American Journal of Engineering Research (AJER)
e-ISSN: 2320-0847 p-ISSN : 2320-0936
Volume-4, Issue-9, pp-168-175
www.ajer.org
Research Paper Open Access

# The Effect Of Chemical Treatment On Tensile Properties Of Soil Retted Entada Mannii Fibres.

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***ABSTRACT :***This research work was carried out to investigate the influence of chemical treatment on tensile properties of soil extracted Entada mannii *fibre.* Entada mannii *fibre was extracted by soil retting method and after which the fermented fibres were washed with distilled water and dried in an oven. The dried fibres obtained were treated with sodium hydroxide (NaOH) and potassium hydroxide (KOH) solutions and percentages of their constituents were determined. The fibre strands were characterized using the scanning electron microscope while the tensile properties were determined using instron universal tensile testing machine.The tensile results revealed that, fibres treated sequentially with (KOH) behaves superiorly than the NaOH treated and untreated fibres. The alkaline treatment also enhanced the removal of lignin and hemicellulose in treated fibres which may be detrimental to the interfacial bonding strength compared to untreated fibres. However, the results of SEM analysis revealed rough surfaces due to removal of impurities, waxes and the fibre constituents (lignin and hemicellulose) on treated while the untreated revealed smooth surface with the presecence of the constituents.*

***Keywords -****Chemical treatment; Soil retting; Extracted fibre; Constituents; Fermented*

## I. INTRODUCTION

In recent years, natural fibres appear to be the outstanding materials which come as the viable and abundant substitute for the expensive and nonrenewable synthetic fibres [1]. The use of plant fibres as reinforcements of polymer is attracting the interest of industries and researchers because of the wonderful potentials and their unique properties [2]. Natural fibres like sisal, banana, jute, oil palm, kenaf and coir has been used as reinforcement in thermoplastic and thermoset composite for various engineering applications [3]. They are environmentally friendly, fully biodegradable, abundantly available, renewable, cheap, low density, specific strength and stiffness compared to glassfibres, carbon and aramid fibers [4]. Natural fibres properties are influenced by many factors, including plant type and variety, growth conditions, and the method used to extract the fibre bundles [5]. Among natural fibres for composites are the bast fibers, kenaf, flax, ramie and hemp extracted from the stems of plants due to their very good mechanical properties [6].

However, one of the bast plant with great potentials and that has not much received attention from researchers is *Entada mannii bast fibre which b*elongs to the family (Oliv.) Tisser. leguminous mermosaesae, liana plant. The plant is 2 to 3m high semi-climber which grows in the tropical forest of Nigeria, Gabon and Madagascar [7]. They show extreme variations in mechanical properties which includes; the stiffness in the elastic range of bending, torsion, and tension as well as other properties in the nonelastic range up to failure, toughness, extensibility, and critical strain to mention a few make them suitable for use in composites reinforcements [8-9]. Extraction of the fibres from the bast by conventional method of retting is considered for this study. Retting is a microbial process that breaks the chemical bonds that hold the stem together and allows separation of the bast fibres from the woody core. The two traditional types of retting are soil retted and water retting [9]. To extract fibres for industrial uses, stems are retted to separate fibre from non-fibre stem issues. In this process, bast fibre bundles are separated from the core, epidermis, and cuticle and are also separated into smaller bundles [9-11] .Currently, soil retting is the primary process used for the industrial production of bast fibres nevertheless water retting is still carried out in some place [12]. In soil retting, stalks are laid on the ground, and pectins are attacked by pectinolytic microorganisms, mainly aerobic fungi [13]. Soil retting is most popular in Europe although it is strongly dependent on the geographical location, produces coarser and lower quality fibres than those produced using water retting technique [14]

Hence, the use of natural fibre in composite reinforcement developed some drawback due to pertinent characteristics such as fibre incompatibility, fibre aggregations that occur during processing and compounding showing a poor fibre-matrix adhesion due to presence of the fibre constituents such as lignin, hemicellulose, wax and impurities [15]. The adoption of chemical treatment on natural fibres which removes these fibre constituents improvises the compatibility of the fibre and the matrix and better surface adhesion [16]. Alkaline treatments such as potassium hydroxide and sodium hydroxides applied to fibre surface during treatment is called mercerization [17]. They promotes the removal of the constituents, impurities and oil soluble in alkaline solutions thereby reduced the fibre diameter and level of aggregations of the fibres, improved the mechanical properties and exposed the fibre surface and becomes rougher [18].

