



EFFECTS OF SURFACE MODIFICATION ON THE PROPERTIES OF AMPELOCISSUS LEONENSIS FIBER.

Ejikeme, Patrick C.N.^{*1}, Ejikeme, Ebere M.², Onyia, Ogechukwu Sandra³

1,2 and 3 - Department of Chemical Engineering, Enugu State University of Science and Technology, Enugu, Nigeria

Abstract - Effects of surface modification of natural fiber through chemical treatment on the properties of *Ampelocissus Leonensis* fiber were studied. The natural fiber (*Ampelocissus Leonensis*) was treated with sodium hydroxide, acetic anhydride, nitric acid and zinc chloride. Effects of chemical concentrations and duration of treatment on the properties of the natural fiber were investigated. The results showed that fiber treated with chemicals exhibited better aspect ratio, lower water absorption capacity and brighter micrographs in comparison with the untreated *Ampelocissus Leonensis* fiber. The Fourier transform infra-red (FTIR) analysis indicated presence of new moieties which were introduced by the chemicals during treatment. The chemicals were therefore able to alter the physical, chemical and morphological properties of the fiber.

KEYWORDS: *Ampelocissus Leonensis*, chemical treatment, morphological properties, waterabsorption

1.0 INTRODUCTION

Polymers have in recent time been playing vital roles in engineering applications and utilizations. This position is taken because of the advantages these polymers have over the conventional materials for same engineering applications (Lipsa and Sweta 2007). These polymers are normally used in the form of composites to improve the overall property of the polymers. Composites are a combination of two or more different materials to achieve certain properties different from each material on its own. One of the materials acts as the reinforcement while the other acts as the matrix. The reinforcement is embedded into the matrix to improve its mechanical and physical properties. (Irene et al 2010). Natural fibres which are endowed with great advantages like biodegradability, light weight and recyclability have swayed the attention of researchers in their favour as reinforcement in composite

production. Because of these adorable properties, natural fibres have been increasingly adopted to replace synthetic fibers in composite production for industrial and engineering applications (Nural and Ishak 2012). Therefore developing natural fiber reinforced polymer composites becomes very attractive for both researchers and Industrialists alike. In addition to the health hazards, high density and non-recyclability associated with synthetic fibers such as glass, carbon and aramid as reinforcements in polymer materials (Herrera and Valdez 2005), synthetic fibres also possess the problem of disposal at the end its useful life. The advantage of natural fibres over synthetic ones ramped-up the choice of natural fibres and made synthetic fiber very unattractive.

Over the years several natural fibres have successfully been used as reinforcements in polymer materials. Natural fibers like bamboo

(Shibata et al 2007), Kenaf (Elshekeil et al 2012) and bagasse (Cao et al 2006), Flax (Bei Wang 2004) hemp (Bledzki et al 2004) have been studied as reinforcements and fillers in polymer composites. However these natural fibers could not be said to be without problems as reinforcements in polymer materials for composite production. The performance, stability and durability of fiber reinforced composite materials depend greatly on the development of coherent interfacial bonding between the fibre and the polymer matrix. However, there is visible lack of good interfacial bonding between the natural fibre and the polymer matrix. This is because the hydrophilic cellulose nature of the fibers and the hydrophobic nature of the polymer make them incompatible for the attainment of strong interfacial bond (Mwaikambo and Ansele 1999, Elshekeil et al 2012). The fiber's structural compositions like cellulose, hemicelluloses, pectin, lignin and waxy substances amply gave them out as favourably disposed to high moisture absorption from the environment. This situation leads to poor bonding with the matrix material (Doan et al 2006). The above condition is significantly responsible for the development of very weak bonds between the reinforcements and the polymer materials.

Certain chemical treatments of the natural fibres are needed to enhance the bond ability of the fibres as reinforcements in polymer composite materials. This is because the Hydroxyl group as a derivative of the cellulose and lignin contents of natural fibres make them very amenable to chemical modification. The chemical modification activates the hydroxyl groups thereby introducing new moieties that can effectively interlock with the polymer matrix. This informed the use of chemical treatment to advance the development of these moieties in natural fibers for the anticipated effective interlocking with the polymer materials.

In this work *ampelocissus leonensis* fiber was treated with sodium hydroxide, acetic anhydride, nitric acid and zinc chloride to evaluate the effect of these treatments on the

physical, chemical and morphological properties of the reinforced polymer composite.

2.0 MATERIALS AND METHOD

2.1 Materials:

The natural fibre *ampelocissus leonensis* was obtained from Olo in Ezeagu Local Government Area of Enugu State, South East Nigeria.

The chemicals, namely, Sodium Hydroxide, acetic anhydride, nitric acid and zinc chloride were of analytical grade and were obtained from De-Cliff integrated company Ltd, Enugu, South East Nigeria.

2.2. METHOD

2.2.1 Extraction of Fiber

The natural fibre was extracted from the plant stem. The stems were traumatized heavily and soaked in water for two weeks. These items were brought out and thoroughly washed and further soaked in water for another one week. The fiber now in strands was washed in distilled water and air dried at room temperature to a constant weight.

