

Characteristics of Activated Carbon from Cassava Stump Prepared at High Temperatures Using Physical Reactivation and Rapid Procedure

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Abstract

Cassava is one of the top-five most exported agricultural products of Thailand. After harvesting the crop there are an estimated 5.5 million metric tons yearly of cassava stumps left in the field. The farmer generally gets rid of such waste by burning that causes environmental pollution. To solve this problem, a highly efficient, commercial-scale furnace was developed to convert useless cassava stumps into high-value activated carbon. The furnace was run for 30 minutes, operating at a high temperature of about 1,200 °C with physical activation. The characteristics of the activated carbon from cassava stumps produced by this furnace were studied. The BET surface area value was 110.96 m²/g compared to the Langmuir surface area value of 118.16 m²/g. The BET value is satisfactory for this burning process. An aromatic hydrocarbon functional group was found in the activated carbon sample using FT-IR spectroscopy. The physical structure of the sample was studied using scanning electron microscopy. The life cycle assessment of this research was assessed based on gate-to-gate boundary production of activated carbon from cassava stumps. Converting cassava stumps into activated carbon will reduce greenhouse gas emissions. The emission factor of the cassava stump furnace is 0.0985 ton CO_{2eq}.

Keywords: Activated carbon; Adsorption; Cassava stump; Environmental cycle assessment; Waste to energy

1. Introduction

In 2020, Thailand exported 2,537,027 metric tons of fresh and dried cassava worth USD 546 million making Thailand the largest cassava exporter in the world. Each year, the yield from cassava is 30 million metric ton (Mmt), with the cassava stumps as agricultural waste accounting for about 5.5 Mmt (Viriyathonphan, 2017). Thai farmers have generally paid disposal expenses to get rid of this waste to landfill or by burning. In landfill, the stumps take about three years to decompose, resulting in possible mold problems and the development of mutant diseases. Therefore, the farmers generally burn the cassava stump in the open air which causes

environmental pollution. To solve this problem, there been research into converting the useless cassava stump into high value activated carbon (AC) not only to reduce global warming but also to decrease imports of AC.

In Thailand, there has been some research into AC, mostly produced from agricultural materials since the 1900s. Due to the various applications of AC, the critical factor for effective AC products is the type of furnace and activation technique (Mopoung, 2015). Nowadays, a smokeless kiln called a gasification furnace is widely used (Khongkrapan, 2016). Thai researchers prefer chemical activation rather than physical

activation because the former can utilize a small furnace at lower temperature and can be investigated in the laboratory. However, the disadvantages of chemical activation are that it takes a long time to produce the AC, it is difficult to prepare the chemical solution, and there may be problems with the chemical residues (Phuangchik, 2015). Furthermore, there has been little interest in Thailand in the AC production process using cassava stumps as the raw material.

Conventional furnaces in the production process usually have the operating time for both carbonization and the activation process of at least 2 hours (Tippayawong *et al.*, 2017). A single step gasification furnace for commercial scale was designed in the current research to run at a maximum temperature of 1,200 °C with physical activation occurring in a total operating time of 30 minutes. The short operating time saves a lot of energy, resulting in a low production cost. The AC production capacity from this furnace was 10 metric ton per day. According to the standards and market demand, the characteristics of the AC from cassava stumps were investigated and compared to samples from relevant research. In addition to studying the properties of AC, an environmental impact assessment of the AC production from cassava stump was considered. The evaluation results should help to improve and develop the AC production process, to obtain a better quality of AC, and to reduce the impact on the environment.

2. Materials and methods

2.1 Sample preparation

To prepare the sample, the moisture content of raw material was reduced to 19 – 20% using sun drying for 3 days. The dirt was removed from the raw material before it was placed in the furnace. After the furnace had been sealed, the temperature of the furnace was continuously raised from room temperature to 1,200 °C in 25 minutes and maintained at 1,200 °C for 5 minutes. The total time for carbonization and activation processes was 30 minutes. After that, the activated carbon was sieved and cooled at room temperature without oxygen for 24 hours.