Therefore this research work aimed at assessing the effect of soil retting extraction method on the mechanical properties of *Entada mannii* fibre.

## II. MATERIALS AND METHOD

### *2.1 Materials*

The *Entada mannii* plant stem wasobtained from Ikare Akoko, Ondo state, Nigeria. The *Entada mannii* plant was identified at the Federal College of Forestry Herbarium, Ibadan, Nigeria. NaOH and KOH were used to separate the fibre from their constituents.

### *2.2 Method*

#### *2.2.1 Extraction of fibre (Soil retting)*

The harvested *Entada mannii* stem plant was extracted by conventional soil retting method as shown in Figure 1.*Entada mannii* fibres were stripped from the stem bundles and buried in moist environment for 20 days during which the bark tissues undergo fermentation and the fibres were separated from the bark bundle as strands.However, the fibres obtained were washed with distilled water in order to separate fibre strands from undesirable foreign matters and dried in an oven at 50 $^{0}$C for 48 h. The fibre were characterized by employing scanning electron microscope while the fibre constituents and mechanical properties were also evaluated.

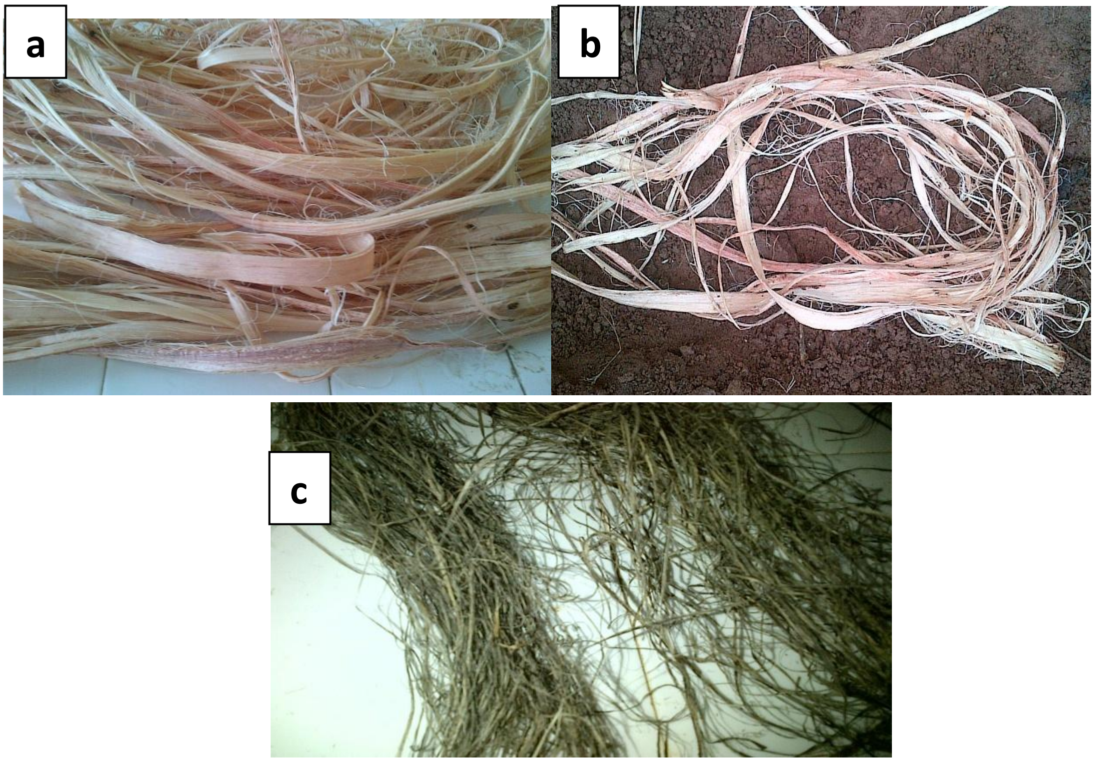


Figure 1 (a) Hand stipped fiber from the *Entada mannii* stalk (b) Burying of the fibers in a moist environment (c) Soil retted fibers after 20 days.