2.2.2. Proximate Analysis of the Fiber

Moisture content, ash, wax, lignin, hemicelluloses and cellulose content of the untreated and treated fibre were done according to work done by Ejikeme et al (2014a).

2.2.3 Chemical treatment of the *Ampelocissus Leonensis* Fiber

Sodium hydroxide, acetic anhydride, nitric and zinc chloride treatments were done according to work done by Ejikeme et al (2014b).

2.2.4 Determination of the physical and mechanical properties of the fibre

2.2.4.1 Diameter Determination

Electronic digital was used to measure the diameter of the fibres. At least twenty single fibre strands of all the different plant fibre species were measured and the average diameter was calculated using equation;

$$D = \frac{D_1 + D_2 + \dots + D_{20}}{20}, \quad (1)$$

where D is the fiber diameter.

2.2.4.2 Aspect ratio

The aspect ratio of the fiber was calculated from the average diameter using ASTM (D1037-12) as in the equation below.

$$\text{Aspect ratio} = \frac{\text{Fiber length}}{\text{Fiber diameter}} \quad (2)$$

2.2.4.3 Density of the fibers

Archimedes principle was used to determine the density of the treated and untreated fibers according to work done by Morton, (1980). In this method, the samples of the fiber were selected and bound into a bundle and its mass measured on a digital weighing balance. The volumes of these fixed mass of the fibers were measured using standard displacement technique. In this method, the weighed fiber bundles were immersed in 100mls measuring cylinder filled with water. The displaced volume was measured with a measuring cylinder. The density was determined by dividing the mass of the fibre bundle by its displaced volume using equation (3) below. The process was observed for both the treated and untreated fibers.

$$\text{Density (g/cm}^3\text{)} = \frac{M}{V} \quad (3)$$

where M is the mass of the fiber bundle and V is the volume of the fiber

2.2.4.4 Water absorption

Humidity chambers (desiccators) were set up at 100% humidity using distilled water. The *ampelocissus Leonensis* fiber (untreated, alkaline treated, acetic anhydride treated, nitric acid treated and zinc chloride treated) was dried in a hot air oven at 60°C for 24hrs, weighed and then placed in a humidity chamber. The humidity chamber was held at room temperature. After 24hrs, the fiber bundle was taken out from the humidity chamber and excess water was carefully mopped with filter paper. Finally, the weight of the fiber was measured and the water absorption was calculated by weight difference.

$$\text{Water absorption (\%)} = \frac{m_a - m_d}{m_d} \times 100 \quad (4)$$

where m_a is the mass of the sample after exposing to water and m_d is the mass of the dry sample.

2.2.5 Surface characterization.

2.2.5.1 Scanning Electron Microscopy (SEM) analysis

Scanning Electron Microscopy (SEM) of the fibers was done on the untreated and treated fibers to study their surface textures.

2.2.5.2 Fourier Transform Infrared Spectroscopy

The surface functional groups and structure were studied using Fourier transform infrared spectroscopy (FTIR). The FTIR spectra of the untreated and treated fiber were scanned at a wavelength of 500–4000nm to obtain its spectra notifications.

3.0 RESULTS AND DISCUSSIONS

3.1 Chemical composition of untreated and treated fiber.

Natural fibre consists of cellulose, hemicelluloses, lignin, wax, water content and water soluble substances. These compositions differ with the type of plant and even with different species of the plant.

The chemical structure of cellulose is the same for all natural fibres, however there may be variations in the cellulose content due largely to varying degrees of polymerization of the cellulose or length of the polymer chains of the cellulose. The degree of polymerization of cellulose has a significant effect on the mechanical properties of a fibre as it is directly related to the molecular weight (Mohanty et al., 2000). When the cellulose has a higher degree of polymerization and molecular weight, the fibre will have greater strength properties (Mohanty et al., 2000). Table 1 shows the chemical composition of the untreated and treated natural fiber.

Table 1. Chemical Compositions of the *Ampelocissus Leonensis* fibres:

Parameters	Untreated	Sodium hydroxide treated	Acetic Anhydride treated	Nitric acid treated	Zinc Chloride treated
Cellulose (%)	43.22	63.94	58.77	55.04	50.62
Hemi cellulose (%)	28.221	15.76	19.66	20.91	24.17
Lignin (%)	34.910	10.87	13.73	19.88	22.92
Wax (%)	0.01	0.00	0.00	0.00	0.00
Moisture content (%)	5.50	3.76	4.88	5.5	5.3

From Table 1, it can be seen that *Ampelocissus Leonensis* has high cellulose content. This high cellulose expresses its degree of polymerization, thus explaining the fibre's effectiveness when used in composite production. Hemicellulose is a fully amorphous branched polymer with a selection of sugar molecules as monomeric unit (Cutter, 2008). They have significantly lower molecular weight with low degree of polymerization making it weak polymer when compared to cellulose (Pott, 2004). From Table 1, it can be seen that *Ampelocissus Leonensis* fibres has high cellulose content and low hemicellulose content and as such has high tensile strength while any fibre with high hemicellulose contents, has low cellulose content with attendant low tensile strength. This was linked to the fact that hemicellulose has low degree of polymerization which is directly related to the tensile strength. Lignin is the most complex polymer among naturally occurring polymers. It functions as a structural support material in plant by filling the spaces between the cellulose and hemicelluloses region, cementing them together and decreasing the permeation of water across the cell walls (Cutter, 2008). The aromatic nature of lignin, high carbon and high hydrogen contents make it hydrophobic (Mohanty et al., 2000; Pott, 2004). Fiber with high lignin content has low moisture content and that with low lignin content has high moisture content.