2.2 Methods

The current experiment was carried out in accordance with Thailand Industrial Standard Institute (TISI) 900 - 2004, in which the apparent density and BET value are determined as AC efficiency (Institute, 2004). The efficiency of activated carbon determines the price of activated carbon in the market. This standard covers all four types of AC: granules, powder, pellets, and sticks.

2.3 Apparent density

The apparent density was determined according to the American Water Works Association (AWWA) Standard B600-05 for the powdered AC method (Association, 2010). The apparent density of AC is defined as the weight in grams per cubic centimeter (g/cc³) of the AC in air. In the experiment, 10 g AC at room temperature was placed in a desiccator for testing. After that, the AC was transferred to a 50 mL or 100 mL graduated cylinder, until the entire sample was transferred. AC was left for 5 – 10 minutes to settle (Association, 2010). The volume of the settled AC was noted. The apparent density on a dry basis was calculated using equation 1:

$$\text{Apparent density} = \frac{(\text{Weight of sample in grams}) \times (100\% \text{ moisture})}{(\text{Volume of sample in cubic centimeters}) \times 100} \quad (1)$$

2.4 Surface area adsorption

Nitrogen adsorption experiments based on Brunauer-Emmett-Teller (BET), BET Surface area analyzer (Micromeritics, 3Flex Surface characterization) were used to study the consistency of the surface area related to the iodine number. The aims of the BET technique are to analyze the pore size diameter and to calculate the surface area of the AC and the pore volume of different types (micropore, mesopore, macropore). The BET model applies nitrogen adsorption at different pressures at the temperature of liquid nitrogen (77 K). The surface area according to BET was determined based on the product of the cross-sectional area of the nitrogen molecule. The BET surface area was calculated using equation 2 (Brunauer *et al.*, 1938). The pore volume was determined by the commonly used nitrogen adsorption isotherm data.

$$A_s = \frac{A_m N_A}{V_m(s+y_0)} \times 10^{-18} \text{ m}^2/\text{nm}^2 \quad (2)$$

Where

A_m = cross sectional area of the adsorbate in nm^2

N_A = Avogadro Constant (6.02214129 x 1,023 mol⁻¹)

V_m = molar volume on a mass basis

S = Slope ($\text{cm}^3/\text{g STP}$)

y_0 = Y -intercept ($\text{cm}^3/\text{g STP}$)

2.5 Fourier-transform infrared spectroscopy

Fourier-transform infrared spectroscopy (FT-IR) was used to determine the elements and chemical compounds present on the surface of the AC and their properties that promoted the adsorption capacity of the AC. FT-IR was performed on a PerkinElmer spectrophotometer operating in the transmittance method. The spectra were operated in the MID-IR range (400–4,000 cm^{-1}) with a spectral resolution of 4 cm^{-1} . The samples were pressed into KBr pellets. The ratio of sample-to-KBr was 1.4:100 (0.0007 g: 0.05 g).

2.6 Scanning electron microscopy

Scanning electron microscopy (SEM), Quanta 450 FEI, was used to study the order and porosity structure of AC and compared with the quantitative measurement of the adsorption properties of AC. The physical structure was evaluated using a high vacuum voltage and the secondary electrons mode at magnifications of 1,500 \times and 2,000 \times .

3. Results and Discussion

3.1 N_2 adsorption isotherms and physical properties

The properties of the AC resulting from apparent density and BET model analysis are shown in Table 1. The apparent density of AC provides a good indication of its absorbency. The apparent density also relates to the BET model. The total surface area is an important indicator of the efficacy of the AC. The AC from the cassava stumps had an apparent density of 0.3715 g/cc^3 and a BET surface area of $110.96 \pm 0.73 \text{ m}^2/\text{g}$.

