



Trade Science Inc.

Materials Science

An Indian Journal

Full Paper

MSAIJ, 8(1), 2012 [09-14]

Characterization of Okhuen (*Brachystegia Nigerica*) wood as a potential reinforcement for polymer composites

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Received: 15th June, 2011 ; Accepted: 15th July, 2011

ABSTRACT

Okhuen (*Brachystegia Nigerica*) wood is a species of legume in the fabaceae family. It is found in Southern Nigeria and Cameroun and elsewhere for construction purposes and fire wood. For their use as reinforcement in polymer composites production requires the understanding of their chemical composition, microstructure and thermal behavior. In this work an attempt was made to investigate the morphology of Okhuen wood through scanning electron microscopy and X-ray diffractometer, their thermal behavior through thermogravimetric analysis, their functional groups through Infra red spectrometry. The various results obtained are comparable to those of other common wood fibers, and confirm that Okhuen wood saw dust show some potential as reinforcement in polymer matrix composites.

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KEYWORDS

Okhuen wood;
Morpology;
Thermal and Functional
Group.

INTRODUCTION

Wood is a complex substance that contains cellulose, hemicellulose, lignin and extractives. The quantity of these components varies from species to species and affects the properties of the wood. The type and the amount of extractives determine the possible use of wood, as the oxidation of extractives tends to increase the acidity of wood and promote degradation^[1], because extractives such as terpenes, terpenoids, phenol, lignans and tannins are very good light absorbers^[2].

On the other hand, the thermal stability of wood is determined by cellulose, hemicellulose, and lignin contents. The low and high stability of wood at low and high temperatures can be attributed to the amount of cellulose, hemicellulose and lignin present^[3]. Wood with

higher cellulose content also contains more hydroxyl groups at the wood surface, which improves the interfacial adhesion between wood and thermoplastic matrix via the compatibilizer. High cellulose content can, however, badly affect the water absorption (WA) rate because of the increased amount of free hydroxyl groups. This is an important factor because WA usually affects mechanical properties and dimensional stability, thereby reducing the scope of application of these materials^[4]. In many cases, the addition of different wood species to the same polymer matrix can lead to different mechanical properties.

The effective use of wood-based particles composites (WPC) and fibers as fillers or reinforcements in thermoplastic composites requires a fundamental understanding of the structural and chemical characteris-

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tic of wood^[5]. One of the key factors of natural fiber thermoplastic composites is thermal degradation^[6]. Furthermore different wood species have different anatomical structures. These structural differences govern the use of these materials in WPC^[7]. Reported that differences in morphology, density, and aspect ratios across wood species account for varying reinforcement properties in thermoplastic composites.

Neagu et al^[8] investigated the stiffness contribution of various wood fibers to composite materials. They observed a correlation between lignin content and longitudinal young's modulus, and an optimal lignin content range for maximum fiber stiffness was recorded for softwood Kraft fibers. Several attempts have been made to correlate wood-based particles and fiber properties to WPC properties^[9-10].

Wheat straw has the same basic components as wood, cellulose, lignin, and pentosan, but the percentages of the components are different. Straw contains 29-35% Cellulose, 16-21% Lignin, and 26-32% Pentosan, whereas coniferous wood contains 40-45% Cellulose, 26-34% Lignin, and 7-14% Pentosan^[11]. The properties of lignocellulosic fibers mainly depend upon their chemical composition. Chemical composition of fibers depends on various factors. It varies with geographic location, climate, type of fibers and soil conditions. Hardly any literature exists on Okhuen wood^[12, 13], studied tropical timbers but did not dwell on morphological and thermal characterization of Okhuen wood which is relevant when thinking to using the fibers as reinforcing/filler in composite manufacture.

Brachystegia Nigerica are known to exist in west Africa and Congo. The colour is light brown to medium dark brown with high luster. The average density of Okhuen is the same as that of white Oak and mechanical properties are very similar. *Brachystegia Nigerica* is a species of legume in the Fabaceae family. It is found in Southern part of Nigeria and the South – West province of Cameroun. The Okhuen wood was chosen because of its abundance at most of the saw mill in Nigeria in recent times and also as Iroko and other prominent trees are diminishing due to deforestation. Based on the above-mentioned situation, the study described in this work intends: to fully exploit the potential of Okhuen wood as reinforcement/filler in thermoplastic

composites production. Hence the aims of the research work include the morphology examination, thermogravimetric analysis, Fourier transform infrared spectrometry (FTIR) and X-ray diffraction (XRD) analysis of Okhuen wood sawdust.

