CHARACTERIZATION OF ALKALI TREATED LUFFA CYLINDRICA FIBRE REINFORCED rLDPE COMPOSITES

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ABSTRACT

Bio-composites are now sought for as a result of their importance. However most bio-composites reinforced with natural fibres exhibit certain limitations when not treated, these including unfavourable moisture absorption characteristics, weak mechanical properties and poor interfacial bonding between the reinforcement and the matrix. In this research, luffa cylindrica fibres were treated with sodium hydroxide (NaOH) concentrations for 24 hours under room temperature and pressure conditions. The characterizations of the fibre was performed using X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD) and Fourier Transform Infra-Red Spectroscopy (FTIRS), while Scanning Electron Microscopy (SEM) was performed on the developed composite. The percentage moisture absorption and the bulk density of the developed composite were determined for the crushed and uncrushed luffa cylindrica fibre reinforced composite. The treated composites showed a crystallinity index of 65.30%. The results from the characterizations showed that the fibres were effectively modified.

Keywords: LC fibres, NaOH concentration, rLDPE, sustainability

1.0 INTRODUCTION

The quest for sustainable and eco-friendly engineering materials is increasing. Traditional synthetic polymers today especially those used for short term engineering applications pose harm to man and the environment due to the non biodegradable nature of such polymers. To promote the sustainability requirement of such non biodegradable materials, some researchers have developed bio-composites filled with natural fibres [1-4].

Luffa cylindrica fruit which has excellent natural interlocking fibres is one of such natural fibres with potentials to enhance and improve the properties of bio-composites when used as filler. Several researches have been carried out on the development of bio-composites and studies showed that matrix-to-reinforcement interaction still remains a key issue as regards the enhancement of mechanical properties [5], [6], [7], [8]. The hydrophobic nature of matrices and the hydrophilic nature of natural fibres reduces the bonding strength and hence results in poor mechanical properties [9], [10], [11]. Researchers have reported that modification of surfaces of natural fibres is necessary in improving the matrix to filler interaction of bio-composites [12], [13], [14]. Among

other forms of chemical treatments, mercerization has been found to be a cheaper and more effective means of modification of natural fibre surfaces. [15], [16].

The choice of recycled low density polyethylene (rLDPE) as matrix material in this research was made as a result of the sustainability attribute of engineering materials sought for by engineers and manufacturers in recent times. In most parts of the world, LDPEs from packaging materials are discarded indiscriminately and are found as litters on city ways, dumpsites and landfills. Knowing the effect of such materials on the ecosystem has lead to many researches on how rLDPE can be put to some other use after serving a given purpose after a period of time [17] so as to reduce the effect of such non biodegradable materials on the ecosystem. The use of polymer composites filled with natural organic fillers, in replacement of mineral inorganic fillers is of great interest in view of reduction of petroleum based, non renewable sources, and in general in a more intelligent utilization of environmental and financial resources [18]. Also, according to La Mantia and Morreale [10], the use of recycled polymers in place of virgin ones

ensures improved cost efficiency and eco-sustainability. The cost of collecting, cleaning and treating this waste LDPEs are minimal when compared with the overall cost of producing synthetic polymers and their composites putting into consideration their adverse effect on the environment.

Despite the positive results presented by several researchers as related to enhancing the compatibility between natural fibres and their matrices, the engineering applications of bio-composite materials are still limited. The properties attained from past research in the literature and from this research as related to NaOH treatment of natural fibres offer a positive and promising future for the use of natural fibre reinforced composites in sustainable engineering applications. This research studies and characterizes the effect of sodium hydroxide treatment with concentrations between 0 and 0.25M in steps of 0.05M on luffa cylindrica fibre and the developed composite in relation to promoting better interfacial interaction between bio-composite materials and hence aids its suitability for use as substitute materials for engineering applications.

2.0 EXPERIMENTAL METHODS

The luffa cylindrica (LC) fibres used in this research were obtained from Kogi State in Nigeria. All sample preparations were carried out at room temperature. The LC was cut open and the seeds and core removed. The LC mat was then cut to approximately 25 x 25 mm length by breath and immersed in NaOH solution prepared with varying concentrations of 0.05 M, 0.10 M, 0.15 M, 0.20 M and 0.25 M with a batch volume of 1600 mL for each. The LC fibres were treated for a period of 24 hours after which they were thoroughly rinsed with distilled water until a neutral pH of the rinsing solution was attained using a digital meter. The LC fibres were dried under direct sunlight and stored appropriately. The treated LC fibres were characterized by Fourier Transform InfraRed Spectroscopy (FTIRS), X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) before the composite development. The FTIRS was carried out using the Shimazu FTIR-8400S

spectrophotometer to detect the molecular vibration so as to analyse the distribution of the functional group in the samples.

