CHARACTERISATION OF MODIFIED-CARBONISED *Eucalyptus globulus* SAWDUST FIBRE FOR POLYMER COMPOSITE REINFORCEMENT

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Abstract: This research focuses on the characterisation of raw and treated carbonised *Eucalyptus globulus* sawdust (EGS) fibre. The micro-structural properties that were analysed include the elemental composition, morphology and functional groups of the EGS fibre. This EGS fibre was optimally mercerised at 3% NaOH concentration for 2 h, after which, 100 g of the treated fibre was carbonised at 500 °C for 2 h. Result from the elemental composition analysis reveal that carbon recorded the highest percentage weight concentration among the other 11 elements detected from the raw EGS sample. This may be due to the EGS specie and inadequate carbonisation process. In the treated carbonised EGS sample, increase in weight concentration of some elements like aluminium zinc and magnesium was observed. Similarly, there was emergence of some transitional metals in the treated carbonised EGS sample. This is as a result of the quantitative purification of the crude raw EGS fibre, initiated by the mercerisation and carbonisation process. The morphology of the treated carbonised sample reveal absence of white patches of impurities with smaller particle sizes. Furthermore, the functional groups as revealed by the Fourier transform infra-red (FTIR) analysis of the treated carbonised fibre shows a drastic reduction in the hydroxyl (OH) group and emergence of an amine (N-H) group with an adsorption character. All of these are in comparison with the raw carbonised EGS fibre. The property of treated carbonised EGS fibre is suitable for application in the production of hybrid polymer composite of good mechanical and thermal property.

Keyword: *Eucalyptus globulus* fibre, carbonised, treated, functional group, morphology, elemental composition.

Introduction

Over three decades now, ceramics, plastics, composite materials, and natural fibre have become emerging materials of interest. Natural fibres which are used as reinforcement in composites can be obtained from animals, plants, as well as from some geological processes. This greatly influences the resulting properties of composites through their reinforcing orientation. When plant fibres are in use as reinforcement, the cellulose component present in it, help to enhance strength and integrity, which can be improved upon by modification through chemical treatment. This has led researchers to explore the advantages that can be obtained from the modification of the chemical composition of plant fibre for specific purposes (Government *et al*., 2019; Kadolph, 2002).

The specie/nature of plant fibre, growth conditions and the methods of chemical composition determination are key factors that influence the chemical composition and properties of plant fibre. Even among fibres of the same kind, there are variations. This is because the overall structure and composition of plant fibre is fairly complex (Amar *et al.,* 2005, Rakesh *et al.,* 2011). According to Bledzki *et al* (2008), plant fibre by nature's design, are composite materials. With the exception of cotton, plant fibres are composed of cellulose, lignin, hemicellulose, extractives (waxes and oil) and some water soluble compounds. Nevertheless, the first three components are predominant in composition. It is worthy to note that, these fibres in their raw form, modified state by physical/chemical process, as well as in carbonised form, can be used as reinforcement in polymer composite production.

A polymer composite is a multiple-phased material in which a polymer matrix is reinforced with fibre, fillers or both to give better performance properties which is not possess by the individual constituting material (Chawla, 2013). In this case, natural fibres such as bamboo, hemp, jute, sawdust and locust bean husk, can be used to reinforce a particular polymer matrix of interest such as polyethylene. Similarly, fillers such as calcium carbonate, ash particulate and clay, can also be used as additives to reinforce polymer resins to obtain better improved mechanical properties. Some of the advantages and outstanding properties of polymer composite are; high strength, design flexibility, light weight, thermal resistance, and good corrosion resistance. These unique properties have necessitated the need for polymer composite to be widely adopted for use in diverse fields such as civil engineering constructions, aerospace, automobile, ship building and in military hardware (Parnas, 2001; Omoyeni *et al*., 2016).

Hassan *et al.* 2012 in Araoye (2015) corroborated the observations of other researchers on the fact that, the use of carbonised particulate as a reinforcing material imparts good strength in composites produced from polymer matrices. These carbonised particulate creates a strong interfacial bond of appreciable strength for effective load transfer. In this research, treated carbonised EGS fibre particulate was produced and characterised to determine its modified property for application polymer composite production.

