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Effect of chemical treatments on the properties of oil palm petiole fiber composites

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ABSTRACT

The effect of chemical treatments with acetic anhydride, potassium chlorate, sodium hydroxide and zinc chloride on the properties of Oil Palm Petiole Fiber composites was studied. The untreated and chemically treated Oil Palm Petiole Fiber (OPPF) was used in fabricating natural fiber/HDPE composites using injection molding machine. The chemical treatments on OPPF enhanced its chemical compositions and morphology. It was observed that the cellulose content at the peak for different treatment are 58.4%, 56.7%, 55.2% and 51.5% at 4wt% concentration for NaOH, $KClO_3$, $C_4H_6O_3$ and ZnCl respectively. The composites produced with chemically treated fibers performed better than the untreated one. The highest tensile strength of 44.2 Mpa, 39.1 Mpa, 35.6 Mpa, 31.2 Mpa and 25.5 Mpa was obtained at fiber content of 29 wt% for OPPF treated with NaOH, $KClO_3$, $C_4H_6O_3$, ZnCl and untreated OPPF respectively. Water absorbed by the untreated fiber was high compared to the chemically treated fiber. The chemical treatment created a better interfacial bonding and could be responsible for the improved mechanical properties.

***Keywords*:** Oil palm petiole fiber (OPPF), acetic anhydride, potassium chlorate, sodium hydroxide, zinc chloride, chemical composition, High density polyethylene, tensile strength

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1. INTRODUCTION

The increased knowledge and campaign in environmental safety and utilization of renewable materials towards greener society has resulted to the use of natural fibers as reinforcement materials in composites (Satyanarayana et al, 2009). Natural fibers are gaining importance and the interest of many researchers, material scientists and industrialists since they are renewable and degradable. A composite is made up of several parts or elements. It is always made from two or more different materials which when combined are stronger and superior than those individual materials used.

Boluk et al (2012) stated that a composite is a material that contains two or more distinct constituents or phases, and the amount of the minor phase must be of reasonable proportion (~5%), so that the composite properties are much different from those of the constituents.

They are also made bearing in mind a particular use like enhanced strength, reduce cost, light weight, efficiency or durability. Polymeric composites are polymers that have been filled with natural or synthetic compounds. Dlamini (2012) mentioned that natural fibers used for polymer composites are obtained from biomass, forestry and agricultural residues. Dayanidhi et al, (2019) stated that natural fiber composites can be much cost effective material for application in building and construction areas (walls, ceiling partition, window and door frame), furniture (chair, table, tools), storage devices (bio-gas container, post boxes), electronic devices (outer casing of mobile phone) and other miscellaneous applications (helmets, suitcase) etc. Composites from natural fibers are gaining importance due to they are very cost effective, non-carcinogenic and biodegradable thus they are potential materials for replacing synthetic fibers which are of high cost (Abilash and Sivapragash 2013; Jawaid et al, 2011), they are not biodegradable and create severe ecological and health hazards for the workers using them.

However, the main disadvantages of natural fiber composite are the relative high moisture absorption, comparatively poor fiber/matrix interaction and relatively lower durability. Jawaid et al (2014) opined that the weaker interfacial or adhesion bonds between highly hydrophilic natural fibers and hydrophobic, non-polar organophilic polymer matrix, leads to considerable decrease in the properties of the composites and thus significantly obstructs their industrial utilization and production.

Also natural fibers have a wide variation in diameter and length, which in turn affects expected mechanical behavior of the composite (Jighesh et al, 2016). So for any polymeric composite to have a substantial reinforcement and good properties, there must be a homogeneous distribution of the reinforcing component, orientation, good adhension and relatively high aspect ratio from the natural fiber to be used.

So many researchers have mentioned several approaches to supplement this deficiency in compactibility that will result in better properties. Kalia et al (2009) stated various surface modification techniques and / or the use of coupling agents that could be used to remedy the problem of low interfacial or adhesion bonding between the natural fibers and polymer matrix. Bongarde and Shinde (2014) mentioned that the surface of natural fibers can be modified and this can be achieved by physical, mechanical and / or chemical treatments.

Oil palm is available in huge amounts in most part of Nigeria and the frond is littered as waste after harvesting the palm oil fruits. According to Mahdavi et al (2010), petiole of natural plant has a very good aspect ratio and aspect ratio is one the factors that affect properties of natural fiber composites. The effect of chemical treatments on the OPPF was used in evaluating the properties of the composite.

2. EXPERIMENTAL

2. 1. Materials and methods

Materials used for this study are fiber (OPPF) and High density polyethylene (HDPE). The HDPE was purchased from the Indorama Eleme Petrochemicals Limited, Port-Harcourt, Nigeria. The OPPF was prepared from the petiole of oil Palm frond by mechanical method (traumatization) / water retting for proper and better fiber separation.

a b

Figure 1(a,b). (a) Oil palm tree and **(b)** mechanically extracted oil palm petiole fiber (OPPF)

The fibers extracted were then washed and cleaned thoroughly with very clean water which removed most of the impurities the fibers. The clean fibers were then dried under the sun for two days before chemical treatment (surface modification).

2. 2. Surface modification

In order to improve the fiber-matrix adhesion, four different chemicals; Acetic anhydride ($C_4H_6O_3$), Potassium chlorate ($KClO_3$), sodium hydroxide (NaOH) and zinc chloride (ZnCl) were used to alter their surface chemistry. The fibers extracted were divided into five (5) different equal portions by weight. One portion was set aside as the control, the other four (4) portions were used for surface modification. The four chemicals with different concentrations by weight percent were prepared by dissolving the pellets in distilled water at room temperature for different lengths of time with different mass / volume ratio.

2. 2. 1. Treatment with Acetic Anhydride

The OPPF was soaked in distilled water for an hour, filtered and placed in acetic anhydride solution of different concentration 2, 3.5 and 5 wt%, with time of 8, 16 and 24 hrs and 2, 4 and 6 g/l solution-to-fiber ratios. The fibers were then washed with distilled water then

sun dried for 48 hours (Bledzki et al, 2008; and Nadanthangam et al, 2013). The same procedure was used for the other three chemical treatments (potassium chlorate, sodium hydroxide and zinc chloride).

2. 3. Characterization

The chemical composition of OPPF was determined using TAPPI Standard Test Method T204 cm-17 for extractives, TAPPT Standard Test Method T203 cm-99 for α cellulose and lignin content by TAPPI Standard Test Method T222.

Fourier transform infrared (FTIR) spectroscopic analysis was done with Buck scientific M530 USA. The morphology of DPPF was carried out with JEOL JSM 7500F Scanning Electron Microscope (SEM). The particle size reduction of OPPF was carried out with a laboratory mill made by Christy and Norris Ltd, Chelmsford, England. The OPPF was milled to fine powder. The same process was repeated continuously till the fiber particle size of 150 μm was gotten.

2. 4. Composites Production

The OPPF, HDPE and coupling agent (Maleic Anhydride Polyethylene – MAPE) were mixed physically in a bowl and then poured into injection molding machine where the proper mixing and melting occurred. The mechanical test samples were formed inside the molds produced with vertical milling center CNC (model 750, manufactured by BAO JI, Laber Precise Industries Co. Ltd).

Then a press was used for compression process. The different ratio that was used for the composite production is shown on Table 1.

Table 1. Ratio formulation for composite production.

Quantity of fiber (%)	HDPE (%)	Coupling agent (%)
11	87	2
20	78	2
29	69	2
38	60	2
47	51	2

2. 4. Test procedures

Universal Testing Machine (Testometric 2500kgf Rochdale England of serial no 52978 was used for mechanical properties of the composites at cross-head speed of 50 mm/min according to ASTM D638 for tensile strength, ASTM D790 for flexural test and ASTM D6110-18 for Chrpy impact test . All these were carried out at standard laboratory atmosphere.

