Investigation of the Potentials of Oil Palm Frond Fibre for Thermal Insulation of Food Flasks

1AKABA ABDUL T. 2ADEMOH NUHU A and 3AUWAL MUHAMMED

1,3Department of Mechanical Engineering, Kaduna Polytechnic, Kaduna.

2Department of Mechanical Engineering

Federal University of Technology, P. M. B. 65, Minna

Nigeria

ABSTRACT

Natural fibers obtained from oil palm fronds at Offa, Nigeria were investigated for reinforcement of heat-resistant lining for hot food flasks. Frond fibre was subjected to 5% NaOH chemical treatment to impose better surface property and used to reinforce polypropylene matrix as composites with 10%, 20%, 30% and 40% fibre loading. Treated frond fibre, polypropylene matrix and samples of fabricated composites were subjected to standard chemical, mechanical and physical tests including density/hardness, impact/tensile strengths and thermogravimetric/thermal conductivity analyses following standard procedures. The test equipment included injection molding machine, Instron tensile tester, Tinius impact tester, Rockwell hardness tester, Perkin Elmer instrument and Lees disc apparatus; accessed in workshops/laboratories of Kaduna Polytechnic, Ahmadu Bello University, Zaria, Engineering Materials Development Institute, Akure and Federal University of Technology, Minna. The characterization result showed the composite was nontoxic to humans if it contacted food in the flask. The physical, mechanical and thermal properties showed that only 20%-40% fibre loading was beneficial after which performance declined. Mechanical properties of frond reinforced composites (density, hardness, impact and tensile strength) were adequate for required service condition. The decomposition temperature of the composites was about 480°C which was above 350°C-435°C for most conventional domestic insulators, 290°C for pure treated frond fibre and 4350°C for polypropylene matrix. Thermal conductivity reduced from 0.0035Wm⁻¹K⁻¹ to 0.0025Wm⁻¹K⁻¹ for 20%-40% 10mm thick fibre loaded composites. For 50mm thick conventional domestic insulators, it was 0.11-0.23Wm⁻¹K⁻¹ for polypropylene matrix, 0.066Wm⁻¹K⁻¹ for rock wool, 0.036Wm⁻¹K⁻¹ for glass fibre and 0.03Wm⁻¹K⁻¹ for polystyrene, confirming the superiority of the frond reinforced composites as minimized thermal conductivity is the most critical determinant of material suitable for thermal insulation.

Keywords: Palm frond fibre, Composite, Thermal insulator, Food flask, Thermogravimetric analysis.

1.0 INTRODUCTION

Oil palm tree belongs to agricultural species called elaeis guineensis. It is perennial oil tree crop with high economic potential due to many products derivable from it. Among products from the tree are assorted fibres derivable from trunk, bunch, fruit mesocarp and frond that may be used for industrial applications. After harvest of palm fruits most biomass fibre generated from the mesocarp is dumped to waste away causing nuisance in farms. The fibres create impediment to farming activities or are at times incinerated releasing green house gases causing atmospheric problem. Several studies showed that rather than burn off, they can be put to industrial use like replacements or compliments to synthetic fibres which are more difficult to bio-degenerate after use and disposal. Bio-fibres like oil palm are eco-friendly, biodegradable, cheap and readily available as farm waste. The raw properties are close to those of synthetic fibres before treatment which makes them find wide use in production of polymeric composites for component parts of varied engineering system. Moreover natural fibres are known to have series of advantages over synthetic fibres in areas like low density/cost, non-toxicity quick biodegradability, environmental friendliness and good mechanical, thermal/electrical insulation properties (Shehu et al, 2014). Study showed applicability of natural fibres is mostly
limited by factors like poor wettability, incompatibility with some polymeric matrix, non-mixing with some binders and high moisture absorption ability solution to which is provided by chemical/biological treatment to make them function like synthetic fibre (Abdul Khalil et al, 2012).

Like other natural fibres, oil palm frond is economically and ecologically considered useful as alternative to reinforcement fibre in polymeric composites due to low density and cost as compared to synthetic type (Ademoh and Olaniyekun, 2015). High moisture absorption ability due to its hydrophilic nature adversely affects mechanical properties such as flexural strength, modulus, fracture toughness; causes delamination, poor interfacial bond between resin matrix and fibre which reduces performance in service. Treatment on natural fibres depends on targeted end use. It is usually done before use to reinforce polymeric composite to make them less hydrophilic for wettability of binder for better mechanical properties. After treatment, frond fibre may be used in particulate reinforced composites (PRC) which are types of reinforcements having all dimensions roughly equal; used to raise high temperature performance, reduce friction, shrinkage; increase wear resistance and stiffness rather than improve strength as particles share load with matrix to lower extents than synthetic fibre (Rana et al, 1998). Fibres are readily derivable from different sections of palm tree like trunk, frond, fruit flower bunch and fruits as shown in figure 1 (Abdul Khalil et al 2012). This work is mostly interested in fibres generated from fronts of oil palm tree. Figure 2 shows a branch of palm frond detached from tree (Ademoh and Olaniyekun, 2015). Palm frond fibre contains high percentage of cellulose that has good potential as natural fibre resource, although its application accounts for small amount of total biomass reinforcement in polymer composite products. Studies showed it can be used as an ineffective reinforcement filler in thermoplastic and thermostetting materials (Khalil, et al 2008).