Materials and Method

Chemical treatment of EG sawdust fibre and carbonisation

The prepared EG sawdust fibre were dried in an oven for 24 h at 105°C to achieve a constant weight. A 100 g weight of sawdust each was taken and treated with sodium hydroxide based on the experimental design input factors. The treated sawdust was washed with enough distilled water until the pH of the solution attains neutrality to modify the sawdust fibre in order to enhance better bonding and interaction with the polymer matrix. Thirteen (13) runs were carried out for the sawdust treatment. The 12th run was observed to have the highest optimum response of cellulose content. This process was captured in Olowokere *et al*., 2022. The treated EGS fibre was carbonised in a furnace at 500^oC for two hours, to form char and ash particles. This was stored and used as one of the reinforcing fillers for hybrid composite production (Olowokere *et al*., 2022; Muhammad *et al*., 2011).

Micro Structural Analysis

The following techniques were used to determine and analyse the micro structural properties of the reinforcing carbonised EGS fibre needed polymer composite production.

Determination of the elemental composition and morphology of EGS fibre

SEM- EDX (Phenom ProX Generation 5) machine was used to show the morphology or surface chemistry of the untreated, treated reinforcing fibre-ash, clay filler and hybrid polymer composite, alongside the corresponding elemental composition of the fibre-ash.

Determination of the functional groups present in the carbonised EGS fibre

The observations and changes in the functional group present in the raw, treated reinforcing materials and hybrid polymer composite was determined and captured by Fourier Transform Infrared (FTIR) spectroscopy machine of model NICOLET 155 thermo scientific Nicolet corporation, madison USA. The FTIR spectra of the samples were analyzed in the range of 4000 cm^{-1} to 500 cm^{-1}.

Results and Discussion

The micro structural properties comprising the elemental composition, morphology and functional groups of the raw and treated carbonised EGS fibres were analysed respectively using SEM–EDX and FTIR. Table 1 presents the elemental composition of the EGS fibre.

Table 1: Elemental Composition for Untreated and Treated Carbonised *Eucalyptus globulus* Sawdust Fibre

Elemental Composition for Untreated Carbonised EGS			**Elemental Composition for Treated Carbonised EGS**		
Element Symbol	**Element Name**	**Untreated Weight Conc. (%)**	**Element Symbol**	**Element Name**	**Treated Weight Conc. (%)**
C	Carbon	87.68	Na	Sodium	57.59
Ca	Calcium	3.17	Ca	Calcium	10.16
I	Iodine	2.55	Zn	Zinc	8.61
Sn	Tin	1.75	Al	Aluminium	5.55
Zn	Zinc	1.69	V	Vanadium	3.94
Ag	Silver	0.87	Cr	Chromium	3.45
Al	Aluminium	0.53	Mn	Manganese	3.31
Na	Sodium	0.52	Mg	Magnesium	2.98
Mg	Magnesium	0.37	Si	Silicon	2.48
Si	Silicon	0.35	Ti	Titanium	1.94
S	Sulphur	0.26			
P	Phosphorus	0.26			

Elemental Composition for Untreated and Treated Carbonised EGS

The percentage (%) elemental composition of carbonised raw (untreated) EGS in Table 1 is expressed in a decreasing order - Carbon (C) >Calcium (Ca) > Iodine (I) > Tin (Sn) > Zinc (Zn) >Silver (Ag) > Aluminium (Al)>Sodium (Na) >Magnesium (Mg) > Silicon (Si) >Phosphorus(P). Carbon has the highest composition of 87.68%, while sulphur and phosphorus are the least with 0.26%. The result obtained here is different from the report of Naik *et al*. (2002), Udoeyo and Dashibil (2002) and Udoeyo *et al*. (2006). The high carbon content may be an indication of inadequate carbonisation of the raw fibre (Bhaskar, 2016). The elemental composition and concentration of wood ash is dependent on the wood type/specie, growth conditions, hydrodynamic of the furnace and combustion temperature (Chowdhury *et al*., 2014).