The results showed that chemical treatment of the fibre resulted to the reduction in the lignin, hemicelluloses and wax content of the fibre and increase on the cellulose content.

3.2 Mechanical properties of untreated and treated natural fibre

Table 2 shows the mechanical properties of the untreated and treated natural fibre. From the table, it is observed that the fibre has high tensile strength. The fibre's tensile strength was as a result of good degree of polymerization of the cellulose content of the fibre. This means that the fibre is ductile and can experience plastic deformation and possible necking before fracture.

The elastic modulus was used to ascertain the stiffness of the fibre. The elastic modulus of the fibre was high. Elongation at break which is the maximum possible extension of the fibre before it breaks is considerably high. Elastic elongation is of decisive importance because composite products without elasticity would hardly be usable. The result obtained showed that the fibre must be able to deform in order to withstand high loading and must also return to shape. It is recorded that *Ampelocissus Leonensis* has low tensile strength and high elongation at break. All the mechanical properties increased as a result of chemical treatment except elongation at break for the fiber treated with nitric acid and zinc chloride. The decrease can be attributed to the fibrillation of the fibre as a result of treatment which made the fibre to be less ductile. This was confirmed on the values of the aspect ratio on Table 3 and on the diameter of the fibre after treatment. As a result of fibrillation, the diameter of the treated fibre reduced thereby increasing the aspect ratio.

Table 2. Mechanical Properties of untreated and treated *Ampelocissus Leonensis* fiber:

	Tensile Strength (Mpa))	Elastic Modulus (Mpa)	Ultimate Elongation At Break (%)
Untreated	67.67	422.81	8.0
NaOH Treated	123.34	770.6	9.0
Acetic Anhydride Treated	103.75	648.2	8.5
Nitric Acid Treated	89.54	559.4	7.3
Zinc Chloride Treated	84.59	528.5	7.0

Table 3. Effect of chemical treatments on fiber length, diameter and aspect ratio

Samples	Average Length (mm)	Average diameter (mm)	Aspect ratio (L/D)
Untreated	100.00	0.58 – 0.60	169.6
NaOH Treated	100.00	0.54 – 0.56	181.8
Acetic Anhydride Treated	100.00	0.57 – 0.59	172.4
Nitric Acid Treated	100.00	0.56 – 0.58	175.4
Zinc Chloride Treated	100.00	0.55 – 0.57	178.6

3.3 Structural analysis of the fiber

Scanning Electron Microscopy (SEM) was used to study the surface morphology of the untreated and treated fibre. Figure 1 shows the SEM of untreated and sample treated with sodium hydroxide. It was observed that the surface of the untreated sample was rough; showing presence of wax, lignin and some extractives but the one treated with sodium hydroxide was smooth. The smoothness indicated that the treatment was effective in removing the extractives and lignin content of the fibre. Fourier Transformed Infrared Spectroscopy (FTIR) analysis was used to characterize the untreated natural fiber and that treated with various chemicals.

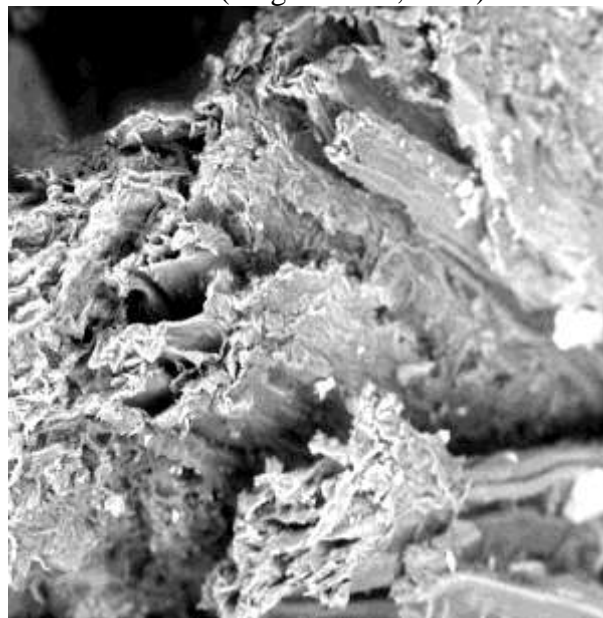
Figure 2 shows the functional groups present on the untreated and fibre treated with different chemicals. All the spectra revealed broad and intense peak at 3400cm^{-1} . This band suggested hydrogen bonded O–H stretching vibration from the cellulose and lignin structure of the fibre (Oh et al., 2005; Kondo 1997). The peak around 1636cm^{-1} present on all the fibre represents the C=O of the hemicellulose (Yan et al., 2009). The bands at 1457cm^{-1} , 1374cm^{-1} , 1418cm^{-1} , 1269cm^{-1} and 1031cm^{-1} were characteristics of C–H, C–O deformation, bonding or stretching vibrations of many groups in lignin and carbohydrates (Yokoi et al., 2003; Ishiada et al., 2007; Meszaros et al., 2007). The peaks around 668cm^{-1} , 517cm^{-1} and 407cm^{-1} can be said to be originated from the β - glucosidic linkages between the sugar units in the hemicelluloses and celluloses (Bledzki et al., 2010).