3. RESULTS AND DISCUSSION

3. 1. Chemical composition of the fiber

Chemical composition of the untreated OPPF is shown in Table 2. It gave a good result with high yield of cellulose (39.83%), hemicelluloses (31.04%) and lignin (21.15%). The presence of high cellulose content is an indication that OPPF is a potential natural fiber resource, and makes it suitable for application in polymeric composites (John et al, 2008).

Table 2. Chemical composition of untreated OPPF

Chemical composition	OPPF
Celluose (%)	39.83
Hemicellulose (%)	31.04
Lignin (%)	21.15

The Chemical composition of the OPPF varied significantly with the different chemicals. It was observed that cellulose content increased with increase in the chemical concentrations. This result shows that NaOH has high extractive properties. The chemically-treated OPPF with NaOH gave the highest yield of cellulose. Alkalization reduces drastically the hemicelluloses content of a natural fiber and exposes more reactive cellulose on the fiber surface (Kalia et al 2009). Jayaramudu et al (2009) mentioned that NaOH treatment removes some hemicelluloses, lignin, glue and other extractives in fiber bundles and yields higher percentage of α-(alpha) cellulose in natural fibers. The effect of chemical concentration on the cellulose yield is presented in Figure 2.

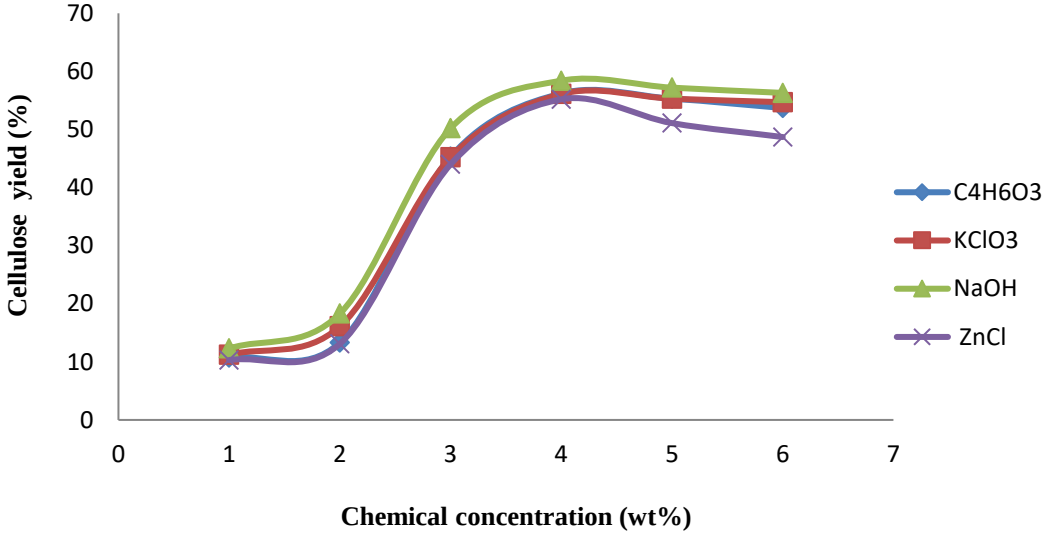


Figure 2. Cellulose yield versus concentration for chemically-treated OPPF

A lag was noticed between chemical concentrations of 1 to 2wt%. This might be as result of inactivity between the functional groups of OPPF and chemicals used. At 4 wt% concentration, the maximum yield of cellulose is 58.4%, 56.7%, 55.2% and 51.2% for OPPF treated with NaOH, $KClO_3$, $C_4H_6O_3$ and ZnCl respectively. Beyond 4wt% concentration, there was a slight decrease which might be due to high concentration of chemical that could cause an excessive extraction of lignin and hemicelluloses and thus damage the ultimate cells walls of the fibers. This is in agreement with report of Kalia et al (2009).

3. 2. Functional groups of untreated DPPF

The functional groups of untreated and chemically-treated OPPF are presented in Figure 3. It was observed that there is a peak at 3200 cm^{-1} which corresponds to hydroxyl stretching vibrations in hemicelluloses and/or cellulose. Also there is a peak at 1700 cm^{-1}. It corresponds to carbonyl group from either carboxylate groups or ester linkages in pectin.

The peak located at 1600 cm^{-1} wave number belongs to C=C bonds or aromatic bonds from lignin. But comparing the intensities of untreated and chemically-treated OPPF, it was seen that there were structural changes and increase in the vibrations after the treatments with the four chemicals used. Some of the lignin and hemicelluloses have been removed thereby exposing more of the hydroxyl group in the cellulose making the active sites in the fiber ready for reactions. The peak at 1700 cm^{-1} corresponding to carbonyl group from either carboxylate groups or ester linkages in pectin disappeared in the treated fibers.

Chemical treatment reduced hydrogen bonding due to removal of the hydroxyl groups by reacting with the chemicals. This is in agreement with the report of Abidi and Herath, (2017). The peak located at 1600 cm^{-1} belongs to C=C bonds or aromatic bonds from lignin decreased in treated fibers.

There is an indication of in-plane bending vibrations of the $-CH_2$ and -CH groups of cellulose (Prasanna and Velmourougane, 2017).