For the alkaline treated carbonised EGS, the elemental composition in a decreasing order shows that sodium is having 57.59%, while titanium recorded 1.94%. More number of elements was recorded in the raw carbonised EGS than the treated sample. C, I, Sn, S and P which were present in the raw EGS were found to be absent in the treated EGS. Leaching of these elements during alkaline treatment is responsible for the absence of these elements. Moreover, element like Tin is poisonous to health. Upon alkaline treatment of the raw EGS with NaOH, it was also observed that elements like Ca, Zn, Al, Na, Mg, and Si increase in their percentage weight compositions. This increase is due to the quantitative purification of these crude elements, which are now exposed by the alkalisation process. Consequently, the oxide components of Al_2O_3 and MgO in the treated ash will help to increase the fire resistance property of the hybrid polymer composite. Similarly, ZnO is also known to have a strong resistive effect to weathering (Layth *et al.*, 2015; Rudi *et al.*, 2019). Furthermore it was also observed that new transitional elements such as Vanadium, Chromium, Titanium and Manganese emerged in the treated carbonised EGS fibre. These emerging elements must have been crude and unrefined elements buried and covered by the impurities or non-cellulosic components present in the raw EGS, prior to the alkaline treatment process. Also, chemical transformation and reaction of the mercerisation process may also be responsible. The oxides of these transitional elements such as titanium oxide are known to be good ultra-violet ray absorbers.

Morphology of Raw and Treated Carbonised EGS Fibre

Figure 1 and Figure 2 presents the morphology of raw and treated carbonised EGS fibre.

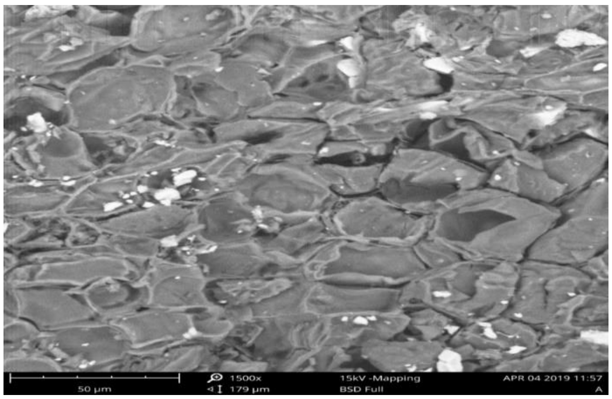

Figure 1: SEM Image for Carbonised Raw (Untreated) EGS Fibre

The morphology of the untreated EGS ash in Figure 1 is characterized by large patterns of regular shaped carbonised particles. Some few impurities were observed in form of white patches. A cursory look at the image shows that the particles sizes are larger in this case compared to the treated carbonised EGS particles in Figure 2. This is because the lignin content which offers rigidity and thermal stability to wood fibre is still relatively high in the untreated EGS fibre (Ilyas *et al.*, 2017).

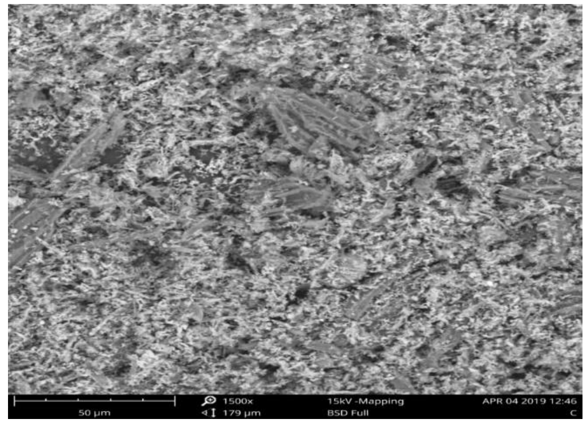

Figure 2: SEM Analysis for Carbonised Treated *Eucalyptus globulus* Sawdust

The SEM analysis of the treated EGS ash is characterized by smaller fine needled-like particles compared to the untreated EGS ash. This is because the treated EGS is less stable under heat during carbonisation as a result of the drastic reduction in its lignin content which was washed off during the alkaline treatment process. Ilyas *et al.*, (2017) observed that treated fibres experiences decrease in diameter, density and moisture content after alkalisation. This will also lead to reduction in particle sizes after carbonisation at 500^{o}C.