After the treatment with sodium hydroxide, the bands at 1732cm^{-1} and 1245cm^{-1} attributed to the stretching vibrations of C=O and C–O groups respectively disappeared. These kinds of groups are present in lignin and hemicelluloses structures. This disappearance can be attributed to the removal of lignin and hemicelluloses components of the fibre. The band at 1318cm^{-1} is related to the vibration $\nu(\text{C}–\text{O})$ of esters, ethers and phenols groups attributed mainly to the presence of waxes in the epithermal tissue (Herrera – Franco and Valadarez – Gorizalea 2005) and the

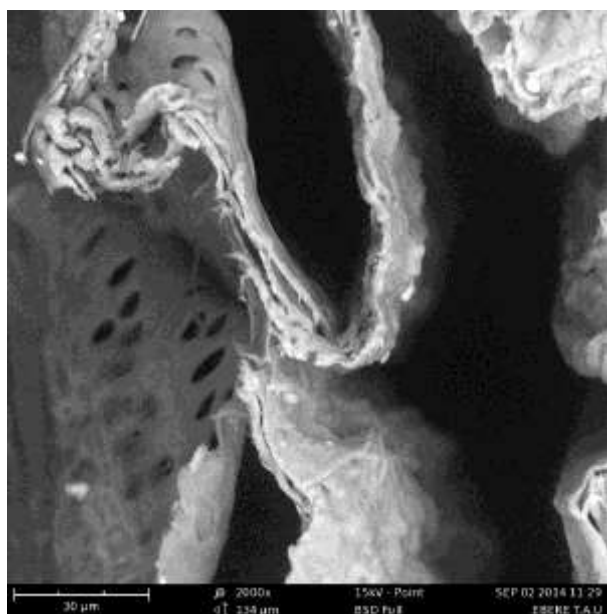
disappearance of this band on the treated fibre results from the removal of those waxes.

After acetylation reaction, new acetyl groups were added to the cellulose as indicated by vibration at 1732cm^{-1} and 1734cm^{-1} (C=O). The spectrum of unmodified cellulose showed an absorption peak at 1374cm^{-1} attributed to the C–H bending vibration. After esterification, the added contribution of acetyl (C–CH₃) stretching vibration intensified this absorption peak (Mokaloba and Batane, 20014).

The nitric acid used for the treatment tends to oxidize the hydroxyl groups from cellulose in the fibre surface to carboxyl groups giving the fiber a soft cationic potential (Rayaes et al., 1998; Shukla and Pai, 2005). This oxidation was confirmed by the spectra of the nitric acid treated fibre at 1732cm^{-1} and 1734cm^{-1} . The carbonyl signal initially present was from lignin and hemicelluloses, after the treatment, the formation of carboxyl group was observed, whose axial vibration of C=O intensified the peak. Between 1370 and 1390cm^{-1} , the absorption peaks referred to a symmetrical and asymmetrical deformation of C–H in cellulose and hemicelluloses groups. In the spectra of the treated fibre, these bands were more accentuated. This can be an indicative of larger exposition of cellulose and hemicelluloses on the fibre surface (Brigida et al., 2010).

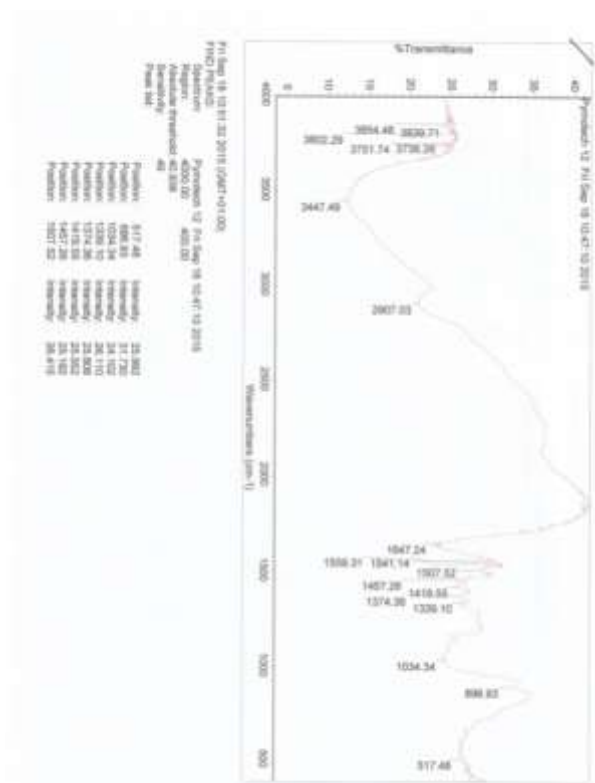


(a)