Due to the physical activation at high temperatures, the characteristics of the AC in the current research were expected to have a high surface area based on type II adsorption isotherms and multilayered adsorption on the surface of the material, which may differ depending on the sample material (Thommes *et al.*, 2015). The adsorption curve increases when the relative pressure increases by almost 1, instead of a constant amount, when the relative pressure increases. As shown in Figure. 1, the AC sample had mesopores and narrow ranges of hysteresis loops formed caused by the condensation of nitrogen gas in the pores. The hysteresis loops were of the open type, which is a common feature of AC where there are mesopores and micropores combined. The characteristics indicating micropores could also be observed as the adsorption volume increased rapidly at low relative pressures.

Table 1. Parameters characterizing porous structure of AC calculated from nitrogen adsorption/desorption isotherms.

Parameters	Cassava stump
Apparent density (g/cc^3)	0.3715
BET surface area (m^2/g)	110.96 ± 0.73
C-value	361.83
Langmuir surface area (m^2/g)	118.16 ± 1.18
Micropore area (m^2/g)	41.79
Micropore volume (cm^3/g)	0.0214

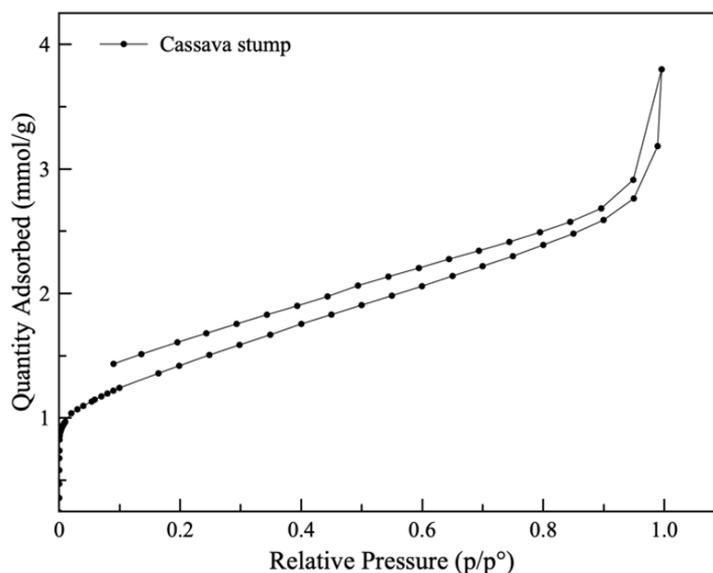


Figure 1. Nitrogen adsorption isotherm of cassava stump AC

Table 2. Surface area and volumes of micropores and mesopores of AC sample

Sample	S_{BET} (m^2/g) [*]	Micropores			Mesopores		
		S_{mic} (m^2/g) ¹	V_{micro} (cm^3/g) ²	D_p (nm) ³	S_{meso} (m^2/g) ⁴	V_{meso} (cm^3/g) ⁵	D_p (nm)
Cassava stumps	110.96	41.79	0.021	0.5002	66.68	0.071	4.2855

^{*} S_{BET} = BET surface area (m^2/g)

¹ S_{mic} = surface area of micropores (m^2/g)

² V_{mic} = micropore volume (cm^3/g)

³ D_p = pore diameter (nm)

⁴ S_{meso} = surface area of mesopores (m^2/g)

⁵ V_{meso} = mesopore volume (cm^3/g)

Table 2 shows the cassava stump AC had a BET surface area value of 110.96 m^2/g which is quite low. This can be explained by the porous structure of the AC produced from softwood (such as cassava stumps) is destroyed by high temperature resulting in fewer micropores in the material.

The cassava stump AC had a surface area ratio of micropore-to-mesopore of about 38:62. From the results, most of the surface area of the cassava stump was occupied with mesopores. The surface area value depends on the surface area of the micropore, which indicates the adsorption capacity.

Figure 2(a) shows the distribution of the micropores, demonstrating the relationship between the nitrogen adsorption volume and the pore size. The pore sizes were mostly in

the range 0.45 – 0.50 nm, which is the range of the highest adsorption volume. For the distribution of mesopores in Figure 2(b), the maximum adsorption volume was in the range 4.00 – 4.20 nm pore size.