The behaviour of a natural fibre is largely dependent on the functional groups it possesses. The FTIR Spectrum for raw (untreated) EGS ash is presented in Figure 3.

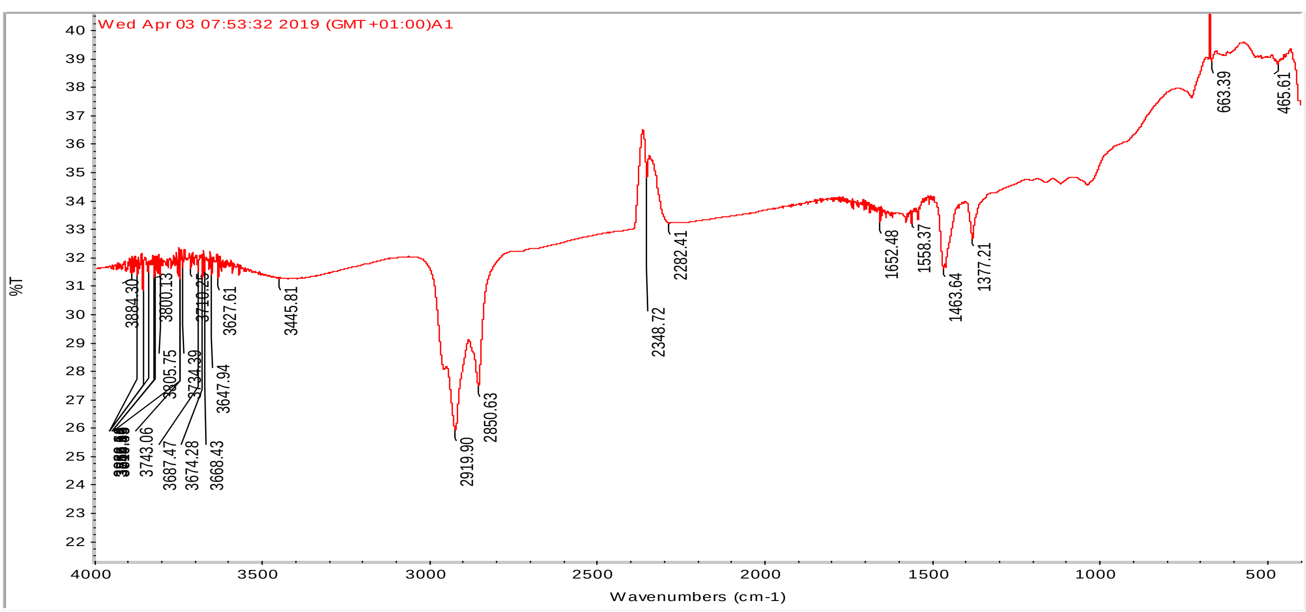

Figure 3: FTIR Spectrum for Raw Carbonised EGS fibre

The untreated carbonised EGS has higher amount of OH groups peaks ranging from 3878.58 – 3445.81 cm^{-1}. There are alkane C-H peaks at 2919.90 and 2850.63 cm^{-1}. All of these are in comparison to the raw EGS fibre. Two nitrile $C\equiv N$ groups were also observed in the EGS ash at 2348.72 and 2282.41 cm^{-1}. This is absent in the raw EGS fibre. The presence of C=C aromatic ring of lignin at 1652.48 and 1558.37 were detected in the carbonised EGS. These are indications of the high thermal stability of lignin at 500°C compared to the thermal stability of other components (cellulose and hemicellulose) of the EGS fibre. This is similar to the observation by Ilyas (2017) on sugar palm fibre. Ishak *et al*. (2012) assert the fact that the thermal degradation of lignin extends to as high as 900°C.
Mercerisation of natural fibre in most cases, initiates a variation in the functional group of the fibre. The FTIR Spectrum for treated carbonised EGS is presented in Figure 4.

