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# Characterization of cassava peelings as a precursor for biochar preparation

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**Abstract:** The growing world population is leading to an intensification of agricultural practices. The consequence of this is the production of large quantities of agricultural waste. However, this lignocellulosic waste can be valorized into useful substances in order to reduce the use of chemical fertilizers in agriculture. The aim of this study is to characterize cassava peelings with a view to their possible use in agriculture. The physical, chemical and thermal properties of cassava peelings were examined by SEM, XRD, XRF, CHNSO analysis, FTIR and TGA/DTG. These cassava peelings have low ash content (2.8%); low moisture content (5.5%); high volatile matter content (76.2%) and high fixed carbon content (15.5%). Acceptable levels of carbon (45.19%), nitrogen (1.19%), hydrogen (6.18%), sulfur (0.05%), chlorine (0.045%), oxygen (43.73%) and low ratios of O/C (0.96) and H/C (0.13) were found. SEM revealed a fibrous and porous morphology. Potential toxic elements (As, Cd, Co, Cr, Cu, Ni, Pb, Zn, Ti, Mn, V) determined by XRD in this biomass were almost trace. Peaks of crystalline and amorphous cellulose and oxides were observed. Hydroxyl, carbonyl and primary amine groups were also identified in this biomass. These results show that cassava peels can be used as potential raw materials for biochar production for agricultural purposes.

## 1. Introduction

Intensive agriculture generates 11.4 billion tons of biomass per year worldwide (Meza-Sepúlveda *et al.*, 2021). The consequence of this practice is the production of large quantities of agricultural residues. Agricultural biomass is made up of resources containing non-fossil organic carbon from living plants, animals, algae, micro-organisms or organic waste streams (Zörb *et al.*, 2018). It is the result of the photosynthesis process that ensures the constitution and maintenance of its structural elements such as cellulose, hemicellulose or lignin, which form the rigid skeleton of plant biomass. According to Roy and Dias (Roy and Dias, 2017), these polymers contain a large amount of carbon.

The latter comes from $CO_2$ taken from the atmosphere during photosynthesis, giving them a high energy potential. From a regulatory point of view, European Program Law n°2005-781 on Energy Policy Guidelines; defines biomass as the biodegradable fraction of products, waste and residues from agriculture, including plant and animal substances from land and sea, forestry and related industries, as well as the biodegradable fraction of industrial and household waste (Kumar *et al*., 2015).

In general, the quantity of agricultural residues exceeds food production (in terms of edible crop parts). More than half of the absolute dry matter of the global harvest consists of agricultural residues such as cereal and legume straws, stems, leaves and shoots from tubers, oilseeds, sugar plants and vegetables, as well as litter from fruit and nut trees (Smil, 1999; Zörb *et al*., 2018; Bouknana *et al*., 2014). These agricultural residues have similar characteristics. Their basic compositions are generally described as lignocellulosic (Inyinbor *et al*., 2017). Nevertheless, the chemical compositions of these residues can vary depending on their sources.

Huge quantities of agricultural waste generated by plant production represent a danger to the environment and human health if not properly managed (Tonini *et al*., 2018). Practices to reduce or eliminate agricultural residues include on-farm burning, which has become very common, particularly in developing countries (Andini *et al*., 2018). Burning waste leads to a loss of soil moisture, which reduces the moisture available for crop seeding and negatively affects germination. Burning also affects soil fauna, particularly the population of earthworms and beneficial insects (Devi *et al*., 2017). This practice, which generates greenhouse gases ($CO_2$, CO, $CH_4$, $N_2O$, $SO_2$), aerosols, particulates, smoke, volatile organic compounds and radioactive gases, exacerbates atmospheric chemistry on a global and regional scale (Devi *et al*., 2017). In addition, smoke emissions can lead to health risks such as lung infections, chronic bronchitis and asthma (Kumar *et al*., 2018). Another common practice of farmers is to leave their agricultural waste in the fields. Notwithstanding, this practice seems harmless, accumulated waste can contain infested plant material leading to the spread of pests and diseases that promote crop loss (Devi *et al*., 2017). Indeed, various factors, such as the lack of highly skilled agricultural labor, the absence of waste management policies in some regions and the poor implementation of waste management policies in other places, further intensify the environmental burden of unmanaged agricultural waste (Ramya *et al*., 2017).