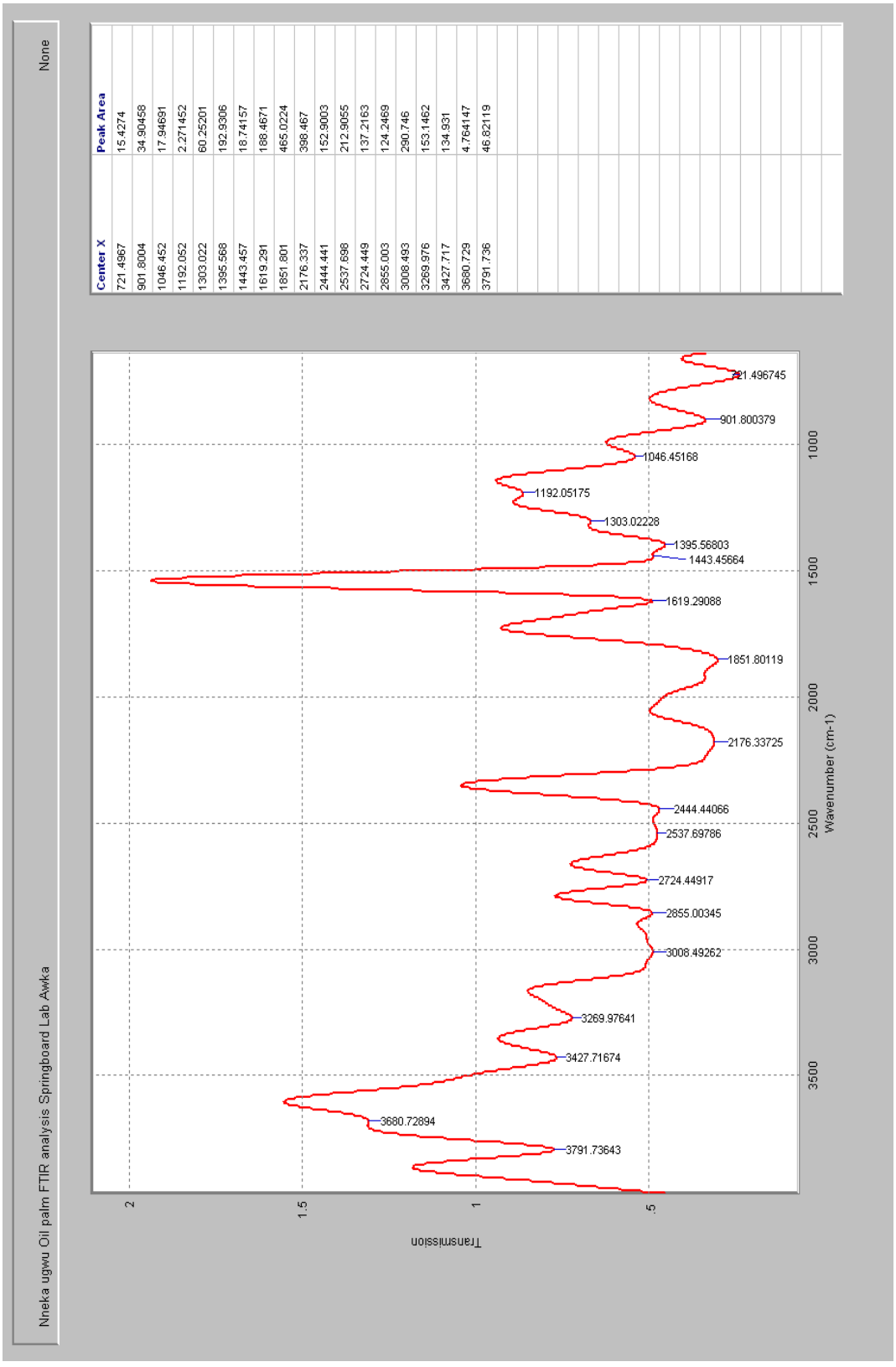
3. 3. Morphology

The SEM micrograph of the untreated and chemically-treated OPPF is presented in Plate 1. It is observed that micrograph of the untreated OPPF has presence of wax, oil, and surface impurities but the chemically-treated micrographs showed successful removal of the surface contaminants. They also have more exposed surface that will be readily available to react with the matrix (HDPE) than the untreated one.

3. 4. Water absorption rate

The water absorption rates of composites made with both treated and untreated OPPF are presented in Figure 4. It was observed that the water absorbed by the composites made with untreated fibers was high compared to the chemically-treated composites. This might be due to possible removal of hemicelluloses during the chemical treatment resulting to a more hydrophobic fiber as hemicelluloses is the most hydrophilic component of fiber (Saheed and Jog 1999).

It might also be due to better interfacial bonding between OPPF and HDPE after the chemical treatment. Chemicals activate the hydroxyl groups or introduce new molecules that can effectively interlock with the matrix (Ali et al 2017).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Nneka ugwu Oil palm FTIR analysis Springboard Lab Awka
Center X
Peak Area
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901.8004 34.90458
1046.452 17.94691
1192.052 2.271452
1303.022 60.25201
1395.568 192.9306
1443.457 18.74157
1619.291 188.4671
1851.801 465.0224
2176.337 398.467
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2537.698 212.9055
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3269.976 153.1462
3427.717 134.931
3680.729 4.764147
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721.496745
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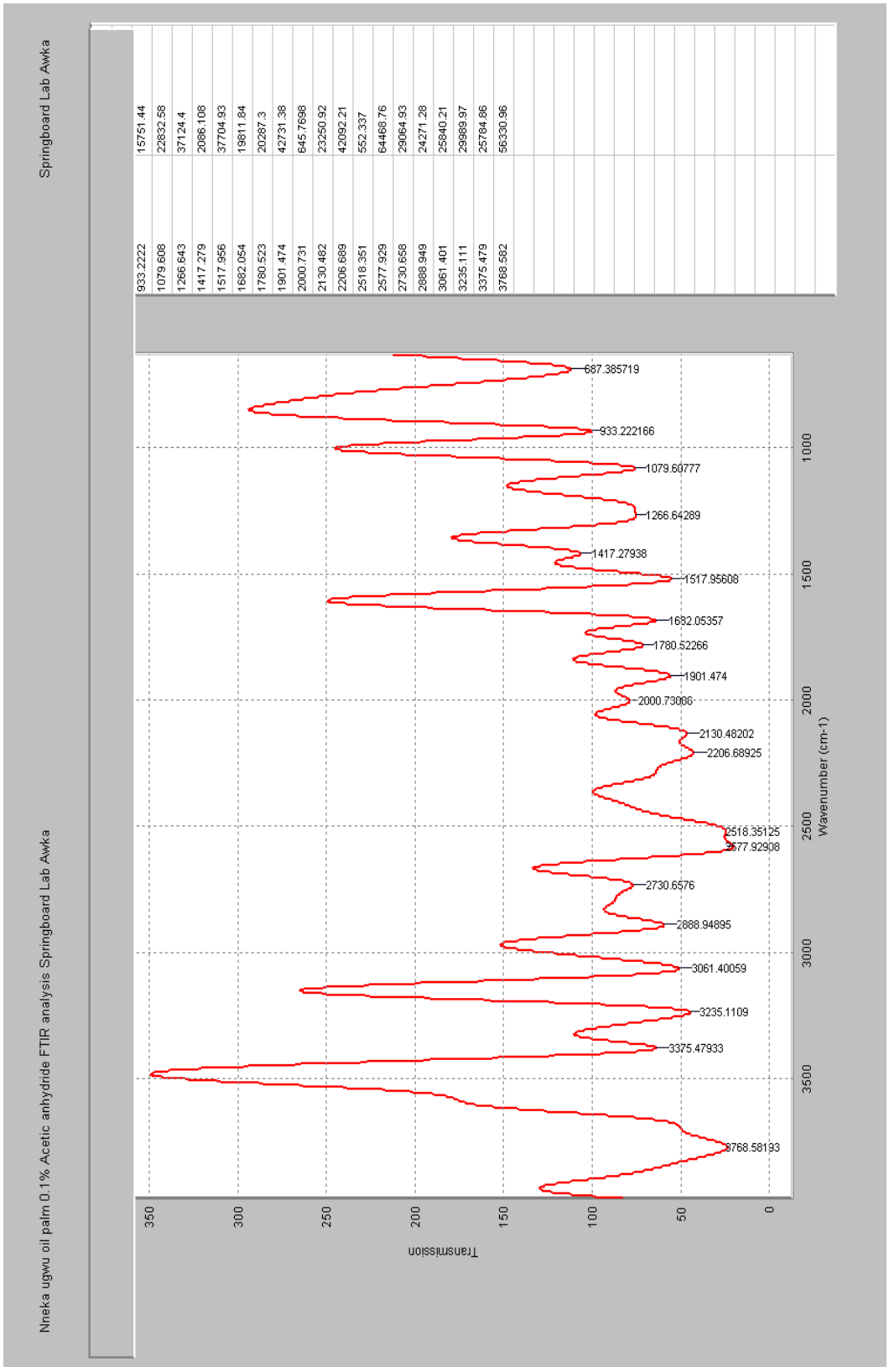
2444.44066
2537.69786
2724.44917
2855.00345
3008.49262
3269.97641
3427.71674
3680.72894
3791.73643
Transmission
2
1.5
1
.5
Wavenumber (cm-1)
1000
1500
2000
2500
3000
3500

(a)


