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Effect of Heat Treatment on the Chemical Composition of *Pentaclethra Macrophylla* Pod

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ABSTRACT: In this work, the effect of heat treatment on the chemical composition of powdered Pentaclethra Macrophylla pod was studied. The Pentaclethra Macrophylla pod was picked from the bush, cleaned manually, soaked and washed with distilled water to further remove the dirt particles. The washed Pentaclethra Macrophylla pod was 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. The dried Pentaclethra Macrophylla pods were crushed to powdered form using a locally fabricated crushing and grinding machines and later sieved. After sieving, the pod was divided into two samples with one carbonized and the other un-carbonized. The carbonized sample was packed in an earthen pot and covered with a lid. This was placed in a heat treatment furnace, heated to 950°C and held for three hours at the carbonization temperature under inert atmosphere. The furnace was turned off and 24hrs cooling was allowed for the furnace to cool to room temperature. The carbonized powdered Pentaclethra Macrophylla pod in the earthen pot was taken out of the furnace using a pair of laboratory tongues for further cooling. The chemical composition of the heat treated Pentaclethra Macrophylla pod was determined using XRF, FTIR and proximate analysis. The results showed that Pentaclethra Macrophylla pod has alumina Al_2O_3 and silica SiO_2 as the dominant metallic oxides. The results also showed that the heat treated Pentaclethra Macrophylla pod had higher composition of fixed carbon and lesser moisture and volatile matter. Therefore, Pentaclethra Macrophylla pod can be used as a refractory material and as reinforcement in composite development.

KEYWORDS: Pentaclethra Macrophylla pod, heat treatment, carbonization, chemical composition.

1.0 INTRODUCTION

Pentaclethra, Hogan, (2012) stated is a genus of flowering plants in the legume family, Fabaceae and belongs to the mimosoid clade of the subfamily Caesalpinioideae. Some genus members of these plants LPWG (2017) observed occur in the Central American region; for example *P. macroloba* is the dominant tree in certain seasonal swamp forests in coastal areas of the Isthmian–Atlantic moist forests.

As was noted by Oboh (2011), *Pentaclethra* comprises three species, with two of these species commonly found in Africa and the other in South America. The two African species are *Pentaclethra Macrophylla* Benth and *Pentaclethra eetveldeana*. *Pentaclethra eetveldeana* differs from *Pentaclethra Macrophylla* Benth by its smaller leaflets and simple hairs. The American specie is known as *Pentaclethra macroloba* and yields timber traded as 'gavilán' and is an important medicinal plant Oboh (2011).

Pentaclethra Macrophylla Benth known as Akpaka, Ukpaka or Ugba (in Igbo) tree is a tropical plant that mainly grows wild but currently being domesticated. Also, it is known by various names by different ethnic nationalities in Nigeria. As was reported by Oly-Alawuba and Anunukem (2018), in Yoruba it is called *Apara apagha*, *Okpaghan* in Urhobo, *Okpagha* in Bini, and *Ugbe* in Esan and Ukana in Efik, Chinasa and Chinwe (2016). Mostly, the tree is known as 'oil bean' tree Adeyemi et al. (2017) and predominantly found in Africa, hence, referred to as African oil bean tree. It is a multipurpose leguminous tree belonging to the family and sub-family of Leguminosae and Mimosoideae Oly-Alawuba and Anunukem (2018) respectively.

Also, *Pentaclethra Macrophylla* Benth is a native plant in the tropical regions of Africa and as was noted by Chinasa and Chinwe (2016), Okwu, (2011) and Okwu and Aluwuo, (2012) the tree is found mostly in the Southern rain forest zone of West Africa growing wild. Corroborating the works of Chinasa and Chinwe, (2016), Okwu, (2011) and Okwu and Aluwuo, (2012), Akachuku and Tombere (2015) in their various opinions also hinted that the plant grows in the forest zone of West and Central Africa, from Senegal to south eastern Sudan and to Angola and also to the Island of principle and Sao Tome.