### *2.2.2 Fibre surface treatment*

*Entada mannii* fibres were treated with 5% NaOH and 5% KOH solutions differently in a shaker water bath at 50 $^{0}$C for 4 h. The insoluble residue was delignified at pH 3, washed with distilled water in order to remove mineral traces and dried in an oven at 60 $^{0}$C for 48 h in order to removes fibre moisture. The untreated fibres were left as control.

### *2.2.3 Determination of the fibre constituents*

In general natural fibers are hydrophilic in nature and they absorb or release moisture depending on environmental conditions. Amorphous cellulose and hemicellulose that are present in the natural fiber are mostly responsible for the high moisture absorption, since they contain easily accessible hydroxyl groups which give a high level of hydrophilic character to fiber [19]. In order to determine the fiber constituents by gravitational method [20]: Lignin, Hemicellulose and waxes contents on the *Entada mannii* fibres has to be removed in order to enhance proper bonding between the fibre and the matrix.

Lignin content

2.5 g of *Entada mannii* fibres was weighed mixed with 75% sulphuric acid concentration and the mixture was kept in an ice bath and stirred continuously for 12 h.The residue was filtered with purpling cloth and washed severally with hot water to remove the acid left and transferred to a crucible. The fibre residue was oven dried at 105 $^{0}$C for 2 h and cooled in a desiccators.

Cellulose Content

2 g of the treated and untreated fibres were weighed and dried in an oven at 100 $^{0}$C for 3 h and 25 ml of distilled water was added .1 ml of concentrated nitric acid was added and the mixture was placed in a thermostat water bath and stirred continuously at 80 $^{0}$C . The fibre residue obtained was placed inside a weighed crucible in an oven at 105 $^{0}$C for 20 h. The final residue obtained was weighed and cooled in a desiccator. The percentage of cellulose was calculated by the measuring weight difference.

Hemicelluloses Content

*Entada mannii* sample of 0.5g was weighed into a beaker while 24% KOH and NaOH solution was added to the beaker and stirred continuously for 2 h. The sample mixture was filter with purpling cloth, washed with additional KOH and NaOH solution and the filtrate was collected into another separate beaker where alcohol was added to the sample precipitate formed. The precipitated hemicelluloses were isolated by centrifuging for 10minutes. The samples were dried in oven for 2 h at 105 $^{0}$C and later transferred into desiccators and allowed to cool for 30 minutes and weighed. The weight of the precipitate was recorded and the percentage was calculated.

### *2.2.4 Determination of the tensile properties of the fibre*

The single fibre pull out test was performed on treated and untreated single fibre according to ASTM-638D [21] test standards.This was done by fixing the sample on the grips of the machine after which will operated automatically. Tensile testing machine equipped with a 5 KN load cell up to a strain of 8% at a cross-head speed of 50 mm/min. 6 specimens were tested and their average fibre tensile strengths and modulus were obtained.

### *2.2.5 Fibre Characterization*

Scanning electron microscope of Model JEOL JSM-7600F was used for the morphological characterization of the *Entada mannii* fibre surface. The fibres were clean thoroughly, air-dried and coated with 100 Å thick irradium in JEOL sputter ion coater at 15kV.

## III. RESULTS AND DISCUSSION

### *3.1 Chemical Treatment*

The effect of chemical treatment on soil retted treated and untreated Entada *mannii* fibres are presented in Fig. 2.It is observed that KOH and NaOH treatment removed fibre constituents that could be responsible for poor fibre –matrix interfacial adhesion from the fibre surface. The percentage of cellulose in the *Entada mannii* treated fibre increases from 62.76 %untreated fibre to 66.95 % NaOH and 69.06 % KOH which increases by 7% and 10% respectively. It is evident that, after treatment, the cellulose percentage increased with KOH treatment and which effectively removes adhesives in the fibre cell wall, such as lignin, pectins and hemicelluloses covering the cellulose. Hence this enhanced the fibre surface energy and interfacial adhesion with the matrix.

The fibre surface after treatment revealed pores and hole within the fibre which could be attributed to the removal of hemicellulose and lignin. This effect became more pronounce in the KOH treated than NaOH and untreated fibres. Generally alkali treatment influences all the constituents of natural fibres due to presence of hydroxyl group undergo surface modification by chemical treatment improve the properties of the fibres [22-23].