At present, there are no quantitative, global methods for accurately estimating the potential of biomass worldwide as a raw material or energy source (Dutuit and Gorenflot, 2008). However, many researchers are looking to develop innovative bioproducts that can contribute to environmental and energy issues, as well as to future demand for food, soil and water (Veiga *et al*., 2017). With this in mind, new technologies have been developed aimed at transforming different biomasses, into higher value-added products. These can be used to retain nutrients in the soil (Glaser *et al*., 2002). Another strategy is to optimize the thermal treatment process in the procedures by which biomass is transformed into biochar, such as in boilers and industrial furnaces, reducing gas emissions generating specific biochar structures with desirable properties for use in poor soils (Veiga *et al*., 2017).

Côte d'Ivoire has a strong cassava-based diet (`attiéké, placali, attoukou, gari) (Patricio Mendez del Villar *et al*., 2017). Annual cassava processing generates 1,250,000 tons of wet cassava peelings in Côte d'Ivoire (Patricio Mendez del Villar *et al*., 2017). Cassava peel represents around 8% to 15% of the cassava root, on a dry basis (Howeler, 2001). These cassava peels are little used in animal feed and are very little valued. Mineral contents have been reported in satisfactory proportions, with a carbon content of 48.7% by weight and, a significant percentage of sodium, calcium, potassium and nitrogen (Kongkiattikajorn and Sornvoraweat, 2011). Little is known about the physical, chemical and thermal characteristics of these cassava peelings for agricultural purposes.

In this study, the physical, chemical and thermal characterizations of cassava peelings were examined in order to assess their potential as a candidate for biochar production. The reason for choosing this raw material is due to its abundance, low cost and year-round availability.

## 2. Materials and Methods

### *2.1 Raw material*

Cassava peelings (**Figure 1**) were purchased from `attiéké´ producers in Yamoussoukro, central Côte d'Ivoire.

**Figure 1.** Cassava peelings (Hamissou *et al.*, 2023)

These peels were washed with water to remove cassava residues, then sun-dried for 7 days. The dried cassava peelings were then crushed and sieved to obtain grain sizes of 250 µm or less (**Figure 2**), and stored in plastic bottles for analysis.

**Figure 2.** Cassava peeling powder (CPP)

### 2.2 Characterization of cassava peel powder

### 2.3 *Determination of moisture content, volatile matter and fixed carbon content*

Moisture and ash contents were determined according to Khouloud (2020). Two grams (2 g) of the sample placed in a crucible is oven-dried at 105°C until its mass stabilizes. After cooling in a desiccator, the sample is weighed. The moisture content (MC) is given by **Eqn. 1**:

$$MC(\%) = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \qquad \textbf{Eqn. 1}$$

With :

$m_1$ = empty crucible mass (g) ;

$m_2$= mass of crucible with sample before drying (g) ;

$m_3$= mass of crucible with sample after drying (g).

With regard to ash content, the sample is placed in a muffle furnace heated to 575°C under air for 3 hours until ash is obtained without the presence of black spots (carbon residues). After cooling, the combustion residue is weighed to determine the ash content (AC) of the sample from **Eqn. 2**:

$$AC(\%) = \frac{m_3 - m_1}{m_{2-}m_1} \times 100 \qquad \textbf{Eqn. 2}$$

With :

$m_1$ = empty crucible mass (g);

$m_2$= mass of crucible with sample before thermal oxidation (g);

$m_3$= mass of crucible with sample after thermal oxidation (g).