In Nigeria, subsistent farmers in agrarian communities of Southern and Middle belt regions mostly cultivate the plant Chinasa and Chinwe (2016) and see the plant as possessing good soil enhancing characteristics Oly-Alawuba and Anunukem (2018) stated. Though, literature is dearth in any known established plantation of this tree, the tree thrives better in Southern parts of Nigeria. In South-East Nigeria, the tree grows in most parts of the area. Adeyemi et al. (2017) were of the opinion that domestication of this tree will create large plantations that can be sources of raw materials to support food processing industries like canned food industry. The tree prefers a medium/loamy acid and well drained soil and as was reported by Adeyemi et al. (2017) a forest elevation of about 0-500 m and an annual rainfall of about 1500-2700 mm. The tree can be grown from stake cuttings, seedlings, and direct seed sowing or by budding. Shoot cuttings produce seed after four years. The tree can grow to about a height of 21m and to about 6 m in diameter branching low down and forming a spreading crown. The stem bark is grayish to dark-reddish brown, flaking off in irregular patches. The leaves with stout compound stalk Paul (2013) stated stretches to about 20-45 cm long and covered with rusty hairs. The leaves Chinasa and Chinwe (2016) expressed enhance soil fertility. Farmers protect this specie on farms because its open crown does not severely affect crop growth and because some trees are leafless during the growing season. March to April is usually the major flowering period for this plant, though, there are few species that does so between June and November.

The high content of nectar in the flowers mostly attracts honeybees, Voorhoeve (1965). The flowers Okoye (2016) opined are usually yellow or pinkish white with sweet smell. Fruiting is throughout the year and this may be attributed to large persistent woody pods that can stand harsh climatic changes. The seeds which are about 7 cm long are mostly oval in shape and are enclosed by a black/brown pericarp or shell. According to Madukasi *et al*. (2015) the seeds mostly are picked from the ground in the wild. Although there are instances where the seeds may be picked from homes gardens, farmsteads depending on where the tree is located. The number of seeds in the pod Madukasi *et al*. (2015) contended depends on the length of the pod and the size of the seeds.

The pods which are about 40- 50 cm long and 5-10 cm wide house the seeds. Each pod contains between 6-10 flat flossy brown seeds. The pods are hard, dark-brown and woody in nature, and curls up as it dries. The pods split explosively when fully mature to disperse the seeds by scattering them indiscriminately. The dispersed seeds are harvested by gathering them from within and afar from the tree. Most of the split pods fall to the ground while some may remain attached to the stalk on the tree. The empty dry pods litter the environment indiscriminately thereby constituting environmental nuisance. In most cases, the empty dry pods Chinasa and Chinwe (2016) opined are used as fuel for cooking and for charcoal making and this Oboh and Ekperigin (2014) argued is usually done in uncontrolled manner with attendant significant contributions to ozone layer depletion and gross environmental degradation.

Heat treatment though, a metallurgical process in the processing of materials, has its origin as an ancient art man widely employs is his determination to improve the performance of materials in their practical applications. Presently in metallurgical practice, heat treatment represents a range of techniques involving thermal, mechanical and chemical treatments by which the desired properties of structural materials are obtained to suit a variety of applications, Rajan et al. (1988).

Carbonization, a heat treatment process in this instance, involves converting the agro waste (*Pentaclethra Macrophylla* pod) to char (charcoal). The charring process (making of charcoal) is known as pyrolysis, is a chemical decomposition of the pod by heating in the absence of oxygen. During the carbonization of *Pentaclethra Macrophylla* pod, volatiles amounting to 70% of the mass of *Pentaclethra Macrophylla* pod on dry weight basis were released to the atmosphere, yielding 30% of *Pentaclethra Macrophylla* pod mass of charcoal. The volatile released during the carbonization process consists mainly of methane, CO2 and wide range of organic vapors. The carbonization temperature ranges between 400^oC and 850^oC sometimes reaches 1000^oC, Chanap (2012).

In this study, carbonization temperature of about 900^oC, was used to study the effects temperature has on the composition of *Pentaclethra Macrophylla* pod with the view of achieving better *Pentaclethra Macrophylla* pod management in terms of utilization, storage and disposal. This will ensure a better friendly environment and produce a quality product for industrial application.