Oil palm frond fibres may be used in fibrous reinforced composites (FRC) where fibers are not directly applicable engineering materials due to their small cross-sectional dimensions but rather embedded in matrix materials to form fibrous composites so that the matrix serves to bind fiber together, transfer load to fibers, protect them against environmental attack and damage on handling (Ademoh and Olaniyekun, 2015). Fiber reinforced composites contain fillers that have length higher than cross-sectional dimension. Fibrous reinforcement provides more physical than chemical means of changing materials to suit various engineering demands (Warner, 1995). Hybrid composites are those incorporated with two or more types of fillers especially fibers in a single matrix; commonly used to improve service properties and lower cost of synthetic composite (Mondadori et al, 2008). A general rule of mixture is applied in the formulation of hybrid composites so as to optimize the benefits of individual constituent in the product. To use this rule, volume fractions are calculated from the density of individual constituent in the formulation bearing in mind the targeted properties which in turn is based on required service conditions. Volume fractions are computed using equation 1:

\[ V_j = \frac{v_j}{V} \]  

where \( V \) =volume fraction of constituent; \( v_j \)=volume of individual constituent and \( v \)  = volume of total constituent (always equal to 1). Theoretical target density of hybrid composite is then determined from equation 2 by using symbols \( \rho_i \), \( \rho_j \), and \( \rho_k \) as densities of each constituent as follows:

\[ \rho_{\text{composite}} = \frac{\rho_i v_i + \rho_j v_j + \rho_k v_k}{V} = V_i + V_j + V_k \]  

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\[ V_i, V_j \] and \( V_k \) are the volume fractions of each constituent in composite. Based on this flexibility in multiple formulation methods, fibre reinforced composites have advantages of high corrosion/fire resistance; high toughness/mechanical damping; excellent tensile/fatigue strength and low cost/density over other competing material (Tiesong et al, 2008). Fibre reinforced composites can easily be formulated for use in multidisciplinary applications like for thermal insulation as proposed in this work. A thermal insulator is single material, mixed materials or composites placed in-between two or more surfaces reduce heat flow from a point to another. Evaluation of its effectiveness is based on such properties as thermal conductivity which depends on physical and chemical structure of base materials. Thermal conductivity is a measure of flow of thermal energy through unit thickness of a material under given temperature gradient. Relationship of variables under steady state condition is proved by Fourier’s Law. The modes of heat transfers in fibrous insulators include solid fiber conduction, still air conduction, convection and radiation. Changes in conductivity in heat transfer were observed to vary with increased filler in fibrous installation. Convection decreased significantly due to impediment of convective current; radiation gradually decreased because of scattering and emission at higher density (Al-Nasearawi, 2008). Lower thermal conductivity gave better heat insulation. Thermal insulators can be made fibrous, cellular, flake like or granular. Its application can be anywhere from wearing...
It is the flexibility and adaptability of the material that allows it to fit to the common shapes found in industrial processes especially as piping and furnace insulation. Typical installations are cryogenic, HVAC and moderate temperatures.

2.0 MATERIALS AND METHODS
2.1 Materials
The raw materials used in the research work included sodium hydroxide pellets distilled water and graded polypropylene (PP) used as polymer matrix were purchased from standard laboratory chemical dealer at Kaduna. Fiber was generated from fronds of oil palm trees at commercial plantation in Offa, Nigeria. MFI for polypropylene was made as 1-12 g/10 min (at 230°C; maximum of 2.16 kg) with density 0.9 g/cc. Figure 3 shows the macro appearance of polypropylene as matrix to produce research specimen prepared with equipment including injection moulding machine (Model EC1300SX), tensile test machine (Instron Model 3360 series test system), impact test machine (Tinius Olsen Model 899) and hardness test machine (Rockwell B scale-HRB). Other equipment included Perkin Elmer instrument (Model 4000) and Lee’s disc apparatus; accessed at Kaduna Polytechnic, Engineering Materials Development Institute, Akure, Federal University of Technology, Minna and Ahmadu Bello University, Zaria, Nigeria.

2.2 Methods
2.2.1 Fibre Treatment:-The spine fibres extracted out of raw palm fronds as shown in figure 2 were naturally retted for four weeks in water at room temperature in preparation for further treatment. Retted fiber was manually cut to average length of 5 mm and subjected to chemical treatment.

Fiber Surface Modification:-Prior to the chemical treatment, bundles of retted fibers were scoured in mild detergent solution at room temperature for 2 hours to remove dust and other impurities. Thereafter, the fibers were washed in distilled water and air dried for two days. Figures 4 and 5 showed the physical states of the fond fibres before and after cutting them into some recommend sizes for the treatment.