Hemicellulose was hydrolyzed by the action of KOH and NaOH solutions, whereas lignin was removed. Removal of hemicellulose and lignin increased the relative amount of cellulose contents on the treated fibres and thus NaOH and KOH treatments dissolved a portion of hemicellulose and lignin constituents from the fibres. NaOH play the critical role in removal of lignin by means of alkaline cleavage in the lignin, which may be accompanied by condensation reactions. Lignins can be completely attacked and removed without any residue left on the fibre after NaOH treatment, but the rate of lignin removal is dependent on the NaOH concentration [24, 25].

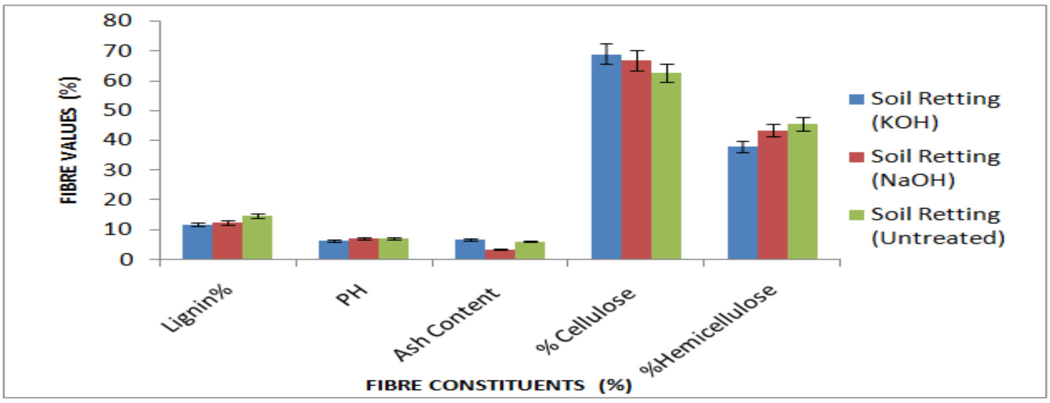


Figure 2 Plot of *Entada mannii* fibreConstituents and PH Values for both treated and untreated fibres.

### *3.2 Tensile Strength*

Figure 3 show the response of the fibres to tensile strength of treated and untreated soil retted *Entada mannii* fibre. It is observed that KOH and NaOH treated fibre have the highest tensile strength of 1.6 5N/mm2, 1.33 N/mm2 and least with the untreated fibre of 0.8 N/mm2.The increase in tensile strength is attributed to removal of hemicellulose and lignin constituents covering the fibre surface which provided strength, stiffness and structural stability of the fibre as the cellulose content increase. Srinivasa et al. [26] also reported that, the areca fibres were treated in a solution of potassium hydroxide (KOH) and alkali treatment NaOH removed hemicellulose, waxes, impurities and lignin from the surface of natural fibers with increase in their tensile properties.

The agglomeration of fibre constituents occurs in the untreated fibre with the presence of these constituents and impurities thereby reduced surface energy and tensile properties of the untreated fibre. Hence, the untreated fibres exhibited lower tensile strength as compared to the treated fibers resulting in easy deformation of fibre micro fibrils during tensile loading and offer a likely poor fiber-matrix interface adhesion.

