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Effect of Carbonization Temperatures on the Proximate Composition of Powdered Pentaclethra Macrophylla Pod

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ABSTRACT: Pentaclethra macrophylla (African oil bean) pod is an abundant lingo-cellulosic agro-residue in West Africa with growing interest as a precursor for bio-char, adsorbents and friction materials. However, the influence of carbonization temperature on its proximate composition, which governs fuel quality and materials performance, remains poorly documented. This study investigates the effect of carbonization temperature on the moisture, volatile matter, ash and fixed carbon contents of powdered P. macrophylla pod. Dried pods were cleaned, oven-dried at 110 °C, pulverized and sieved, then divided into un-carbonized and carbonized fractions. Carbonization was performed in an inert atmosphere at 600 and 950 °C using an earthen pot in an electric furnace with 3 h soaking time followed by controlled cooling. Proximate analysis was conducted according to ASTM E871, E872 and E1755 to determine moisture, volatile matter and ash contents, while fixed carbon was obtained by difference. Increasing carbonization temperature markedly reduced moisture and volatile matter, while enhancing fixed carbon and ash contents relative to the raw pods. The higher temperature (950 °C) produced chars with significantly higher fixed carbon and lower volatiles than 600 °C, indicating improved suitability for high-temperature applications such as friction materials, refractories and solid bio-fuels. The resulting temperature–composition relationships provide a basis for selecting carbonization conditions tailored to specific energy, environmental and composite applications of P. macrophylla pod–derived carbon materials.

KEYWORD: Pentaclethra macrophylla pod, Carbonization, Temperature, Fixed carbon

1. INTRODUCTION

The conversion of agricultural residues into bio-char or carbonized products is increasingly promoted as a sustainable strategy for waste management, carbon sequestration, energy generation, and value-added materials production (Iloabachie, 2022; Iloabachie et al., 2023; Michalak et al., 2019; V.C et al., 2014; Iosr et al., 2015). Carbonization (pyrolysis in limited or no oxygen) alters moisture, volatile matter, fixed carbon, and ash contents—the core proximate composition parameters that control fuel quality, sorption capacity, and suitability for applications such as adsorbents, friction materials, and refractories (Sahoo et al., 2021; Aller et al., 2017; Zhao et al., 2017; Iloabachie et al., 2023; V.C et al., 2014; Areti et al., 2024; Muigai et al., 2020).

Many studies have shown that increasing pyrolysis temperature generally decreases yield, moisture, and volatile matter, while increasing fixed carbon, ash, aromaticity, and thermal stability in a wide range of biomass-derived biochars (Aller et al., 2017; Zhao et al., 2017; Iloabachie, 2022; Iloabachie et al., 2023; Michalak et al., 2019; V.C et al., 2014; Areti et al., 2024; Iosr et al., 2015; Muigai et al., 2020).

Pentaclethra macrophylla (African oil bean) pod is an abundant agro-waste in West Africa. Its pod biomass contains significant levels of alumina and silica, and heat treatment at high carbonization temperatures increases fixed carbon while reducing moisture and volatiles, suggesting potential as refractory material and reinforcement in composite and friction applications (Iloabachie et al., 2022; Almutairi et al., 2022; Klasson, 2016). The African oil bean (Pentaclethra macrophylla) pod has gained attention as a low-cost precursor for bio-based materials, including brake pads, adsorbents, bio-char and other carbonaceous products (Iloabachie, 2022; Ogbeh et al., 2024; Nsude et al., 2022; García et al., 2013; Aller et al., 2017). Its lingo-cellulosic nature and relatively high fixed carbon and low ash contents make it attractive for thermo-chemical conversion and biomass valorization (Iloabachie, 2022; Ogbeh et al., 2024; Aller et al., 2017; Özbay et al., 2021). Proximate analysis (moisture, volatile matter, ash, fixed carbon) is a key tool for assessing such biomass and predicting its suitability for applications in energy, adsorbents and composites (Odetoye & Ocheni, 2022; Ahmed et al., 2021; Racero-Galaraga et al., 2024; Pérez et al., 2024; Iosr et al., 2015). For P. macrophylla, recent work has characterized the chemical composition and proximate properties of raw pods and isolated cellulose, highlighting substantial cellulose and lignin contents and favorable fixed carbon and low moisture/ash for bio-fuel and adsorbent applications (Iloabachie, 2022; Aller et al., 2017).

Heat treatment/carbonization at a single high temperature (~900–950 °C) has been shown to increase fixed carbon and reduce moisture and volatile matter in powdered pods, suggesting potential as refractory material and composite reinforcement (Iloabachie, et al., 2022). Activated carbons and adsorbents derived from P. macrophylla pods have also been optimized for iodine adsorption and pollutant removal, confirming the suitability of this feedstock for high-surface area carbon materials (Venkatesh et al., 2022).