During the sample preparation for the XRD characterisation, the various luffa samples were placed in an oven to reduce their moisture content and aid it to be ground to very fine particle size with an average grain size of between 5μm and 25μm. The samples after being finely ground to pass through 63 microns were homogenised and the average bulk composition was determined. The powdered sample was prepared using the sample preparation block and compressed in the flat sample holder to create a flat, smooth surface that was later mounted on the sample stage in the XRD cabinet. The sample was analysed using the reflection-transmission spinner stage and using the theta-theta settings. Two-theta starting position was 0.00483 and the end was 75.000 with a two-theta step of 0.025 at 3.57 seconds per step. The generator voltage used was 45KVA and the tube current was 40 mA. The scan range of -0.0002^o to 74.99997^o was recorded. Fixed divergent slit size of 1° was used and the goniometer radius was 240mm. The intensity of diffracted x-rays was continuously recorded as the sample and detector rotated through their respective angles. The formula that was used to obtain the crystallinity index is stated in equation 1.

$$CI\,(\%) = \frac{I - I1}{I} \times 100 \qquad (1)$$

Where CI is the crystallinity index

I is the maximum peak intensity of diffraction which is attributed to both amorphous and crystalline fractions

I1 is the small peak intensity attributed to the amorphous fraction

The Epsilon 5 panalytical model was used to carry out the elemental analysis. The samples were placed in the computer programmed XRF and the conditions for trace elemental analysis were set to give the result.

Some of the LC fibres were crushed before the composite development to study the effect of the orientation on the composite. Recycled LDPE used for satchet water packaging obtained in form of waste was

cleaned of dust and impurities. Compounding of the LC fibres and the rLDPE was done using a two roll mill with roller temperatures set to 150 °C and a constant matrix-to-fibre formulation by weight of 4:1 was maintained all through the composite development process. All samples were cured on a hydraulic press at a temperature of 150 °C at 13.7895 MPa for a period of 3 minutes. Tests and characterisation carried out after the composite development include the moisture absorption test, bulk density determination and the Scanning Electron Microscopy (SEM). For the moisture absorption tests, the test specimens were immersed in distilled water at room temperature for 24 hours in accordance with the ISO 62 procedure for testing water absorption of plastic materials. Samples of the developed composites were weighed using a digital weighing balance before immersing them fully in water. The samples were removed, cleaned and weighed again after 24 hours. The percentage moisture content for each sample was calculated using the formula in equation 2.

$$M_c = \frac{W_f - W_i}{W_i} \times 100 \qquad (2)$$

where,

M_c = percentage moisture absorbed (%)

W_f = weight of composite in grams after 24 hours immersion in water (g)

W_i = weight of composite in grams before immersion in water (g)

The density determination was done in accordance with the ISO 1138 standard (method B) at room temperature. A pycnometer was used to determine the density of the luffa reinforced rLDPE composites. The formula used in the calculation of the density is as stated in equations 3.

$$\rho = \left[\frac{(W_2 - W_1)}{\{(W_4 - W_1) - (W_3 - W_2)\}} \right] \times \rho_{water} \qquad (3)$$

where,

ρ = density of the luffa fibre reinforced rLDPE (g/cm^2)

ρ_{water} is the density of water (g/cm^2)

W_1 is the weight of the empty, clean and dry pycnometer (g)

W_2 is the weight of the pycnometer containing the sample (g)

W_3 is the weight of the pycnometer containing the sample and the water (g)

W_4 is the weight of the pycnometer containing the water (g)

The SEM characterisation was carried out using an accelerating voltage of 15KV and was performed on the untreated LC fibre reinforced composite, the 0.1 M treated LC fibre reinforced composites and the 0.25 M treated LC fibre reinforced composite at magnifications of 1000, 3000 and 5000.

3.0 RESULTS AND DISCUSSION

Results from the density determination and moisture absorption test are presented in Table 1. The 0.15 M treated sample with crushed fibres gave the least water absorption property compared with other samples. The moisture absorption result showed that the developed composite has the tendency of absorbing and retaining moisture when exposed to applications operating under high humidity conditions and this may be as a result of the loss of properties of rLDPE during the recycling process involved in developing the composite. However the alkali treatment was beneficial in reducing the percentage moisture absorption as the 0.05 M, 0.15 M and 0.20 M NaOH concentrations reduced the water absorption characteristic of the crushed developed composite while the 0.05 M, 0.10 M and 0.20 M NaOH concentrations reduced the water absorption characteristic of the uncrushed fibre developed composite when compared with the sample whose fibres were untreated. This confirms the report made by [19],[20] that exposure of fibres to NaOH treatment causes dissolution of non-cellulosic cementing substances; hemicelluloses and lignin which reduces moisture absorption by the fibre. They however stated that too long an exposure will degrade the structural cellulose and therefore the alkali treatment must be carefully controlled. It was observed that the luffa fibres also absorbed water along its path from the point of exposure on the surface of the composite.

