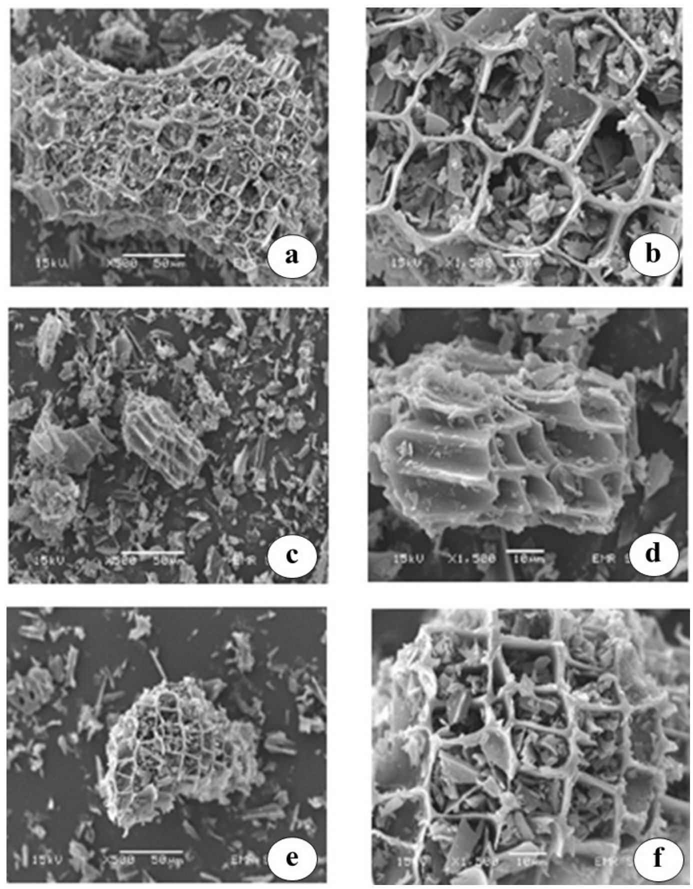

Fig. 2 The porous surface of activated carbon is stimulated at 500 °C with (a) the portion 1:0.5 at 500-time enlargement, (b) the portion 1:0.5 at 1,500-time enlargement, (c) the portion 1:1 at 500-time enlargement, (d) the portion 1:1 at 1,500-time enlargement, (e) the portion 1:1.5 at 500-time enlargement, and (f) the portion 1:1.5 at 1,500-time enlargement.

The quality of activated carbon produced using different proportions of charcoal to phosphoric acid at 1:0.5, 1:1, and 1:1.5 under 600 °C, are shown on Figure 3 (a-f)., A 500X magnification (see Figure 3-a, 3-c, and 3-e) shows less inorganic matter and smaller porous surface. There appear to be more porous surface under temperatures of between 600-800 °C [18]. A 1,500X magnification (see Figure 3-b, 3-d, and 3-f) shows the surface to be cleaner with a few inorganic matters and the porous surface smaller. This is because the phosphoric acid stimulation was done between 400-700 °C. It was expected the porous surface will become larger at 700-900 °C. [19].