Mercerization Treatment:-Mercerization is an alkali treatment process that subjected vegetable fibres to interactions with fairly concentrated aqueous solution of strong base in order to produce great swelling that result to changes in fibre structure, morphology, thermal and mechanical properties (Bledzki A.K and Gassan J., 1999). It was conducted by immersing palm frond fiber in 5% sodium hydroxide (NaOH) for 1 hour at room temperature. John et al (2008); Ademoh and Olanipekun (2015) showed 4-5% NaOH was most effective range of concentration for this type of fibre as higher concentrations caused delignification resulting to fibre surface damage. The frond fiber was washed with distilled water and under continuous stream of running water until total removal of NaOH, dried at room temperature for 24 hours and stored.

Figure 3:-Macrostructure of the Polypropylene
2.2.2 Fabrication of Composites:-Different grades of reinforced composite samples were prepared using compositional mixture of polypropylene matrix and treated fond fibre filler presented in table 1. Dried treated fond fibres were crushed, ground and vibrated in BS standard sieve to obtain particle sizes 50µ, 75µ and 100µ that permitted close interparticulate mix and good compaction with granular polypropylene material. Each mixed specimen was separately introduced into hopper, moved to the mixing chamber and compounded in the injection moulding machine. In the chamber, composite was heated to a temperature range of 150°C-180°C and sucked to die head. With the aid of pump, mixture was transferred into mould which was thereafter closed with a pressure setting of 100 bars. Specimen was allowed to cure within the chamber for 20 minutes in accordance with ASTM D-3039 standard used by Singha et al (2008). Tensile, impact and hardness specimens were prepared according to ASTM D-3039. Similar procedures were followed to fabricate the samples presented in table 1. Figure 6 shows a representative thermal composite sample produced with the 200g capacity injection moulding machine equipped with 75°C cooling system.

Table 1 Compositional percentage constituents of the reinforced composite specimens

| Sample Code/Composition | A   | B   | C   | D   | E   |
|-------------------------|-----|-----|-----|-----|-----|
| Polypropylene Polymer Content in Composite (%) | 100 | 90  | 80  | 70  | 60  |
| Fond Fibre Content in Composite (%) | 0   | 10  | 20  | 30  | 40  |

2.2.3 Mechanical and physical Testing of Fibres:-The tensile properties of both treated and untreated fibres were determined using Instron universal test machine (model 3396). Tests were conducted on five carefully selected representative samples each of treated and untreated palm frond fibre. Result obtained is presented in table 3. A digital micrometer screw gauge was used to measure the diametric thicknesses while a well calibrated meter rule was used to measure the length of the fibres.

For density test, dried samples of treated and untreated fibres were weighed and recorded as w. Distilled water was poured into a measuring cylinder with initial volume noted and recorded as V₁. Weighed dried fibre was put into measuring cylinder with distilled water and final volume was noted and recorded as V₂. This was repeated for each sample and density was calculated using equation 1 (Khalil et al 2011).

\[
\frac{W}{V_2-V_1} \times 1000 \text{Kg/cm}^3 \quad \text{................................. (3)}
\]

2.2.4 Chemical Analysis of Fibre:-Chemical analysis of fibre was carried out to enable determination of percentage composition of cellulose, hemicellulose, lignin and ash content in treated and untreated fibres. In accordance with procedures used by Ververis et al (2007), 0.7g of ground frond fibre was hydrolysed by boiling in 5ml of 72% H₂SO₄ solution for \( \frac{41}{2} \) hours. The suspension remaining after the hydrolysis was filtered. Residue was oven dried at 105°C for 24 hours, left to cool in desiccators, weighed and recorded as W₁. The dried sample was transferred into pre-weighed porcelain crucible, placed in furnace, heated at 600°C for 5 hours, allowed to cool, weighed and recorded as W₂. Percentage ash content was determined and acid insoluble lignin was calculated by difference in recorded weight (i.e. W₁-W₂). A volume of 5ml of filtrate was measured from the hydrolysis containing sugar released from cellulose and hemicelluloses, neutralized with 10% concentration NaOH, stirred and diluted to 100ml. Glucose C₁ and reducing sugar C₂ concentration in filtrate were determined using glucose peroxidase assay kit using dinitrosalicyclic acid (DNS) methods respectively. This was done by determining rates of absorption with spectrophotometer. Solutions used to determine C₁ and C₂ were formulated as practiced by Ademoh and Olanipekun (2015):
1ml of sample (filtrate) + 2ml of reagent A
1ml of glucose (Standard) + 2ml of reagent B
1ml of sample (filtrate) + 1ml of DNS + 2ml of H2O
1ml of glucose (Standard) +1ml of DNS + 2ml of H2O
The solutions were heated up in boiling water bath for 5 minutes and absorbance was determined using spectrophotometer at 540nm. Thus values of C1 and C2 were determined.

Percentage composition of cellulose and hemicellulose in starting material was calculated as follows:

%w/w cellulose content = (0.9/0.96) x C1 x (V/M) x α x 100
%w/w hemicellulose content = (0.88/0.93) x (C2-C1) x (V/M) x α x 100 (Ververis et al, 2007); where:
0.9 is the coefficient of the molecular weight ratio of the polymer and monomer hexose;
0.96 is saccharification yield; 0.88 is the molecular weight ratio of the polymer and monomer pentose;
0.93 is saccharification yield of xylene to xylose; C1 is the determined glucose concentration in (g/L);
C2 is the determined reducing sugar concentration in (g/L); V is the total volume of sugar solution (L);
M is the dry weight of the fibre (g); α is the dilution factor. (For this analysis dilution factor was 0.05).