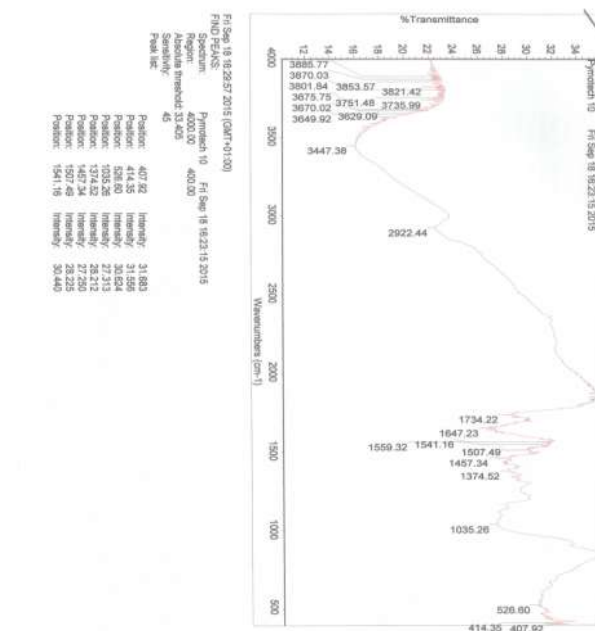


(b)

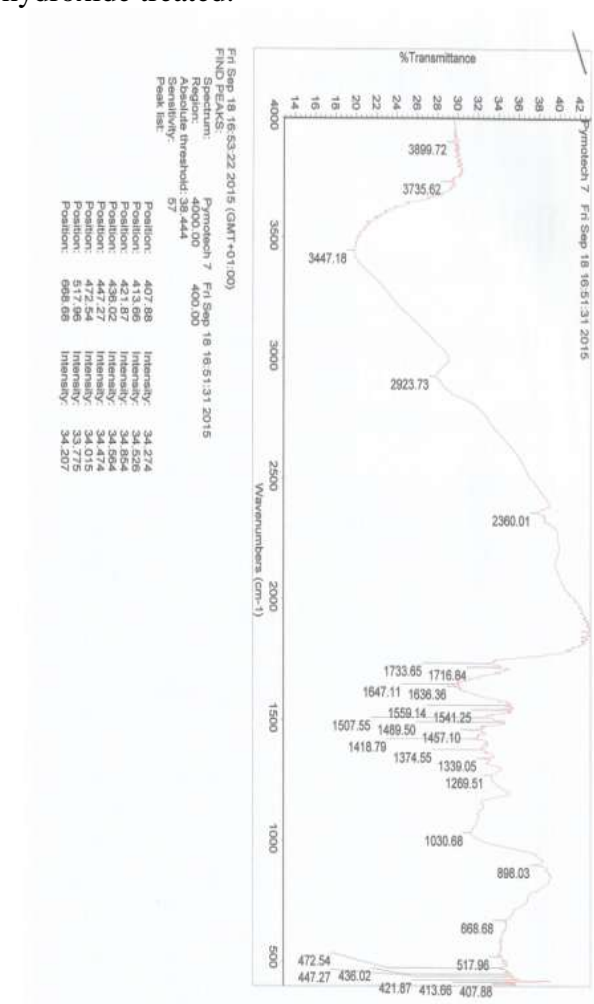
Figure 1. SEM micrographs of *Ampelocissus leonensis* fibre (a) untreated (b) sodium hydroxide treated.



