FTIR CHARACTERISATION AND CHEMO- MECHANICAL DISTINCTION OF RETTED KENAF FIBRES.

CARACTERIZACIÓN FTIR Y DISTINCION QUIMIO-MECANICA DE FIBRAS RETRASADAS DE KENAF.

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ABSTRACT

Inadequate information on the binding characteristics of kenaf biomass at the molecular level has adversely affected the modifications of its cellulose-hemicellulose-lignin structure. This study was undertaken to assess the characteristic features and chemo-mechanical properties of kenaf fibres retted under three different media namely: tank, stream and ribbon. Fourier transform infra-red (FTIR) spectra analysis as well as the ASTM standard for tensile tests were applied. The results showed that the broad bands occurred at the range of 3312- 3420 cm^{-1} for all the retted fibres were due to the presence of hydroxyl (–OH) group while the peaks were obtained at 1635.76, 1635.34, and 1730.69 cm^{-1} for stream, tank and ribbon retted fibres respectively. However, tank retted fibres had the most broad band intensity at 3419.78cm^{-1} while ribbon fibres had the highest absorption peak of

1730.69cm^{-1} corresponding to C=O stretching of the acetyl group in hemi-cellulose. Stream retted Kenaf fibres had the highest tensile strength followed by the tank retted fibres while ribbon fibres had the least. Furthermore, there was no significant difference between stream and tank retted Kenaf fibres in terms of tensile modulus and this was significantly higher than that of ribbon fibre. The α-cellulose content of stream retted fibres was slightly higher than that of ribbon fibres while tank retted fibres had the least. From the results, tank retted Kenaf fibre was ranked as 'the best fibre' with the most intensive broad bands and least in lignin and hemi-cellulose content which were regarded as the impurities, gummy and waxy materials, responsible for an easy deformation of the fibre cellular networks.

Keyword: Kenaf fibre, spectra, chemo-mechanical properties.

RESUMEN

La información inadecuada sobre las características de unión de la biomasa de kenaf a nivel molecular ha afectado negativamente a las modificaciones de su estructura de celulosa-hemicelulosa-lignina. Este estudio se realizó para evaluar las características y propiedades quimio-mecánicas de las fibras de kenaf en tres medios diferentes, a saber: tanque, corriente y cinta. Se aplicó el análisis de espectros de infrarrojo por transformada de Fourier (FTIR) y el estándar ASTM para ensayos de tracción. Los resultados mostraron que las bandas anchas ocurrieron en el rango de 3312-3420 cm-1 para todas las fibras enredadas debido a la presencia del grupo hidroxilo (-OH) mientras que los picos se obtuvieron a 1635.76, 1635.34 y 1730.69 cm-1. para la corriente, el tanque y las fibras de la cinta trenzadas, respectivamente. Sin embargo, las fibras en tanque tenían la mayor banda de intensidad a 3419,78 cm-1, mientras que las fibras de cinta tenían el mayor pico de absorción de 1730,69 cm-1 correspondiente a C = O de estiramiento del grupo acetilo en hemicelulosa. Las fibras de Kenaf reticuladas tenían la mayor resistencia a la tracción, seguidas por las fibras del tanque, mientras que las fibras de la cinta tenían la menor. Además, no hubo una diferencia significativa entre la corriente y el tanque de las fibras de Kenaf en términos de módulo de tracción y esto fue significativamente más alto que el de la fibra de cinta. El contenido de α-celulosa de las fibras reticuladas fue ligeramente mayor que el de las fibras de la cinta, mientras que las fibras reticuladas tuvieron el menor contenido. De los resultados, la fibra Kenaf en tanque se clasificó como "la mejor fibra" con las bandas anchas más intensas y menos contenido de lignina y hemicelulosa que se consideraron como

impurezas, materiales gomosos y cerosos, responsables de una deformación fácil del redes de fibra celular.

Palabra clave: fibra de Kenaf, espectros, propiedades quimomecánicas.

INTRODUCTION

The use of sophisticated facilities in agro-fibre analyses is becoming rare due to its high cost and unstable electricity in most developing countries. The distinctive features of agro-fibres (kenaf fibre in particular) is of great concern to the up takers/end users. The ultimate quality of kenaf fibre used either for composite reinforcement, weaving or non-woven materials depends largely on its structural composition. Hence, many studies have emphasized on the lack of available information regarding the behaviour of tensile strength fabrics using properly aligned agro-fibres (Ismail and Hassan, 2014, Zampaloni *et al*. 2007, Aji *et al*., 2009). In the light of this, Sapuan *et al*. (2006) emphasized on the tensile and flexural behaviours of woven banana fibre reinforced composites while Khan *et al.* (2016) investigated the influence of woven structures and direction on the mechanical behaviour such as tensile, flexural and impact tests. From this analogy, one could quickly pre-empts the existing wide knowledge gap of the kenaf fibre structure among the kenaf growers, up takers and industrialists in some countries. Few researches seemed to have focused on this direction, although, Omenna and Oduwaye (2017) highlighted on the pivotal roles of kenaf in boosting the gross domestic products (GDP/GNI) and its impacts in World paper consumption. It is disheartening to note that kenaf with its multi-potentials for rope, produce bags, pulp, paper, fibre glass, fortification of compressed earth blocks and phytoremediation is yet to gain a befitting publicity in most countries like Nigeria. Hence, the objectives of this study were to bridge the knowledge gap by providing fundamental data for future studies on the use of Fourier Transform Infra-Red (FTIR) to determine: molecular orientation, hydrogen bonding, and linkaging of functional groups as well as to assess the chemical and mechanical properties of kenaf fibres retted naturally in three media without any chemical treatments. It has recently been found that FTIR was the most promising technique to examine the changes in the chemical compositions of natural fibres due to inherent defect but there is no literature record of this kind for kenaf fibre.