Springboard Lab Awka
Nneka ugwu oil palm 0.1% Acetic anhydride FTIR analysis Springboard Lab Awka
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1417.279 2086.108
1517.956 37704.93
1682.054 19811.84
1780.523 20287.3
1901.474 42731.38
2000.731 645.7698
2130.482 23250.92
2206.689 42092.21
2518.351 552.337
2577.929 64468.76
2730.658 29064.93
2888.949 24271.28
3061.401 25840.21
3235.111 29989.97
3375.479 25784.86
3768.582 56330.96
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933.222166
1079.60777
1266.64289
1417.27938
1517.95608
1682.05357
1780.52266
1901.474
2000.73086
2130.48202
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2518.35125
2577.92908
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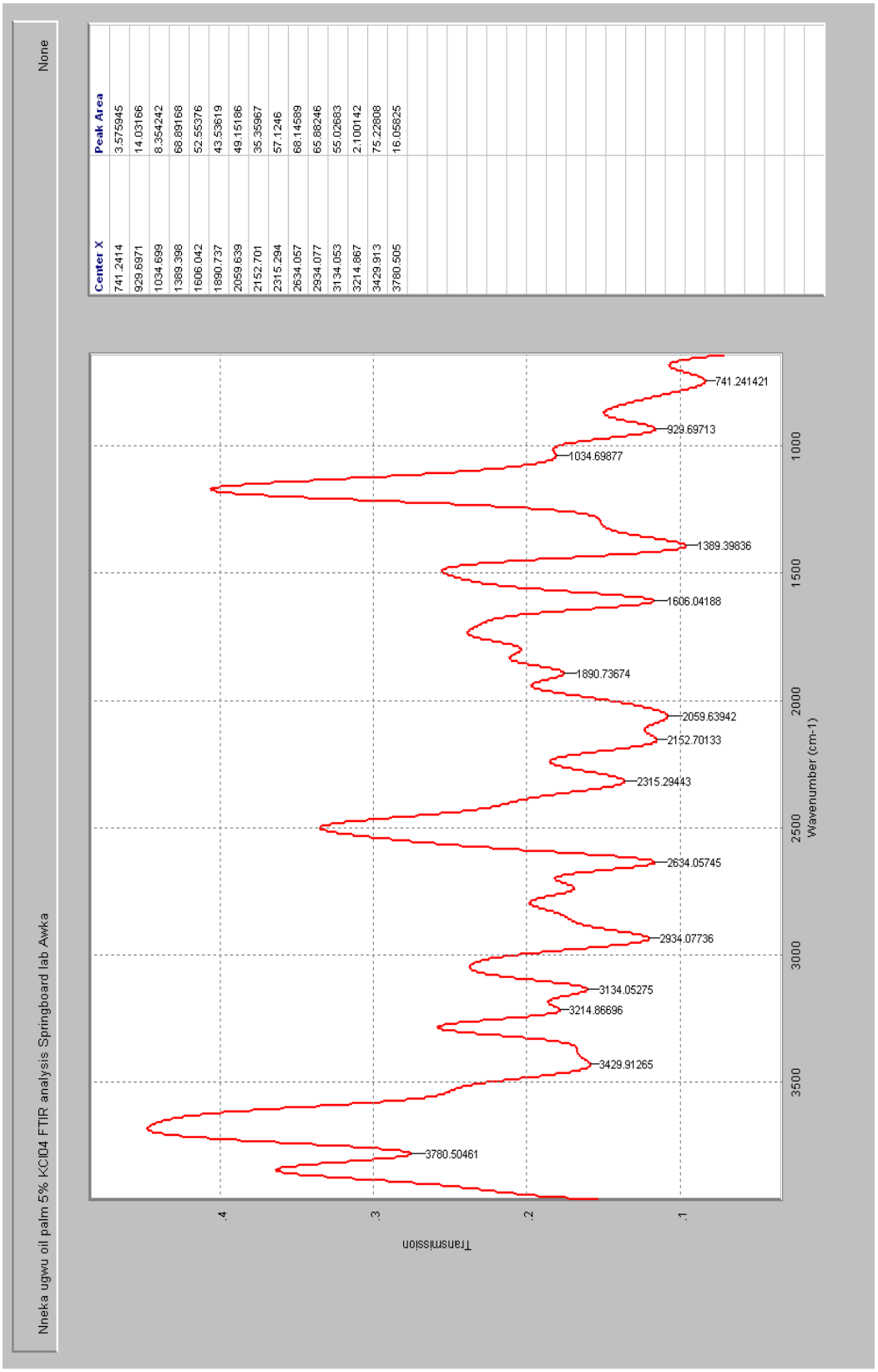
Transmission
Wavenumber (cm-1)

(b)


None
Nneka ugwu oil palm 5% KOH04 FTIR analysis Springboard lab Awka
Center X
Peak Area
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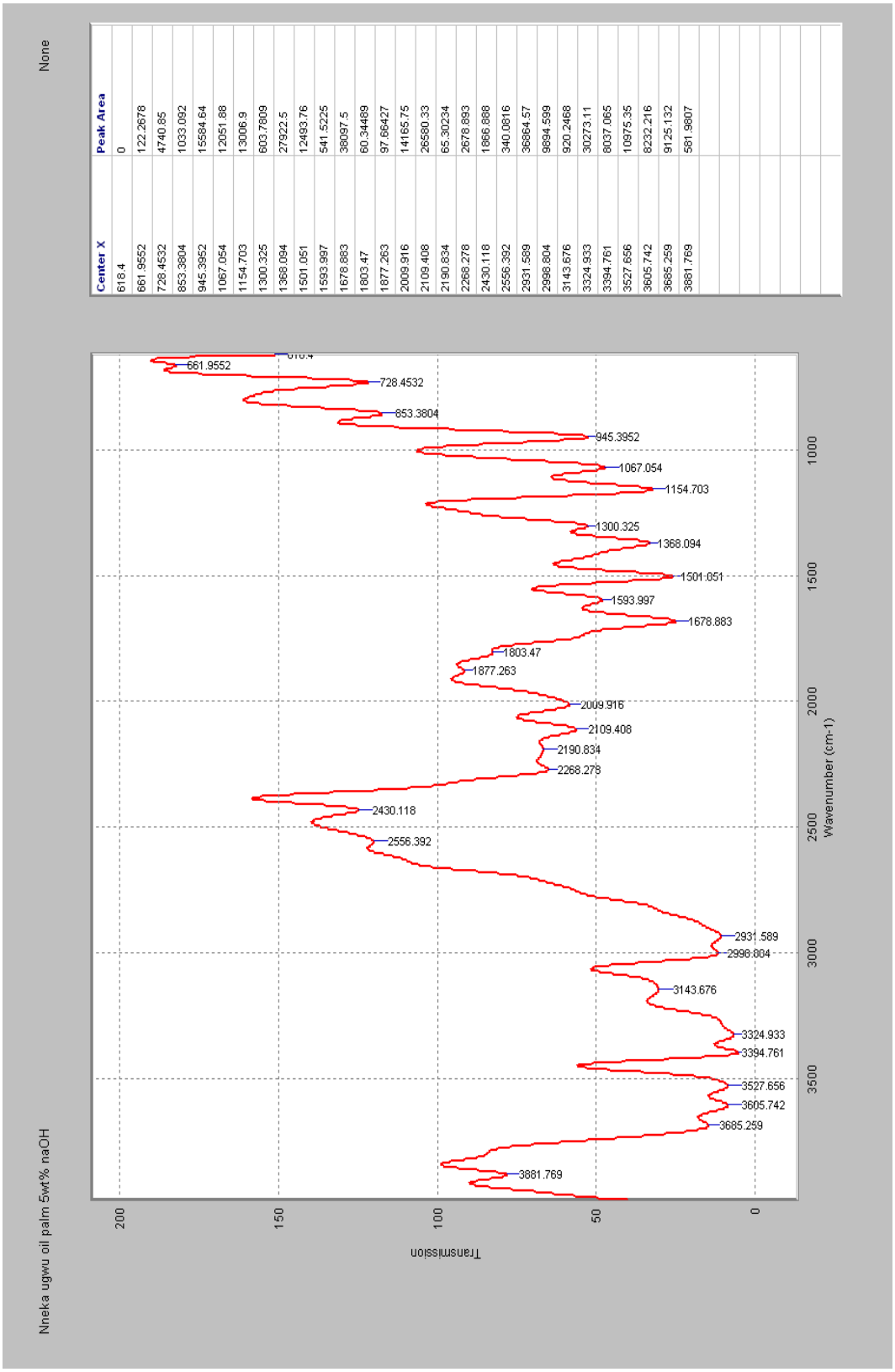
3.575945
929.6971
14.03166
1034.699
8.354242
1389.398
68.89168
1606.042
52.55376
1890.737
43.53619
2059.639
49.15186
2152.701
35.35967
2315.294
57.1246
2634.057
68.14589
2934.077
65.88246
3134.053
55.02683
3214.867
2.100142
3429.913
75.22808
3780.505
16.05825
741.241421
929.69713
1034.69877
1389.39836
1606.04188
1890.73674
2059.63942
2152.70133
2315.29443
2634.05745
2934.07736
3134.05275
3214.86696
3429.91265
3780.50461
Transmission
.4
.3
.2
.1
Wavenumber (cm-1)
1000
1500
2000
2500
3000
3500