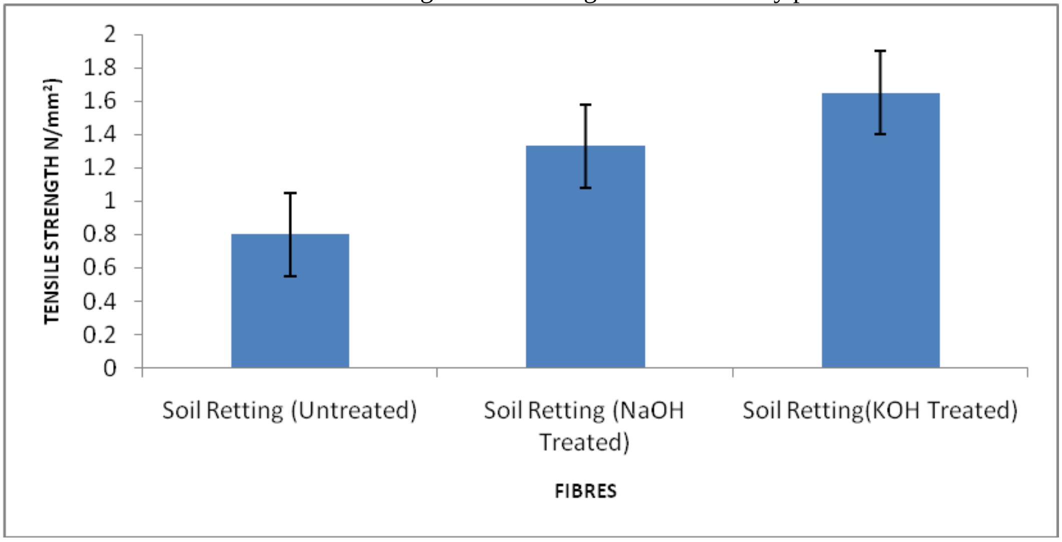


Figure 3 Tensile strength for both treated and untreated fibers

### 3.3 Young's Modulus of the Fibers

The results of the young's modulus of elasticity of the fibres are shown in Figure 4. Young's modulus is the slope of the stress-strain curve within the range of proportionality before yield. At the yield stress, a large amount of deformation takes place at constant stress [27-28]. The soil retted fibres treated with KOH gave the best young modulus of 59.8 7N/mm2 followed by fibres treated with NaOH of 55.53N/mm2 and have the least value of 42.95 N/mm2 of untreated fibre. Alkaline treatment influences surface modifications, stiffness and improved the porosity of the *Entada mannii* fiber due to the hydroxyl group within the fibre. However, untreated fibres shows a lower young modulus due to the prescence of the moisture within the fibre. Natural fibres are hydrophilic in nature and have tendency to absorbed moisture than treated fibres. It is also evident that untreated fibre surface is found to be smooth due to the presence of lignin covering the fibre surfaces.

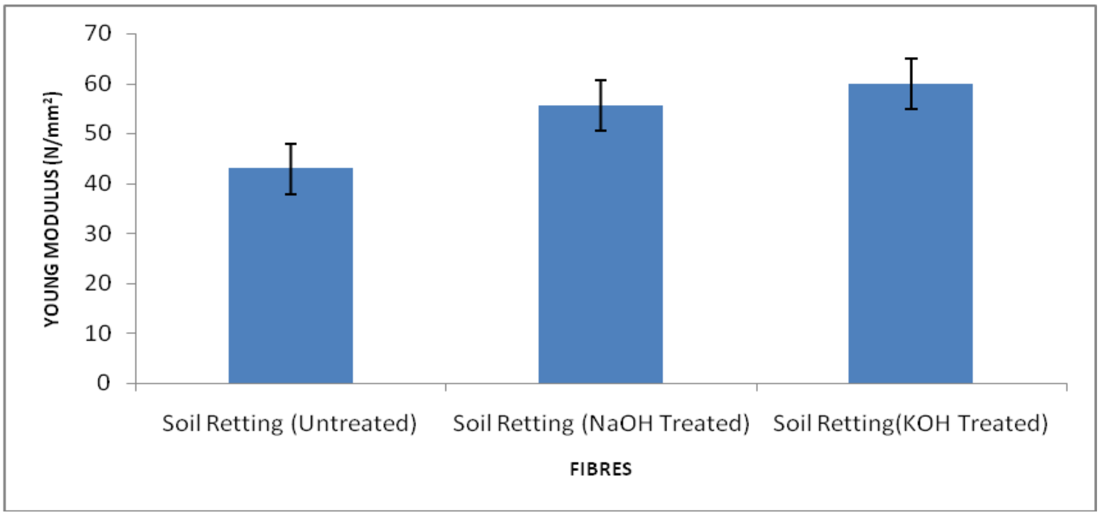


Fig.4 Young modulus against fibers for both treated and untreated fiber

### 3.4 Elongation at break

The elongation at break for the treated and untreated soil retted *Entada mannii* fibres are shown in Figure 5.The elongation at break or strain is expressed as the ratio of the total deformation to the initial dimension of the material body in which the force is applied [29]. An increase in elongation for the KOH treated was demonstrated compared to the NaOH and untreated fibres. This indicated that higher elongation of fibres indicated higher ductility and lower elongation indicates lower ductility of materials [30]. The elongation at break for the fibres experienced a significant drop with untreated fibres as a result of the impurities and waxes within the fibre as compared to the treated fibres. Hence the stiffness is interrupted and lead to the brittle nature of the untreated fibre.