It is worthy of note that FTIR has been mostly successful in accurate analysis of both major (cellulose, hemicellulose and lignin) and minor (mineral, pectin, waxes) constituents of natural fibres. Cellulose, which acts as the reinforcing material in the cell wall, is the main

constituent of kenaf fibre. The cellulose molecules are laid down in micro fibrils in which there is extensive hydrogen bonding between cellulose chains, producing a strong crystalline structure (Hinterstoisser and Salemen, 2000).Characterization of the hydrogen bonds in cellulose by using various techniques, among which FTIR has proved to be one of the most useful methods. FTIR was used in the hydrogen bonds analysis, determination of structures and chemical compositions, and the morphological characterization for natural fibres like kenaf fibre (Krouit *et al*.2008). Hinterstoisser and Salmén (2000) recently used DMA-FTIR to investigate OH stretching vibration regions between 3700 and 3000 cm^{-1} in the cellulose, and it was established that FTIR was able to examine the nature of molecular chains, crystallinity and their correlations with various bonds.

Kenaf (*Hibiscus cannabinus*, L) is a herbaceous annual plant that belongs to the family of Malvaceae. Kenaf possess two kinds of fibres and each of these fibres has different potentials to be explored. For instance, Shamsuddin *et al*. (2016) highlighted that kenaf core fibre was used to produce activated carbons (AC). The long fibre contains approximately thirty percent (30%) of the total volume of kenaf plant whereas the short fibre represents the remaining of seventy percent (70% (Kuroda *et al*. 2005)). The stem of kenaf plant consists of the outer bark (bast) and inner core, both contain fibrous components. Recent studies have demonstrated the capability of kenaf core and bast fibres to enhance bioremediation and adsorption process (Using *et al*. 2010, Chowdhury *et al*. 2012).

MATERIALS AND METHODS

Sample preparation: All kenaf samples used in this experiment were harvested at 10 weeks after planting (WAP). Freshly harvested kenaf plants were manually defoliated and then were tied in a replicate consisting of six kenaf stems. Each treatment was carried in three replicates of kenaf samples. Retting is the process by which the fibre is removed from its stem with the help of chemicals or microbes present in the retting water (Dhanalaxmi and Jyoti, 2012).

Retting procedures: tank retting: Triplicate of kenaf stems were soaked in an improvised plastic tank containing 150 litres of water. The texture of kenaf stems were checked daily by touching with fingers as to determine when the retting is completed. Retting was adjudged to have been retted completely when the kenaf bast were very soft and can easily be loosened from the core stems.

Ribbon retting: The same quantity (in triplicate) of kenaf stems were decorticated into ribbons (using a decorticator) and the ribbons were steeped in an improvised plastic tank containing 100 litres of water. The ribbon texture was also assessed daily until it became soft to remove from the core stems. Ribbon retting reduces the volume of water required from 1:20 to 1:10 substrate liquor ratio.

Stream retting: Triplicate of kenaf stems was immersed in a slow-running stream and the kenaf stem bundle was supported with heavy stones. The soaked kenaf stems were assessed daily until it became soft to remove from the core stems.

All kenaf stems from the three media (namely: tank, ribbon and stream) were duly retted in fourteen (14) days. After completion of the retting, kenaf fibres were manually removed from the core stems, washed properly, sundried and stored at room temperature for further analysis.

Fourier Transform Infrared (FTIR) characterization of retted kenaf fibres: the spectra characterization of kenaf fibres retted from three media namely tank, stream and ribbon was conducted using Fourier Transform Infra-red (FTIR) spectrophotometer, machine model-IDS- 6500 Thermo Perkin-Elmer (Perkin-Elmer, Norwalk, CT, USA).The analysis was run using the potassium bromide (KBr) pellet technique in which the KBr pellet samples were prepared by mixing about 1.50 to 2.00mg of finely grounded kenaf fibres with 200mg KBr (FTIR grade) in a vibratory ball mixer for 20s.The 13mm KBr pellets were prepared under vacuum in a standard device under a pressure of 75kNcm^{-2} for 3min. Powdered fibres pelletized with potassium bromide were used for recording the spectra. Transmittance was measured over a range from 1000 to 4000 per wavenumber (cm^{-1}). The FTIR spectra curve was automatically generated. The wavenumber of peak positions corresponding to the degree of hydrogen bond in the region. This is with a view to extensively investigate the chemical composition of kenaf bast, cell wall structure and its fibre composites. FTIR spectra peaks of the kenaf fibre from each treatment were used to determine the molecular orientations by comparing their values with the standard assigned Infra-Red (IR) bands for wood cell walls and natural fibres as prescribed by Kataoka and Kondo(1998).