2.2.5 Composite Characterization:- Characterization of newly formulated thermal insulator composite was done to assess its ability to withstand physical, mechanical and thermal stresses it will encounter in serve and preservation of heat within vessel by minimizing thermal conductivity to limit rapid heat loss. Performance of natural fibre based composite is controlled by properties of fibre-matrix adhesion and bonding (Singha et. al. 2008). Primary requirement for effective use of reinforcement properties of natural fibres is good interfacial bond (adhesion) between polypropylene matrix and fiber. Interfacial adhesion or bonding nature has significant effect on strength of fibre reinforced composites (Ismail et. al. 2004).

**Tensile Strength:** It measured the maximum applied load/stress that a specimen could withstand before breakage. Tensile strength highly depended on adhesion between fiber and matrix, fiber length and fiber loading (Chai et al. 2009). It is important as material will subjected to tension by weight of its content. The test was conducted on computerized universal machine in accordance with ASTM D-3039 standard method. A sample of 10cm length was clamped into the two jaws of machine. Each end of jaws covered 2cm width of sample as in figure 7. Tensile strength was measured over gauze length with initial reading set at zero, stressing was done at a constant strain rate of 10mm/min until failure occurred and load with extension curves drawn. The experimental results were instantaneously read from machine and recorded.

**Impact Test:** The impact resistant of the palm frond reinforced insulator composite was investigated with an Izod impact tester to determine its toughness. The notched izod impact strength of the specimens was evaluated using an impactometer manufactured by Tiniusolsen, USA in accordance with ASTM-D-256 with a notch depth of 2.54 mm and notch angle of 45° with results recorded in Joules per meter. Notches on specimens illustrated in figure 8 were made by a specimen notch maker attached to the instrument.

**Hardness Test:** The test was done with Rockwell hardness tester on B scale (HRB), according to ASTM D-224 standard to measure material resistance to surface indentation, friction and wear. Samples were prepared according to the ASTM method where the base and load bearing surfaces were made parallel. A standard block with hardness 101.2HRB was first tested to assess condition of machine as a routine check. A hardened steel ball indenter was forced onto specimen surface via minor load of 98N.
followed by major load of 980N to make indentation as shown in figure 9. Three indentations were made across length of a specimen with average hardness taken instantaneously and recorded.

Figure 9: Rockwell hardness test sample.

Thermo-gravimetric Analysis, (TGA):-Analysis was conducted to predict thermal behavior by providing basic information on the material thermal stability. Thermo-gravimetric analysis (TGA) and differential thermal analysis (DTA) studies on the newly fabricated composites samples were conducted on Perkin Elmer analyzer at heating rate of 10ºC/min under nitrogen atmosphere with flow rate of 20ml/min. TGA characterized decomposition/thermal stability of materials under variety of conditions. Principally change in thermal stability was valued in terms of %weight loss as function of temperature. Simultaneously DTA compared the precise temperature difference between a sample and an inert reference material while both are heated. DTG was a thermal analysis done to study rate of material weight changes versus temperature upon heating. Results were used for simplified study of recorded weight loss versus temperature.

Thermal conductivity:-Thermal conductivity analysis was conducted on composite specimens with 40mm diameter by 10mm thickness as in figure 10 using Lee’s disc apparatus. A brass disc (B) was hung from a stand with help of 3 strings. On the brass disc (B), a sample disc (S) was placed. Metallic disc (M) was placed on top of sample disc (S). On the metallic disc, a heating chamber (H) with facility for in and out passage of steam created. 2 holes were made in brass and metallic discs for insertion of thermometers.

Before test, mass of brass disc (B) was measured on digital balance. Diameter of specimen was measured with vernier caliper and thickness was measured with a micrometer screw gauge. Heater (H) was started by sending steam through the heating chamber of apparatus. Temperatures $T_1$ and $T_2$ were recorded at regular intervals of 5 minutes until steady state was reached by when steam supply was cut off. The upper metallic disc (M) and sample disc (S) were then removed. Steam was again passed to heat the brass disc to 100ºC above steady state temperature $T_2$. Thereafter, the heating chamber was removed to allow brass disc to cool. Temperature was noted after every 30 seconds until it fell to about 100ºC from steady state temperature $T_2$. A graph was drawn with the cooling time set as abscissa and temperature of brass disc as ordinate. A tangent was drawn at steady state temperature $T_2$, the slope of which gave rate of cooling ($\frac{\delta T}{\delta t}$) at the steady state temperature $T_2$. Value of ($\frac{\delta T}{\delta t}$)$_{T2}$ was the slope of the temperature time graph at steady state temperature. This and temperature differential ($T_1$-$T_2$) were determined from the result and graph as:

$$\frac{\delta T}{\delta t} = 4.103 \times 10^{-3} \text{C/s}; \text{ and } (T_1-T_2) = 336\text{K}.$$  

Computation was based on the fact that thermal conductivity of material is its property that describes rate at which heat flows within it for given temperature change. The rate of heat conducted through specimen or sample was then given as in equation 3:

$$Q = KA \frac{(T_2-T_1)}{L}.$$  

where: $L$ was thickness of sample; $A$ was the cross sectional area of sample; $K$ is thermal conductivity; $Q$ is rate of heat transfer and ($T_1$-$T_2$) was the temperature difference.