Parallel studies have optimized carbonization and activation conditions for P. macrophylla pod–derived activated carbons, demonstrating strong effects of temperature and activation parameters on iodine number, yield, surface area and aromaticity (García et al., 2013). More broadly, biochar and biomass studies consistently report that increasing carbonization/pyrolysis temperature raises fixed carbon, heating value and aromaticity, while lowering volatile matter, hydrogen and oxygen contents, with strong consequences for fuel quality, stability and adsorptive performance (Ronsse et al., 2013; Chukwuneke et al., 2019; Iosr et al., 2015; Okoro et al., 2024). Thermo-gravimetric approaches and modified proximate protocols have also been proposed to better distinguish volatile and fixed carbon fractions in biomass and bio-chars (Odetoye & Ocheni, 2022; Ahmed et al., 2021; Pérez et al., 2024).

Despite this promise, systematic information on how varying carbonization temperature affects the proximate composition of P. macrophylla pod is sparse. Existing work typically adopts a single high carbonization temperature (≈900–950 °C) to compare carbonized versus un-carbonized pods (Iloabachie et al., 2022; Almutairi et al., 2022; Klasson, 2016), without mapping trends in moisture, volatile matter, ash, and fixed carbon across a controlled temperature series.

In contrast, detailed temperature–composition relationships are well documented for other residues such as crop stalks, wood, macroalgae, and mixed agricultural wastes (Aller et al., 2017; Zhao et al., 2017; Iloabachie, et al., 2022; Iloabachie et al., 2023; Michalak et al., 2019; V.C et al., 2014; Areti et al., 2024; Iosr et al., 2015; Abdullah et al., 2023; Muigai et al., 2020). There is no systematic study on how varying carbonization temperature (rather than a single temperature) affects the proximate composition of Pentaclethra macrophylla pod specifically. Existing work on this biomass either considers only raw vs. one carbonized condition (Iloabachie, et al., 2022), focuses on chemical/structural characterization or cellulose isolation (Aller et al., 2017), or targets activated carbons where temperature is varied but proximate trends are reported mainly in relation to adsorption indices rather than as a dedicated temperature–composition map for the raw pod feedstock (García et al., 2013).

Extensive temperature–proximate relationships established for other biomasses and bio-chars notwithstanding, (Ronsse et al., 2013; Chukwuneke et al., 2019; Iosr et al., 2015; Okoro et al., 2024), the temperature-dependent evolution of moisture, volatile matter, ash and fixed carbon in P. macrophylla pod over a controlled carbonization temperature range remains undocumented. This lack of temperature-resolved proximate data for P. macrophylla pod limits rational selection of carbonization conditions tailored to specific uses (e.g., high fixed carbon and low volatiles for fuel or friction materials versus tailored ash and porosity for adsorbents).

Carbonization is often used to enhance the structural properties of bio-fiber material. The carbonization process involves converting the biomass to char (charcoal). The charring process (making of charcoal) is known as pyrolysis, which is chemical decomposition of the shell by heating in the absence of oxygen Iloabachie et al., (2018). During the carbonization of coconut shells, volatiles amounting to 70% of the mass of coconut shells on dry weight basis were released to the atmosphere, yielding 30% of coconut shell mass of charcoal Iloabachie et al., (2018). The volatile released during the carbonization process consists mainly of methane, CO_2 and wide range of organic vapors Iloabachie et al., (2018).

The present study addresses this gap by evaluating the effect of different carbonization temperatures on the proximate composition of Pentaclethra macrophylla pod, providing temperature–property relationships needed to optimize its utilization in energy, materials, and environmental applications. The temperatures used in this work are 600°C and 950°C

2. MATERIAL AND METHODS

2.1 Material

The materials used in this research work are dry Pentaclethra macrophylla pod and distilled water.

2.2 Methods

2.2.1 Processing of Dry Pentaclethra Macrophylla Pods

The dry Pentaclethra Macrophylla pods were manually cleaned by removing dirt particles on the surface, washed with distilled water and sun dried for about six hours. This was followed by ovum drying at a temperature of about 110°C for three hours to achieve a constant weight and allowed to cool Iloabachie et al., (2025). The dried Pentaclethra Macrophylla pods were pulverized to powdered form using a locally fabricated crushing and milling machines and later sieved. It was divided into two portions and one portion carbonized and the other un-carbonized.

2.2.2 Carbonization of Dry Pentaclethra Macrophylla Pods

The carbonization temperatures selected were 600°C and 950°C. The powdered Pentaclethra Macrophylla pods were separately packed into an earthen pot and covered with a lid. This was placed in a heat treatment furnace model KGVB kohaszat gyarepito vallalat, heated differently to 600°C and 950°C and held for three hours at the carbonization temperatures under inert atmosphere at Scientific Equipment Development Institute, SEDI, Akwuke, Enugu. The furnace was turned off and 24hrs cooling was allowed so that the furnace comes to room temperature. The carbonized powdered Pentaclethra Macrophylla pods in the earthen pot were taken out of the furnace using a pair of laboratory tongues Iloabachie et al., (2026) for further cooling.