A summary of the various reports in the literature regarding cassava stumps and the experimental conditions is shown in Table 3. In Thailand, cassava stump is one form of agricultural waste that is processed into AC. However, generally, chemical activation agents, such as H_3PO_4 , $ZnCl_2$, and KOH , are used to produce AC. Chemical activation takes longer to produce AC than physical activation because there must be a chemical soaking process. Furthermore, there may be chemical residues in the AC. However, the chemical activation will generate a high surface area

at a low activation temperature. The current study used physical activation (a one-step gasification method) with the activation agent being air at high temperature, with the whole process taking 30 minutes for AC production. The BET surface area of the current work was higher than from using physical activation but a different activation agent (98 m²/g) and higher than that from using chemical activation with ZnCl₂ (92 m²/g), as shown in Table 3. This experiment produced AC using an industrial-scale charcoal furnace with a production capacity of 10 metric tons per day. The reference data in Table 3 were produced on a laboratory scale.

Figure 3 shows the period of the nitrogen adsorption isotherms for the AC from cassava stump. Since the period of nitrogen adsorption isotherms was short, the porosity structure of the cassava stump sample was mainly macropores due to the macropores only acting as conduits to mesopores or micropores.

3.2 FT-IR analysis

The physical and chemical properties of the AC were investigated as functional groups, polarity, and electrostatic charge. These properties are essential parameters for determining the adsorption of various underlying substances, including some heavy metals. These were used in the study of FT-IR spectroscopy.

Figure 4 shows the FT-IR spectra of AC made from cassava stumps. The samples were analyzed using a gasification process at high temperatures up to 1,200 °C.

Almost all the chemical functional groups had disappeared; however, some functional groups were present. The bands between 3,500 and 3,400 cm⁻¹ corresponding to (-OH) group were evident. The (-OH) group was presented due to the AC absorbing moisture (Thammee *et al.*, 2012). The group of bands at 1,430, 1,084, and 875 cm⁻¹ of the samples referred to aromatic C-C stretch (in-ring) (Sulaiman *et al.*, 2012), silica (Si-O) (Thammee *et al.*, 2012) and oop C-H bending bands (Tho *et al.*, 2021), respectively. In addition, an aromatic hydrocarbon was found on the surface of the cassava stump sample, having a characteristic odor and being a non-polar covalent molecule. Therefore, it is easily soluble in non-polar solvents. And silica was found at 1,084 cm⁻¹ due to the AC was activated at high temperature.

3.3 SEM analysis

The physical structure of the sample was studied using scanning electron microscopy at magnifications of 1500× and 2000×. Figure 5 shows the notably porous structure of the AC from cassava stumps. The large pores were square, with a thin cell wall, which affected the adsorption efficiency of AC, with relevant factors being:

1. The wood in cassava stumps is soft with a thin cell wall structure. Therefore, in the carbonization stage, almost macropores occur.
2. The temperature of carbonization and physical activation are too high for a short time, which destroys the pore structure of the AC and removes smaller components.

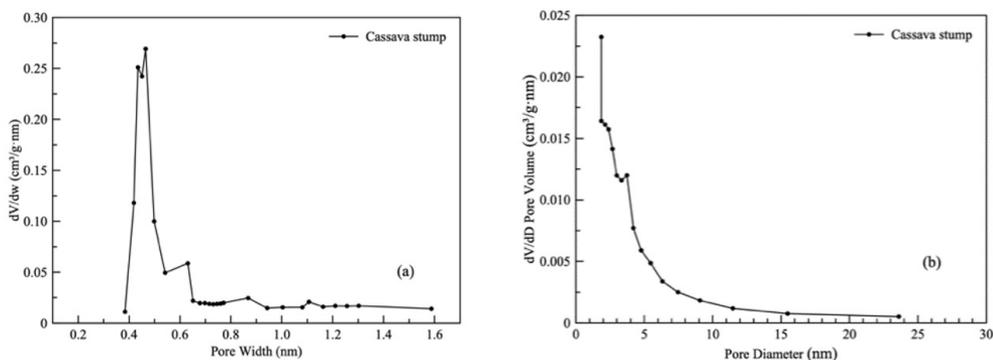


Figure 2. Pore distribution curve of AC from cassava stumps: (a) micropore distribution; (b) mesopore distribution

Additionally, more macropores are generated than micropores (Poolprasert, 2020). The greater numbers of macropores in the AC give it high sensitivity with a low efficiency of adsorption (Temyarasilp, 2008).