Volatile matter content was determined using the method described by Abderahim (2019). 1.00 g of PEM is introduced into a porcelain crucible. The sample is then heated in the absence of air at 900°C for 7 min in a muffle furnace. After cooling in a desiccator for 10 min, the sample is weighed again. The percentage of volatile matter (VM) is determined by measuring the mass loss of the sample after subtracting the mass due to moisture. The fixed carbon content (FC) is calculated from the moisture, ash (mineral matter) and volatile matter contents. It is obtained from the following equation (Zohra, 2019):

$$\mathrm{FC}\ (\%) = 100 - (\mathrm{MC} + \mathrm{AC} + \mathrm{VM}) \qquad \textbf{Eqn. 3}$$

### *2.4 Elemental analysis*

The contents of carbon (% C), hydrogen (% H), nitrogen (% N), sulfur (% S) and chlorine (% Cl) were determined according to the procedures described in European standard EN 15104:2011. Analyses were performed in a universal analyzer (Elementar Vario MACRO-Cube). Oxygen content (% O) was quantified by difference according to the formula below:

$$\mathrm{O}\ (\%) = 100 - (\%\ \mathrm{C} + \%\ \mathrm{H} + \ \%\ \mathrm{N}\ + \%\ \mathrm{S} + \ \%\ \mathrm{AC}) \qquad \textbf{Eqn. 4}$$

### *2.5 Determination of trace metals and oxides*

The determination of trace metals and oxides in the sample was carried out by X-ray fluorescence spectrometry (XRF) using a Newton XL3T spectrometer. The analysis consisted in determining the trace metallic elements and oxides contained in the sample.

### *2.6 Determination of crystalline structure*

The crystallinity of the biomass powder was analyzed using a German SIEMENS X-ray diffractometer. The analysis was carried out to determine the nature of the crystalline and amorphous species present on the surface of the materials.

### *2.7 Determination of surface functional groups*

Fourier transform infrared spectroscopy was used to identify the surface functional groups present in the biomass sample. This analysis was carried out using a German micromeritics spectrometer in the infrared region over a range from 650 to 4000 $cm^{-1}$.

### *2.8 Analysis of biomass surface morphology*

The surface morphology of the sample was observed using a Japanese HITACHI-4800 spectrophotometer.

### *2.9 Thermogravimetric analysis (TGA)*

In order to study the thermochemical behavior of the biomass used, (ATG) and (DTA) analyses were carried out. The thermal behavior of the sample was carried out using a Dutch-made Perkin Elmer ATG 4000 analyzer, under a nitrogen atmosphere, with a heat ramp from 10 ºC min-1 to 950 ºC.

## 3. Results and Discussion

### *3.1 Physical characteristics of cassava peelings*

The physical and chemical analyses carried out on the raw material justify its quality for biochar production. Table 1 shows some of the physical characteristics of cassava peelings.

**Table 1.** Basic physical characteristics of cassava peeling powder

| Parameters | Values |
|---|---|
| Moisture content (%) | 5.5 |
| Volatile matter (%) | 76.2 |
| Ash content (%) | 2.8 |

The moisture content in the biomass studied is low (5.5%). This is lower than that found by Flores *et al*., (2017), who found a content of around 7% in agricultural residues. This low moisture content in biomass favors biochar production, as it not only reduces thermal energy but also shortens the time required and facilitates the pyrolysis process (Tripathi *et al*., 2016). In addition, an ash content (2.8%) of less than 10% was determined in the biomass studied. This low ash content indicates that cassava peelings can have an excellent adsorption capacity, as ash is an inactive filler which reduces the performance of an adsorbent by obstructing the pores and crowding the exchange surface (Diop *et al*., 2022). This precursor is therefore acceptable for biochar production. Ash contains metals and inorganic matter such as P, mainly in the form of oxides in biomass residues. It defines the quality of a biomass in combustion, determining its non-combustible content (Nieto-Delgado *et al*., 2011). Thus, a high ash content could negatively affect the yield of biochar production (Pereira *et al*., 2014; Li *et al*., 2017 Tomczyk *et al*., 2020;). Volatile matter content is another important parameter, as it gives an indication of the reactivity and ease of ignition of an organic material. According to Canales-Flores and Prieto-García, (2016), volatile matter content is the parameter responsible for pores in the structures of carbonaceous materials. Indeed, during biomass pyrolysis at high temperatures, volatile matter is released, leading to the formation of porous structures (Canales-Flores and Prieto-García, 2016). In this study, a high volatile matter content (76.2%) was found. This indicates that the agricultural biomass in the study is a good candidate for pyrolysis, since it will gradually release volatile matter in a