Table 1: Physical properties of the developed composite

Property	NaOH Concentration Treatment for the LC fibres reinforced Composite (Moles)					
	0	0.05	0.10	0.15	0.20	0.25
S.G. of LC	0.3	0.3	0.4	0.3	0.3	0.4
Density of crushed LC sample (g/cm³)	0.81	0.98	1.39	0.74	0.88	0.98
Density of uncrushed LC sample (g/cm³)	0.98	0.85	0.91	1.47	0.78	0.87
Moisture Absorption of Crushed LC sample (%)	13.6364	13.2075	24.2857	7.5000	10.1124	15.1515
Moisture Absorption of uncrushed LC sample (%)	17.9245	17.3913	15.7895	18.6047	11.9048	19.3182

From researches, it has been reported that sodium hydroxide treatment of natural fibres decreases the fibre density [19], [20]. However from the results of the specific gravity of the luffa fibre obtained in this research, we note alternating values with slight difference in the value of their specific gravities. Density values for the developed composites as obtained in this research showed that the LC/rLDPE composite is very light with most of the uncrushed fibre samples showing lower density values when compared with the untreated fibre sample except for the 0.15 M treated fibre sample which showed a higher density value of 1.47g/cm³. For the crushed sample, the 0.15 M treated specimen gave the least density value of 0.74g/cm³.

Figures 1 and 2 show the spectra obtained for the various alkali treated LC fibres. The peaks less than 690cm⁻¹ (C-Br stretch) showed the presence of alkyl halides. KBr was introduced to the luffa fibre in conducting the test.

The untreated fibres showed the presence of C-H bond (839.06cm⁻¹) of the aromatic out of plane bend. C=O stretch (1717.67cm⁻¹) and O-H stretch band were also observed.

The broad absorption band at the 3650cm⁻¹ to 3250cm⁻¹ region is a characteristic of polymeric association of –OH groups and hydrogen bonded –OH stretch vibration present in carbohydrates (cellulose and hemicelluloses) and lignin [14], thus confirming the presence of the –OH groups present in the untreated fibre and the reduction of these carbohydrates in the treated as indicated by the peaks of the treated fibres. The 0.10 M and 0.25 M treated LC fibres showed a significant reduction in cellulose, hemicellulose and lignin.