3. Using air as the gas medium and a high temperature for the physical activation process results in a lot of ash that can clog the pores and reduce adsorption.

Table 3. Comparisons of porosity characteristics of AC from cassava stump materials in of current work compared with other literature

Reference	Activation method	Activation temperature (°C)	Total process time* (h)	Iodine number (mg/g)	S _{BET} (m ² /g)	V _{tot} (cm ³ /g) ¹	V _{micro} (cm ³ /g)
<u>Cassava Stump</u>							
Present work	Physical (air)	1,200	0.5	434.07	110.96	0.10	0.02
(Thammee <i>et al.</i> , 2012)	Chemical (H ₃ PO ₄) 85%	700, 800	12 – 14	609.6	-	-	-
(Aupparee, 2016)	Chemical (fly ash solution)	900	24 – 32	655.44	-	-	-
(Wongnate, 2011)	Chemical (ZnCl ₂)	500	1.5 – 3.5	725	91.5	-	-
(Wongnate, 2011)	Physical (CO ₂ with microwave)	385 W	1.5 – 3.5	690	98.6	-	-

* The total process time is the period from the sample in the dry state through soaking the sample in a chemical agent for activation, and then the carbonization period until the end of the activation process. ¹V_{tot} is total pore volume (cm³/g)

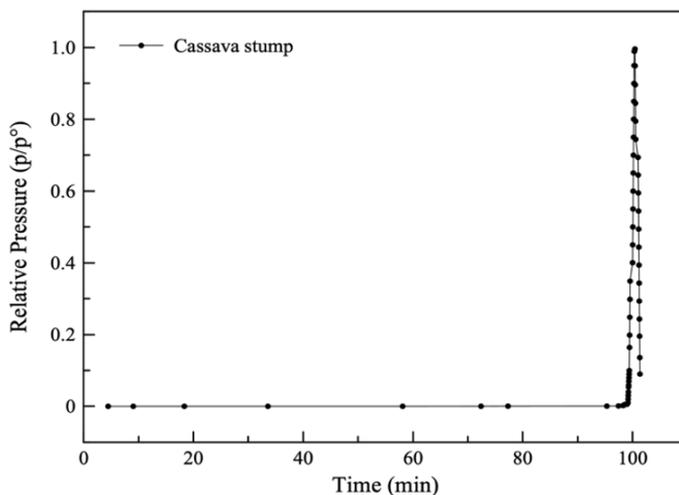


Figure 3. Period of nitrogen adsorption isotherms for AC from cassava stumps.

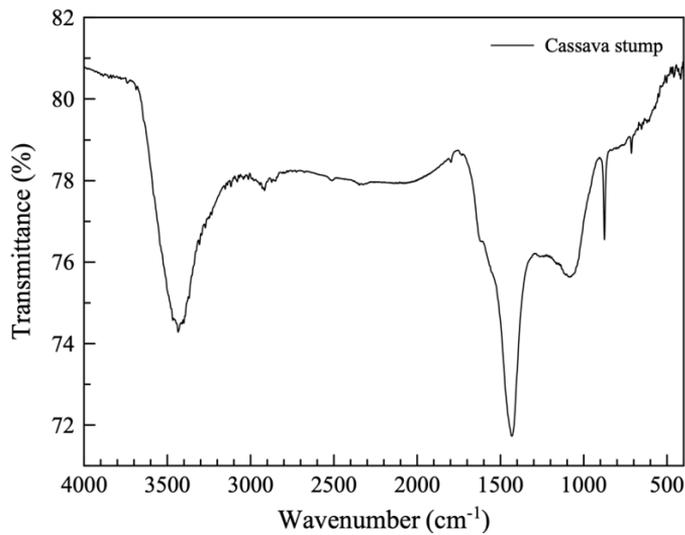


Figure 4. FT-IR spectra (KBr pellets) at 1,200 °C of AC from cassava stump.