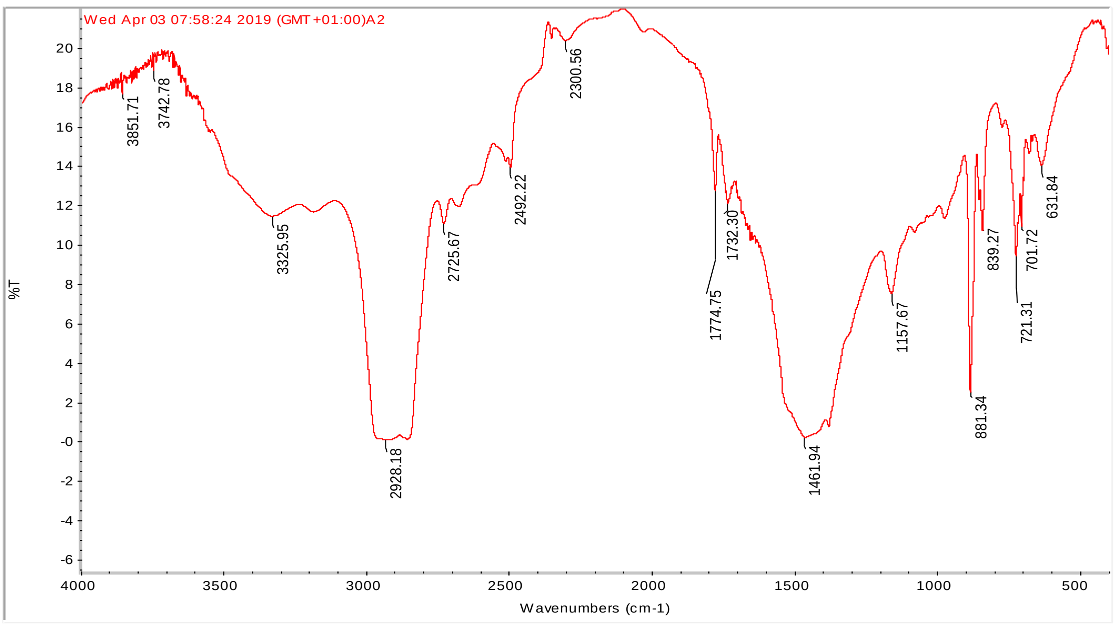

Figure 4: FTIR Spectra for Treated Eucalyptus Globulus Sawdust Ash

Figure 4 shows that there is a drastic reduction in the number of OH peaks present in the carbonised treated EGS. This is an indication that the multiple OH group peaks that characterized the hemicellulose and cellulose have been broken down and washed off during the alkaline treatment process. The leaching of these OH groups which was initiated by the alkalisation process was further promoted during the carbonisation process. This is a sign that hemicellulose is less stable under heat as reported by Ishak *et al.* (2012) and Ilyas (2017). There is an emergence of amine-N-H group peak at 3325.95 cm^{-1}. This N-H character confers an adsorbent property on the carbonised treated EGS. This same observation is reported by Alhassan *et al.* (2017), on activated carbon from sugarcane bagasse. Broad C-H and C=C aromatic ring were recorded at 2928.18 and 1461.94 respectively against the sharp peaks noticed from untreated carbonised EGS.

Conclusion

This research was able to characterise the properties of raw and treated carbonised EGS fibre for hybrid polymer composite application. The mercerisation and carbonization of the EGS fibre had a positive modifying effect on the elemental/oxide composition, morphology and functional group of the natural fibre. There was improvement in the alumina, silica and magnesium content, as well as emergence of titanium metal from the treated carbonized EGS fibres. All of these components provided the needed properties that placed a prominent role in imparting thermal and ultra violet (UV) retarding effect, on hybrid polymer composites with good mechanical strength. Furthermore, the mercerisation and carbonization process confers an adsorptive property on the EGS fibre with hydrophobic character for polymer composite application.

Conflict of Interest

There is no conflict of interest between the authors.

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