2. MATERIAL AND METHODS

2.1 Material

The materials used in the research work were dry ground *Pentaclethra Macrophylla* pods and distilled water.

2.2 Method

2.2.1 Raw Material Preparation and Processing

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^oC for three hours to achieve a constant weight and allowed to cool. The dried *Pentaclethra Macrophylla* pods were crushed to powdered form using a locally fabricated crushing and grinding machines and later sieved. The sieving was carried using a set of sieves arranged in descending order of fineness in accordance with BS1377:1990 standard as was reported by Rajan et al., (2013) at the Civil Engineering Department soil laboratory of Institute of Management and Technology, Enugu.

2.2.2 Surface Modification of Powdered *Pentaclethra Macrophylla* Pods by Carbonization

The carbonization temperature selected was 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 to 950ºC and held for three hours at the carbonization temperature 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 Chanap, (2012) for further cooling.

2.2.3 **Chemical Analysis of Raw Materials**

The ground *Pentaclethra Macrophylla* pods was analyzed to determine its chemical composition. The chemical analysis was carried out at the Department of Geological Sciences, Gwombe State University, Nigeria using X-ray Fluorescence (XRF) analysis technique, Fourier Transform Infra Red FTIR, at the Chemical Engineering Department of Ahmadu Bello University, Zaria, Kaduna State and proximate analysis at Scientific Equipment Development Institute, SEDI. Akwuke, Enugu.

2.2.4 X-ray Fluorescence (XRF) Analysis

Mini Pal compact energy dispersive X-ray Fluorescence (XRF) was used for the oxide analysis of the powdered *Pentaclethra Macrophylla* pod. The system is controlled by a PC running the dedicated Mini Pal analytical software. This test was done at the Department of Geology, Gombe State University, Gombe.

2.2.5 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopic analysis of the un-carbonized and carbonized samples of the powdered *Pentaclethra Macrophylla* pod was carried out in a Perkin Elmer Spectrometer 2000 FTIR. KBr pellet technique was applied Salmah *et al* (2013). Scanned range was 400-4000 cm^{-1} and resolution for all the infrared spectra was 4 cm^{-1}.The system includes a personal computer with compatible software that provides real-time updates of the spectral profile during sample collection and spectral collection. The Data analysis was performed using appropriate reference spectra. This was done at Chemical Engineering Department, Ahmadu Bello University, Zaria, Kaduna State.

2.2.6 Proximate Analysis

Proximate Analysis is among the characterization methods used to analyze bio-filler materials like *Pentaclethra Macrophylla* pod. This process was used to analyze the pod and the waste car tire. The process provides information on moisture, ash, volatile matter and fixed carbon contents on dry weight base. This 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, Ojha *et al*, (2015) 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.

$$Fixed\ Carbon = 100 - (Ash\% + Moisture + Volatile\ Matter \qquad 1$$

3. RESULTS AND DISCUSSIONS

3.1 XRF Result Analysis of Powdered *Pentaclethra Macrophylla* Pod

The XRF test result of the carbonized and un-carbonized *Pentaclethra Macrophylla* Pod as seen in Tables 1a and Tables 1b showed that it contains Al_2O_3, K_2O, CaO, MgO, SiO_2, Fe_2O_3, and P_2O_5 as dominant oxides. The presence of hard metal oxides like silica-SiO_2, alumina-Al_2O_3 and hematite -Fe_2O_3 (Tables 1a & b) explains the hard nature of *Pentaclethra Macrophylla* Pod. This therefore, indicates the suitability of *Pentaclethra Macrophylla* Pod as particulate reinforcement material in a polymer resin, Iloabachie *et al*. (2017). In addition, SiO_2, Al_2O_3 and Fe_2O_3 act as abrasive materials in brake pad by increasing friction between the pad and the disc and also control friction film build-up. The presence of silicon oxide also helps to reduce the drying shrinkage of the composite material and aids inter particle binding when fused. Hematite (Fe_2O_3) acts mainly as a mild abrasive and functions as friction producer / modifier by improving cold friction. Calcium oxide or lime CaO acts as a filler material and helps to prevent corrosion induced by the presence of Fe-additives. Furthermore, CaO eases processing and helps to raise fade temperatures, Blau in Lawal *et al*. (2019). Potassium Oxide K_2O is an inert filler material which also functions as an insulator hence, improving friction stability and wear resistance. It therefore, helps to control the tribological properties of the brake pad.