controlled manner. This release will lead to carbon enrichment in the precursor. Moreover, it falls within the range reported for other similar precursors used in biochar preparation (69-84%) (Canales-Flores and Prieto-García, 2016).

### *3.2 Chemical characteristics of cassava peelings*

The carbon, hydrogen, oxygen, nitrogen, sulfur and chlorine contents of cassava peelings are shown in **Table 2.**

**Table 2.** Elemental and summary characteristics of cassava peelings

| Parameters | Values |
|---|---|
| Carbon (%) | 45.19 |
| Hydrogen (%) | 6.18 |
| Nitrogen (%) | 1.92 |
| Sulfur (%) | 0.05 |
| Chlorine (%) | 0.045 |
| Oxygen (%) | 43.73 |
| Fixed carbon (%) | 15.5 |
| H/C | 0.13 |
| O/C | 0.96 |

According to Tripathi *et al.*, (2016), carbon, oxygen and hydrogen contents are not only used to predict the calorific value of biomass but are also the main contributors to the energy content of biomass. As table 2 shows, the carbon content (45.19%) is the highest in the biomass studied. This result shows that this biomass could be a good precursor for biochar or activated carbon. Indeed, according to Nieto-Delgado *et al.*, (2011), a good biochar or activated carbon precursor should have a carbon content between 40 and 90%. The oxygen content of this biomass is 43.73% and the hydrogen content is 6.18%. The relatively high carbon and oxygen contents confirm that we are dealing with a woody, agricultural biomass (Flores *et al.*, 2017). Thus, the presence of these elements leads to greater char formation as well as a high calorific value of the materials (Tripathi *et al.*, 2016). In contrast, nitrogen, sulfur and chlorine are present in low quantities, as shown in table 2. This result is favorable, as high nitrogen and sulfur contents in biomass would lead during pyrolysis to the release of gas mixtures (SOx, NOx) that are toxic and pollute the environment (Flores *et al.*, 2017; Tripathi *et al.*, 2016). These results also prove that the feedstock examined in this study is a good precursor for biochar production. Fixed carbon provides important information on biomass quality, as it is the most resistant part remaining in the biochar after pyrolysis (Veiga *et al.*, 2017). It also indicates the amount of non-volatile organic matter present in the biomass and the high calorific value thus directly reflecting the biochar's permanence in the soil. The fixed carbon found in the study biomass is 15.5%. This value is higher than that found by Emiola-Sadiq *et al.*, (2021). The H/C and O/C ratios found in the biomass studied are 0.13 and 0.96 respectively. These are high and similar to those found by Veiga *et al.*,( 2017). Zheng *et al.*, (2013), report that high ratios (H/C and O/C) indicate the presence of aromatic structures in biomasses. Also, when biomass undergoes thermal treatment, it tends to be more carbonaceous due to thermal degradation of hemicellulose, some of the oxygen and aromatic components are lost (Angın, 2013; Lee *et al.*, 2013; Veiga *et al.*, 2017; Zheng *et al.*, 2013). Thus, H/C and O/C ratios tend to decrease with increasing temperature during pyrolysis, giving rise to a material that is more resistant to degradation.