MATERIALS / EQUIPMENT

Brachystegia Nigerica wood sawdust whose local name is Okhuen was collected from local saw mill in Benin City Nigeria, and sieved to 0.420mm particle size using ASTM sieve of mesh 40. The sawdust was dried in an open air and kept in a polythene contain. Equipment used in this research was Scanning electron microscope (SEM), X-ray diffractometer (XRD), Fourier transform infrared spectrometry (FTIR) and DTA/TGA Machine.

METHOD

Chemical analysis of Okhuen sawdust

The chemical analysis of compounds present in the wood was carryout in the pharmacy dept of Ahmadu Bello University Zaria, Nigeria using wet analysis method. Specific density of sawdust at temperature of 32 degree C is 0.272 and density of 0.28g/ml.

The x-ray diffraction (XRD) analysis

XRD analysis of the Okhuen sawdust was carried to determine the various element and phases distribution in the sawdust particles. The test was carried out on a Philips X-ray diffractometer. The X-ray diffractograms was taken using Cu K α radiation at scan speed of 3^o/min^[11].

Microstructural analysis

The scanning electron microscope (SEM) JEOL JSM-6480LV was used to identify the surface morphology of the Okhuen sawdust particles. The surfaces of the sawdust were examined directly by scanning electron microscope JEOL JSM-6480LV. The sample was cleaned thoroughly, air-dried and are coated with 100 Å thick platinum in JEOL sputter ion coater and observed SEM at 20 kV. The sample was sputter-coated with gold to increase surface conductivity. The digitized images were recorded.

TABLE 1 : Identified Patterns List of Okhuen saw dust

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	01-076-0912	31	Keatite, syn	0.000	0.663	Si O ₂
*	00-051-2049	23	Dimethyl-2,6-naphtalene dicarboxylate	0.000	0.089	C ₁₄ H ₁₂ O ₄
*	00-040-1556	20	P-Toluic acid	0.000	0.036	C ₈ H ₈ O ₂

Thermal properties

Thermal decomposition was observed in terms of global mass loss by using a DTA/TGA Instrument TGA Q50 thermogravimetric analyzer. This apparatus detects the mass loss with a resolution of 0.1 as a function of temperature. The Okhuen sawdust particles were evenly and loosely distributed in an open sample pan of 6.4 mm diameter and 3.2 mm deep with an initial sample amount of 8-10 mg. Due to different bulk density, the depth of the sample layer filled in the pan was about 1-2 mm. The temperature change was controlled from room temperature (25±3°C) to 700°C at a heating rate 10°C/min^[8].

Fourier transform infrared spectrometry (FTIR) analysis

Fourier transform infrared spectrometry (FTIR) was carried out on Okhuen sawdust as well. IR spectra of the sawdust were recorded using Perkin Elmer spectrum 100 FT – IR spectrometer in the frequency range 4000 – 400cm⁻¹, operating in ATR (attenuated total reflectance) mode.

RESULTS AND DISCUSSION

The chemical composition of Okhuen sawdust is extremely complex due to the different compounds present at various concentrations. Wood mainly composed of cellulose, hemicellulose, and lignin, plus tannins and some amount of lipids (oils, fats and waxes). The lignin acts as binding agent. The results show that Okhuen wood contains 44.5% cellulose, 20.1% Pentosan, 21.2% lignin, 2.4% extra and 4% ash. The results compared favorably with the chemical composition of some hard wood used as reinforcement in natural composite materials^[1,5].

The XRD pattern of the Okhuen wood sawdust particles reveal that, the major diffraction peaks are

15.93°, 25.05°, and 40.04° and their inter-planar distance, 5.56Å, 3.55 Å, and 2.25Å, and their relative intensity of X-ray scattering are 7.82, 6.86, 88.5, 1.31 and phases at these peaks as: SiO₂, C₁₄H₁₂O₄ and C₈H₈O₂, this revealed that particle has some of the composition of hemicelluloses, cellulose, and lignin that has been confirmed by literature(see Figure 1 and TABLE 1)^[8,9].