(c)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Nneka ugwu oil palm 5wt% naOH
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Peak Area
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661.9552 122.2678
728.4532 4740.85
853.3804 1033.092
945.3952 15584.64
1067.054 12051.88
1154.703 13006.9
1300.325 603.7809
1368.094 27922.5
1501.051 12493.76
1593.997 541.5225
1678.883 38097.5
1803.47 60.34489
1877.263 97.66427
2009.916 14165.75
2109.408 26580.33
2190.834 65.30234
2268.278 2678.893
2430.118 1866.888
2556.392 340.0816
2931.589 36864.57
2998.804 9894.599
3143.676 920.2468
3324.933 30273.11
3394.761 8037.065
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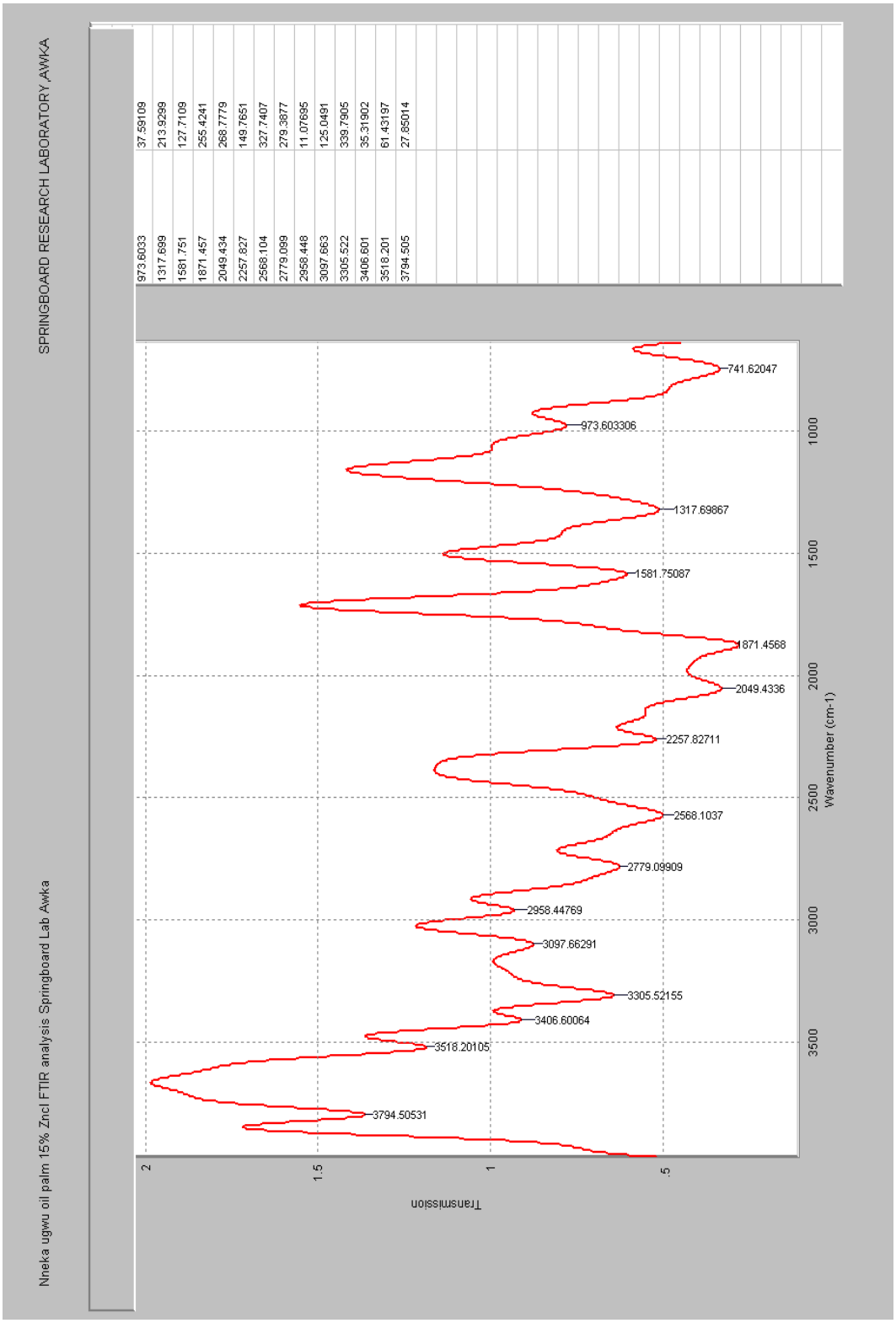
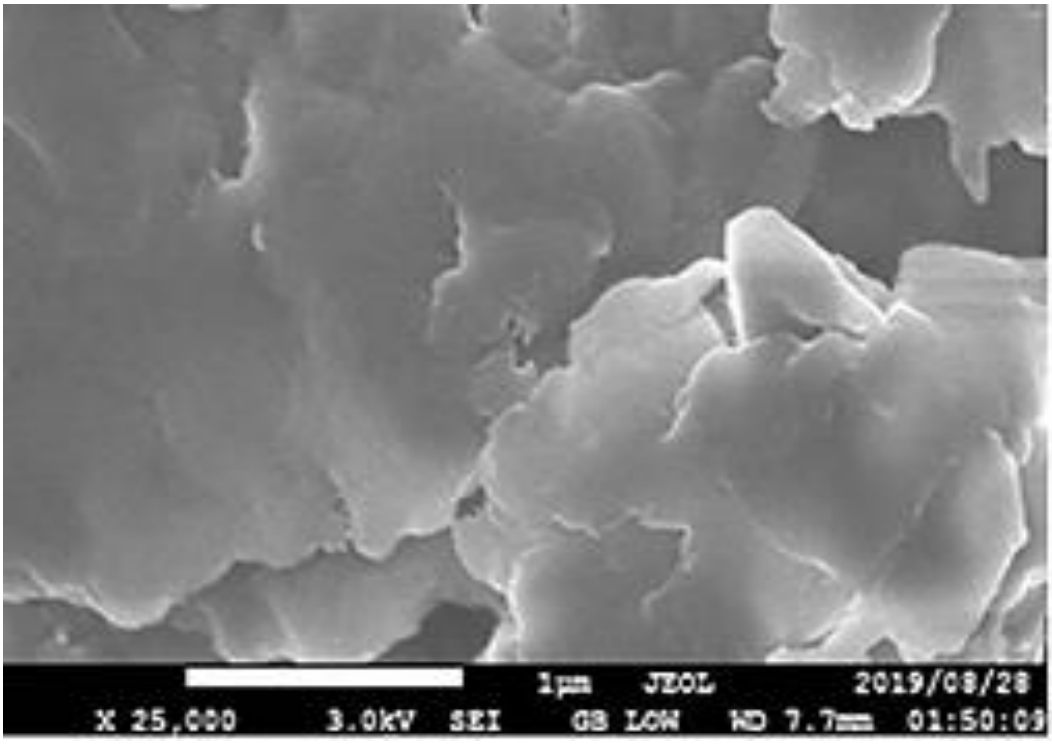
Transmission
Wavenumber (cm-1)

(d)



(e)