The rate of heat loss by the brass disc (B) to the surrounding under steady state was given as.

$$Q = mc \frac{(\delta T/ \delta t)}{T_2}L.$$

where, $m$ was mass of brass disc (B), $c$ was specific heat of the brass disc (B) and ($\delta T/ \delta t$) was it’s rate of cooling at $T_2$. Comparing equations (3) and (4) gave equation 4 as:

$$K = mc \frac{(\delta T/ \delta t)A}{L} \frac{T_2}{T_1}.$$

These were calculated from Lee’s disc method resulting graph based on input value of mass of brass disc (B), specific heat of brass disc, thickness and cross sectional area of sample. Thermal conductivity of palm frond reinforced composites with different loading in table 2 were used to carry out the analysis.
The untreated fibre. It had higher tensile rigidity, cleared impurities adjoining fibre material in service. Mechanical properties of fibre adhesion.

To be noted, there was increase in the cellulose content in the treated oil palm frond (Reza et al., 2013).

Table 4 above, it can be deduced from the result that, there was increase in the cellulose content in the treated oil palm frond fibre in respect of about 36.88%. The hemicelluloses content of the fibre got decreased by 67.43%; lignin content was decreased 58.28%.

Table 2: Temperature and time for determination of thermal conductivities of palm frond composites

| S/N | Fibres                          | Temp. (°C) for 20% Frond content | Temp. (°C) for 30% Frond content | Temp. (°C) for 40% Frond content |
|-----|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
|     |                                 | 5                               | 10                              | 15                              | 20                              | 25                              | 30                              |

Table 2: Temperature and time for determination of thermal conductivities of palm frond composites

| Temp. (°C) for 20% Frond content | 38 | 36 | 34 | 35.5 | 31.6 | 30.9 |
| Temp. (°C) for 30% Frond content | 35 | 32 | 31.5 | 33 | 30.5 | 30.2 |
| Temp. (°C) for 40% Frond content | 32 | 30 | 29.5 | 28.7 | 26.3 | 27.1 |

Data analysis for the thermal conductivity test: From the experimental result,

Sample thickness X=10mm = 0.01m; Diameter of sample d = 40mm = 0.04m;

Cross-sectional area of the sample \( A = \pi r^2 = \pi d^2/4 \);

Constant variables from above equation are:

Mass of brass (B) \( M = 0.097kg \); Cross-sectional area of sample = \( 1.2568 \times 10^{-3} m^2 \);

Sample thickness = 0.01m; Specific heat of brass disc = 0.38KJ/Kg.K

From equations 2 and 3 above of Fourier’s equation of heat transfer, From equation 4 above;

\[ K = mc \left( \frac{\delta T}{\delta t} \right) \frac{L}{A} (T_2 - T_1) \]

Where \( k \) = thermal conductivity of the sample

From the curve of temperature against time the thermal conductivity of the composites were calculated at different fiber constituents. Table 3 showed the values obtained from the experiment.

To calculate the thermal conductivity \( K \) of frond composite at 20% fiber loading is as follows:

\[ K_{20} = \frac{0.097 \times 0.38 \times 4.103 \times 10^{-3} \times 0.01}{1.2568 \times 10^{-3} \times 336} = 0.00358 \text{w/mk} \]

\[ K_{40} = 0.00333 \text{w/mk} \]

\[ K_{60} = 0.00269 \text{w/mk} \]

These computational procedures yielded \( K_{20} = 0.00358 \text{w/mk} \) and \( K_{40} = 0.00269 \text{w/mk} \) that were plotted.

3.0 RESULTS AND DISCUSSION

3.1 Results of the physical and mechanical properties of treated and untreated frond fibre: - Table 3 below shows summary of the results obtained from the physical and mechanical properties tests of the treated and untreated oil palm frond fibre to be used as fillers in the thermal insulator composites.

Table 3: Physical and Mechanical Properties of the Fibres

| S/N | Fibres                          | Length (mm) | Diameter (mm) | Density (X 10^3Kg/cm^3) | Tensile strength (Mpa) | Young Modul (Gpa) | Elongation at break (%) |
|-----|---------------------------------|-------------|---------------|--------------------------|------------------------|------------------|-------------------------|
| 1   | Untreated palm frond            | 653 - 955   | 0.28 - 2.23   | 2.65                     | 9.123-139.673          | 0.343-3.567      | 2.655-4.381             |
| 2   | Treated oil palm frond          | 652 - 953   | 0.26 - 1.33   | 1.78                     | 19.497-459.66          | 0.178-9.412      | 4.924-9.597             |

The density of treated fibres was observed to be lower than that of the untreated oil palm frond fibre by, a reduction of 58.50%. The treatment reduced weight of fibres which is a more desirable property for the material in service. Mechanical properties of untreated palm frond agreed with values obtained from past works by Ademoh and Adesoji (2015); Abdul Khalil (2012). Close observation of the diameters of fibres showed that treated fibre had reduced diameter than the untreated fibre. It had higher tensile strength than untreated fibres with average of 14.38%-53.04% increase in strength of frond fibre. Chemical treatment with NaOH had advantage of removing moisture content from fiber to increase strength, flexural rigidity, cleared impurities adjoining fibre and stabilization of molecular orientation. Alkaline treatment is well-known for surface modification of plant fiber for reinforcing polymer as it removed lignin, hemicellulose, wax and oil that covered fibre surface (Reza, Jamaludin and Abdul Rahman, 2013). It led to fibrillation that caused break down of composite fibre bundles to smaller ones, reduced diameter, increased aspect ratio which developed rough surface topography that gave better fibre/matrix interface adhesion.