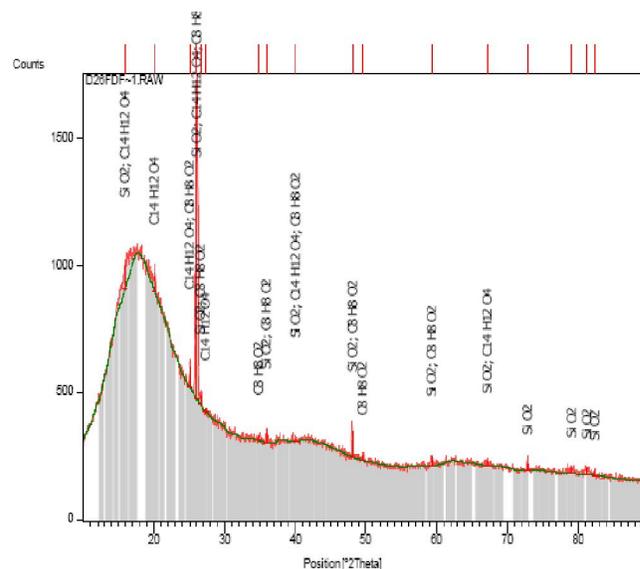


Figure 1 : XRD pattern of Okhuen saw dust

The microstructure of the Okhuen wood sawdust particle reveals that the size and shape of the particles vary; however, they can be sorted into three main groups – prismatic, spherical and fibrous. The prismatic particles consist mainly of C and O. The spherical ones contain C and O as well as H, The fibrous ones consist of only C as a result of the EDS scan in rectangle (see Figure 2-3). These structures are overlapping and are bonded firmly together, by pectin and other non-cellulosic materials. This observation is in par with the earlier findings of^[1,5].

The temperatures of destruction (T_{des}) of the Okhuen wood saw dust, subject to investigation, were deter-

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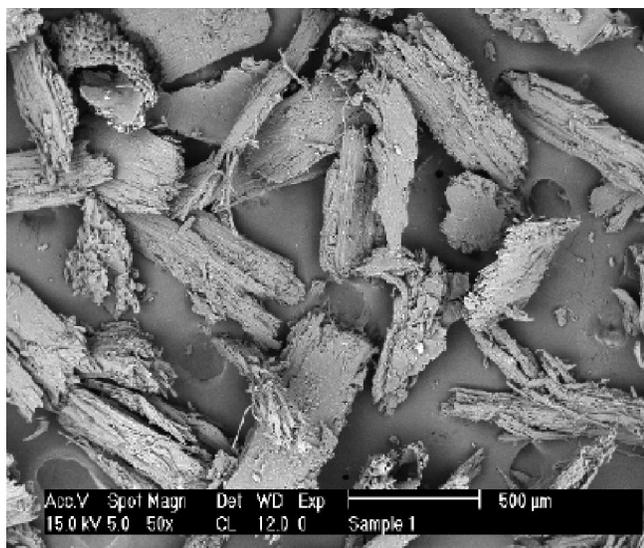


Figure 2a : SEM microstructure of Okhuen wood saw dust(x50)

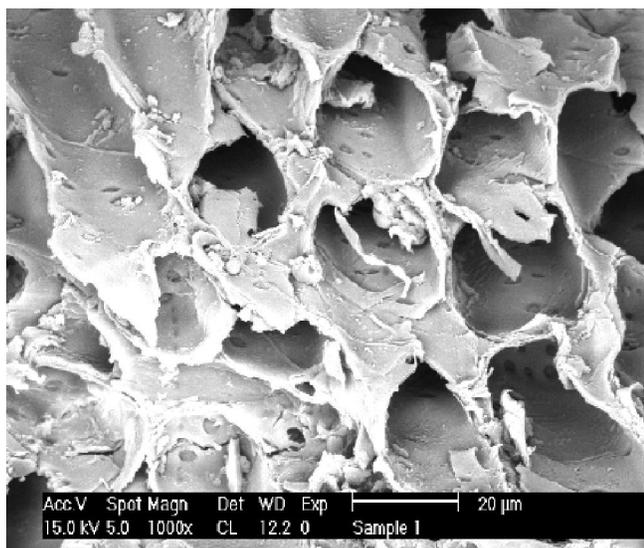


Figure 2b : SEM microstructure of Okhuen wood saw dust(x1000)

