ISSN No:-2456-2165

Treatment Time Influence of Alkali Upon it's Characteristics for the Palm Oil Plant Fiber Composites

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Abstract:- Natural fiber finds better inter-facial interaction with the matrix when treated with chemical agents. With this knowledge, a sodium hydroxide (or Alkali) treatment was used to treat the fiber gotten from Oil Palm Fruit. Between 0 and 6 hours, the fiber got submerged in the solution of alkaline at various intervals. The optimal soaking time was determined through investigating their influence on the physical and mechanical features of the polyester resin composite, reinforced with fiber at various immersion period of time. The composite was prepared by hand-laying it up. At an optimum soaking time of 6 hours, maximum tension strength (8.496 MPa) and impact strength (84.605 j/m^2) were obtained, while the untreated material gave 7.012 MPa and 113.684 j/m^2, respectively. The maximum bending modulus (4487.867 MPa) and strength (104.312 MPa) were obtained at 4 hours of soaking time, as their untreated counterparts gave 41.007 MPa and 5026.187 MPa, respectively. The maximum water absorption (1.096%) and density (1113.860g/cm^3) were determined at 6 hours, while the untreated gave 1.230% and 1099.890 g/cm^3, respectively. The result of the SEM showed better fiber-matrix surface adhesion in the untreated fiber than in the treated fiber. The TGA/DTA results revealed an improvement in the composite's thermal stability at 450°C. The fibers had a higher proportion of crystallinity, according to the XRD test. These findings demonstrated that, at various ideal soaking times, NaOH treatment enhanced the characteristics of oil palm fruit fibers.

Keywords:- *Scanning Electron Microscope, Thermogravimetric Analysis, X-Ray Diffraction, and Oil Palm Fruit Fiber.*

I. INTRODUCTION

A composite material (also known as a blended material or composition) constitutes of a substance formed from multiple constituent materials that possess very different characteristics on their own yet when put together, give rise to a material with attributes that are distinct from the sum of the parts. To distinguish composites from mixes and solid solutions, the individual parts in their final form continue to be distinctive as well as separated.

These composite materials are classified based on their matrix material. Thus, composites are classified into these groups. Composites made from metal, ceramics and polymers. composites of polymers are of two types: Particle- and fiber-reinforced polymeric composites. Fibers and an adhesive are used for manufacturing fiber-reinforced composites. The adhesive holds the individual fibers together in shape and transfers stresses between the reinforcing fibers, whilst the fibers are the reinforcement material and the primary provider of strength. As a result, the loads are transported by fibers across the transverse orientations.

These fibers are used in making fiber composites due to their lightweight, excellent strength-to-weight ratios, corrosion resistance, ease of fabrication, design flexibility, self-lubricating properties, better coefficient of friction, wear resistance, and high durability. The properties of these fibers make them attractive materials as compared to others, and as such, they can be applied in the manufacturing of engineering objects and components such as wall, floor, window, and door frames; brake pads; panels for false ceilings; particle boards; bath units; suitcases; helmets; roof tiles; chairs; tables; etc.[1,2,3]

The polymer matrix is hydrophobic in nature, and the natural fiber, being hydrophilic in nature, leads to poor surface interaction of the matrix with the fiber. This, therefore, causes deterioration in the composites' mechanical characteristics.[4] To introduce compatibility between the

ISSN No:-2456-2165

natural fibers and the polymer matrix, the natural fiber is modified, and the effectiveness of interfacial adhesion is improved. Chemical modification of natural fiber exposes more reactive groups on the fiber surface and thus promotes more efficient coupling with the polymer matrix. These modifications, such as maleated coupling agents, alkali treatment, silane coupling agents, acetylation treatment, isocyanate treatment, and grafting, have been conducted to improve fiber-matrix adhesion.[5]

Hassan et al.[6] looked into the tension and bending characteristics of an epoxy-incorporated palm oil devoid fruit bundle fiber using unidirectional alignment. The fiber-matrix bond in the composite was improved by alkaline treatment of the fiber surface using 1% sodium hydroxide solution. For fiber orientations of 0°, 45°, and 90°, the research was conducted. The outcome demonstrated that the composite will produce greater tensile and flexural strengths the higher the angle of the fiber orientation.

Faizi et al.[7] examined the effects of various soaking times in an alkaline solution on the fiber in oil palm empty fruit bunches (OPEFB). NaOH was applied to the OPEFB fibers for 12 hours, 1 hour, 3 hours, 5 hours, 7 hours, and 24 hours at a concentration of 5%. In comparison to other soaking times and untreated fiber, the results showed that the fiber exhibits the best tensile strength at 7 hours of soaking time.