Phosphorus oxide P_2O_5 was also revealed by the XRF result. Phosphorus and silicon are known to react with alumina (Fe_2O_3) to form aluminum-phosphate/silicate which has appreciable strength above 350^{0}C, Decker (2013). The resulting aluminum-phosphatesilicate bond acts as additional binder in the brake pad composite matrix thereby enhancing its strength. This confirmed the high compressive strength and hardness values obtained in the developed brake pads.

Although, the XRF result of the carbonized and un-carbonized powdered *Pentaclethra Macrophylla* Pod (Tables 1a and 1b) are almost of the same chemical composition, however, the percentage composition of the compounds vary. From Table 1a, it could be seen that while the percentage compositions of SiO_2, Al_2O_3, CaO, K_2O, MgO, and P_2O_5 are 17.9%, 5.96%, 22.5%, 35.7%, 4.79% and 4.09% respectively for the carbonized sample, that of the un-carbonized sample Table 1b are 21%, 7.35%, 19.9%, 20.7%, 3.16%, and 5.04% respectively. Also, it could be observed that 5.09% of Na_2O was detected in the un-carbonized sample but was not detected in the carbonized sample. The observed differences in percentage compositions after carbonization may be attributed to the carbonization process which involves converting the powdered *Pentaclethra Macrophylla* Pod to charcoal. The charring process (making of charcoal) known as Pyrolysis, is the chemical decomposition of the powdered *Pentaclethra Macrophylla* Pod by heating in the absence of oxygen.

Carbonization of powdered *Pentaclethra Macrophylla* Pod results in the release of volatiles amounting to 70% by mass of the powdered *Pentaclethra Macrophylla* Pod on dry weight basis into the atmosphere, yielding 30% of powdered *Pentaclethra Macrophylla* Pod mass of charcoal. The volatile released during the carbonization process consists mainly of methane, CO_2 and wide range of organic vapors.

Iloabachie *et al*. (2018) were of the opinion that the ash content of carbon in an agro-waste is the residue that remains when the carbonaceous materials is burned off which is an indication of the presence of carbon compounds and inorganic compounds in the form of salts and oxides in the agro-waste.

3.2 Proximate Analysis Result

Table 2 revealed that while the fixed carbon content of carbonized powdered *Pentaclethra Macrophylla* Pod is 87.36% that of the un-carbonized powdered *Pentaclethra Macrophylla* Pod is 33.61%. Furthermore, the ash content of the carbonized powdered *Pentaclethra Macrophylla* (2.69%) is much lower than that of the un-carbonized sample (7.11%). Jabit, (2012) had reported that the inorganic material contained in activated carbon is measured as ash content, generally in the range between 2% and 10% and agreed with this work. The volatile matter content of the carbonized sample is about 7.22% while about 41.7% volatile matter was observed in the un-carbonized sample. Chanap (2012) stated that volatile matter contains most of the cellulose, hemi-cellulose, lignin and other organic matter in agro-waste material.

Yang *et al* (2012), argued that the pyrolysis of hemi-cellulose mostly occurs at 220–315 °C, that of cellulose at 315–400 °C, while lignin is more difficult to decompose, as its weight loss happens in a wide temperature range of temperature (from 160 to 900 °C). This possibly explained the lower volatile matter content of the carbonized powdered *Pentaclethra Macrophylla* Pod as indicated in Table 2.

Table 2 also showed that carbonized powdered *Pentaclethra Macrophylla* Pod had the least moisture content of about 2.76% compared to 17.57% of the un-carbonized powdered *Pentaclethra Macrophylla* Pod. Cellulose and lignin Iloabachie *et al*., (2017) hinted contain most of the polar hydroxyl (OH) group in coconut in a bio-fiber material. Yang *et al* (2012) had stated that while cellulose decomposed at about 315–400 °C, lignin decomposed around 160 to 900 °C

This therefore, justified the low moisture content of the carbonized powdered *Pentaclethra Macrophylla* Pod.