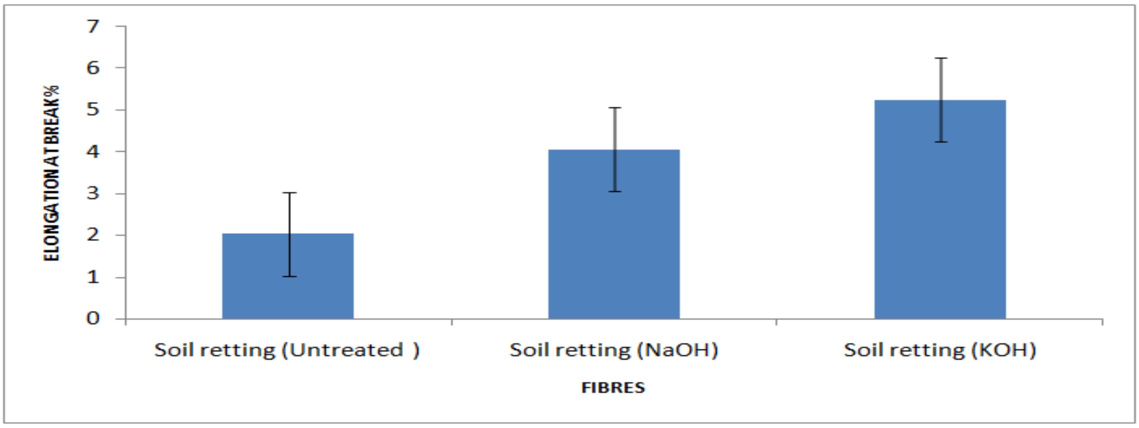


Fig.5 Elongation of fibers for both soil retted treated and untreated fibers

***3.5 The scanning electron micrograph results***

Fig.6a shows SEM micrographs of untreated *Entada mannii* fibres and constituent; cellulose, hemicellulose and lignin. The results show that untreated fibre were black in colour with smoother surface as a result of presence of the lignin, hemicellulose, wax, fatty and dirt than treated fibres. It is evident that external surface features of fibres such as contours, defects and damage and surface layer are deposited on the untreated fibre surfaces. Smooth surface of the untreated fibre connotes a lot of defects and fibre damages were observed on the surface due to prescence of impurities.

Fig. 6b shows the soil retted NaOH treated fibre where surfaces appeared to be rough with increase in the cellulose. Alkaline treatment was found to be very effective in removal of lignin and hemicellulose and thereby increases the roughness of the surface which deposited pores to greater extent. Lignin acts as a cement between fibrils and when removes allows an increase in surface area thereby creating pore on the surface and improves the fibre/matrix adhesion [31]. The rough surface was observed as a result of removal of waxes and oil from the surface and thereby increases the overall roughness as shown in the Fig.6 b and 6 c.The chemical will breakdown the composite fibre bundles into smaller fibres and therefore a rough fiber surface topography is developed [32].

However, the removal of the extractives by KOH treatment in Figure 6c revealed the pore and pits. There is possibilities of Parenchyma cells that are naturally constituent of lignocelluloses fibres and presence of globular protrusion which are fatty deposit called Tylose. Exposing the rough surface with globular marks thereby increases the surface energy. The presence of the pits and globular marks affect the chemical treatment and important for the increase in the effectiveness of the surface area and higher increase in surface roughness of the fibre.