Rice husk-stripped oil palm fruit bunch fiber (OPFBF) polypropylene hybrid composite's physical and mechanical characteristics were examined by Hassan et al.[8] in relation to the impacts of chemical treatment. The findings demonstrate that mercerization and acetylation significantly raised the hybrid composite's tensile strength, impact strength, and hardness values. These two chemical modifications thereby improved the properties of the fiber composite.

Obasi et al.[9], in their study of the characteristics of fiber-reinforced epoxy bio-composite made from oil palm, showed that alkali-treated composites performed better than the untreated ones. OPP fibers at showed that the tensile strength, flexural strength, and impact strength all increased with a 20% fiber loading. The composites, therefore, increased with increasing fiber loading.

Wulan et al.[10] examined the structural enhancements of composite materials manufactured from oil palm fruit stalk fiber through the addition of a Carbon Nanotube. It demonstrated that the composite's tensile strength rose for each weight percent of CNT added.

Potassium chlorate, sodium hydroxide, zinc chloride, and acetic anhydride were used as chemical treatments to examine the effects on the characteristics of composites made from oil palm petiole fiber by Ejikeugwu et al.[11]. Superior interfacial bonding was produced by the chemical treatment, which also led to superior mechanical properties.

Thus, Alkaline, silane, and acetyl treatments improved the bond among the natural fibers and epoxy resin, thereby increasing their tensile strength and tensile modulus, according to investigations on the influence of these treatments on the oil palm fiber (OPF)/epoxy composite. This was further supported by the SEM results, which demonstrated that chemically treating Oil Palm Fiber results in higher characteristics than leaving it untreated due to optimal bonding between the surfaces of the matrix and the fibers.

However, this study concentrated on how different soaking times—2 hours, 4 hours, and 6 hours—of alkalization affected the physical and mechanical features of polyester composites reinforced with Oil Palm Fruit fibers.

II. THE METHODOLOGY AND THE MATERIALS

A. Fiber extraction, and materials used:

The fiber from the palm oil fruit was sourced from a village called Nsukka in Nigeria's Enugu State. Sodium oxide granules were purchased at an industrial shop. A supplier in the state of Enugu in Nigeria delivered the unrefined polyester polymer along alongside the appropriate starter (cobalt naphthalate) and the promoter (methyl ether ketone peroxidase). At the oil-producing plant in Nsukka, Nigeria, the fibers were removed from the fruit after the oil and kernel were taken out. They were thoroughly washed in water with a surface-active agent and sun-dried for 21 hours. The fibers were separated from the whole and weighed to 100 grams, respectively, using the electronic weighing balance for each treatment specimen. The Sodium hydroxide treatment agent was prepared. The standard, 1 mole (40 grams) of NaOH pellets, were properly dissolved and reacted in 100 ml of water. Thus, the alkali solution was prepared using 600 ml of water and reacted with 240 grams of NaOH, which produced sodium and hydroxide ions. During the reaction, lots of heat was emitted, showing the reaction to be exothermic. Hence, 600 ml of solutions of Sodium hydroxide were prepared for each treatment time. Three samples of the separated 100-gram OPFFs were soaked in NaOH for 2 hours, 4 hours, and 6 hours, and afterward sun-dried for 36 hours. The treated OPFF was matted using the conventional random mat method, and the hand lay-up method was adopted for the preparation of the composite. The unsaturated polyester was used as a matrix, cobalt naphthenate as a catalyst, and methyl ethyl ketone as an accelerator. An Acrylic metal sheet coated with a remover of 300 mm x 300 mm x 10mm dimensions is used as a mold where the composite is poured into it. Using the roller, the liquid composite was pressed for the proper spreading of resin and void minimization. Then the mold was allowed to cure for 24 hours and post-cure for 240 hours (10 days) at room temperature.

B. Characterization

The tensile test was performed with the aid of the universal testing machine (UTM) (Model: M500-25CT). The material is held by the grips, and a load is applied until failure occurs. The ultimate tensile test is noted, and the stress against strain graph is plotted. Three samples were prepared for each test, and the experiments were carried out three times. The average (MPa) values were used for discussion. Test specimens were prepared into the required

ISSN No:-2456-2165

dimensions according to the dimensions, gauge length, and cross-head speeds of the testing instrument. The flexural Strength and modulus tests were done in a three-point flexural setup. The test specimen was cut according to the dimensions of the testing machine. The tests were carried out with the Universal Testing Machine (UTM). The test specimens were placed in the machine, and force was applied to them until they fractured and broke. The bending strength at the peak was noted, and stress vs. strain graphs were generated. The Impact Strength test specimens were prepared according to the required dimensions of the testing instrument.