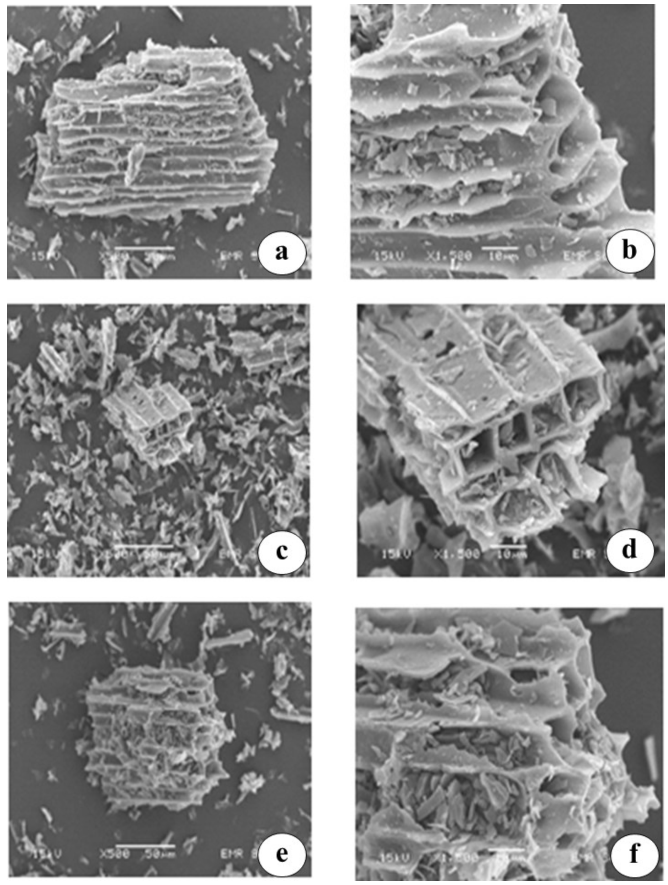

Fig. 3 The porous surface of activated carbon is stimulated at 600 °C with (a) the portion 1:0.5 at 500-time enlargement, (b) the portion 1:0.5 at 1,500-time enlargement, (c) the portion 1:1 at 500-time enlargement, (d) the portion 1:1 at 1,500-time enlargement, (e) the portion 1:1.5 at 500-time enlargement, and (f) the portion 1:1.5 at 1,500-time enlargement.

The quality of activated carbon produced using different proportions of charcoal to phosphoric acid at 1:0.5, 1:1, and 1:1.5 under 700 °C, are shown on Figures 4 a-f. A 500X magnification (see Figure 4a, 4-c and 4-e) shows several unarranged porous surfaces and a few inorganic matters. These are consistent with the prior report [20]. A 1,500X magnification (see Figures 4-b, 4-d, and 4-f) shows that the porous surface was wide, indicating the diffusion of phosphoric acid during the stimulation process [21]. Only a few inorganic matters can be seen, perhaps because the higher temperature destroyed much of the inorganic matters thus making the porous surface cleaner [17].