3.3 FTIR Result

Figure 1a shows the FTIR spectra of carbonized particles of *Pentaclethra Macrophylla* pod. It is evident from Fig. 1a that band shifting around the broad peak at 3336.0 cm^{-1} (83.904) at a transmittance of about 85 and this indicates the possible involvement of hydroxyl group. The peak at 2926.0 cm^{-1} (87.953) at a transmittance of about 88 may be due to the CH stretching that induces vibrations of CH,- CH, and CH_3 groups. The absorption bands at about 1722.0 (89.536) and 1367.9 cm^{-1} (84.554) indicate the characteristic of C ═ C bonds in aromatic rings. The major changes that can be seen from Fig. 1b are at the increments in the C – O carboxyl bands at 1636.3(72.963) and 1289.7 cm^{-1}(69.597). Activity of carboxyl oxygen atoms seem to account for the alterations in these band areas. Visible deviations are in the regions from 3336.0 cm^{-1} (83.904) at a transmittance of about 85 and 2926.0 cm^{-1} (87.953) at a transmittance of about 88 Fig. 1a to 3142.1 (79.902) at a transmittance of 80 and 2937.1 cm^{-1}(79.320) at a transmittance of 78 Fig. 1b which are assigned to the vibrations of N – H and O – H functional groups. The peaks at 1069.7 (65.115) and 1028.7 cm^{-1} (64.256) Fig. 4.3b, may be due to Si – O stretching and bending, which shows the presence of silica.

Table 1a: Chemical Composition of Carbonized Powdered *Pentaclethra Macrophylla* Pod by XRF

Oxides of Element	Al_2O_3	SiO_2	Fe_2O_3	K_2O	MgO	TiO_2	CaO	MnO	Na_2O	P_2O_5
Percentage (%) Composition	4.55	17.9	5.96	35.7	4.79	0.431	22.5	0.128	ND	4.09

Table 1b: Chemical Composition of Un-carbonized Powdered *Pentaclethra Macrophylla* Pod by XRF

Oxides of Element	Al_2O_3	SiO_2	Fe_2O_3	K_2O	MgO	TiO_2	CaO	MnO	Na_2O	P_2O_5
Percentage (%) Composition	7.35	21.0	12.4	20.7	3.16	0.537	19.9	0.216	5.97	5.04

Table 2: Proximate Analysis Result of Powdered *Pentaclethra Macrophylla* Pod (wt %)

Constituents	Fixed Carbon	Volatile Matter	Ash	Moisture
Un-carbonized Powdered *Pentaclethra Macrophylla* Pod	33.61	41.71	7.11	17.57
Carbonized Powdered *Pentaclethra Macrophylla* Pod	87.36	7.22	2.69	2.76