During the testing process, the specimen was loaded into the testing machine and allowed to swing until it fractured or broke. The energy needed to break the material was noted and the impact strength was determined using this expression;

$$G_c = \frac{U}{A}(J/m^2) \quad (1)$$

where; Gc = Impact strength; U = Energy of fracture (in joule); A=Cross section area (in m^2).

For the Water Absorption test, the composite samples were immersed in water at 23°C for 96–120 hours and removed at regular intervals (every 24 hours) up to 96–120 hours, at which point most specimens reached their saturation points. At each point of removal, the specimens are patted dry with a lint-free cloth and weighed. An electronic weighing balance was used to check the weight of water absorbed. The percentage water absorption (%W.A.) was determined with this expression:

$$\%W.A = \frac{Wet.weight - Dry.weight}{Dry.weight} \times 100 \quad (2)$$

The density of the composite $?_c$ was determined using this expression: $?_c = ?_f V_f + ?_m V_m$.

First, the mass of the fiber (M_f) and that of the matrix (Mm) were found using an electronic balance, while the volume of the matrix (Vm) was determined using a measuring cylinder. Secondly, the density of the matrix ($?_m$) was determined using the equation for the density of a substance (Mm/Vm). Then, the density of the fiber was determined with the same equation for density, M_f / V_f. The volume of the fiber (V_f) was determined with the aid of the Archimedes principle since it provides a convenient and accurate method for determining the volume of an irregularly shaped object. Using this principle, the volume of the liquid (kerosene) displaced is equal to the volume of the fiber ($V_k = V_f$). Hence, the fiber was submerged in the kerosene; some portion of the kerosene was displaced into a smaller bowl. The values were substituted into the expression for the density of the composite, and the composite density was determined. The Scanning Electron microscopy (SEM) result of the composites was determined with the use of TESCAN VEGA 3 SBU (2 units). This is important to ascertain the structural changes occurring in a fiber composite upon treatment. The Thermogravimetric Analysis (TGA) and dynamic thermal analysis (DTA) of the fiber composite were conducted with the aid of universal V4.5A TA Instruments for evaluating the thermal stability of the fibers, with a weight percent between 0 and 120% and a temperature range of 0 to 500ºC. X-Ray Diffraction (XRD) tests of the fiber composite were performed with the use of an X-ray diffractometer. The diffraction intensity was between 0 and 90˚ at the 2θ scale (Bragg angle), and at 1 second step time, the intensities were between 0 and 900 counts. Percentage Crystallinity (%Cr.) and Crystalline Index (Cr.I) were calculated with the following expressions;

$$\%Cr = \frac{I_{22}}{I_{22} + I_{18}} \times 100 \quad (3)$$

and

$$C.I = \frac{I_{22} - I_{18}}{I_{22}} \quad (4)$$

Where I_{18} and I_{22} are, respectively, the amorphous and the crystalline strengths at the 2θ scale near 18 and 22.[12]

III. RESULTS AND DISCUSSIONS

- *Effect of Alkali Soaking Time on The Mechanical Properties of the OPFF/Polyester Composite*

From Figure 1, the result showed that the maximum tensile strength (8.496 MPa) and impact strength (84.605 j/m^2) of the composite were obtained at the optimum soaking time of 6 hours. This showed that at 6 hours of treatment, the treated fiber might have experienced a stronger effect of the alkali treatment, which gave rise to higher wettability and a higher increase in the composite's impact and tension properties. However, a maximum impact strength of the untreated material (113.684 j/m^2) outperformed the maximum impact strength of the treated material, whereas the maximum tensile strength of the treated OPFF composite outperformed the greatest strength of the untreated material (7.012 MPa).

From figure 2, it showed that the highest flexural modulus (4487.867 MPa) and strength (104.312 MPa) were obtained at 4 hours respectively. It possibly meant that the effect of alkalization was felt more by the fiber at 4 hours of soaking time and thereby increased the impact and flexural strength to maximum properties. However, at the optimum soaking time of 4 hours, the maximum flexural strength performed better than the untreated (41.007 MPa) composite, while the untreated (5026.187 MPa) flexural modulus performed better than the treated at maximum.