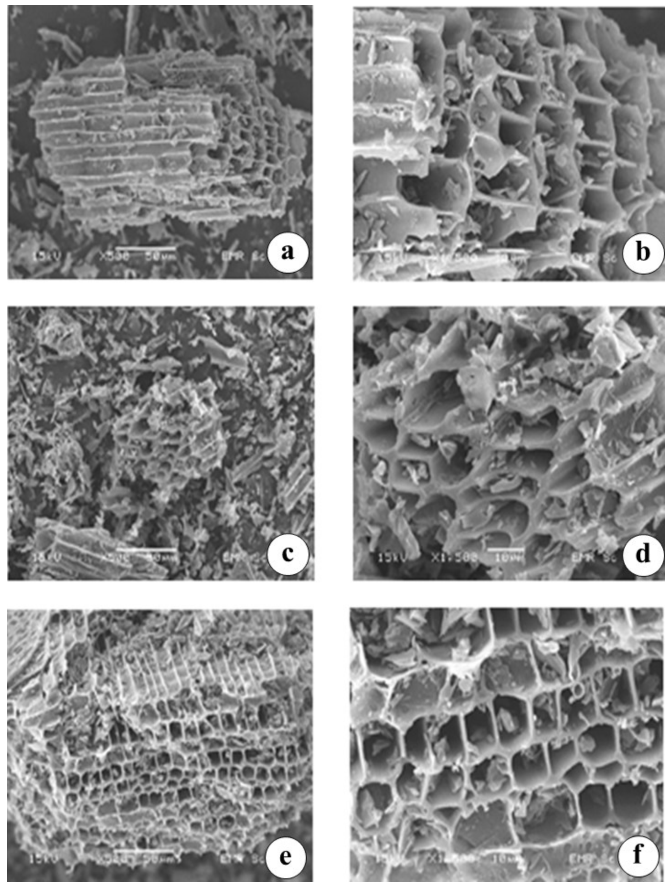

Fig. 4 The porous surface of activated carbon is stimulated at 700 °C with (a) the portion 1:0.5 at 500-time enlargement, (b) the portion 1:0.5 at 1,500-time enlargement, (c) the portion 1:1 at 500-time enlargement, (d) the portion 1:1 at 1,500-time enlargement, (e) the portion 1:1.5 at 500-time enlargement, and (f) the portion 1:1.5 at 1,500-time enlargement.

4.2. Amounts of elements analysis using Energy Dispersive X-ray Spectrometer (EDS/EDX)

An examination of the porous surface of the activated carbon using scanning electron microscopy showed the remaining inorganic matters. The remaining inorganic matters were examined further using an energy dispersive X-ray. spectrophotometer. This can show the amounts of remaining elements on the activated carbon surface. The results are shown on Table 2.

The elemental analysis of the various activated carbon produced at temperatures 400, 500, 600, and 700 °C and at proportions of charcoal to phosphoric acid at 1:0.5, 1:1, and 1:1.5, showed that the activated carbon comprised mostly of carbon. At production temperature of 600 °C, with proportion of 1:1, carbon content was at the lowest at 78.75%. Carbon content at 89.17% was at the highest at production temperature of 700 °C with proportion of 1:1.5. These results conformed to the general estimates that activated carbon contain between 80-90% of carbon. Minimal amounts of oxygen were found due to probably to the removal of silica (SiO2) in the cassava roots [14, 22]. Sodium and chlorine were also present. The sodium came from the sodium hydroxide (NaOH) used to adjust the pH from acidic to neutral before the adsorption examination. Chlorine came from using hydrochloric acid (HCL) used for washing the charcoal after the stimulation process [23]. All these elements were on the activated carbon porous surface.

Table 2 The results of element analysis within activated carbon made from cassava root.

Temperature (°C)	Ratio of charcoal: phosphoric acid	Element (%)				
		C	O	Na	Cl	Si
400	1:0.5	82.82	14.74	0.73	1.83	-0.14
	1:1	79.74	15.22	0.83	4.13	0.08
	1:1.5	82.33	14.23	1.03	2.87	-0.45
500	1:0.5	84.91	10.52	0.89	4.37	-0.68
	1:1	83.33	11.94	1.46	3.47	-0.21
	1:1.5	87.50	9.69	0.21	3.17	-0.57
600	1:0.5	81.66	12.21	1.22	5.24	-0.34
	1:1	78.75	15.42	1.50	4.51	-0.18
	1:1.5	86.54	10.35	0.41	3.35	-0.65
700	1:0.5	86.08	7.15	1.04	6.41	-0.69
	1:1	84.42	9.19	0.97	5.64	-0.22
	1:1.5	89.17	6.98	0.49	3.64	-0.28

4.3. Cadmium adsorption

The standard absorbance values of several cadmium solutions of known concentrations were determined using an Atomic Adsorption Spectrometer (AAS) (see Table 3). The results were plotted to establish a standard graph (see Figure 5) that can be used to determine the concentration of other cadmium solutions by just using the absorbance values for the results of the ASS tests. The Coefficient of Determination or $R^2 \geq 0.995$ in this case [38].

Table 3 Adsorption of standard cadmium solution.