2.2.3 Chemical Characterization of Powdered Pentaclethra Macrophylla Pods by Proximate Analysis

Proximate analysis which provided information on moisture, ash, volatile matter and fixed carbon contents on dry or weight basis Iloabachie et al., (2018) was used to determine the chemical composition of the powdered Pentaclethra Macrophylla. It is one of the characterization methods used to analyze bio-fiber material like powdered Pentaclethra Macrophylla. The proximate analysis was carried out in accordance with ASTM standards E-871, E-1755, E- 872 for moisture at 110ºC, ash at 715ºC and volatile matter at 925ºC using a muffler furnace. The fixed carbon content was determined by subtracting the sum of the values of weight percent of moisture, ash and volatile matter from 100%. It was carried out at Scientific Equipment Development Institute, (SEDI) Akwuke, Enugu.

Fixed Carbon (FC) (%) = 100 - (Ash% + Moisture + Volatile Matter).

3. RESULTS AND DISCUSSIONS

Table 3.1 Proximate Analysis Result of Powdered Pentaclethra Macrophylla Pod (wt %) at 600ºC

Constituents	Fixed Carbon	Volatile Matter	Ash	Moisture
Un-carbonized Powdered Pentaclethra Macrophylla Pod	34.75	40.57	7.11	17.57
Carbonized Powdered Pentaclethra Macrophylla Pod at 600ºC	48.72	29.64	9.31	12.33

Table 3.2 Proximate Analysis Result of Powdered Pentaclethra Macrophylla Pod (wt %) at 950ºC

Constituents	Fixed Carbon	Volatile Matter	Ash	Moisture
Un-carbonized Powdered Pentaclethra Macrophylla Pod	34.75	40.57	7.11	17.57
Carbonized Powdered Pentaclethra Macrophylla Pod at 950ºC	88.42	6.69	2.21	2.68

3.1 Proximate Analysis Result

The proximate compositions of the powdered un-carbonized and carbonized Pentaclethra macrophylla pod are presented in Tables 3.1 and 3.2, respectively. The un-carbonized powder contains volatile matter (40.57%) and fixed carbon (34.75%), with a moisture content of 17.57% and ash content of 7.11%. Ash content is the residue that remains when the carbonaceous material is burned off and indicates the presence of carbon compounds and inorganic components in the form of salts and oxides in the material (Iloabachie et al., 2018), in agreement with the findings of Iloabachie et al. (2022).

Carbonization of the powdered P. macrophylla pod at 600 °C and 950 °C significantly altered its proximate composition by increasing the fixed carbon content and markedly reducing the volatile matter and moisture contents. At 950 °C, the fixed carbon content was 88.42%, and at 600 °C it was 48.72%, compared with 34.75% for the un-carbonized sample. Although fixed carbon increased at 600 °C relative to the raw material, the conversion was less complete than at 950 °C. The increase in fixed carbon content was approximately 154.45% at 950 °C and 40.20% at 600 °C. In general, fixed carbon increases substantially with rising temperature; thus, carbonization temperature enhances the fixed carbon content of powdered P. macrophylla pod, consistent with the observations of Iloabachie et al. (2018).

The volatile matter content of the carbonized powdered P. macrophylla pod at 950 °C was lower than that at 600 °C and in the un-carbonized sample. Volatile matter in a carbonaceous material contains most of the cellulose, hemicellulose, lignin, and other organic constituents (Iloabachie et al., 2018). In thermal analysis, Yang et al. (2006) reported that pyrolysis of hemicellulose usually occurs at 220–315 °C, while cellulose decomposes between 315–400 °C. Iloabachie et al. (2018) further argued that lignin is more difficult to decompose, exhibiting weight loss over a broad temperature range (160–900 °C). These findings may explain the lower volatile matter content of the carbonized powdered P. macrophylla pod at 950 °C.

Increasing the carbonization temperature also led to a reduction in moisture content. The moisture content of the pod powder decreased from 17.57% in the un-carbonized sample to 12.33% at 600 °C and 2.68% at 950 °C. Lignin and cellulose contain most of the polar hydroxyl (OH) groups in coconut shell (Iloabachie et al., 2017) and decompose over a temperature range of approximately 160–900 °C. Consequently, increasing carbonization temperature lowers the water absorption capacity of powdered P. macrophylla pod. Hence, the higher the carbonization temperature, the lower the resulting moisture content.

The ash content of the powdered P. macrophylla pod at 950 °C was about 2.21%, substantially lower than the 7.11% measured in the un-carbonized powder. Jabit (2012) reported that the inorganic material contained in activated carbon is measured as ash content, generally ranging between 2% and 10%, which is consistent with the present findings.

Raw Pentaclethra Macrophylla **pod**

Un-carbonized Pentaclethra Macrophylla **pod**

Carbonized Pentaclethra Macrophylla **pod**

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