From Figure 3, it is shown that the maximum percentage of water absorption (1.096%) of the treated composites was obtained at the optimum soaking time of 6 hours. This might be due to the fiber treatment, which exposed the fiber's chemical properties and thereby absorbed more water after immersion for a total of six hours. It

ISSN No:-2456-2165

therefore revealed that untreated fiber composite (1.230%) obtained a higher value result than the treated composite at maximum percentage water absorption. The treated BFBF composite, therefore, yielded a better result than the untreated composite since the untreated absorbed more water than the treated. The maximum density (1113.860g/cm^3) of the treated composite, as seen in Figure 3, was obtained at 6 hours of soaking time, while the minimum was obtained at 4 hours. At 6 hours of soaking, the maximum density showed better results than both the treated and untreated fiber composite (1099.890g/cm^3). Therefore, the alkali soaking treatment improved the density of the oil palm fiber composite. Generally, Alkali treatment had a significant effect on the composite, as also shown in Table 1.

SEM images showing the six-hour maximum strength and untreated OPFF composites' surfaces are shown in Figures 4 and 5, respectively. In order to observe the adjustments that occur on the interface of the OPFF composites as a result of the alkaline modification, the interfacial structure of its fibers is crucial. The fiber's treated surface didn't appear to differ all that much from the untreated surface. However, the treated composite appears to have more exposure to the fiber chemical properties (hemicellulose and lignin) due to the alkali treatment. It is therefore conclusive that Figure 4 showed a better surface than Figure 5.

Figure 6 displays the Thermogravimetric analysis outcomes for an OPFF composite that was untreated for 6 hours. According to the Figure, the overall change in weight loss took place between 50ºC and 450ºC.The specimen broke down in three distinct stages. The third stage (between 300 and 450ºC) saw the most significant decomposition; the first decomposition took place between 50 and 150ºC, and the second between 150 and 300ºC.The weight loss of the specimens during the first stage, which is caused by the first loss of moisture or solvents connected to the fiber, is around 10%. About 15% of the weight is lost during the second decomposition, which takes place between 150 and 300 degrees Celsius and corresponds to the hemicellulose decomposition. In the third stage, the degradation of the crystalline cellulose caused the majority of the weight loss at temperatures between 300°C and 450°C, which eventually caused the specimens to lose weight by around 70%. The ultimate weight loss occurred at a temperature between 450 and 500ºC as a result of the degradation of the lignin content of the fibers, though it was minimal given that it did not represent the useful life of the fiber. As a result, the TGA results indicated that the fiber composites' thermal stability had increased, and as a result, they can be utilized to process engineering materials at temperatures up to 450ºC.

Analysis was done on the variations in heat deterioration of the fiber composite as indicated in Figure 7. It is clear that the first degradation, or the initial decomposition temperature, took place at a temperature between 50 and 150°C as a result of the loss of moisture or solvents connected to the fiber, which caused the specimens to lose around 10% of their original weight. About 15% of the weight was lost during the second decomposition, which corresponded to the hemicellulose decomposition, which took place at temperatures between 150°C and 300°C.

At a temperature level between 300 and 450 degrees Celsius in the third stage, degradation of the crystalline cellulose caused the majority of the weight loss, which led to a loss of roughly 70% of the specimens' weight.

The ultimate weight loss occurred at temperatures between 450 and 500ºC as a result of the degradation of the lignin component of the fibers, though it was minimal given that it did not represent the useful life of the fiber. Since the material may be used to create engineering components with temperatures between 50°C and 450°C, as shown by the TGA results in Figure 6, the DTA results also showed better thermal stability.

The crystalline and amorphous intensities at the 2θ scale were determined using XRD analysis, as illustrated in Figure 8 of the results. Equations 3 and 4 were used to calculate the Crystallinity percentage (%Cr) and crystalline index (C.I).

From Figure 6 and 7, the amorphous intensity (I18) between 160 and 180 (160≤2θ≤ 180) is 300, and the crystalline intensity (I22) at exact 220 is 360. The crystalline percentage (%Cr) and crystalline index (C.I.) of untreated OPFF are 54.55% and 0.17. The Untreated OPFF has a crystalline index (C.I.) of 0.17 and a crystalline percentage of 54.55%, respectively.

Table 1 Results of the Opff/Polyester Composite's Mechanical and Physical Characteristics.