Mechanical characterization of retted kenaf fibres: the mechanical characterization of Kenaf fibres follows the same methodology as that of cotton fibres. An Instron Testing Machine (UTM model 3369, Norwood NJ, USA) was used to determine kenaf fibre's mechanical properties according to the standard prescribed by ASTMD 3039 (ASTM, 2010).Tensile test on kenaf fibre was performed in an ambient temperature and then quasi-

statically stressed at a constant cross-head displacement of 2 mm/min. During the test, the force applied to the sample and the displacement of its point of application (movement of the cross member) were recorded. The fibre strand response in terms of force and displacement is recorded automatically and then converted into stress versus strain diagram. The essence of mechanical characteristics of kenaf fibre is to determine the molecular orientation which is one of the most important parameters, affecting the physical properties of macromolecular systems.

Chemical characterization of retted kenaf fibres: different retted kenaf fibres for chemical analysis were pulverized in a mortar. The experimental technique used was the Van Soest procedure which outlined that the concept behind the detergent fibre analysis can be divided into less digestible cell walls (containing hemicellulose ,cellulose and lignin) and mostly digestible cell contains starch and sugar. Using four detergents namely: Neutral Detergent Soluble (NDS), Neutral Detergent Fibre (NDF), Acid detergent fibre(ADF) and Acid Detergent Lignin (ADL) to quantify the amount of cellulose, hemicelluloses and lignin (Van Soest and Wine,1967; Fulgencio *et al*., 1983). Following Van Soest and Wine (1967) procedure:

NDF=Hemi-cellulose + Cellulose +Lignin (1)

ADF= Cellulose + Lignin (2)

Where NDF=Neutral Detergent Fibre; ADF= Acid Detergent Fibre

NDF determination followed these steps

1. Grind the air dried kenaf fibre sample to pass 1mm screen
2. Weigh in a crucible 1g of ground fibre sample with 1 mg approximation
3. Add 100ml of neutral detergent solution at room temperature into crucible with 0.5g of sodium sulphite and some drops of n-octanol.
4. Heat to boiling and reflux 60minutes from the onset of boiling.
5. Filter and wash three times with boiling water, then twice with cold acetone.
6. Dry for 8 hours at 105^{o}C and allow to cool in a desiccator.
7. Weigh again and then calculate neutral detergent fibre using the formula:

$$\% \text{ NDF} = \frac{weight\ of\ Crucible + weight\ of residue) - weight\ of\ crucible\ X\ 100}{weight\ of\ fibre\ sample} \quad (3)$$

NDS= %NDS= 100- %NDF

The above steps was repeated for acid detergent fibre(ADF) determination with the replacement of neutral detergent solution with the same volume of acid detergent solution. The percentage acid detergent fibre (ADF) was calculate using the formula:

$$\%ADF= \frac{(weight\ of\ crucible+weight\ of\ residue)-weight\ \ X\ 100}{Weight\ of\ fibre\ sample} \quad (4)$$

RESULTS AND DISCUSSIONS

FTIR characterization of retted Kenaf fibres: Fourier transform infrared Spectroscopy is a widely used analytical technique for the identification of chemical bonds and the characteristics of a given material. The spectra peaks obtained from this test was compared with the assigned band values in Kataoka & Kondo (1998) as to infer or confirm the presence of functional groups or bonds. From the results in Figures 1 – 3, the broad bands occurring at about 3312- 3420 cm^{-1} for all the retted fibres were due to the presence of hydroxyl (–OH) groups while the peaks were obtained at 1635.76, 1635.34, and 1730.69 cm^{-1} for stream, tank and ribbon retted fibres respectively. However, tank retted fibres had the most broad band intensity at 3419.78cm^{-1} which may likely be the characteristic stretching of O-H bonds in cellulose and/or hemi-cellulose while ribbon fibres had the highest absorption peak of 1730.69cm-1 which may likely correspond to C=O stretching of hemi-cellulose. This finding agrees with the literature reports in Table 2. Absorption peaks around 1635 and 1637 cm^{-1}as observed in stream retted kenaf fibres were likely be attributed to carboxylate groups of uronic residues of galacturonic acid while the region between 1000 and 1500 cm^{-1} and absorption peaks at 1398 cm^{-1} and 1121 cm^{-1} may be linked to the presence of C-H bond of cellulose. These results were not significantly different from the data (Table 2) on spectra characteristics of kenaf and flax by Millogo *et al*. (2015). There was no change in the peak around 1730 cm^{-1} except for the ribbon fibres as a result of aromatic skeletal ring vibration of lignin, which may be due to structural changes of the lignin component in kenaf fibres after the decortication. This result agrees with Hamza *et al*. (2016) reported that band at 1737 cm^{-1} may likely corresponds to C=O stretch in aldehydes, ketone group or saturated aliphatic. The weak absorptions between 1500 and 1260 cm^{-1} in ribbon fibres (Figure 4) arose from the aromatic ring vibrations with C–O stretching in lignin. The findings of this study were in agreement with the reported data on FTIR spectra of raw kenaf fibre (KF) by Shamsuddin *et al*. (2016) that bands at 1595 cm^{-1} and 1423 cm^{-1} of raw KF are indicative of aromatic compounds (C-C) stretching in the aromatic ring. Similarly, ribbon retted kenaf fibre has band peak at 1259.93cm^{-1}, this suggests the likelihood of the same functional

characteristics with 1260-1234cm^{-1} of O-H phenolics present in other wood fibres as reported by Bodirlau and Teaca (2009). The spectra characteristics of retted kenaf fibres were not at variance with other assigned Infra-red(IR) band behaviours of wood functional groups as shown in table 1.