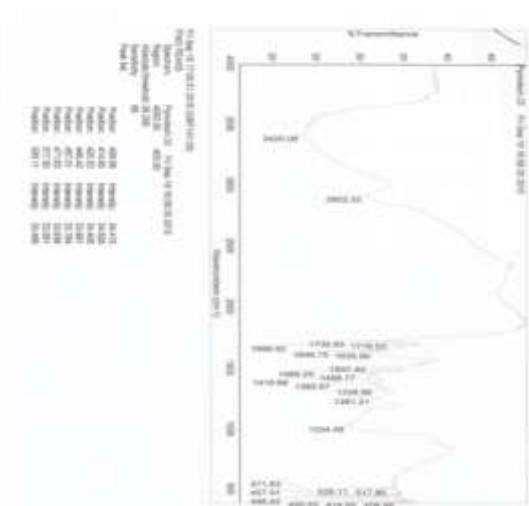
(b) Sodium hydroxide treated



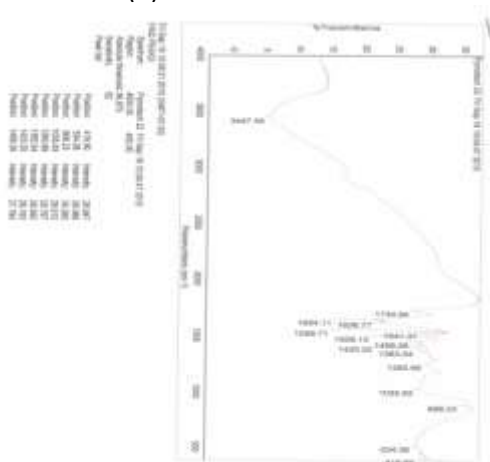
(c) Acetic Anhydride



(a) Untreated



(d) Nitric acid treated



(e) Zinc Chloride treated

Figure 2. FTIR Spectra of the fiber

1. Effect of chemical treatment on properties of the *Ampelocissus Leonensis* fiber

Several chemicals were used to modify the surface of the fibre. The surface modification improves the fibre surface properties (Wang, 2004). Chemical treatments were applied to the fibre with the aim of changing surface tension and polarity through modification of fibre surface (Scandola et al., 2000). The following chemicals; sodium hydroxide, acetic anhydride, nitric acid and zinc chloride were used to treat the fibre.

A: Effect of chemical treatment on the tensile strength of the fiber

The effect of different types of chemicals used on the tensile strength of the fibre was studied to understand the magnitude of variation resulted due to chemical types used. The

chemical effects were studied at optimum chemical concentrations and 50 minutes pre-treatment time. Fig. 3 shows the tensile strength of the fibre treated with different chemicals with the untreated sample serving as control. It is clear from the plots that sodium hydroxide had highest effect on the tensile strength of the fibre. This was as a result of improved fibre surface structure which resulted from the removal of cementing substances like lignin and hemicellulose during the treatment. All the chemically treated fibres had improved tensile strength compared to the untreated fibres. Cellulose gives the strength, stiffness and structured stability to the fibre, and is the major framework components of the fibre. Lignin and hemicelluloses produce the adhesive to hold the cellulose framework structure of the fibre together, reducing the permeation of substances to the cellulose. The chemical treatment used resulted in the removal of certain portions of hemicellulose, lignin and other extractives covering materials. As a result, fibre surface became cleaner as evidenced in the SEM analysis (Fig. 1). In other words, finer surfaces become more uniform due to the elimination of micro voids and thus stress transfer capacity between the ultimate cells improved. This resulted to higher tensile strength when compared with the untreated fibre.

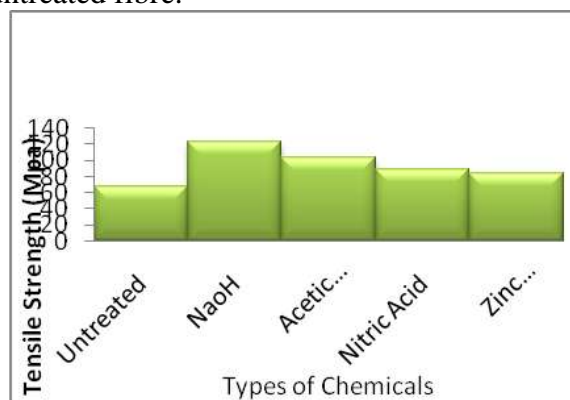


Figure 3: Tensile strength of the untreated and treated fiber

A: Effect of chemical treatments on Water absorption of the fibre

From Fig.4 shows the water absorption of treated and untreated fibre. It was observed that the untreated fibre absorbs more water than the

treated fibres. The water absorption by the untreated fibre is due to the fact that the untreated fibre contains very high percentage of hemicellulose in its structure. Also, in natural cellulose fibers, a hollow cavity called lumen exists in the unit cell of the fibres which provides more path ways for water to diffuse into the fibre. The effect of chemical treatment on the water absorption showed that the chemical treatment process decreased the dissemination of water into the fibre and the fibre became more hydrophobic. This reduction is likely due to the reduction of the hemicellulose components from the raw fibre. Chemical treatments result in the partial dissolution of hemicellulose (Ring et al., 2001). It was observed that the mercerized fiber absorbed the least water compared to the fiber treated with other chemicals. This can be attributed to the removal of the non-cellulose constituents' pectin, lignin, hemicellulose and impurities from the fibre surface by mercerization. The results equally indicated that acetylation process decreased the dissemination of water into the fibre and it became more hydrophobic. This is due to the replacement of hydroxyl groups by hydrophobic acetyl groups. Chemical modification takes place as the acetic anhydride substitutes the cell wall hydroxyl groups with acetyl groups making the surface more hydrophobic (Paul et al., 1977). The same trend was observed on the nitric acid and zinc chloride treated fibre.