The aromatic region related to the lignin is at 1000 cm⁻¹ to 1500cm⁻¹ [21], [22]. The alkali treated fibres did not show the strong absorption at 1735cm⁻¹ (the carboxyl group: -O-C=O) and there was band reduction at 1245cm⁻¹ (C-H). The former is the most reported FTIR alteration due to alkaline treatment [23]. The smaller peaks as noticed in the FTIR spectra for the treated fibres indicate less percentage content of hemicelluloses and is advantageous due to the fact that higher hemicellulose content will reduce the mechanical properties of the LC fibre composite [24]. The peak at band 1600cm⁻¹ indicates the C=C aromatic stretching with conjugate C-C bond and this peak is attributed to lignin content in the fibre [24], [25]. Bands between 800 cm⁻¹ and 1500 cm⁻¹ are specific to cellulose [26], [27] thus confirming the positive modification of the fibres as observed from the spectra of Fig. 1 and 2. The loss in the C=O carbonyl peak at 1725 cm⁻¹ upon NaOH treatments is attributed to the removal of reduced hemicelluloses found on the fibre surface and the change in 1235cm⁻¹ peak associated with the C-O stretching of the acetyl groups of lignin indicating that lignin was partially removed from the fibre surface after the NaOH treatment [28].
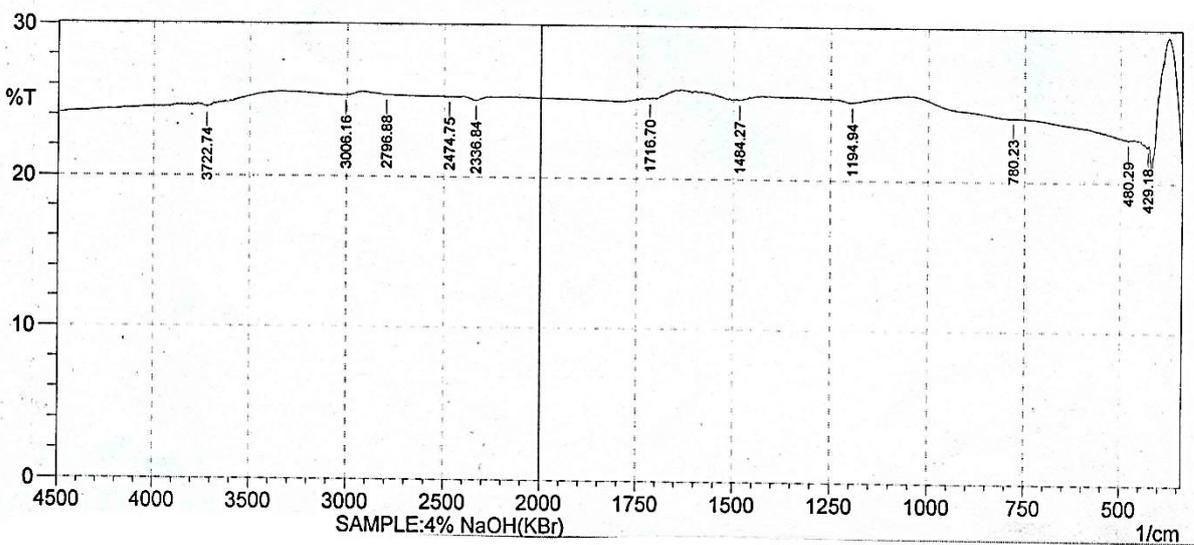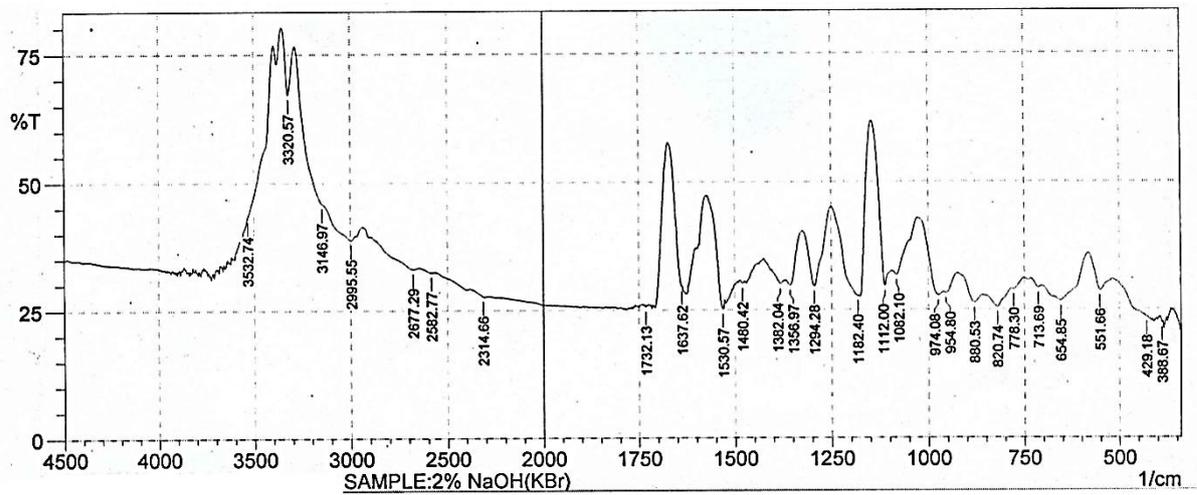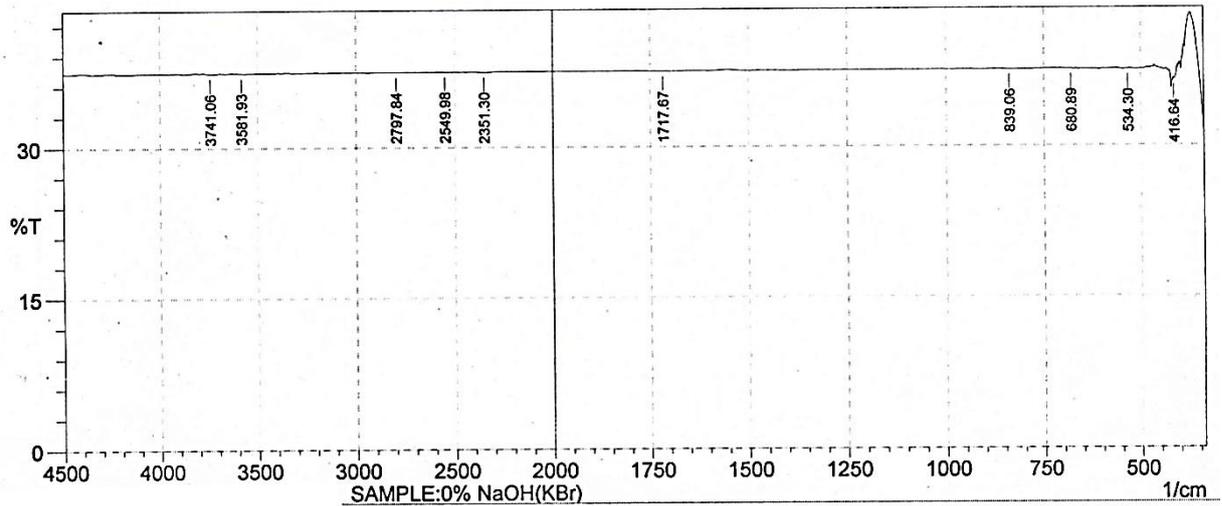
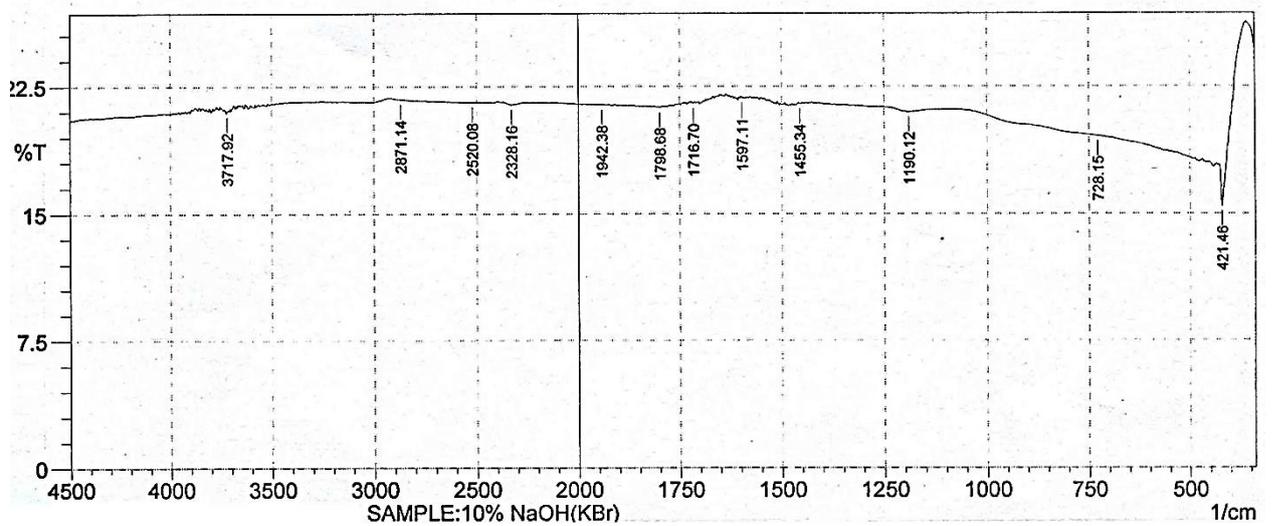*Fig. 1: FTIR Spectra for the untreated LC fibre, the 0.05 M and 0.10 M NaOH treated LC fibre respectively*
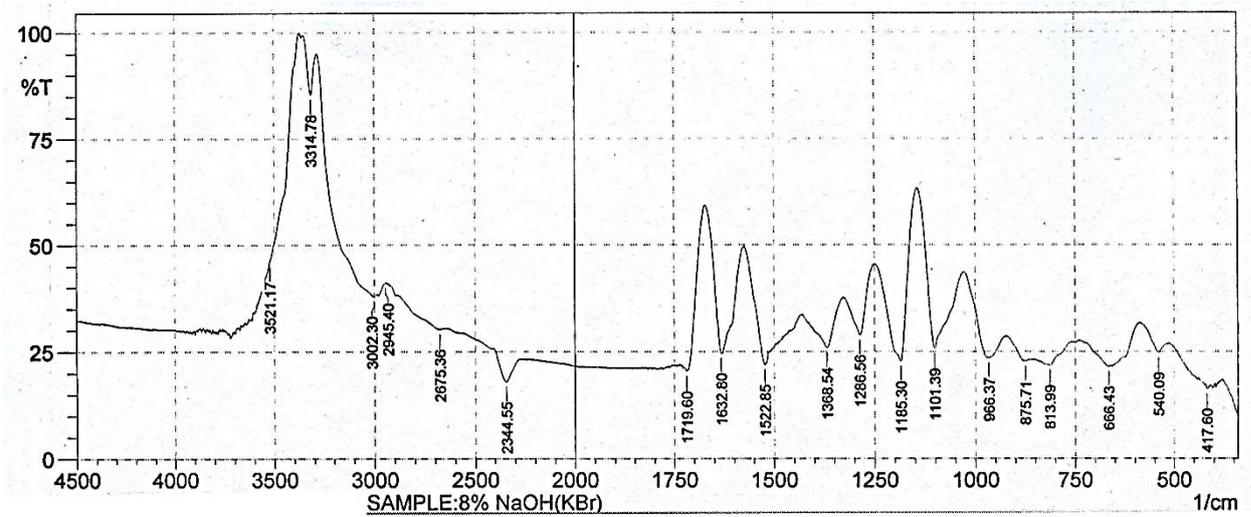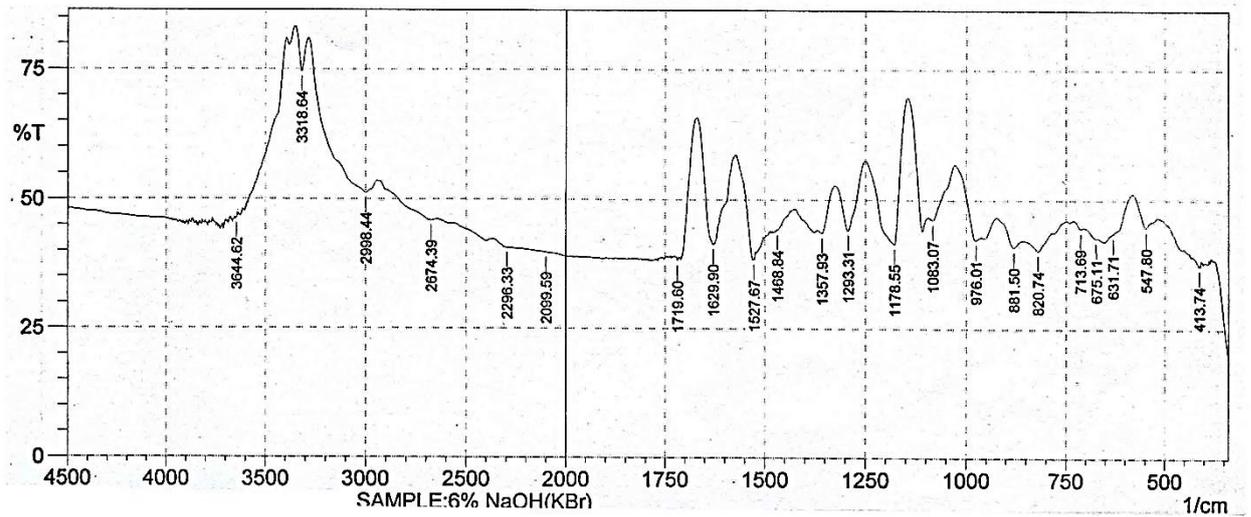
Figure 2: FTIR Spectra for the 0.15 M, 0.20 M and 0.25 M NaOH treated LC fibre respectively