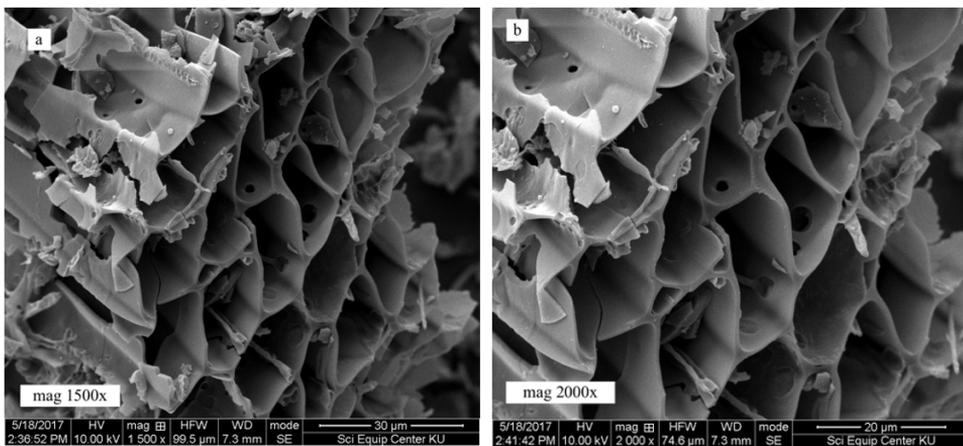


Figure 5. Scanning electron micrographs of AC from cassava stumps at high voltage (10.00 kV): (a) 1500× magnification; (b) 2000× magnification

3.4 Life cycle assessment

The scope of the environmental cycle assessment study for the gate-to-gate production process of AC from cassava stumps applied in the current research is shown in Figure 6 (Arena *et al.*, 2016).

The scope of the environmental cycle assessment was specific to the activities occurring within the manufacturing plant, from the drying process to the production of charcoal in the gasification furnace. The physical activation of AC used heat up to

1,200 °C, combined with compressed air as an activating agent until the process of compaction and sizing of the AC was completed, as shown in Figure. 7. The AC production capacity of the current study was 10 kg of cassava stump raw materials yielding 1 kg of AC. Many incoming resources were used in different quantities in each process, such as electricity, water, air, gas, and fuel. This process released water, CO₂, and CO that affected the environment, in addition to the AC product (Figure. 7).

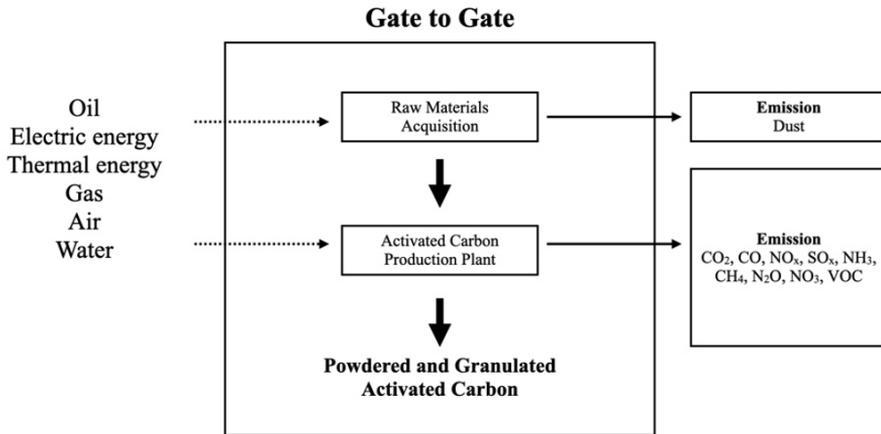


Figure 6. System boundaries for activated carbon production system for environmental cycle assessment analysis