Table 1: Assigned IR bands of the functional groups in wood

Band position(cm^{-1})	Functional group
3450-3400	O-H alcohol
2930-2910	C-H methyl and methylene groups
1740-1730	C=O carbonyls
1640-1618	C=C alkene
1515-1504	C=C aromatic
1462-1425	CH_2 cellulose, lignin
1384-1346	C-H cellulose, hemicellulose
1260-1234	O-H phenolic
1170-1153	O-H(primary& secondary) and aliphatic esters
910	C=C alkenes

(Source: Bodirlau and Teaca,2009).

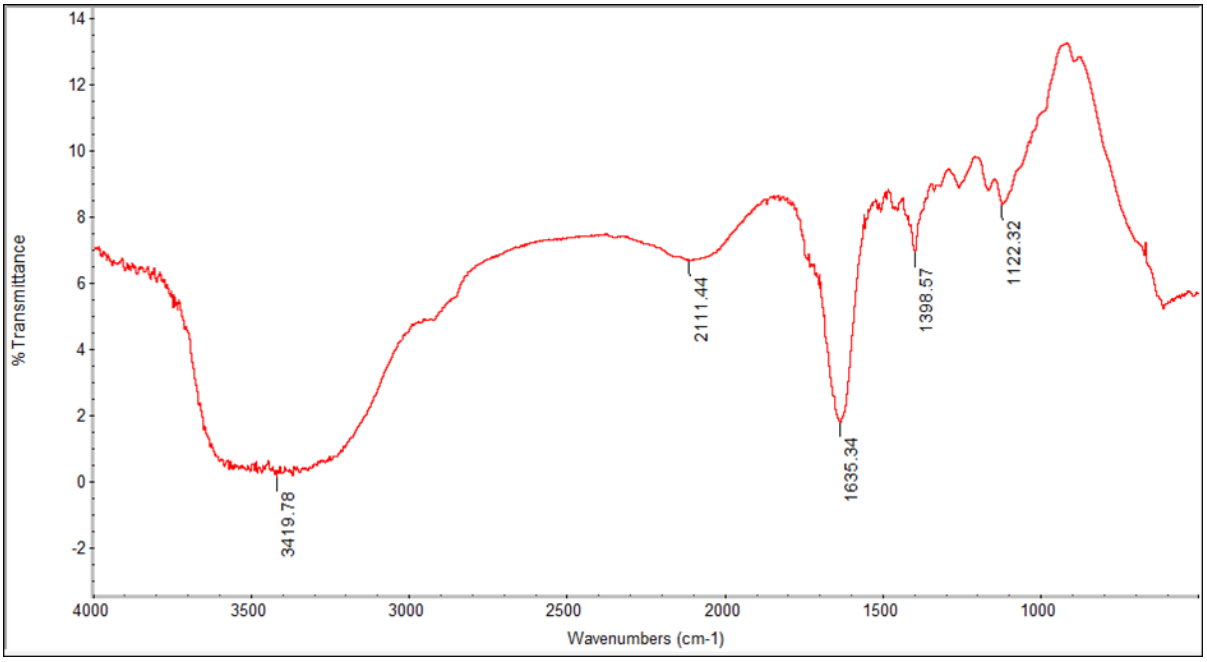

Figure 1: FTIR spectra of tank retted kenaf fibre.