Figure 3. FTIR Analysis of OPPF **(a)** untreated, **(b)** $C_4H_6O_3$, **(c)** $KClO_3$, **(d)** NaOH, **(e)** ZnCl.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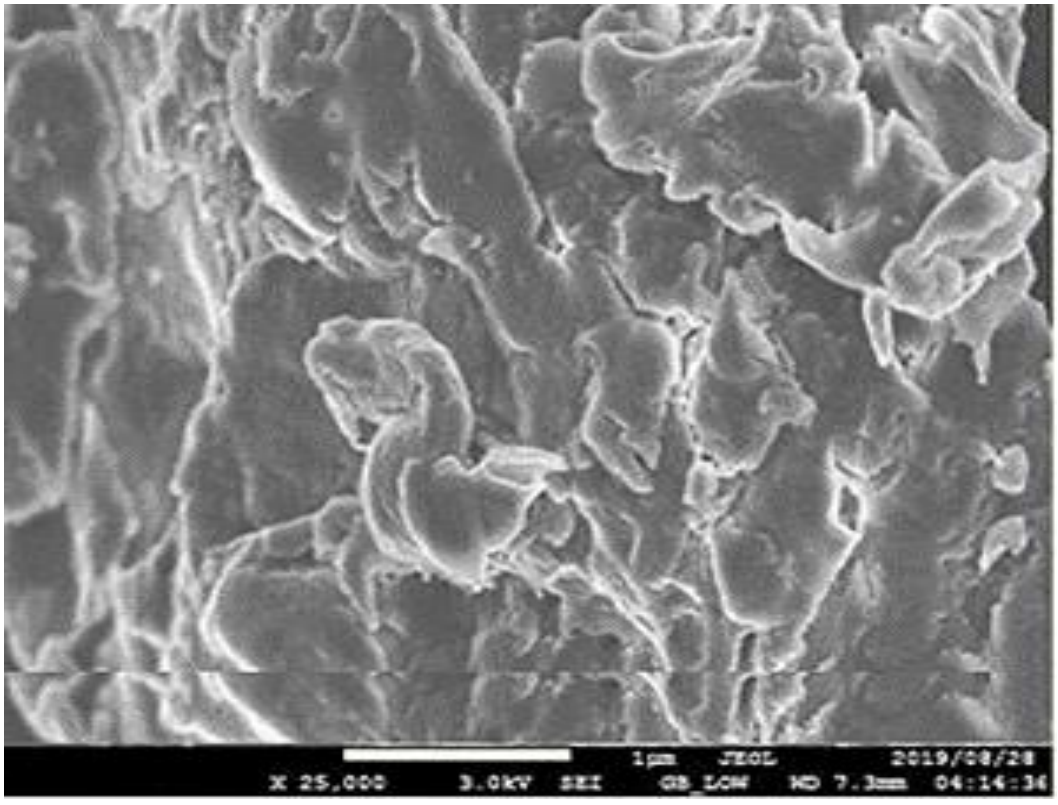
(a)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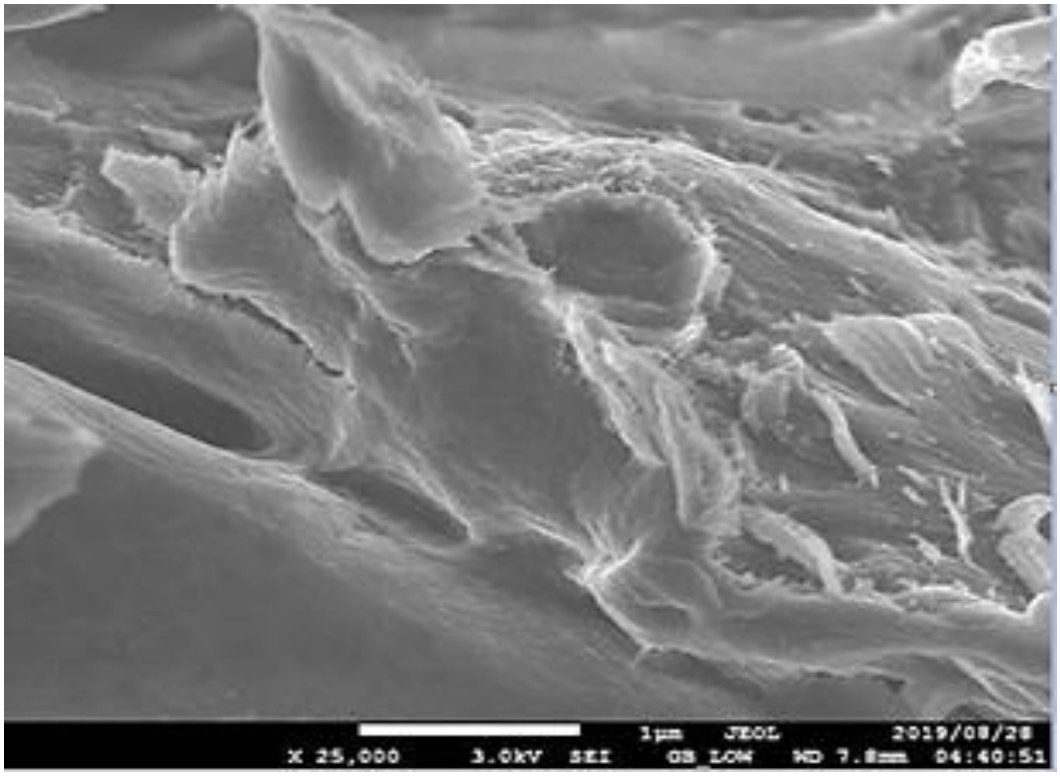
(b)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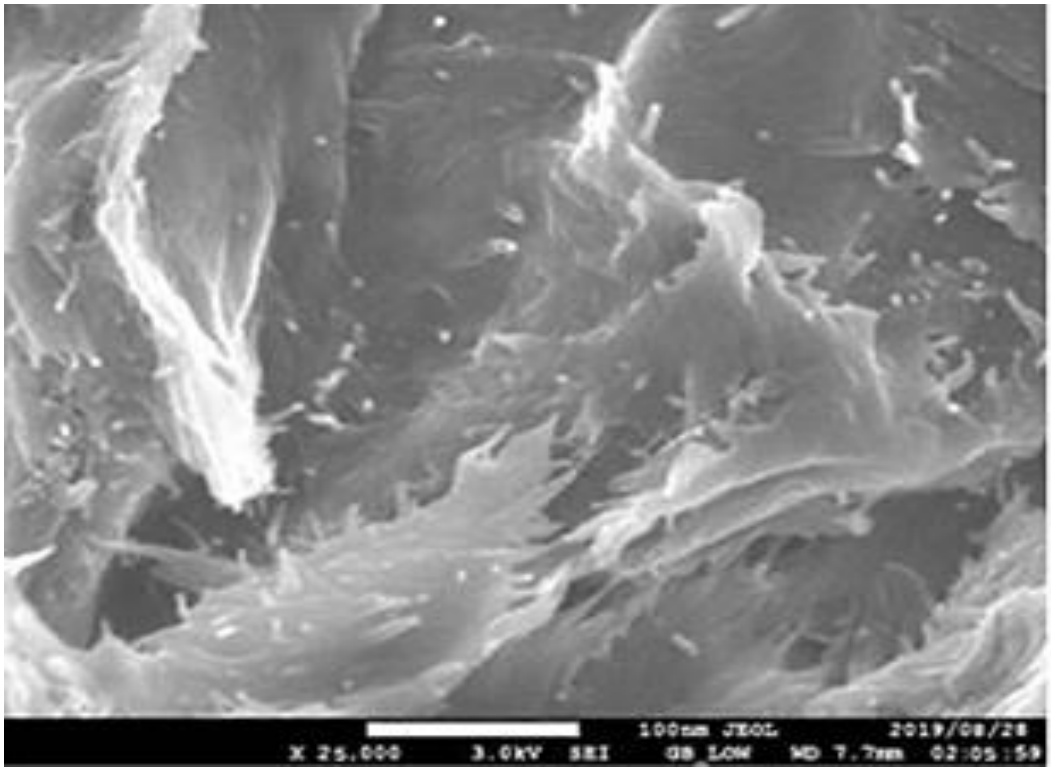
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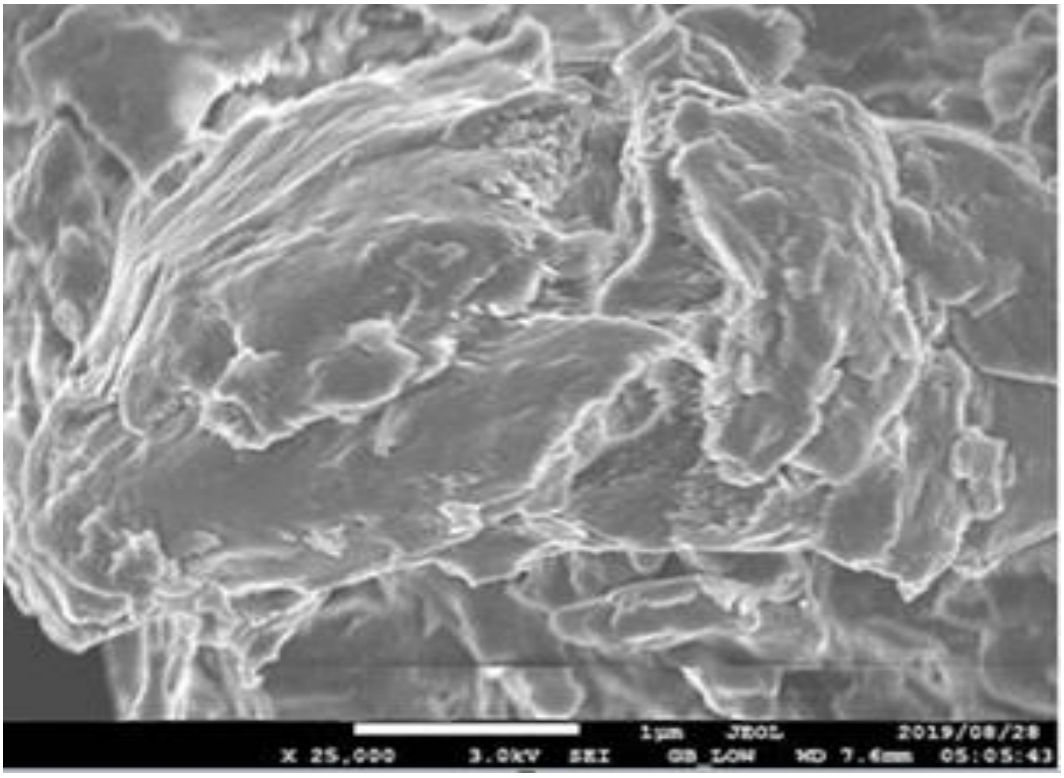
(d)