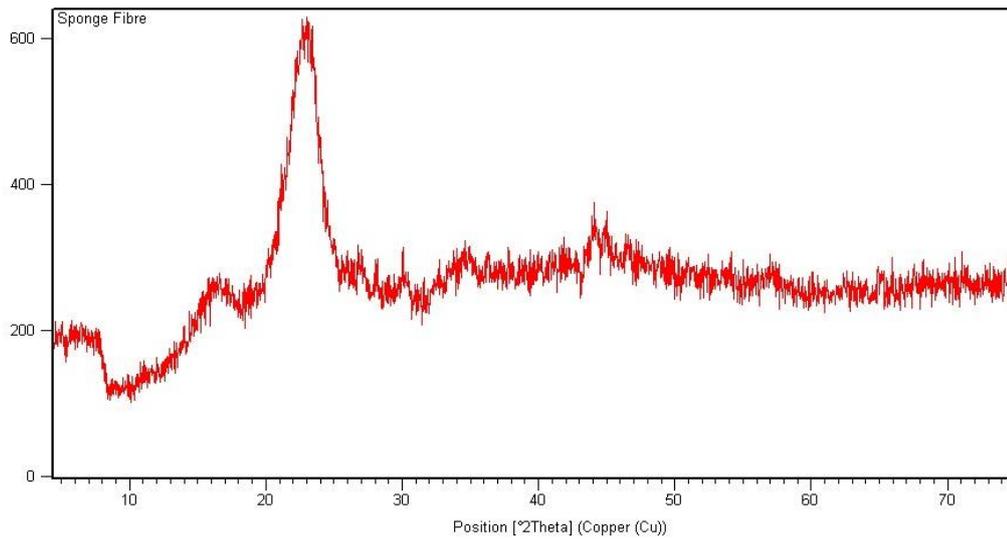Figure 3: X-Ray Diffractogram of the treated LC/rLDPE composites