3.2 Result of chemical analysis of fibre: - The basic chemical constituents of palm frond for treated and untreated fibres as analysed in their natural combined forms are presented in table 4.

Table 4: Result chemical analysis of treated and untreated oil palm frond fibre

| S/N | Fibre                     | Cellulose (%) | Hemicellulose (%) | Lignin (%) | Ash (%) |
|-----|---------------------------|---------------|------------------|------------|---------|
| 1   | Untreated oil palm frond fibre | 59.84         | 17.97            | 19.65      | 2.3     |
| 2   | Treated oil palm frond fibre   | 82.10         | 5.98             | 9.94       | 1.6     |
by 49.42% while the ash content was decreased by 34.45% due to NaOH chemical treatment which removed most of naturally accompanying constituents of fibre that caused undesired properties in the raw material. Mercerization left fibre with higher cellulose content which made it have higher strength and lower hydrophilic nature. It showed increase in cellulose with attendant decrease in hemicellulose, lignin and ash constituent of the treated over untreated fibre. The chemical analysis showed that none of the components of the palm frond fibre is toxic or radioactive; showing its suitability for use in manufacturing heat preservation food flask. Moreover the growing concern of the world on environmental degradation, renewability of raw materials, disposability of used industrial wastes coupled with abundance of palm wastes and quest to increase local content of industrial consumables in Nigeria, there is great potential for use of the fibre for polymer composite reinforcement.

3.3 Result of mechanical properties test on palm frond reinforced thermal insulator composite:- Variation of fibre content in composite to arrive at acceptable compositions with suitable properties for industrial application to ascertain whether these natural fiber reinforced biopolymers possess promising properties to replace traditional composites. The tests included hardness, impact and tensile strength. The result of the analyses on the plain polypropylene used as the matrix in the composite is as in table 5.

### Table 5: Properties of the experimental Polypropylene

| Polymer   | Tensile strength (MPa) | Impact strength (J/m²) | Hardness (HRB - No) | Density (Kg/m³) | Thermal Conduct. (W/m·K) |
|-----------|------------------------|------------------------|---------------------|-----------------|--------------------------|
| Graded PP | 25 – 40                | 20 - 100               | 80 - 100            | 900 - 910       | 0.11 – 0.23              |

The results were set aside and used as control data for comparison and validation of the result generated from analyses done on the newly formulated palm frond reinforced thermal insulator composites.

**Impact test**: Result of the notched izod impact test are as presented in figure 11 for insulator composites with different four different percentage constituents polypropylene and palm frond natural fibers. It should be noted that 10% and above 40% fibre contents were also experimented with and discovered not viable in the preliminary experimental test trials and therefore cleaned up from the results presented.

![Figure 11: Result of impact strength (J/m) for palm frond reinforced thermal insulator composite](image)

The result showed increase in impact resistance with increased fibre content. Plain polypropylene with 0% binder had impact resistance of 20J/m. It increased steadily from 20% fibre (30J/m) to 40% fibre content (40J/m). It showed improvement in property over the virgin material (with no fibre) and thus, increased ability of composite to withstand higher impact load especially in form of weighted solids that may be thrown onto the material during use. Impact resistance of composite is known to be affected considerably by the selection of fiber reinforcement and matrix. For instance Chai et al (2009) showed that adding untreated softwood fiber to reinforce polypropylene caused reduction of impact strength with increased fiber content. A study by Plackeet et al (2003) showed that when fibre was treated, its attendant improved adhesion reaction between fiber and matrix caused considerable increase in impact resistance. Thus, increased impact resistance of composite reinforced with treated fond fibre over plain matrix was due to increased adhesion between fiber and matrix. According to Maryam Talimi (2011), beyond certain fiber content, there was no improvement in impact properties as was observed during initial trials of this work where 40% fibre content was noted as the highest fibre composition for best performance.

**Hardness test**: The hardness of the polypropylene matrix and natural frond fiber based thermal insulator composites were investigated with the result presented in figure 12.
Fiber content increase of 20-40% caused increased hardness from 80HRB no of polypropylene matrix to about 88HRB no at 40% fibre in composite. Increment in hardness of composite was quite significant between 20% and 30% fibre while it was at 30-40% fibre and thereafter decreased. Reason being that as it was for impact strength test result, reduction in hardness at over 40% fibre content was due to reduction in adhesion between frond fibre and matrix which reduced the bond strength holding the two together.