(e)

Plate 1. SEM of OPPF**. (a)** Untreated **(b)** $C_4H_6O_3$, **(c)** $KClO_3$, **(d)** NaOH, **(e)** ZnCl.

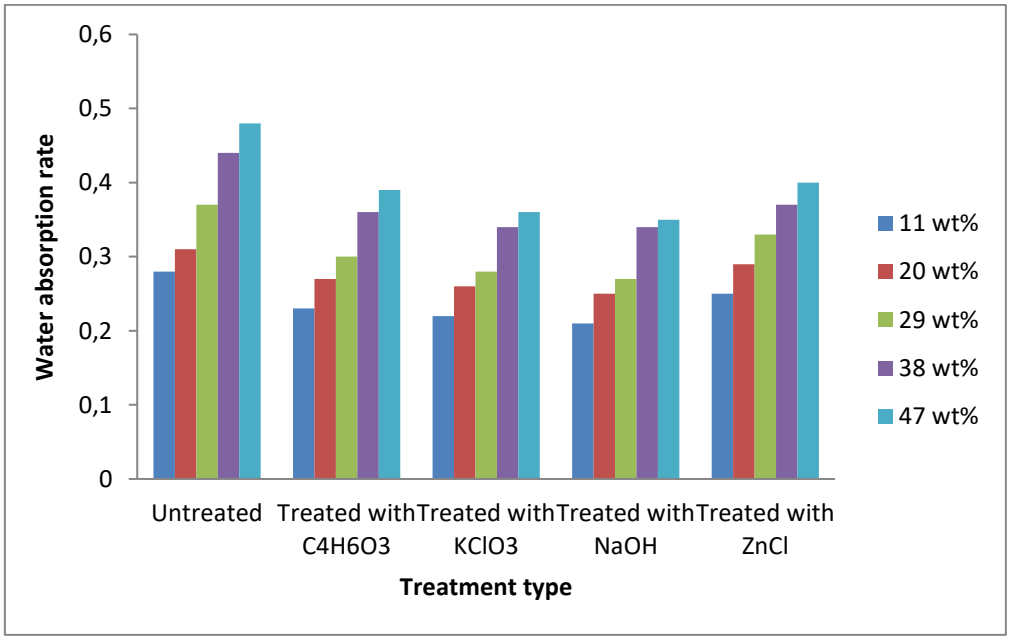


Figure 4. Effect of fiber/polymer ratio on water absorption rate on the composites.

According to Jighesh et al, (2016) water absorbed by fibers in the composite could lead to swelling and dimensional instability and to a loss of mechanical properties due to the degradation of fibers and the interface between the fiber and matrix so chemical treatment on the fibers reduced the issue of swelling and consequently better properties are seen. It was seen also that in the chemically treated fibers, the rate of water absorption increased as the fiber content increases in the composite produced till there were no much increment.

This is similar to the report of Alamril and Low (2012) which stated that the amount of water absorbed increases with fiber ratio of the composite. Tesfamariam et al (2019) stated that the water absorption property of composites reinforced with natural fibers and their derivatives are dependent on the amount of the fiber, fiber orientation, immersion temperature, area of the exposed surface to water; the permeability of fibers, void content, and hydrophilicity of the individual components.

3. 5. Mechanical properties of the composites

Effect of Fiber/polymer ratio on tensile strength of untreated and treated fiber composites is presented on Figure 5. It is seen that tensile strength of the composite made from chemically-treated OPPF has higher tensile strength than that of the one made from untreated OPPF. This might be as a result more exposed surface on the treated fibers that is readily available to react with another surface. It was also observed that as fiber/polymer ratio increased, the tensile strength increased till maximum fiber/ polymer ratio of 29/69 was reached then after this point, there was a decrease in tensile strength.

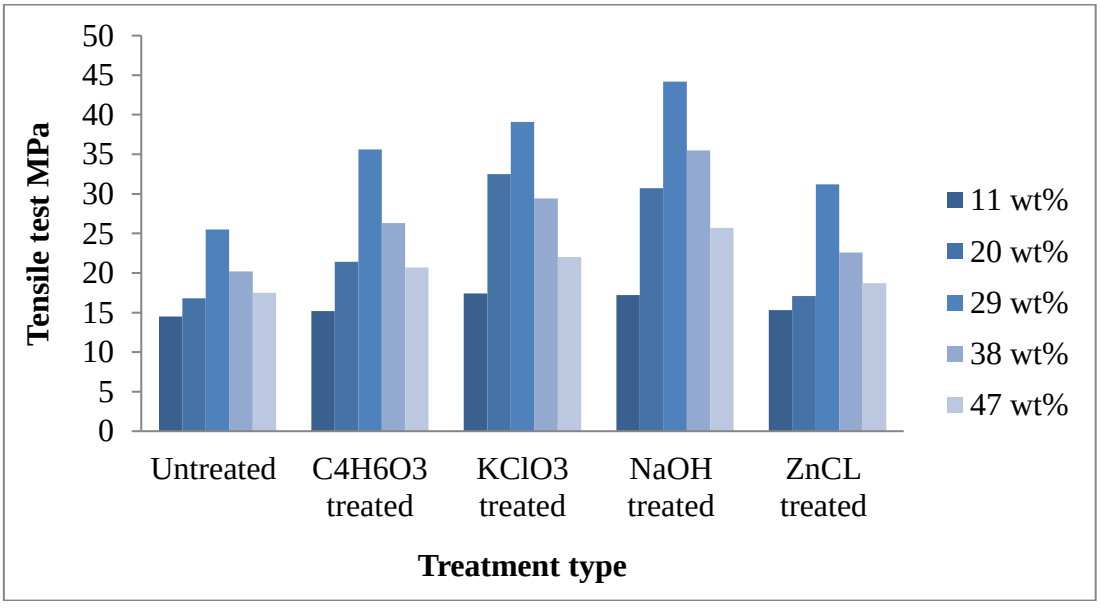


Figure 5. Effect of fiber/polymer ratio on the tensile strength of the composites.