### *3.3 Trace metals and oxides*

**Table 3** shows the results for trace metals and oxides in the raw material. The potential toxic elements (As, Cd, Co, Cr, Cu, Ni, Pb, Zn, Ti, Mn, V) determined in the biomass are virtually trace, as shown in table 3. Silicon ($SiO_2$), potassium ($K_2O$) and calcium (CaO) oxides are the most abundant in the biomass studied (cassava peelings). The existence of these metal oxides could be explained by the use of chemical fertilizers (absorbed by the plants) in cassava fields by farmers on the one hand, and by the physical and chemical properties of the soils on the other.

**Table 3.** Percentages of trace metals and oxides

| Trace metals | Values (ppm) | Metal oxides | Values (%) |
|---|---|---|---|
| **As** | 0.002 | **MgO** | 2.091 |
| **Mo** | 0.002 | **$Al_2O_3$** | 4.841 |
| **Pb** | 0.002 | **$SiO_2$** | 23.059 |
| **Zn** | 0.002 | **$P_2O_5$** | 3.734 |
| **Cu** | 0.002 | **$SO_3$** | 3.329 |
| **Ni** | 0.002 | **$K_2O$** | 28.1 |
| **Co** | 0.002 | **CaO** | 23.76 |
| **Mn** | 0.005 | **$TiO_2$** | 1.424 |
| **Cr** | 0.003 | **MnO** | 0.214 |
| **Ti** | 0.016 | **$Fe_2O_3$** | 3.568 |
| **V** | 0.002 | **ZnO** | 0.0782 |

### *3.4 Analysis of the cassava peeling powder diffractogram*

The diffractogram of cassava peel powder is shown in **Figure 3.** Crystalline and amorphous cellulose peaks were observed near 2θ = 18° and 2θ = 22°. These similar crystalline and amorphous cellulose peaks were observed in mango leaf biomass by Akhtar *et al*., (2016).

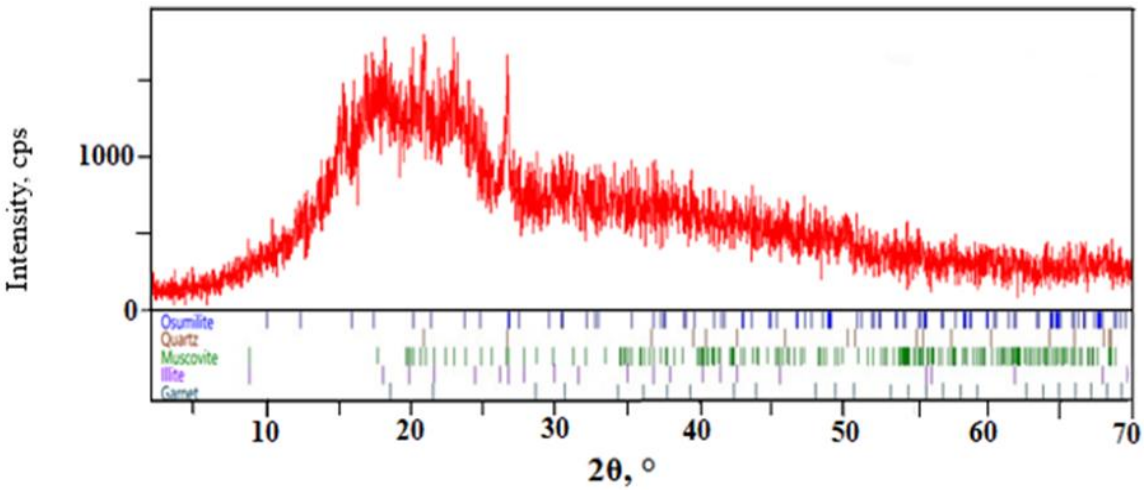


**Figure 3.** Diffractogram of cassava peelings powder.

Peaks located at 2θ = 28° and 2θ = 68° in the biomass correspond to quartz ($SiO_2$). Peaks present at 2θ = 38°, 2θ = 48° and 2θ = 58° could be attributed to the existence of metallic impurities (K, Ca, Mg

etc.) and inorganic components such as potassium oxide($K_2O$), calcium oxide (CaO) and many others (**Table 3**).