Tensile strength:- Tensile strength of the thermal insulator composites was investigated under different palm frond filler constituents; result of which is presented in figure 13. It is observed that tensile strength of pure polypropylene of 25MPa (table 5) was increased by the addition of frond fiber from 20% through 30% to 40%. The values are also higher than tensile strength for untreated (9.14mPa) and treated (19.50) pure frond fibre (table 3) showing that fabricated composite is stronger than any of the constituent being used separately. Increment in tensile strength followed trends in impact and hardness results as it increased significantly with a steeper slope between frond contents of 20% and 30%; while the increment was gradual with a lower slope between 30% and 40% with insignificant effect at over 40% filler content.

The increase in tensile strength was attributable to chemical modification treatment on frond fibers which promoted a level of chemical interaction between front particle and polypropylene matrix by way of close adhesion. Weyenberg et al (2005) reported alkaline treatment gave up to 30% increased tensile strength in flax fiber based epoxy composite. Jacob et al (2009) agreed with this as they asserted that composite strength/physical properties depended on fibers interaction with polymer matrix up to limited amounts. This showed ability of fibre to support stress transmitted from polymer matrix was relatively improved by treatment.

Adequate tensile strength is vital for this type of composite to enable it withstand weight of its content. Increase in tensile strength up to 40% fiber indicated ability of fibre to absorb stress transmitted from polymer matrix. Composite with higher fiber (over 40%) was reported to have greater tendency of filler-to-filler interactions took to form more voids. This initiated crack formation and propagation in composite that led to lower strength when compared with moderate fiber loadings of 20% to 40% (Jacob et al, 2009). Another reason composite with over 40% fiber have poor strength is probably because high fiber content caused more degradation during processing when composite was compounded the friction between fibers raised temperature. Basically, higher fibre in composite causes higher friction that leads to more degradation. As natural fibers have low thermal stability, they will degrade at temperatures above 200°C leading to loss of strength. Thus composite with over 40% fiber were not considered suitable.

3.4 Result of physical property tests on frond reinforced composite:- The physical and thermal properties of frond reinforced composites made for thermal insulation of food flasks were investigated to check its suitability as renewable source of bio-degenerable replacement for synthetic material. Such tests including thermogravimetric analysis (TGA), derivative thermogravimetric graph (DTG), density and thermal conductivity were conducted on standard specimens adopting procedures of past related works.
Result of the density tests on the composites:

The result of density tests on fabricated thermal insulator composites is presented in figure 14. It is observed that density of composites decreased with increasing content of frond fibre due to the fact that the added filler had lower density than the polypropylene.

![Figure 14: Result of density tests of thermal insulator composites with varied palm frond loading.](image)

The density of composite was higher than that of palm frond fibre but lower than values for polypropylene polymer matrix. This is desirable property as high material density will unnecessarily increase weights of flasks that it is used to insulate. When compared with densities of common heat insulating materials like rock wool, glass fibre and polystyrene below it is suitable as it is lighter than all except some rook wool.

**Thermogravimetric Analysis (TGA):**

This analysis (TGA) was used to investigate possible temperature related effects on the properties of palm frond reinforced composite fabricated for use as heat resistant padding in food flasks. Thermal degradation was carried out at programmed rate of 10°C/min from room temperature to 600°C under constant nitrogen flow. Onset temperature (T_o), 50% degradation temperature (T_{50%}), peak degradation temperature (T_P) and degradation temperature range (T_R) were obtained using TGA and DTG curves. Figure 15 is a combined plot of the thermal degradation of treated frond fibers, pure polypropylene and newly formulated thermal insulator frond based composite.

![Figure 15: A plot of the combined TGA of pure palm frond, pure-polypropylene and the composites](image)

The TGA thermograph shows that frond fiber has the first peak degradation temperature of 287°C. This is because devolatilization of moisture in frond fibers occurred at a temperature range of 70–100°C even after frond fiber had been incorporated into the polypropylene matrix. The moisture content might have played a significant role in degradation processes due to the fact that OH group in water was more reactive than OH groups in frond fiber. TGA thermograph may be probably due to chemical reagent used for treatment which had tendency of absorbing moisture from environment. The thermal degradation of frond fiber was due to decomposition of cellulose, lignin and hemicellulose which gave off its volatiles (Abubakar, 2006). Frond based composites started to decompose at about 450°C (onset temperature T_o). Loss in mass in composites in one-step degradation procedure started at about 450°C and continued very slowly until at 480°C beyond which progression occurred quickly to a final decomposition temperature of about 500°C. Pure polypropylene started degrading at decomposition temperature of 435°C while treated frond had peak degradation temperature of 287°C which were both below the start of degradation (450°C) of the composite. This indicated that the thermal stability of frond reinforced composites was over that of pure polypropylene or treated fond fibre to further show benefit of reinforcement process. Comparatively degradation temperatures of most conventional domestic product insulators are in a range 300°C–400°C; also below values of the new composite, thus confirming its superiority and suitability for the purpose.