SOAKING TIME (HRS)	**The Physical and Mechanical Characteristics of an OPFF/polyester composite.**					
	TENSILE STRENGTH (MPA)	***IMPACT STRENGTH (J/M^3)***	***FLEXURAL STRENGTH (MPA)***	***FLEXURAL MODULUS (MPA)***	***WATER ABSORPTION (%)***	***Density (G/CM^3)***
0 (Untreated)	*7.012*	*113.684*	*41.007*	*5026.187*	*1.230*	*1099.89*
2	*4.833*	*38.263*	*62.601*	*4345.246*	*0.797*	*1113.783*
4	5.224	31.711	104.312	4487.667	0.100	1101.870
6	8.496	84.605	51.176	4361.287	1.096	1113.860

ISSN No:-2456-2165

Fig 1 Tensile and Impact Srength of Treated and Untreated OPFF Polyester Composite

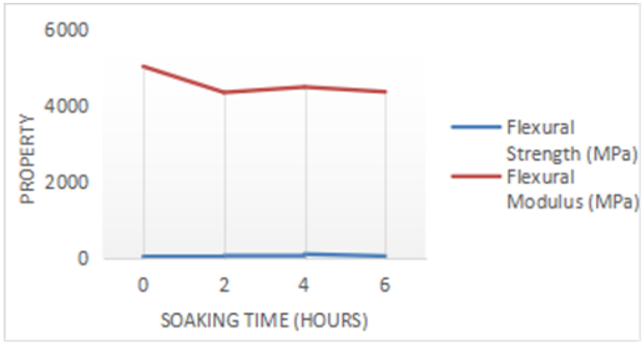

Fig 2 Flexural Strength and Flexural Modulus of Treated and Untreated OPFF Polyester Composite

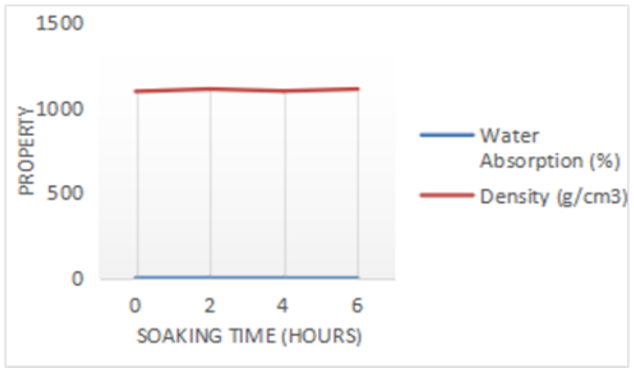

Fig 3Percentage Water Absorption and Density of Treated and Untreated OPFF Polyester Composite

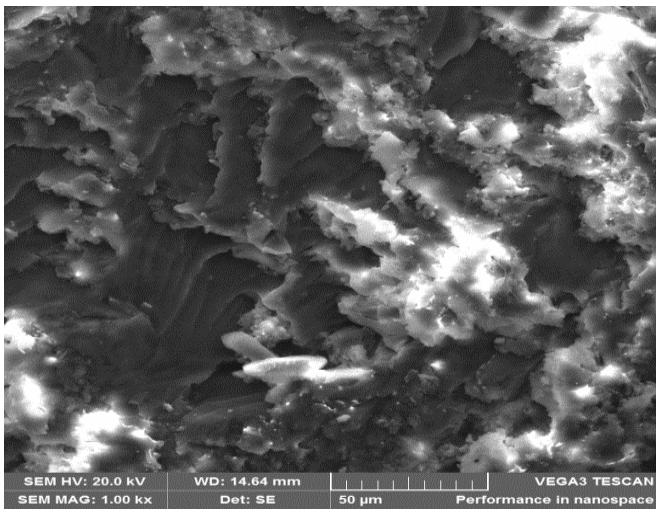

Fig 4 SEM of Fractured Surface at 6hours Treated NaOH OPFF Composite.

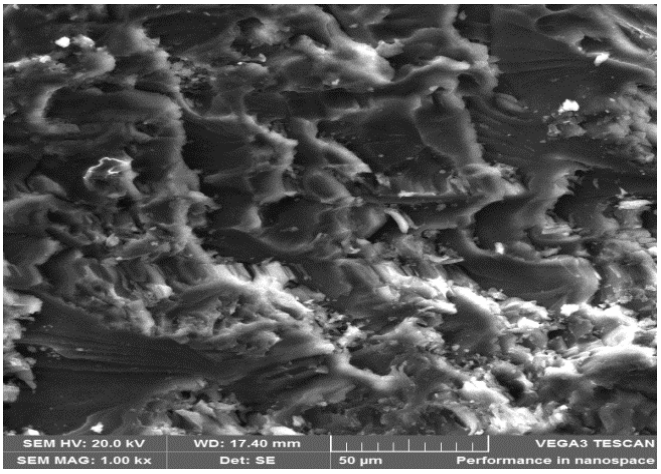

Fig 5 SEM of Fractured Surface at 0hour (untreated) OPFF Composite.