Table 2: Result of the XRF analysis on the luffa fibres

Oxide	0%NaOH	2%NaOH	4%NaOH	6%NaOH	8%NaOH	10%NaOH
SiO_2	16.02	10.08	10.02	10.07	10.12	10.09
P_2O_5	13.50	13.61	13.51	13.90	13.95	13.60
SO_3	9.50	9.53	9.50	9.55	9.99	9.55
K_2O	24.90	25.60	24.90	24.70	24.09	25.60
CaO	15.70	15.80	15.70	15.50	14.97	15.80
TiO_2	0.35	0.34	0.35	0.34	0.33	0.34
MnO	0.77	0.74	0.77	0.75	0.73	0.73
Fe_2O_3	4.75	4.77	4.75	4.73	4.76	4.72
CuO	0.34	0.33	0.34	0.32	0.33	0.31
ZnO	1.50	1.60	1.50	1.60	1.60	1.40
Na_2O	10.47	16.20	16.40	16.20	16.80	16.20
Eu_2O_3	0.77	0.10	0.97	0.99	0.90	0.66
Re_2O_7	0.22	0.31	0.22	0.21	0.31	0.21

The XRD characterisation spectrum is presented in Fig. 3. The treated fibres showed the same pattern of bands in the spectra, thus suggesting that the treatment of the fibre with various concentrations of NaOH does not affect the bulk properties of the fibres. The highest peak occurred at $2\theta = 23.01°$ at an intensity of 639.4 and the small peak intensity corresponding to the amorphous fraction occurred at $2\theta = 18.928°$.

Siquera *et al*. (2010) stated that the crystallinity index of LC fibres treated with 4% NaOH concentration is 81.3% and that a significant increase in the crystallinity index of microfibrillated cellulose and whiskers when compared with untreated fibres is evident [26]. The high crystallinity index of the LC fibres allied to the relatively low lignin and hemicelluloses contents [26].