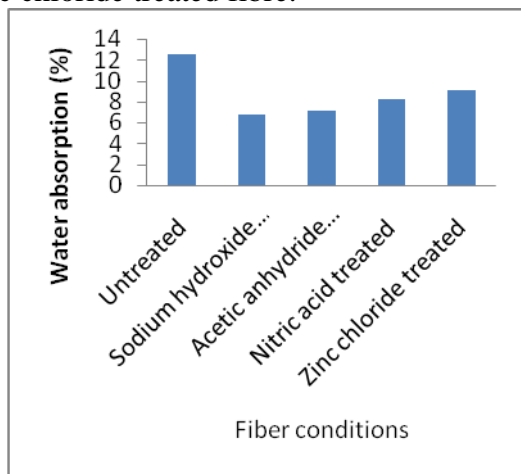


Fig.4: Percentage water absorption for *Ampelocissus Leonensis* fiber

Effect of chemical treatment on the Aspect ratio of the fibre

Figure 5 shows the aspect ratio of the untreated and treated fibre. From the results, it can be seen that the aspect ratio of the treated fibre is higher than the untreated fibre. This is as a result of the effect of the chemical treatment of the fibre led to fibrillation of the fibre bundle, thus, reducing the fibre diameter without affecting the fibre length, thereby increasing aspect ratio (L/D).

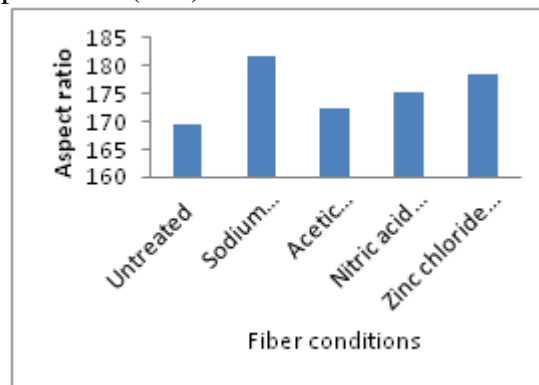


Fig. 5: Effect of chemical treatments on fibres' aspect ratio.

4.0 CONCLUSION

The work has been able to use several chemicals to modify the surface chemistry of natural fibre (*Ampelocissus Leonensis*) and consequently enhancing their properties. It was found that chemical treatment was successful in removing the waxy encrusting substances from the surface of the fibre which ultimately enhanced the studied properties. Alkaline treatment was found to be more effective in removing the surface impurities of the fibre and this was largely responsible for the outstanding improvement in the properties of alkaline treated fibre in comparison with those of other chemicals.

REFERENCES

- ASTM Standard D1037-12: Standard Test Method for evaluating properties of wood base fibre and particle panel materials. ASTM International, West Conshohocken, PA.
- Bei Wang (2004) Pre-treatment of flax fibers for use in rotationally molded biocomposites. A thesis submitted to the college of graduate studies and research department of agricultural

and Bioresource Engineering university of Saskatchewan Saskatoon, Saskatchewan.

Bledzki, A. K., Fink, H. P., Specht, K. (2004). Unidirectional hemp and flax EP- and PP-composites: Influence of defined fiber treatments. *Journal of Applied Polymer Science*, 93(5), 2150-2156.

Bledzki, A.A., Mamun, A.A., Volk, J., (2010). Barley husk and coconut shell reinforced polypropylene composites: The effect of fiber physical, chemical and surface properties”, *Composite Science and Technology*, 70, pp. 840- 846

Brigida, A. I. S., Calado, V. M. A., Goncalves, L. R. B., Coelho, M. A. Z. (2010). Effect of chemical treatments on properties of green coconut fiber. *Carbohydrate polymers*, 79. Pp. 832-838.

Cao, Y., Shibata, S., Fukumoto, I. (2006). Mechanical properties of biodegradable composites reinforced with bagasse fibre before and after alkali treatments. *Composites Part A: Applied Science and Manufacturing*, 37(3), 423.

Cutter, Andrea Gillian (2008) Development and Characterization of renewable resource structure composite Electronic thesis and material dissertation UC San Diego.

Doan, T.T.L., Gao S., Madar, E. (2006), Jute Polypropylene composites. Effect of matrix modification composites *Science and Technology*, 66 (7 – 8), pp 952-66.

Ejikeme, M.E., Ejikeme, P.C.N., Abalu, B.N. (2013). RSM Optimization process for uptake of water from Ethanol water solution using oxidized starch. *Pacific Journal of Science and Technology*. 14(2): 319 – 329

Ejikeme, Patrick. C. N, Onukwuli, D. O., Ejikeme, Ebere M. (2014a). Optimization of Chemical Treatment Conditions for Adenia Lobata Fiber Using CCD. *International Journal of Engineering and Innovative Technology (IJEIT)* Volume 4, Issue 6.

Ejikeme Patrick C. N., Ejikeme Ebere M. Nwosu David C. (2014b). Effect of chemical treatments on ampelocissus Cavicaulis fiber. *International Journal of Application or Innovation in Engineering & Management (IJAIEM)*. Volume 3, Issue 11,

El shekeil, Y. A., Sapuan, S. M., Khalina, A., Zainudin, E. S., Al-shuja, O. M. (2012). Influence of chemical treatment on the tensile properties of Kenaf fiber reinforcement thermoplastic polyurethane composite. *Express polymer letter* vol. 6, No. 12 pp 1032 – 1040.