### *3.5 Fourier transform infrared (FTIR) spectrum of CPP*

The spectrum acquired by Fourier transform infrared spectroscopy **(Figure 4)** provided information on the chemical groups present in the biomass structure.

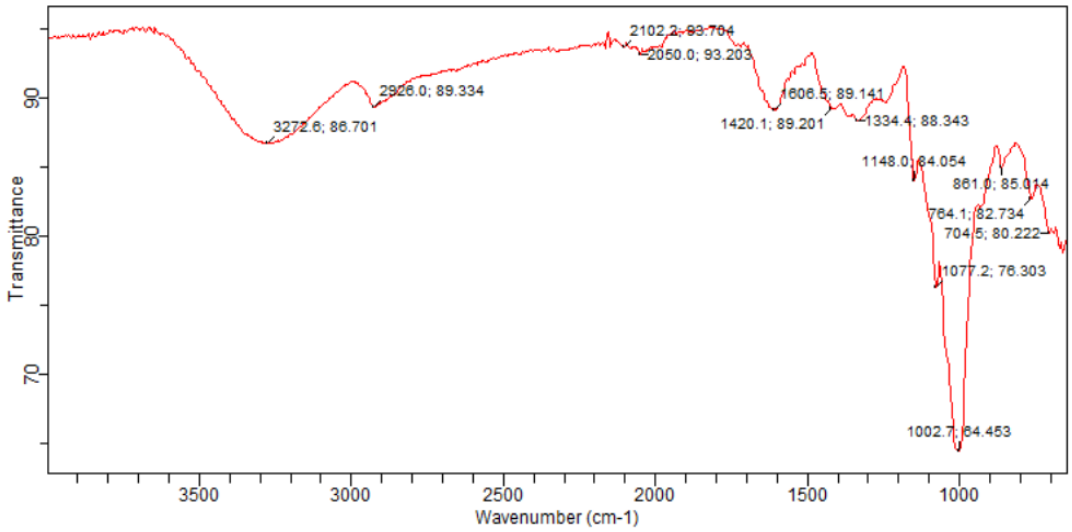


**Figure 4.** Infrared spectrum of cassava peel powder

The band observed in the 3360 -3000 $cm^{-1}$ range represents the hydrogen-related stretching band of OH groups, originating from the glycosidic bonds of cellulose or the hydroxyphenyl, guaiacyl and syringyl groups of lignin (Akhtar *et al*., 2016; Mothé and Miranda, 2009). The peak at 2926 $cm^{-1}$ can be attributed to asymmetric and symmetric C-H stretching in the methyl and methylene groups. This peak also corresponds to the aliphatic parts of cellulose and hemicellulose (Akhtar *et al*., 2016). The band observed at 2102 $cm^{-1}$ can be attributed to Si-H stretching (Flores *et al*., 2017). In addition, the bands in this region are representative of an inorganic hydride or oxide (Bledzki *et al*., 2010). The peak observed in the wavelength range 1650-1580 $cm^{-1}$ corresponds to N-H bending in the primary amine. This result corroborates with that found by Flores *et al*., (2017). The peak at 1420.1 $cm^{-1}$ is attributed to bending in the CO plane (Adeniyi *et al*., 2022; Odeyemi *et al*., 2023). Peaks at 1148 $cm^{-1}$ and 1077.2 $cm^{-1}$ are attributed to stretching in the CO plane (Anas *et al*., 2022, 2021; Odeyemi *et al*., 2023). These oxygenated groups indicate the presence of the lignin faction of cassava peel (Odeyemi *et al*., 2023; Silva *et al*., 2022). Bands at 861$cm^{-1}$; 764.1 $cm^{-1}$, and 704.5 $cm^{-1}$ correspond to the presence of hydrogen alkene in out-of-plane bending mode (Adeniyi *et al*., 2022; Emenike *et al*., 2022; Odeyemi *et al*., 2023).