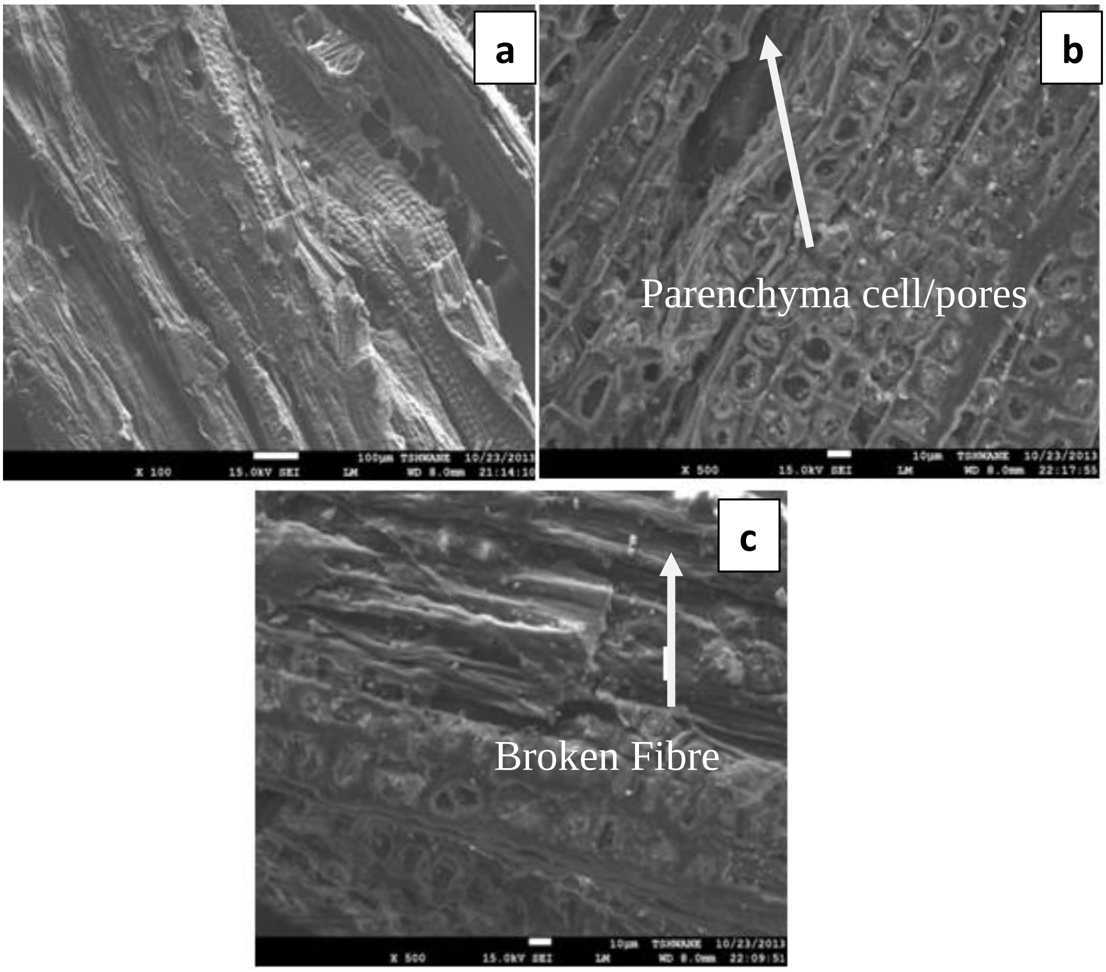


Figure 6 Scanning electron micrograph of both treated and untreated soil retted fibers

# IV. CONCLUSION

The influence of soil retting extraction method and chemical treatment on tensile properties of *Entada mannii*(Olive Tisserant) plant stem fibres was investigated. The results show that:

1. The alkaline treatment plays a significant role in improving the mechanical properties (tensile strength, elastic modulus and elongation at break) of the *Entada mannii* fiber and decreasing the moistures absorption of the fibres.
2. KOH treated fibers gave the optimum tensile strength properties due to the removal of lignin and hemicelluloses from the fibre surface, as compared with untreated and NaOH treated fibers.
3. SEM analysis revealed that treated fiber surface differs after the removal of the lignin and hemicellulose which revealed the roughness of the fibre surface as compared than the smooth surface of the untreated fibers.
4. KOH and NaOH treated fibers show the removal of the lignin and the hemicellulose on the surface for proper adhesion but created pit and voids which could improve of bonding strength of the fibre.

***ACKNOWLEDGEMENTS***

I wish to acknowledge the following organizations for their support; Regional Initiative in Science Education (RISE), Science Initiative Group (SIG) and African Materials Science and Engineering Network (AMSEN). Prototype Engineering Development institute (PEDI-NASENI), Ilesha, Osun State Nigeria is gratefully appreciated for the time and support given to Oluwayomi Balogun.

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