Herrera-Franco, P. J., Valadarez-González, A. (2005). A study of the mechanical properties of short natural-fiber reinforced composites. *Composites: Part B*, 36, 597–608.

Herrera-Franco, P. J., Valadez-Gonzalez, A. (2005). Fiber-Matrix Adhesion in Natural Fiber Composites. *Natural Fibers, Biopolymers, and Biocomposites*, A. K. Mohanty, M. Misra, and L. T. Drzal, eds., CRC Press, Boca Raton.

Irene S., Fashion, Salah Ethaggar, Hatem Elay at (2010). Experimental investigation of Natural fiber reinforced polymer. *Material Sciences and Applications*, 3, pp 59-62.

Ishida, Y., Goto, K., Yokoi, H., Tsuge, S., Ohtani, H., Sonoda, T., Ona, T. (2007). Direct analysis of phenolic extractives in wood by thermochemolysis-gas chromatography in the presence of tetrabutylammonium hydroxide. *J. Anal. Appl. Pyrolysis*, 78, 200–206

Kondo, T. (1997). The assignment of IR absorption bands due to free hydroxyl groups in cellulose. *Cellulose*, 4, 281–292.

Lipsa Mahapatra, Sweta Mohanty (2007). The effect of rain rate on jute fibre composites. Thesis submitted to department of Metallurgical Material Engineering Natural Institute of Technology Rourkela.

Mészáros, E., Jakab, E., Várhegyi, G. (2007). TG/MS, Py-GC/MS and THM-GC/MS study of the composition and thermal behavior of extractive components of Robinia pseudoacacia. *J. Anal. Appl. Pyrolysis*, 79, 61–70.

Mohanty, A. K., Misra, M., Hinrichsen, G. (2000). Biofibres, biodegradable polymers and biocomposites: An overview. *Macromolecular Materials and Engineering*, 276-277(1), 1-24.

Mokaloba, N., Batane, R. (2014). *The effects of mercerization and acetylation* treatments on the properties of sisal fiber and its interfacial adhesion characteristics on polypropylene

International Journal of Engineering, Science and Technology Vol. 6, No. 4, pp. 83-97.

Mwaikambo, L. Y., Ansele, M. P. (1999). The effect of chemical treatment on the properties of hemp, sisal, jute and kapok fibers for composites reinforcement 2nd international wood and natural fiber composites symposium June 28 – 29 in Kassel/Germany.

Nurul Munirah Abdullah, Ishak Ahmed .(2012). Effect of chemical treatment on Mechanical and Water sorption properties of coconut fiber unsaturated polyester from Recycled PET. International scholarly Research Network. Vol 272, article ID 134683, 8 pages.

Oh, S.Y., Yoo, D.I., Shin, Y., Kim, H.C., Kim, H.Y., Chung, Y.S., Park, W.H., Youk, J.H. (2005). Crystalline structure analysis of cellulose treated with sodium hydroxide and carbon dioxide by means of X-ray diffraction and FTIR spectroscopy. Carbohydr. Res., 340, 2376–2391.

Paul, A., Joseph, K., Thomas, S. (1997). Effect of Surface Treatments on the Electrical Properties of Low-Density Polyethylene Composites Reinforced with Short Sisal Fibers. Composites Science and Technology, 57(1): 67-79.

Pott, G. T. (2004). Natural Fibers with Low Moisture Sensitivity. Natural Fibers, Plastics and Composites, F. T. Wallenberger N. E. Weston, eds., Kluwer Academic Publishers, Norwell, MA.

Reyes, J., Peralta-Zamora, P., & Durán, N. (1998). Hidrólise enzimática de casca de arroz utilizando-se celulasas. Efeito de tratamentos químicos e fotoquímicos. Química Nova, 21, 140–143.

Scandola, M., Frisoni, G., Baiardo, M. (2000). Chemically modified cellulosic reinforcements. In Book of Abstracts 219th ACS National Meeting, 26-30. Washington, D.C.: American Chemical Society.

Shibata, S., Fukumoto, I. (2007). Prediction of flexural modulus of the biodegradable composites made from bamboo and kenaf fibers. Key Engineering Materials, 334-335 I, 53.

Shukla, S. R., Pai, R. S. (2005). Adsorption of Cu(II), Ni(II) and Zn(II) on modified jute fibres. Bioresource Technology, 96, 1430–1438.

Wang, H. M., Wang, X., (2004) Bast fiber degumming; Textile, 2, 28-30

Yan, T., Xu, Y., Yu, C. (2009). The Isolation and Characterization of Lignin of Kenaf Fibers, Journal of Applied Polymer Science, Vol. 114, No. 3, 2009, pp. 1896-1901.

Yokoi, H., Nakase, T., Goto, K.; Ishida, Y., Ohtani, H., Tsuge, S., Sonoda, T., Ona, T. (2003). Rapid characterization of wood extractives in wood by thermal desorption-gas chromatography in the presence of tetramethylammonium acetate. J. Anal. Appl. Pyrolysis, 67, 191–200.