### *3.6 Morphology of raw material (cassava peelings)*

**Figure 5** shows the SEM image of cassava peelings. Fibrous and porous structures can be observed, which are suitable characteristics for obtaining carbonaceous materials such as biochars and activated carbons (Canales-Flores and Prieto-García, 2016).

These structures make it easy to decompose or transform biomass during pyrolysis. According to the literature, a good precursor for biochar production should have a porous and fibrous structure, as under these conditions, oxygen can easily diffuse inside the particle during combustion. In addition, volatile matter can be gradually released (Gani and Naruse, 2007). Based on the results of this observation, the raw material could be a good potential precursor for biochar production.

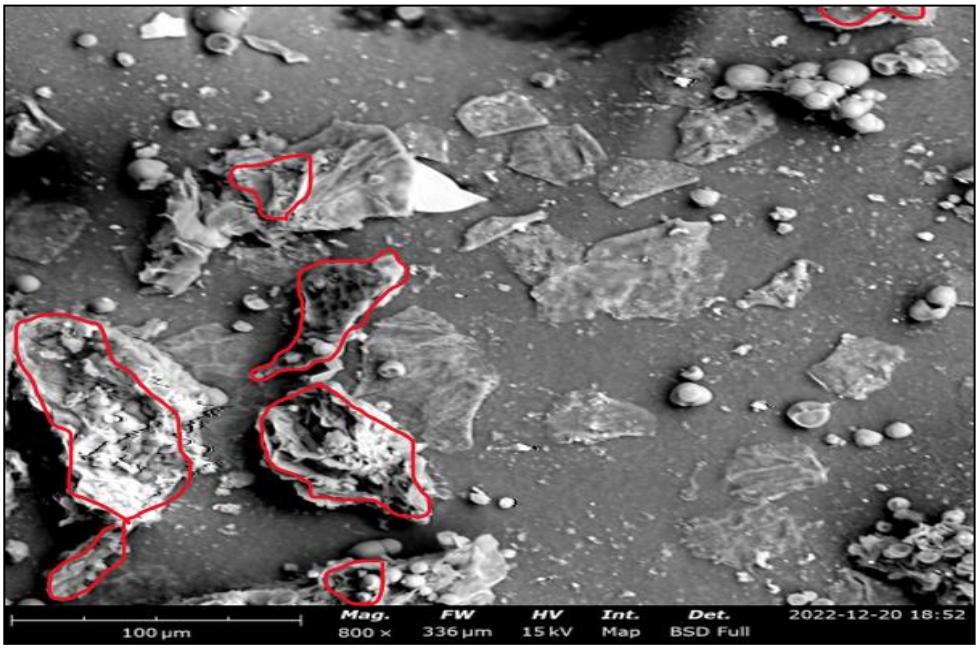


**Figure 5.** Cassava peel powder viewed with a scanning electron microscope (SEM)

### *3.7 Thermograms of CPP*

**Figure 6** shows the results of the thermal analysis carried out on cassava peelings. These results provide valuable information concerning the thermal decomposition (pyrolysis) of the biomass studied in this work.