The calculated crystallinity index for the treated fibres in this research is 65.30%. However, Hassan [32] stated that the effect of the modification of LC fibres on the crystallinity of cellulose in untreated luffa shows typical cellulose I diffraction pattern with reflections at $2\theta=25.5°$ and $2\theta=17.3°$ and crystallinity index of 67% and that the reflection peak at $2\theta=25.5°$, which was derived from the 002 plane of the cellulose I lattice, has been broadened on increasing the concentration of the NaOH used and on increasing the extent of the reaction [27]. Hassan [32] reported that at 20% NaOH concentration (0.50 M), the reflection peak at $2\theta=25.5°$ splits into two peaks at $2\theta=25°$ and $2\theta=23.5°$, indicating the gradual transformation to cellulose II structure. Meanwhile at the same time, the intensity of the peak around $2\theta=17.3°$, which is assigned

to the reflection of the 101 lattice planes of cellulose I, gradually decreased and broadened and became very broad and weak at high NaOH concentration. The crystallinity index using lower concentrations of 5% and 10% of NaOH (0.125 M and 0.25 M) resulted in a crystalline index of 70% while that of 15% (0.375 M) and 20% (0.50 M) NaOH treated LC fibre resulted in a reduced crystallinity index of 62% and 72% respectively [28]. NaOH solution of low concentration (approximately 8% corresponding to 0.20 M) is more suitable for dissolution of the amorphous hemicelluloses while at higher NaOH concentration (15% corresponding to 0.375 M) the crystallinity decreased because of the alteration in the structure of cellulose in the luffa [29]. The XRD results obtained in this research showed a crystallinity index of 65.30% which was maintained for the range of concentrations used excluding the untreated LC fibre.

Laidani *et al*. [29] obtained the crystallinity index of LC fibre as 69±2% at 2θ=22.4°, 16.2° and 15° respectively corresponding to cellulose1 and amorphous cellulose [29]. Deepack et al. [30] in their characterization of LC fibres noted a crystalline index of 52% and a crystallite size of 33.5Å for the raw fibre pre-treated with 0.1M of NaOH for 30 minutes while a crystalline index of 47% and crystallite size of 9.1 was noted for the grafted LC fibres [30]. The peak obtained at 2θ=23.01° corresponds to 002 crystallographic plane of cellulose and the peak at 2θ=18.93° corresponds to amorphous cellulose [13], [31]. According to Parida *et al*. (2013)], the peaks are most prominent and are indicative of high cellulose crystallinity in the treated fibres. The crystallinity index of 4% wt. (0.10 M) NaOH treated LC fibre was seen to be 60.3% [13].

Tanobe *et al*. (2005) also reported a crystallinity index of 59.1 for untreated LC fibres and a mean value of 62.9 for the 2% wt. (0.05 M) NaOH treated LC fibres [33].

From the results obtained in comparison with the literature, it can be said that the crystallinity of the LC fibres was enhanced by the NaOH treatment parameters.

The mineral contents corresponding with the various NaOH concentration treatments of the LC fibres as obtained from the tests are presented in Table 2. It was noted that the various NaOH concentrations did not eliminate any of the oxides present in the LC fibre. However the varying NaOH concentrations slightly altered the percentage compositions of the oxides, though not in a proportionate pattern. In all the samples, potassium recorded the highest presence in the fibre and decreased with increasing concentration except for the 0.25 M NaOH treated fibre which had all the elemental composition almost equal to that of the 0.05 M treated fibre. The minerals noted to be present in the LC fibre are confirmed by the presence of similar minerals in natural sisal as reported by [34] and from the trace elements confirmed by the Scanning Electron Microscopy – Energy Dispersive Spectroscopy (SEM-EDS) results. The XRF results showed the presence of SiO_2, CaO, K_2O and P_2O_5 as the major constituents of the luffa cylindrica with traces of some other oxides as reported in Table 2. Sodium dioxide, iron oxide and alumina are known to be among the hardest substances [1], [35] thus accounting for the mechanical properties exhibited by the fibres. The 0.05 M and 0.25 M NaOH treated fibres had the highest calcium oxide content of 15.80% while for silicon dioxide, the fibre treatments reduced the composition of the oxide from 16.02% (untreated fibre) to between 10.02% (for the 0.10 M NaOH treated LC fibre) and 10.12% (for the 0.20 M NaOH treated fibre). Na_2O attained its optimum value with the 0.20 M treated fibre at a composition of 16.80% as against 10.47% recorded for the untreated fibres.
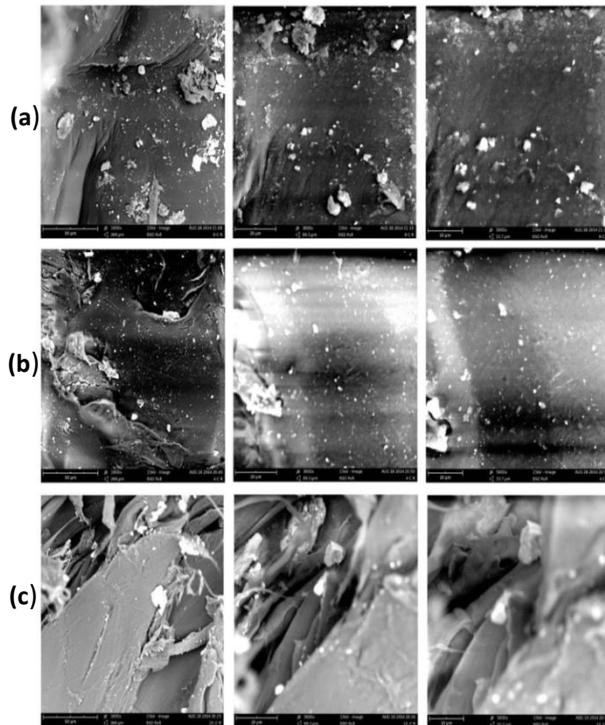
Figure 4: SEM morphology of the crushed sample at 1000x, 3000x and 5000x for the (a) untreated LC fibre (b) 0.10 M treated LC fibre (c) 0.25 M treated LC fibre
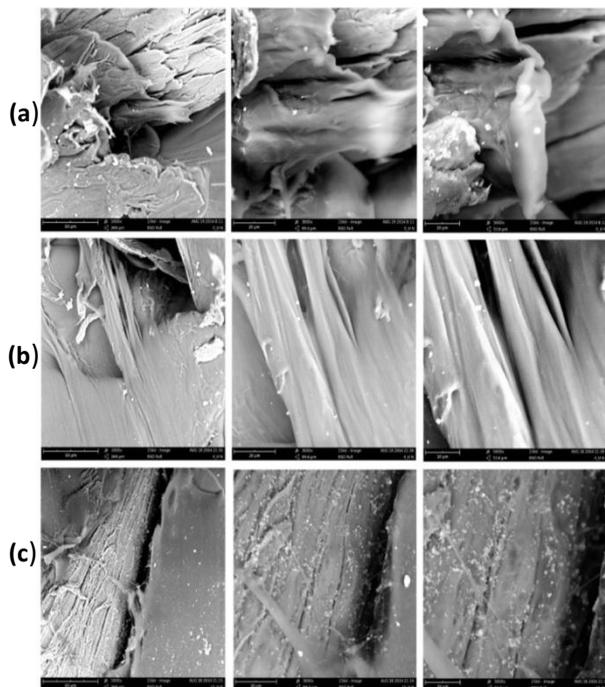