This might be as a result of inefficient fiber/polymer bonding at a higher fiber ratio that can lead to fiber–fiber contact, which results in inefficient fiber–matrix bonding and a lower stress transfer between fibers and the matrix (Al-Khanbashi et al (2005) or as a result of inadequate fiber to reinforce the matrix of the composites. It could also be due to poor

wettability, high porosity and voids which results to a weak interface and inefficient stress transfer or non-uniform stress transfer due to fibers touching each other within the matrix. Weight fraction ratio of fiber to polymer has a great effect on the mechanical properties of a fiber. The composite made from NaOH-treated OPPF gave the highest tensile strength of 44.2 MPa then $KClO_3$ (37.1), $C_4H_6O_3$ (35.6), and ZnCl (28.2). Treatment of natural fibers with NaOH makes the fiber surface to be rough leading to improved interfacial bonding between fiber and matrix.

Goud and Rao, 2011) opined that alkalization causes fiber bundle breakages (fibrillation) and this increases the effective surface area available for wetting by the matrix which leads to a better fibre/matrix interface due to the reduction in fiber diameter, increased fiber aspect ratio and rough surface topography hence improved mechanical properties.

3. 5. 1. Flexural strength

Flexural strength of untreated and treated composites is presented in Figure 6. It is observed that flexural strength of OPPF/HDPE composite was and increased with increase in fiber content up to 29 Wt%, then there was a decrease in flexural strength at a higher fiber contents. The reduction could be as a result of inadequate filling of HDPE resin into the OPPF during production of the composites causing fibre/fibre interaction instead of fibre/matrix interaction. Mahdavis et al (2010) stated that petiole of palm fibers have high length thus, high flexrual properties. Like the tensile strength, the composite made from NaOH-treated OPPF gave the highest flexural strength of 45.4MPa then $KClO_3$ (42.8), $C_4H_6O_3$ (38.6), and ZnCl (35.12).

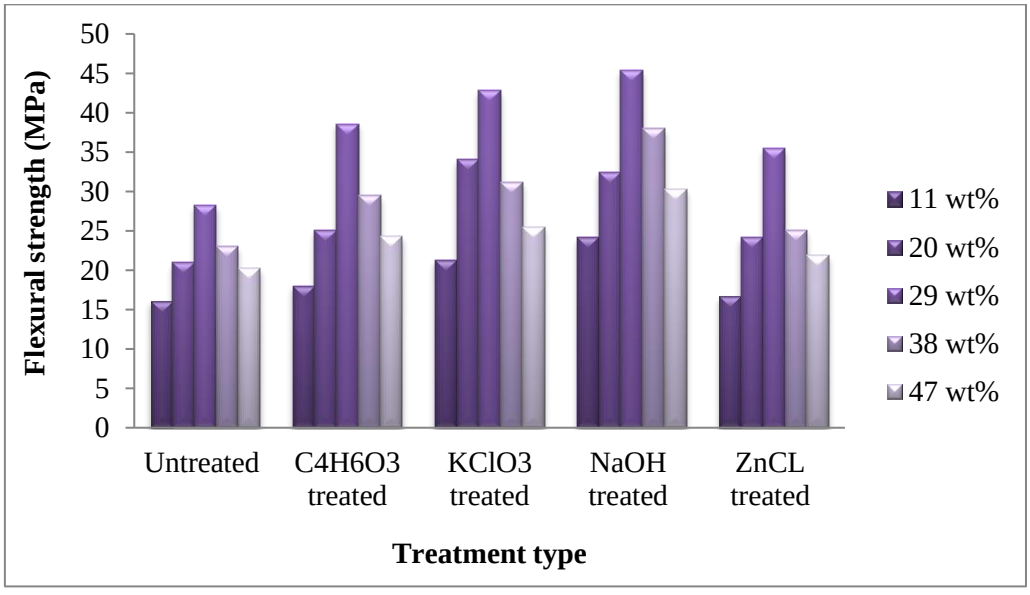


Figure 6. Flexural strength of untreated and treated SPPF/HDPE composites.

3. 5. 2. Impact strength

The impact strength of untreated and treated composite with variation in filler content of the fiber is shown in Figure 7. It is observed that the impact strength increased steadily with

increase in filler content for both untreated and treated composites till 29wt% after which there is a slight reduction in the impact strength. This may be as a result of the existence of weak interfacial interaction between the filler and matrix for higher filler concent beyond 29 wt%. Bengtsson et al (2007) opined that natures fibers behaves like stress concentrates in a polymer matrix thereby reducing the crack initiation energy and consequently the impact strength of the composites. Composites made with NaOH treated OPPF gave the highest impact strength of 55.1 J/m, then $KClO_3$ (54.7 J/m), $C_4H_6O_3$ (48.5) and ZnCl (43.5 J/m). This might be as a result of influence chemical treatment has on the interfacial bonding between fiber and matrix. Also the chemical removal of cementing material exposed the OH groups on the fiber surface, thereby enhancing a better bonding between the OPPF and HDPE. So the treatments of the OPPF enhanced the compatibility and improved the ability of the composite to dissipate energy during fracture.

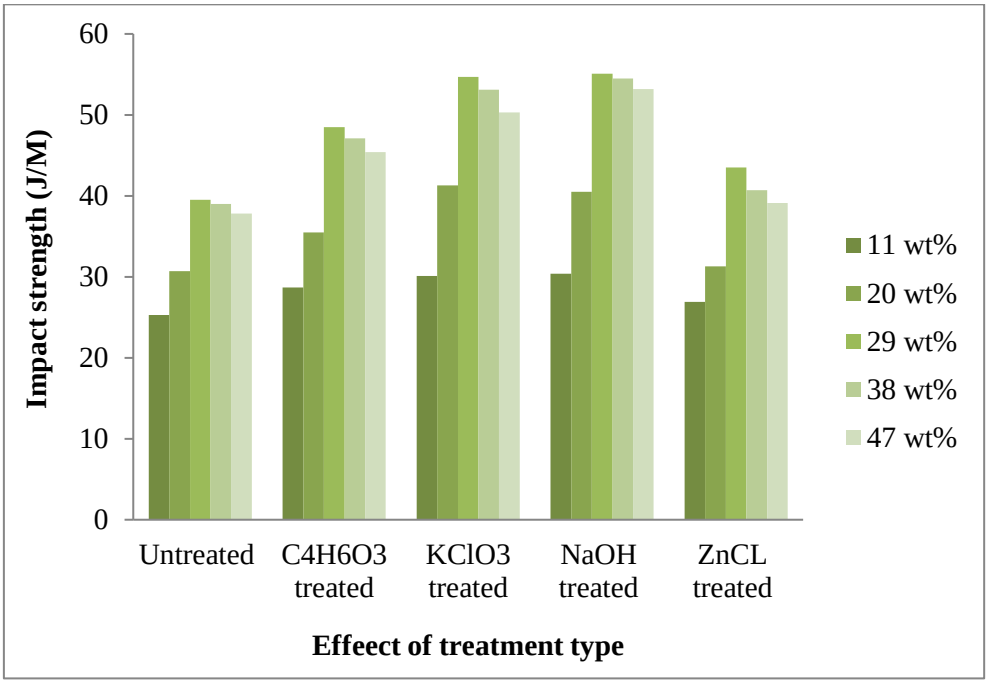


Figure 7. Impact strength of untreated and treated SPPF/HDPE composites.

4. CONCLUSIONS

From the experimental results and analysis of the data, the following conclusions were made:

i. The percentage yields of cellulose on the fiber were high, especially after the chemical treatment.
ii. From the FTIR result, it is observed that there were structural changes after chemical treatment. Some of the functional groups were removed thereby making the active sites (hydroxyl group) in the fiber ready for reactions.
iii. Surface modifications carried out on the OPPF, enhanced the morphological and chemical properties of the fibers. When surfaces of both treated and untreated fibers

were compared, the treated one has more exposed surface readily available for reaction than the untreated fiber.

iv. Water absorbed by the untreated fiber was higher than the chemically-treated fiber.

v. The mechanical properties of OPPF reinforced high density polyethylene composites increased as the fiber content increased, until it got to an optimum level where it started decreasing.

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