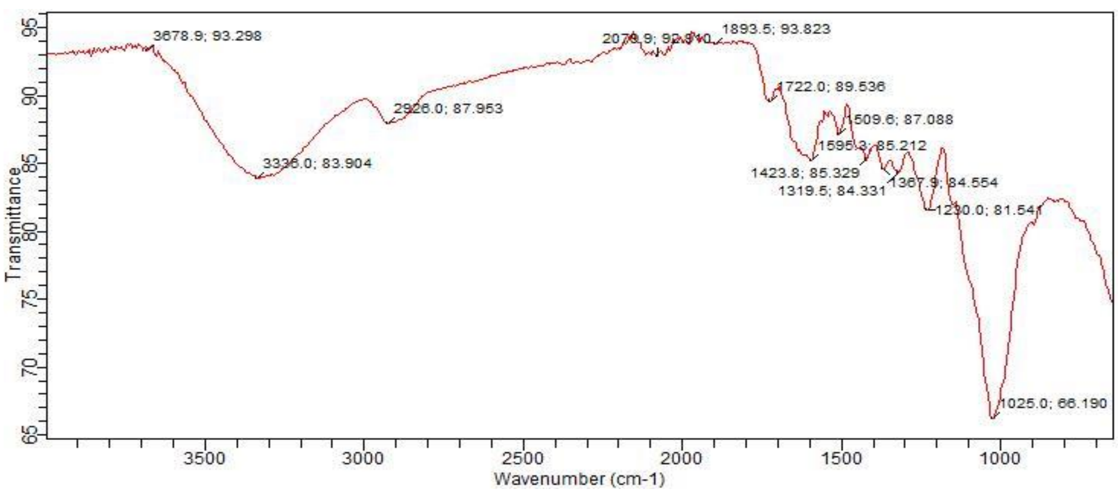

Fig. 1a: FTIR Spectra of Un-carbonized Particles of *Pentaclethra Macrophylla* Pod

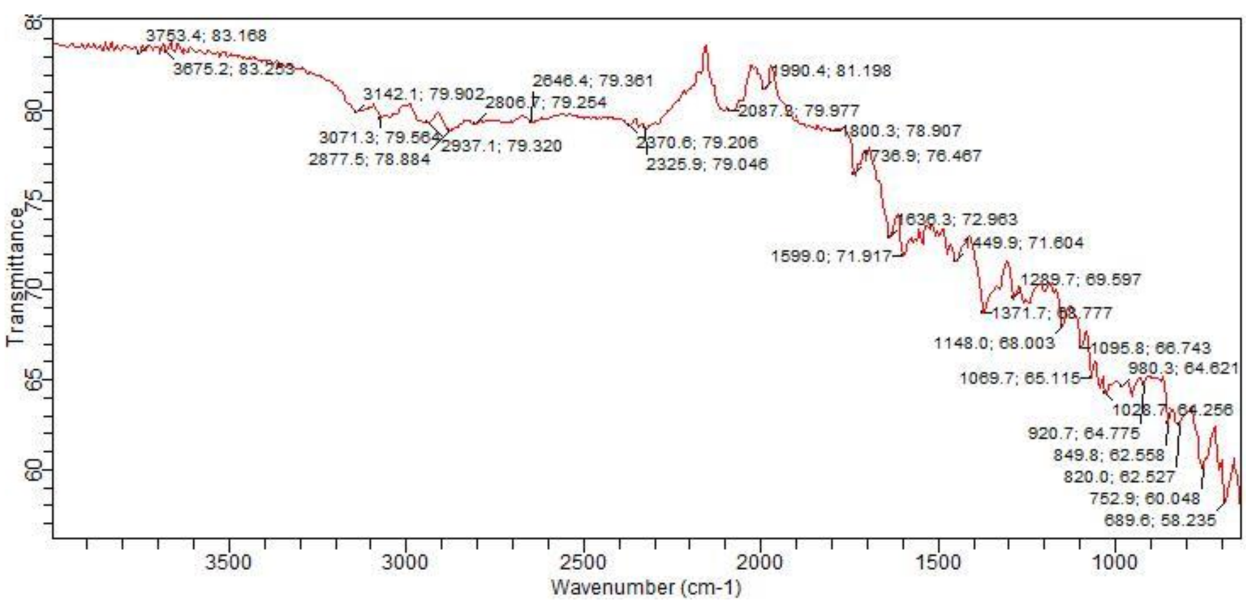

Fig. 1b: FTIR Spectra of Carbonized Particles of *Pentaclethra Macrophylla* Pod

4. CONCLUSION

The effect of heat treatment on the chemical composition of powdered *Pentaclethra Macrophylla* pod has been studied; hence, the following conclusion can be made:

- Although, powdered *Pentaclethra Macrophylla* pod contains mainly alumina Al_2O_3 and silica SiO_2, the carbonized i.e. the heat treated sample had lesser amount of the two major constituents which may be attributed to removal of volatile matter and moisture as a result of heat treatment.
- Heat treatment increases the fixed carbon content of powdered *Pentaclethra Macrophylla* pod.
- The presence of alumina and silica in reasonable amount showed that powdered *Pentaclethra Macrophylla* pod can be used as a refractory material.

***Pentaclethra Macrophylla* Pod.**

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