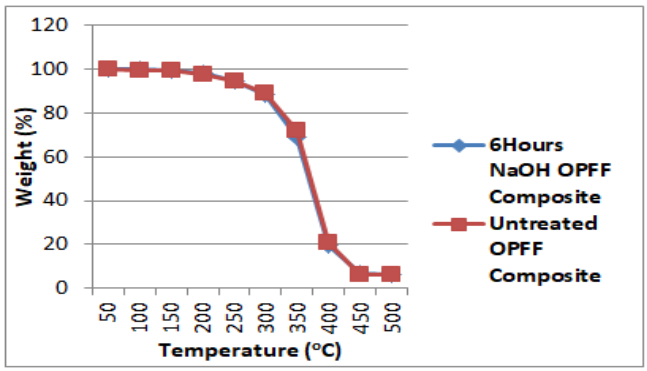

Fig 6 TGA of the 6hours NaOH Treated and Untreated OPFF Polyester Composites

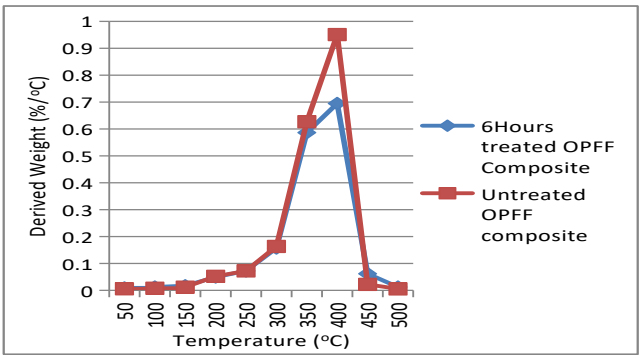

Fig 7 DTA of 6Hours NaOH Treated and Untreated BFBF Polyester Composites

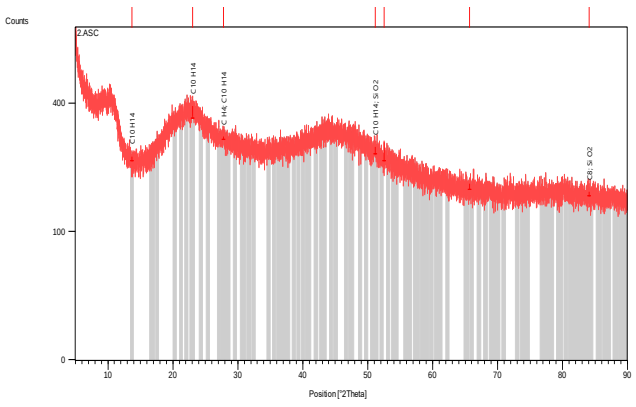

Fig 8 XRD Analysis of Untreated OPFF Polyester Composite

ISSN No:-2456-2165

IV. CONCLUSION

In this study, fiber from an oil palm fruit had been strengthened with a polyester composite and alkali treatment at varied treatment times of 2, 4, and 6 hours.

According to the findings, the ideal soaking duration of 6 hours produced the highest tension and impact strengths, respectively. At the ideal soaking time of 4 hours, the maximum bending strength and modulus were reached. The ideal soaking time of 6 hours produced the highest percentages of water absorption and density, respectively. The treated fiber composite had better fiber-matrix surface adhesion than the untreated, according to the SEM results. The TGA/DTA results showed improved thermal stability, while the XRD of the untreated fibers obtained a greater percentage of crystals. Therefore, with the exception of bending strength and modulus, which occurred at the penultimate treatment time, the NaOH treatment of Oil Palm Fruit Fiber improved the physical and mechanical characteristics of the composites at varied optimal soaking times.

ACKNOWLEDGMENT

The authors would like to thank God for His provision throughout this work and also the Liquefied Natural Gas (LNG) Laboratory of the University of Nigeria, Nsukka, for their support in providing the equipment.

- *Conflict of Interest Statement*

No conflicts of interest were disclosed by the author(s) with regard to the research, writing, or publication of this paper.

- *The Financing*

The author(s) of this paper did all of the research, writing, and/or publishing on their own initiative without any assistant financially.

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