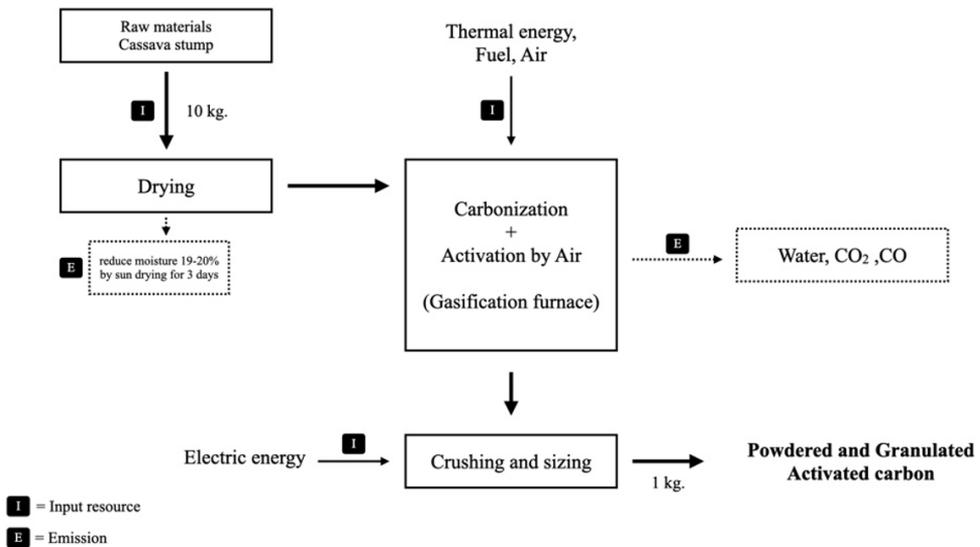


Figure 7. AC production

The furnace was an innovation created by combining the principle of pyrolysis with the gasifier technique. The gasifier process reduces greenhouse gases by using volatile substances as fuel to increase the temperature of the furnace during combustion. The volatile substances in cassava stump (up to 75%) consisted of hydrogen (H₂), methane (CH₄), and carbon monoxide (CO). This innovative process decomposed at least 80% of the non-carbon dioxide (CO₂) greenhouse gas emissions produced using a conventional AC furnace.

The pyrolysis process in the furnace started at a normal temperature; then, at 300 °C, the cassava stumps released volatile

substances. The heat in the furnace with the remaining oxygen caused the fuel gases in the volatile substances (such as CO, H₂, CH₄) to react chemically. Therefore, CO₂ inside the furnace is involved in the combustion of the volatile substances containing carbon as a component. The chemical reaction $CO + CH_4 = CO_2 + H_2O$ shows that carbon dioxide was generated in the furnace from the gasifier technique. Hydrogen, methane, carbon monoxide, and water vapor acted as fuel gases for combustion in the furnace. The fuel gases increased the temperature inside the furnace to greater than 1,200 °C, reducing greenhouse gases by at least 7% of the raw material weight without using any

fuel or external energy. The cassava stumps were completely converted into AC, so the combustion process terminated.

The properties of the AC from wood vary according to the burning characteristics of different furnaces. The cell wall composition of the wood is primarily organic compounds, with carbon as the main molecular constituent. When wood undergoes pyrolysis, carbon and solids in the wood are converted to fixed carbon.

The volatile substances found in AC were approximately 7 – 30% by weight, which is much less than in wood; when the AC was heated above 750 °C, various gases were produced, such as carbon dioxide (about 9%), carbon monoxide (about 20%), hydrogen (about 64%), and other hydrocarbons (about 7%). The ash, which is part of the inorganic component that remains after being heated above 950 °C, is about 0.5 – 10%, based on the detection of inorganic substances in AC. Neutron activation analysis identified small quantities of inorganic compounds, such as Al, Br, and Cu. The assumed composition of the cassava stumps used to investigate its potential as fuel for heat and electricity generation are provided in Table 5.