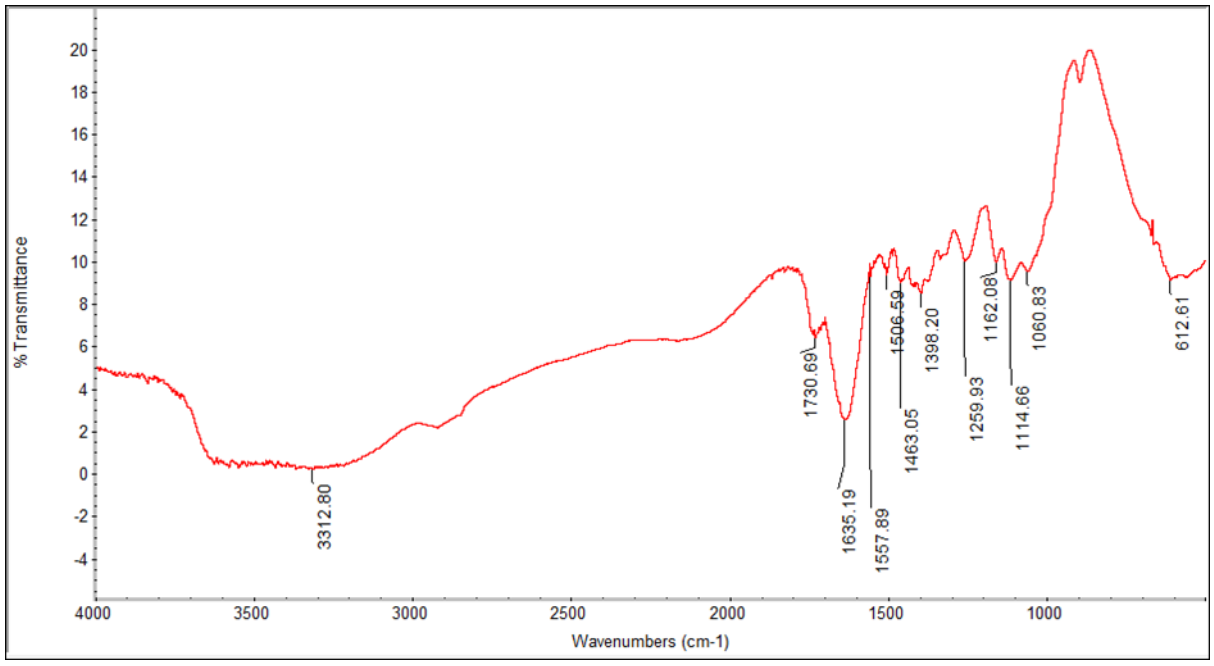

Figure 2: FTIR spectra of ribbon fibre.

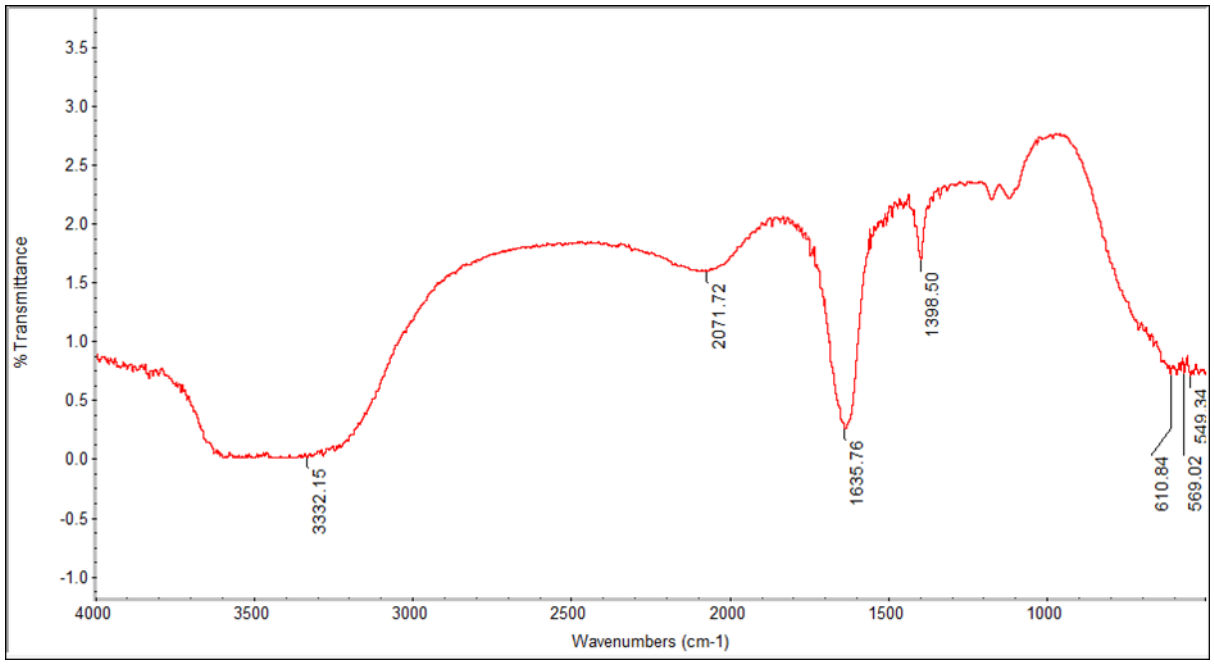

Figure 3: FTIR spectra of stream retted kenaf fibre.

Table 2: Reported main infrared absorption bands of Kenaf and flax.

Wave number Cm^{-1}	Designation
3400-3345	Characteristic stretching vibrations of O-H bonds of Cellulose an hemi-cellulose
2915	Stretching bonds of C-H of cellulose and hemi-cellulose chains
2848	Stretching of –CH2 bonds of cellulose and Hemicellulose
1725	Stretching of C = O bond of carboxylic acids present in pectins and in acetyl groups present in the hemicelluloses
1625	Shear bond vibration-OH characteristic of free water
1503	Cycles aromatiques de la lignine pour la fibre de Kenaf)
1420	Vibration of the characteristic carbonyl bond pectins
1360	shear vibration of C-H bonds of cellulose and hemicellulose
1310	Stirring of the two H-C bonds of CH groups of cellulose and hemicellulose
1230	Characteristic peak of lignin (Kenaf)
1162	Stretching of C-O bonds of acetyl groups (found in hemicellulose or pectins)
1108	skew distortion of the group C-O-C
1032	Stretching of the C-O bond
890	characteristic vibration of the β- binding cellulose