Figure 5: SEM morphology of the uncrushed sample at 1000x, 3000x and 5000x for the (a) untreated LC fibre (b) 0.10 M treated LC fibre (c) 0.25 M treated LC fibre

SEM micrographs for the untreated, 0.1 M and 0.25 M treated samples of the crushed and uncrushed luffa fibre reinforced rLDPE composites were observed and taken under varying magnifications. All images of the

crushed luffa fibres showed fragments of the fibres, their dispersion in the matrix, and a poor matrix to fibre adhesion in general compared with the uncrushed fibres. There was not much interconnectivity between them compared with the images got from the uncrushed samples with similar magnifications; hence the reason for the better mechanical properties exhibited. Presence of lignin was evident from the SEM images for the untreated luffa fibres for both the crushed and uncrushed samples. The 0.1M treated uncrushed sample showed better matrix to fibre adhesion characteristics. The 0.25M treated sample for both the crushed and uncrushed samples showed damaged fibres owing to the attack on the fibres by the NaOH concentration. The 0.25M treated sample also showed characteristics of excess delignification. Fig. 4 shows the morphology of the crushed luffa fibre reinforced rLDPE composite and Fig. 5 shows the morphology of the uncrushed luffa fibre reinforced rLDPE composite. The SEM images of the crushed samples showed poor interfacial interaction between the rLDPE matrix and the luffa fibre reinforcement.

The images of the 0.1M and 0.25 M NaOH treated fibre composites showed that the treatment increased the surface roughness which helps for better interlocking between matrix and fibres [36]. As noticed in the images, the NaOH treatment removed the waxy and gummy substances observed in the untreated LC fibres. Also, alkali treated fibres showed the formation of scratches due to the removal of the lignin and hemicelluloses [31]. These characteristics shown by the treated fibres improved the adhesion of the fibre with the matrix. Tanobe *et al.* [37] in their research reported that their "SEM results indicated that the chemical treatments given to the luffa fibres did not produce the intended characteristics in a homogeneous way, including the uniform increase of roughness, cleaning of the fibre surface and activation of the surface functional group", as also noticed with the 0.1 M NaOH treated LC fibre composite in this research which otherwise would have contributed to enhance the

mechanical properties as reported by some authors.

4.0 CONCLUSION

- The results from the characterisation showed that alkali treatment effectively modified the surfaces of the LC fibres each concentration used had its own specific impact on the LC fibre. The LC fibre treated with 0.25 M exhibited poor properties due to damaged fibre as a result of excess delignification.
- The physical test and SEM results showed that the uncrushed LC fibre composite samples gave better properties when compared with the crushed LC fibre samples for the constant matrix to fibre formulation ratio of 4:1.

Due to the excellent light weight and sustainability characteristics of the developed composites, they can effectively serve as substitutes to synthetic polymers in the automobile industries as materials for vehicle dashboard and door panels; in the electrical/electronic industries as casings for phones and mobile electronics; and in the aerospace industries for aircraft interior components. Therefore, ease of processing, low production cost, less harm to operators, benefits to the environment, and abundance in nature of the LC/rLDPE composite materials give these bio-composites a clear cost advantage over synthetic composites.

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