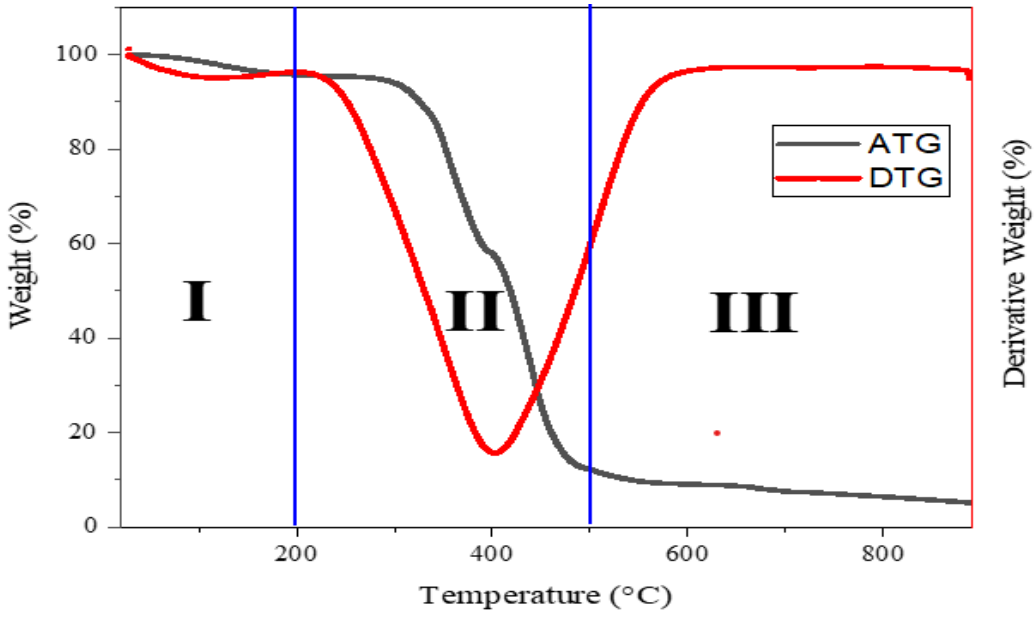


**Figure 6.** ATG/DTG curve for cassava peelings

**Figure 6** shows three phases of mass loss in the biomass studied. The first phase (between 30°C and 200°C) is generally attributed to the loss of water and evaporation of certain volatile compounds in the biomass (Emiola-Sadiq *et al*., 2021; Flores *et al*., 2017). The second phase (200°C and 500°C) is produced when the biomass is completely devoid of moisture. It could be attributed to the degradation of hemicellulose as well as a small amount of cellulose (Veiga *et al*., 2017). This is the main pyrolysis region where most of the sample mass is lost (Emiola-Sadiq *et al.*, 2021). These results are in agreement with those found by some authors who indicate that hemicelluloses, the least thermally stable compounds, decompose at rather low temperatures (200°C -250°C) (Flores *et al*., 2017; Ousmaila *et al*., 2018; Raveendran and Ganesh, 1998). According to the same authors, cellulose decomposes in a narrowed temperature range of 300°C- 400°C and 325°C- 375°C. The third phase (III) observed could be attributed to lignin decomposition and further degradation of coal residue to ash (Emiola-Sadiq *et al*., 2021). These results are in line with those reported by Gani and Naruse, (2007) who indicate that the thermal behavior of biomass depends on its own components such as cellulose and lignin content.

## Conclusion

Cassava peelings were characterized to determine their chemical, physical and thermal compositions in order to assess their potential as feedstock for biochar production. On the basis of the results obtained, the biomass samples show suitable chemical, physical and thermal characteristics to be considered as a good biochar precursor. Carbon content above 40%, ash content below 5%, moisture content below 10%, volatile matter content above 75%, fixed carbon content (15.5%), porous, fibrous morphology and chemical functional groups were found. Potential toxic elements determined in the biomass were almost trace, and peaks of crystalline and amorphous celluloses and metal oxides were observed. Thermal decomposition of the biomass took place in three stages attributed respectively to water loss and evaporation of certain volatile compounds (30°C-200ºC); degradation of hemicellulose and a small amount of cellulose (200°C -500ºC); decomposition of lignin (500°C -800ºC) and further degradation of charcoal residues to ash. These results show that the selected biomass has acceptable characteristics and can be considered a good potential precursor for biochar production for agricultural and environmental purposes. This research proposes viable, environmentally friendly solutions for disposing of agricultural waste and transforming it into value-added products such as biochar.

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