(Source: Millogo*et al*., 2015; Shin *et al.,*2012)

Mechanical characterization of retted Kenaf fibres: stream retted Kenaf fibres had the highest tensile strength followed by tank retted fibres while ribbon fibres recorded the least. Contrariwise, stream retting method is environmentally unfriendly as it pollutes the water bodies and endangers the users at the downstream (Omenna *et al*., 2016). In the recent times, the use of stream retting is being discouraged despite its unprecedented results

(Omenna *et al*., 2016). Stream retted fibre recorded the highest tensile strength (1220MPa) followed by the tank retted fibre (605.83MPa) and ribbon fibre had the least tensile strength (130.36MPa). Conversely, stream retted Kenaf fibres had similar tensile modulus (tenacity) with tank retted fibres and this was significantly higher than that of ribbon fibre (Table 3). However, the range for tensile strength obtained from ribbon and tank retted fibres were only significantly lower than the reported data (700- 900MPa) for the kenaf fibre strength by Laibi*et al*. (2017) but this discrepancy could be attributed to variation in length of the kenaf fibre, difference in the climate, soil type and plant species.

Chemical composition of retted kenaf fibres: the α-cellulose content of kenaf fibres from each treatment, determines the cell wall framework, and pectin is located between the cellulose microfilaments of the cell wall. The results in Figure 4 shows that the α-cellulose content of stream retted fibres (SRF) was slightly higher than that of ribbon retted fibres (RBF) while tank retted fibres (TRF) had the least α-cellulose content. Cellulose has become an important raw material for the pulp and paper, textile, and fibrous chemical industries. The observed α-cellulose content of all the treatments were in agreement with the data reported on kenaf fibre by Shin *et al*. (2012). However, the range of values obtained in this study for α-cellulose content were significantly lower than those reported on kenaf fibre used in building compressed earth block by Laibi*et al*. (2017). Although, Millogo *et al*. (2015) recorded 70% α-cellulose content of kenaf fibre from Burkina Faso that was used in reinforcement of earth blocks as shown in table 4. There was no emphasis on the type of retting methods used to produce the kenaf fibres. By and large, the α-cellulose content of all retted Kenaf fibres was in the range of 60-62%, this makes Kenaf fibre highly compatible than the reported cellulose content of 40-52% for softwoods and 38-56% for hardwoods (Ververis *et al*., 2004).

Table 3: Mechanical properties of retted Kenaf fibres

Sample	Tensile strength(MPa)	E-Modulus (GPa)
Ribbon fibre	130.36c	2.34b
Stream retted fibre	1220.00a	7.30a
Tank retted fibre	605.83b	7.33a

Mean values with the same letter in a column are not significantly different ($P<0.05$)

Table 4: Comparison between the chemical compositions of kenaf fibres from this study and literature.

Fibre	%Cellulose	%Hemi-cellulose	%Lignin	Reference
RBF	61.38	22.3	15.88	This study
TRF	60.85	21.85	14.69	This study
SRF	61.86	22.60	17.19	This study
Other reports on Kenaf fibres	73	18	6	Laibi*et al*.,2017
,,	70	19	3	Millogo*et al*.,2015
,,	53±4	18±1.4	8±1.2	Godin *et al.,*2010
,,	58±1	22±1	17.5±1.3	Akil*etal*., 2011
,,	45-57	21.5	8-13	Jonoobi*et* a*l.,*2011
,,	60.8	19.2	14.7	Shin *et al*.,2012
,,	31-39	21.5	15-19	Bledzki and Gassan,1999

Where RBF=ribbon kenaf fibre, TRF=tank retted kenaf fibre, SRF=stream retted kenaf fibre