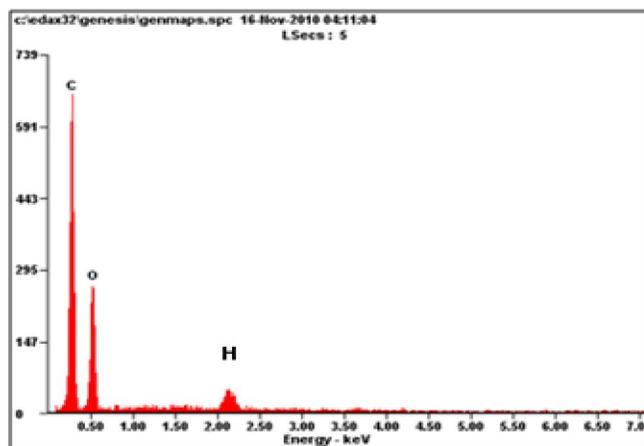


Figure 3 : EDS of Okhuen wood saw dust

mined from DTA curves. DTA data were recorded on “Derivatograph OD 102”, at heating rate of 10°C/min in argon. The results of the DTA/TGA scan of the Okhuen wood saw dust is shown in Figure 4. From the Figure the TG/DTA curve shows three weight loss steps, while their composition occurs in two stages.

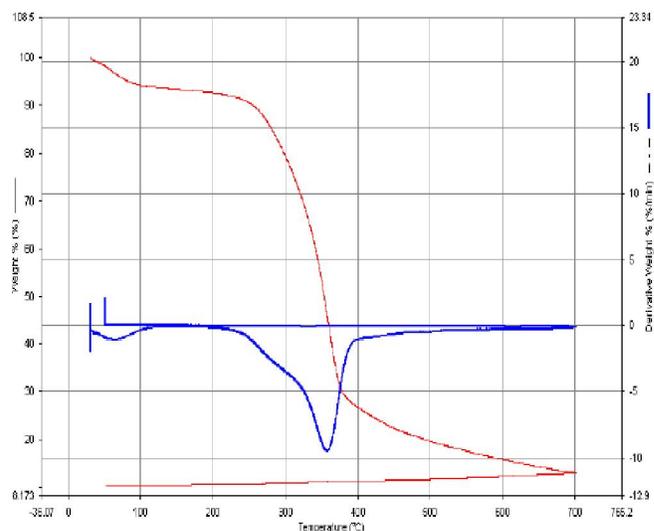


Figure 4 : DTA/TGA Curve of Okhuen saw dust

The initial weight loss (~ 9%) observed between 30 and 200°C is attributed to the vaporization of the water from the wood, while degradation of the wood started at higher temperature, precisely after 250°C. Above this temperature, the thermal stability of Okhuen wood gradually decreased and degradation of the wood occurred. Temperature between (250°C) to (300°C) is associated to the thermal depolymerization of hemicelluloses, pectin and cleavage of glycosidic linkages of cellulose (weight loss 16.5%), while corresponds to the degradation of α - cellulose present in the wood (weight loss 55.6%). Decomposition of lignin occurs slowly due to its complex structure within the whole temperature range. In fact the lignin is composed of aromatic rings with various branches^[1].

DTA curve shows that the temperature of maximal decomposition/ destruction was 351°C (see Figure 4). The presences of endothermic effects in Okhuen saw dust sample are results of two processes – dehydrogenation and evaporation of some non-cellulosic materials. This conclusion was confirmed by the decreased mass of the sample. DTA curve also confirmed these results. In an inert atmosphere, the final products of the degradation of cellulose consist in carbonaceous resi-

dues and possible un-degraded fillers. On the analogy of these results it was assumed that the total burning/ degradation of the residual Okhuen wood saw dust (dehydrogenation) took place in this temperature interval (400-600°C), In the last temperature interval the mass loss was minimal. This last step (accompanied by the evolution of CO₂ only)^[11-12].