All volatile substances, such as hydrocarbons, become gaseous in the furnace and complete combustion turns

them into very stable gas. An emissions factor is a representative value that attempts to relate the quantity of a pollutant released to the atmosphere with an activity associated with the release of that pollutant. Therefore, it is used as a reference to determine the emission factor (EF) defined as the weight of CO₂ produced by activity/ natural weight of CO₂ in units of g CO_{2eq} or kg CO_{2eq} or t CO_{2eq}.

Normal AC has an EF value of 1.0054 kg CO_{2eq} (Ecoinvent, 2.2 IPCC 2007 GWP 100A Update 24Sep 2012).

The fixed carbon value of AC from cassava stumps had a lower EF value due to removing volatiles substances.

Therefore, when burning the cassava stumps in this furnace with an input of s 10 kg of dried cassava stumps and an output of 1 kg of AC, the total remaining carbon content in the furnace was 4.612 kg. Thus, the remaining carbon burned was 3.612 kg, meaning that if 1 kg of AC was converted to 3.666 kg CO₂, then 10 kg of AC would produce 36.666 kg CO₂. Therefore, the EF of the cassava stump furnace was 3.612 / 36.666 = 0.0985 kg CO_{2eq} or 0.098 t CO_{2eq}. The EF of the AC is the amount of CO₂ produced by burning / amount of naturally occurring CO₂ = 3.612 / 3.666 = 0.985 kg CO_{2eq}.

Table 5 Assumed composition of cassava stump raw material.

Substance	Mass basis (%)
Moisture	1.8
Fixed carbon	14.0
VOC	75.8
Density	238.0
Heat energy	14,734.8
Element	
Carbon	46.12
Hydrogen	7.55
Oxygen	44.83
Sulfur	1.13

4. Conclusion

This study investigated the characteristics and parameters of AC produced from cassava stumps prepared using physical activation in a specially designed gasification furnace with a total operating time of 30 min. The physical and chemical properties of AC were tested. The efficiency of absorption was measured using the BET method, the pore structure of the AC was studied using SEM, and FT-IR was used to analyze the chemical compounds on the surface of the AC. The AC from cassava stumps had multilayer adsorption indicating the structure consisted mainly of macropores. The FT-IR results indicated an aromatic C-C stretch (in-ring) function group on the AC surface. The quality of the AC was consistent with other reports in terms of the characteristics of cassava stumps. Although the AC made from cassava stumps in this research had slightly lower adsorption quality than the AC prepared using other furnace designs, the current process had the shortest production time. Overall, the results indicated that the AC made from cassava stumps was suitable for applications such as water treatment and bleaching. This study was the first to use the air-activated preparation method with cassava stumps. This innovative development should reduce the amount of bio-waste and pollution from combustion and increase the value of the cassava waste. Furthermore, this project contributes to sustainable development goals: 1) no poverty; 3) good health and well-being; 7) affordable and clean energy; 12) responsible consumption and production; 13) climate action; and 17) partnership for goals (Development, 2015).

The AC produced in this study is a commercial product suitable for use in various industries. However, this study was undertaken in a laboratory and the raw materials were made into AC and the characteristics of plant species are likely to influence the adsorption properties of the AC. In addition, the temperature used to activate the carbon may be too high for the short production time, resulting in the AC having relatively less surface area. It may be necessary to use steam and air to increase the surface area of the AC, while still using the same time for the production process.

Using combustion to convert cassava stumps into AC by this process will reduce greenhouse gas emissions. Normally, the emission factor of charcoal is 1.0054 kg CO_{2eq} (Ecoinvent, 2.2 IPCC 2007 GWP 100A Update 24Sep 2012). The emission factor of the cassava stump furnace was 0.0985 t CO_{2eq}, meaning that 1 t of cassava stumps when burned in the developed cassava furnace emitted only 0.0985 t CO₂ greenhouse gas. Furthermore, the emission factor of the AC products was 0.985 kg CO_{2eq}, is lower than the charcoal value, which means that using this AC as a raw material would reduce the emission factor in an organization's production process.

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