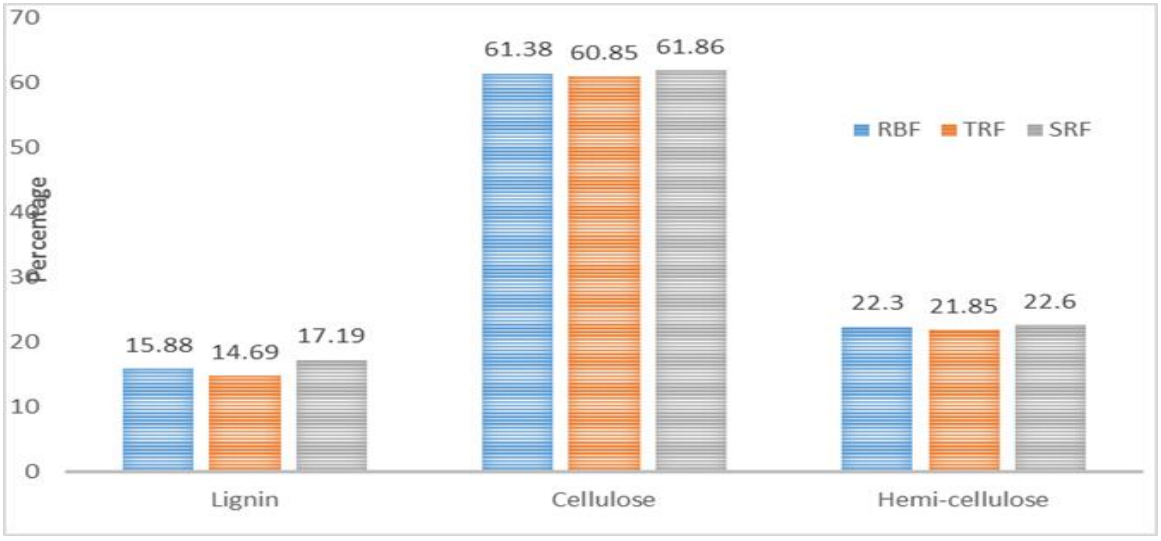

Figure 4: Chemical compositions of retted kenaf fibres

Key: RBF= Ribbon kenaf fibre, TRF=Tank retted fibre, SRF=Stream retted fibre.

The level of hemi-cellulose obtained from this study was not significantly different from the literatures (Jonoobi *et al*., 2011; Godin *et al*., 2010; Bledzki and Gassan, 1999). But these retting methods have little or no impact on the kenaf hemi-cellulose decomposition. From the result, comparable values were recorded for the hemi-cellulose content of stream retted fibres (SRF) and ribbon fibres (RBF) whereas tank retted fibre (TRF) had least. The hemi-cellulose content of Ribbon and Stream retted fibres tallied with the data (22±1%) reported by Jonoobi *et al*. (2011) while Tank retted fibre (TRF) had hemi-cellulose content which was similar with those recorded by Akil *et al*. (2011) and Laibi *et al.* (2017).The type of retting methods used by these duos were not clearly stated as their works focused on the physico-chemical and mechanical properties of the composites.

The lignin content ranges from 14 - 17.2% had placed Kenaf fibres at the high end of the normal range (11-27%) reported for non-woody biomass and this was far below the ranges of 24-37% reported for softwoods or 17-30% for hardwoods (Bodirlau and Teaca, 2009).The finding of this study gives credence to the published data on kenaf fibres by Shin *et al*. (2012) and by Laibi *et al*. (2017). On the contrary, the observed lignin content of all the different retted kenaf fibres was significantly higher than those (3- 8%) reported in the literatures (Akil *et al*., 2011; Millogo *et al*., 2015; Godin *et al*., 2010). However, the lower the lignin content the better the fibre quality and vice versa. From this result, the tank retted fibres appeared to be the best followed by ribbon fibres in terms of Klason lignin content as shown in Figure 4.

As conclusion, among the three media used for kenaf retting, tank retted kenaf fibre exhibited intensive broad-bands with the least lignin and hemi-cellulose content. It can be concluded that tank retting produced the best fibres. It was also more economical and eco-friendly than ribbon and stream retting.

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REFERENCES

Adapa, P.K., Karunakaran,C., Tabil, L.G.,& Schoenau G.J. 2009.Potential applications of infrared and Raman spectromicroscopy for agricultural biomass. *Agr. Eng. Int.: the CIGRE J. Manusc.,* 11:1-25

Aji I., Sapuan S., Zainudin E. & Abdan K. 2009. Kenaf fibres as reinforcement for polymeric composites: a review. *Int. J. Mech. Mat. Eng.,* 4: 39-48

Akil, H.M., Omar M.F., Mazuki A.A.M., Safiee S., Ishak Z.A.M, & Abu Bakar A. 2011. Kenaf fibre reinforced composites: A review. *Mat. & Design.* 32: 4107-4121.

ASTM (2010) International Standard of Testing and Materials Annual Book of ASTM standards. Section 15, New York. USA

Bledzki K.A. & Gassan J. 1999. Composites reinforced with cellulose based fibers. *Progr. Polym. Sci.,* 24: 221-274.

Bodirlau, R. & Teaca, C.A. 2009. Fourier transform infrared spectroscopy and thermal analysis of lignocellulose fillers treated with organic anhydrides. *Rom. Journ. Phys., Bucharest,* 54, 93–104

Chowdhury Z.Z., Zain S.M., Khan R.A., Ahmad A.A. & Khalid K. 2012. Application of response surface methodology (RSM) for optimizing production condition for removal of Pb (II) and Cu (II) onto kenaf fibre based activated carbon. *Res J Appl. Sci. Eng Technol*. 4: 458-465.

Dhanalaxmi, R.K. & Jyoti, V.V. 2012.Influence of retting methods on quality of mesta fibre. *Indian J. Nat. Prod. Res.,* 4: 178-183.

Fulgencio S.C., Jaime C. & Juan G.R. 1983. Determination of hemicelluloses, cellulose, and lignin contents of dietary fibre and crude fibre of several seedhulls. *Data Comp.*, 177: 200-202

Godin B., Ghysel, F., Agneessens, R., Schmit T., Gofflot S.,Lamaudière S., Sinnaeve G., Goffart J.P., Gerin, P.A. & Stilmant, D. 2010. Détermination de la cellulose, des hémicelluloses, de la lignine et des cendres dans diverses cultures lignocellulosiques dédiées à la production de bioéthanol de deuxième génération/Cellulose, hemicelluloses, lignin, and ash contents in various lignocellulosiccrops for second generation bioethanol production. *Biotechnol. Agron. Soc. Environ*., 14: 549-560

Hamza, U.D., Nasri N.S., Amin, N.S., Mohammed J. & Zain, H.M. 2016. Characteristics of oil palm shell biochar and activated carbon prepared at different carbonization times. *Desalin Water Treat*. 1-8.