Concentration (ppm)	Absorbance (nm)
0	0.0015
0.5	0.1269
1	0.2068
2	0.4357
3	0.5986
4	0.8711

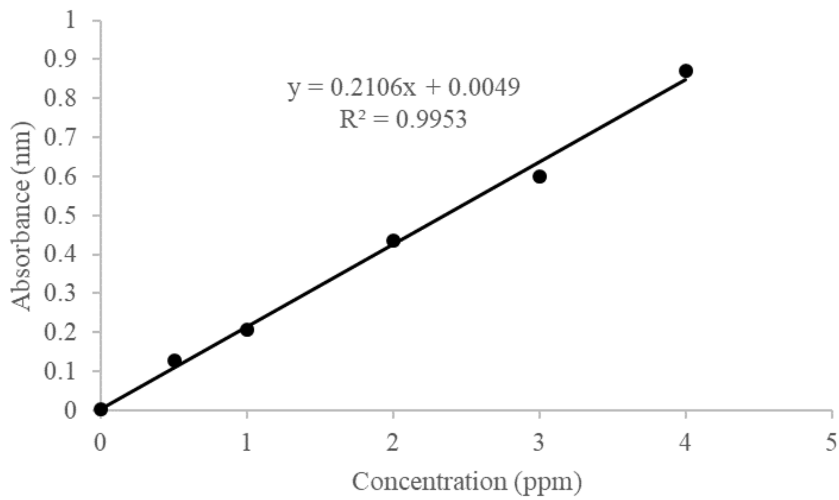

Fig. 5 Standard graph of cadmium solution.

Cadmium is a heavy material widely used in industries; hence, it can easily contaminate the environment. Several studies have been attempted to demonstrate the effectiveness of activated carbon in adsorbing cadmium [21, 22, 24] to prevent cadmium contamination and its adverse health and environmental impacts. The percentage of cadmium adsorbed by different activated carbon produced from cassava roots at temperatures of 400, 500, 600 and 700 °C, and at proportion of charcoal to phosphoric acid at 1:0.5, 1:1, and 1:1.5, were examined. The detailed results shown on Table 4.

Table 4 10-ppm Cadmium adsorption by activated carbon made from cassava root at several temperature levels and proportions.

Temperature (°C)	Ratio of charcoal: phosphoric acid	Removal (%)
400	1:0.5	99.50
	1:1	99.92
	1:1.5	100.00
500	1:0.5	100.00
	1:1	100.00
	1:1.5	100.00
600	1:0.5	100.00
	1:1	100.00
	1:1.5	100.00
700	1:0.5	100.00
	1:1	100.00
	1:1.5	100.00

The study showed that activated carbon made from cassava roots at every temperature and proportion levels, can absorb all the cadmium in water except at 400 °C and at proportion levels of 1:0.5 and 1:1. Activated carbon produced at this temperature and proportion levels showed adsorption of only up to 99.50 and 92.92%, respectively. This may be because the temperature was not high enough. Higher temperature can change the structure of activated carbon which can result to better adsorption [25].

5. Conclusion

Activated carbon burnt at 700 °C with the charcoal to phosphoric acid ratio of 1:1 produced the best chemical properties because of lower values of moisture, ash, and volatile matter content, but higher fixed carbon content at more than 80%. The studies show that this activated carbon can adsorb nearly 100% of cadmium contained in an aqueous solution. The amorphous structure examination using scanning electron microscopy (SEM), showed numerous pores with some inorganic matters on the surface. This conformed to the results on the analysis of the amounts of substances in the activated carbon, using the energy-dispersive X-ray spectroscopy (EDX), which gave carbon content at 84.42%. percent of carbon. These analysis and studies showed that cassava roots are good raw material for activated carbon, and therefore can be a good option for the utilization and disposal of agricultural wastes.

6. Suggestions

(1) There should be more stimulations with other kinds of chemical substances such as zinc chloride, because the lower temperature level can eradicate volatile matters from the porous surface well.

(2) There should be utilization of other materials that contain a good chemical quality, high carbon and low moisture, ashes and volatile matter, in order to increase the surface to adsorb.

(3) There should be more studies about the temperature of carbonization and stimulation to be suitable with the raw material because burning at numerous temperature levels affects the substance eradication on the porous surface and adsorption.

(4) There should be more studies about the pore size of activated carbon that might be related to the adsorption of other heavy metals including cadmium.

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