The endothermic effects observed in the temperature range indicate above were probably as a result of the double bonds formed in the Okhuen wood saw dust backbone, cross linkage of the dehydrogenation wood macromolecules and continuing oxidation of the products from its thermal degradation. Finally, is worthy of note that the thermal analysis curves reveal that Okhuen wood is stable until around 350°C. This is in agreement with values of some other wood material reported in literature^[1,6]. This temperature is higher than many agro-waste currently used as a reinforcement for polymer composites and board composites.

The chemical structure of the components of Okhuen wood sawdust was analyzed using Perkin Elmer spectrum 100 FTIR- ATR. The main absorbance peaks of interest in this study have been identified and depicted in Figure 5. FTIR spectrum of wood absorption bands of functional groups characteristic of lignocellulosic compounds are cellulose, hemicelluloses and lignin. Such components are mainly consisting of alkenes and aromatic groups and various oxygen containing functional groups (ester, ketone and alcohol). In TABLE 2, is shown the peaks, intensity functional groups and stretches. The highest peak at 3399.53cm⁻¹ is charac-

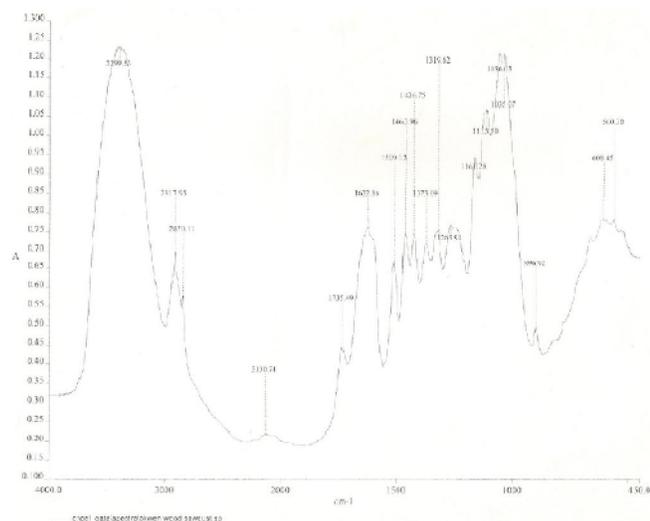


Figure 5 : ATR-FTIR spectrum of Okhuen(Okhuen) sawdust.

TABLE 2 : Infrared Spectrometer Result of Okhuen Sawdust

Cm	INTENSITY	FUNCTIONAL GROUP	STRETCHES
3399.53	Medium	Amine/hydroxyl	N-H stretch and O-H stretch for amines, amides and hydroxyl compound
2917.95	Medium strong	Alkenes, aromatic	C-H stretch for hydrocarbon
2850.11	Weak	Alkenes	C-H stretch
2850.11	Medium weak	Aromatics	C-H stretch
2130.71	Variable medium	Nitriles	CN stretch vibration
1735.40	Strong	Esters and Lactones	C=O stretch Vibration
1622.86	Medium strong	Amides and Lactones	C=O stretch Vibration
1509.13	Medium	Conjugated Phenyl Ketones	C=O conjugated
1463.96	Variable	Alkenes and aromatics	C=C bond
1426.75	Variable	Alkenes and aromatics	C=C bond
1373.69	Strong	Esters, amides and ketones	C-O stretch
1319.62	Strong	Carbonyl group	C-O stretch
1265.94	Strong	Carbon	C-O deformation of esters, amides, ketone
1100.00	Strong	Carbonyl group for esters, amides ketones	C-O deformation for esters, amides, ketones
1056.05	Strong	Alkyl groups	C-H deformation on bonds of alkyl groups
896.92	Medium	Alkyl groups	C-H deformation
560.30	Strong	Methyl group	bond of methyl groups

teristic of amine/hydroxyl N – H stretch and O – H stretch. While the absorbance at 560.30cm⁻¹ correspond to methyl group and C – H deformation bond of methyl group. This results obtained are in par with the result obtained for the SEM, XRD and the elemental analysis.

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CONCLUSIONS

Sawdust extracted from Okhuen (*Brachystegia Nigerica*) was characterized by electron and FTIR. Their thermal degradation behavior was fully investigated through TGA/DTA curves, morphology by SEM/EDS and XRD analysis. The various results obtained are comparable to those other common wood and Lignocellulosic fibres, confirm that these Okhuen wood saw dust show some potential as reinforcement in polymer matrix composites.

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