Hinterstoisser, B. & Salmén, L. 2000. Application of dynamic 2D FTIR to cellulose.Vibrational Spectroscopy, Vol.22, No.1-2, pp. 111-118,

Ismail, A.E, & Hassan M. 2014. Low velocity impact on woven kenaf fibre reinforced composites. *Appl. Mech. Mat.,* 503-6.

Jonoobi M., Harun J., Tahir P.M., Shakeri A., SaifulAzry, S. and Makinejad M.D. (2011).Physicochemical characterization of pulp and nanofibers from Kenafstem. *Mater.Lett*. 65: 1098-1100.

Kataoka, Y. and Kondo, T. (1998). FT-IR Microscopic Analysis of Changing Cellulose Crystalline Structure during Wood Cell Wall Formation. *J. Polym. Sci.,* 31: 760-764.

Khan, G.M.A, Terano, M., Gafur, M.A, & Alam M.S. 2016. Studies on the mechanical properties of woven jute fabric reinforced poly(l-lactic acid) composites. *J. King Saud Univ. – Eng. Sci.,* 28:69-74.

Krouit, M., Bras, J. & Belgacem, M.N. 2008. Cellulose surface grafting with polycaprolactone by heterogeneous click-chemistry. European Polym. J., 44: 4074-4081

Kuroda, K.I., Nakagawa-Izum, I.A., Mazumde, B.B., Ohtani, Y. & Sameshima, K. 2005. Evaluation of chemical composition of the core and bastlignins of variety Chinpi-3 kenaf (Hibiscus cannabinus L.) by pyrolysis-gas chromatography/mass spectrometry and cupric oxide oxidation. *Ind Crops Prod*. 22: 223-232.

Laibi, B.A., Gomina, M., Sagbo E., Agbahoungbata, M., Poullain, P., Leklou N. & Sohounhloule K.C.D. 2017. Physicochemical and mechanical characterization of Benin's Kenaf fibres and its effect on the building compressed Earth Blocks (CEB) mechanical properties. *Res. J. Chem. Sci.*, 7: 6-15

Millogo Y., Aubert J.E., Hamard E. & Morel J.C. 2015. How Properties of Kenaf Fibres from Burkina Faso Contribute to the Reinforcement of Earth Blocks. *Materials,* 8: 2332-2345.

Omenna, E.C. & Oduwaye, O.F. 2017. Effect of variation in urea concentration used in retting ,on the chemical and mechanical properties of kenaf fibres. *Journal of Experimental Agriculture International*.16 (4):1-11

Omenna, E.C., Adeniyan O.N., Ejigbo, E.A., Oduwaye, O.F. & Ezaka, E. 2016. Comparative effect of chemical and stream retting on the kenaf fibre quality. Agr. Biol. J. North America 7: 275-283.

Roslan, M.N., Ismail, A.E, Hashim M.Y., Zainulabidin M.H. & Khalid S.N.A. (2014). Modelling analysis on mechanical damage of kenaf reinforced composite plates under oblique impact loadings. *Appl. Mech. Mat.,* 1324-8.

Sapuan S.M., Leenie, A., Harimi M. & Beng Y.K. 2006. Mechanical properties of woven banana fibre reinforced epoxy composites. *Mat. Design.* 27: 689-693

Shamsuddin M.S.,Yusoff, N.R.N. & Sulaiman, M.A. 2016. Synthesis and characterization of activated carbon produced from kenaf core fibre using H3PO4 activation, *Proc. Chem.* 19: 558 – 565

Shin, H.K., Jeun J.P., Kim, H.B. & Kang, P.H. 2012. Isolation of cellulose fibres from Kenaf using electron beam.*Radiat. Phys. Chem.*, 81: 936-940.

Taufik R.S., Adibah, M .N.F., Muhamad M.R., & Hasib H. 2014. Feasibility Study of natural fibre composite material for engineering application. *J. Mech. Eng. Sci.* 6: 940-948.

Using, S., Core, A.K, Jani, S.M., Khiew, P.S. & Chiu, W.S. 2010. Removal of Copper (II) ions from aqueous fibres adsorption. *Sci. Technol.* 28: 10-16

Ververis,C.,Georghiou, K., Christodourlakis, N. & Santas, S.R. 2004. Fibre dimension, lignin and cellulose content of various plant materials and suitability for paper production. ELSEVIER,Int'l J. 245-254.

Van Soest P.D. & Wine R.H. 1967. The use ofdetergents in the analysis of fibrous feed II. A rapid methodfor determination of fiber and lignin. *J. Assoc. Off. Agric. Chem.,* 50: 50-55.

Zampaloni, M., Pourboghrat, F, Yankovich S.A., Rodgers, B.N., Moore, J. & Drzal, L.T. (2007). Kenaf natural fibre reinforced polypropylene composites: A discussion on manufacturing problems and solutions. *Comp. Part A: App. Sci. Manufact.* 38:1569-80.