Figure 16 shows derivative thermograph (DTG); plot of derivative weight (%/min.) against temperature for polypropylene matrix (red curve) and thermoplastic composite of varied fibre loading (blue curves).
TGA in figure 15 for frond fibre and polypropylene resin used to form thermal insulator composite was studied as function of % weight loss against increasing temperature. At the start of DTG presented in figure 16, depolymerization and dehydration took place between temperature ranges of 191°C-230°C followed by cleavages of C-H, C-C and C-O bonds in cellulose portion of fibre (Singh et al, 2008). Initial decomposition temperature (IDT) and final decomposition temperature (FDT) of oil palm fibers had are between range 250-400°C. However the initial decomposition temperature of the polypropylene resin was 435°C and its final decomposition temperature was 490°C. In differential thermogravimetric analysis of natural fibre reinforced composite have, in addition to exothermic peaks endothermic peaks at different temperatures when compared with parent polymer matrix (Singha et al, 2009). Inspection of magnitude and location of peaks in DTA/DTG curves showed there was change in thermal behaviour of the polymer when reinforced with frond indicating decreased degradation process of bio-composites with added fibre.

This is very beneficial for intended application as thermal insulator. Result showed that the composite is more thermally stable than raw polypropylene resin. Beydokhti et al (2006) showed most conventional synthetic materials have lower decomposition under thermal load than natural fibre reinforced composite. Singha et al (2008) Stated that for composite to effectively bear load, fibre and matrix must interactively cooperate. The cooperation of natural fibre and polymer here depended on presence of good interfacial strength which was based on surface topology of frond fibre. The interface acted as binder and transferred load between matrix and fibre. The available interfacial area played major role in determining strength of composite because each fibre formed individual interface with matrix. Cellulose was a major component in frond fibre with strong hydroxyl groups in structure that formed hydrogen bond within macromolecule itself and the hydroxyl groups of polymer. In fact during fabrication of composite fibre acted as carriers of load, transferred stress from matrix along reinforcements to give good mechanical and thermal properties.

**Thermal conductivity:**-Lee’s disc apparatus was used to determine conductivity of frond fiber reinforced composite by measuring initial and final temperatures ($T_1$&$T_2$) of sample and computing the result shown in figure 17. The outcome was used to evaluate performance of the composites for thermal insulation.

The figure showed variation of thermal conductivity of composites against percentage fibre contents. It is observed that thermal conductivity decreased with increased fibre loading in composite just as reported by Dedeepya et al (2012) who measured mechanical properties like tensile strength, tensile modulus and thermal conductivity of typhangustifolial fibre reinforced composite with universal strength test machine and guarded hot plate apparatus respectively. Figure 18 showed that increasing frond fibre from 20% to 30% sharply decreased thermal conductivity; but the decrease was not significant at 30%-35%; tended to stabilize or start to increase at 35-40% fibre. Low thermal conductivity is a very desirable property for thermal insulating materials. Comparing result of frond reinforced composites with pure polypropylene (table 5), showed the composite had lower thermal conductivity and further proved effectiveness of the reinforcement process. Thermal conductivity of composite was better than for conventional materials like glass fiber, rock wool and polystyrene in table 6 (Abubakar et al, 2005). Each composite was
found to be lower and more preferable than conductivity of conventional materials. The table shows existing insulator materials in the table all have higher thermal conductivities than frond fibre reinforced composite.

### Table 6: Comparative thermal conductivities of selected insulation materials.

| Material     | Thermal Conductivity i.e. k (Wm⁻¹K⁻¹) | Density (kg/m³) |
|--------------|----------------------------------------|-----------------|
| Rock Wool    | 0.016-0.026 (50 mm thick)              | 9.6-32          |
| Glass Fiber  | 0.036                                 | 64-140          |
| Polystyrene  | 0.03                                  | 29-56           |

Moreover thickness of tested conventional insulation materials was 50mm while that of frond reinforced composite was 10mm. Al-Naseerawi (2008) had noted that increasing thickness of natural fibre based composite decreased thermal conductivity. Thus the composites are not only suitable for use as insulator lining but also provide better insulation at lower weight and cheaper cost for hot food carrying flasks.

### 4.0 CONCLUSION

This work showed the potentials of biodegradable natural fibre generated from fronds of oil palm tree as an effective reinforcement for polypropylene polymeric material for fabricating heat resistant linings for heat bearing food flasks. Characteristics of the composite showed it is non toxic and wouldn’t pose food poisoning danger to humans if it gets in contact with contents of flask. Physical, mechanical and thermal properties showed that increased fibre loading was mostly beneficial between 20% and 40% after which composite performance depreciated as reported in past related works. The mechanical properties of palm frond based composites including hardness and impact/tensile strength were adequate for required service conditions. Density is one of the important physical properties considered for such materials. Result here wasn’t only within acceptable limit but was better than those of some conventional thermal insulators. Decomposition temperatures of composites were above the 350°C-435°C for most conventional domestic insulators. Conductivity reduced from 0.0035Wm⁻¹K⁻¹ to 0.0025Wm⁻¹K⁻¹ for 10mm thick frond reinforced composites of 20-40% fibre but values for 50mm thick conventional thermal insulators are 0.066Wm⁻¹K⁻¹ (rock wool), 0.036Wm⁻¹K⁻¹ (glass fibre), 0.03Wm⁻¹K⁻¹ (polystyrene); 0.11- 0.23Wm⁻¹K⁻¹ (polypropylene). This confirmed superiority of frond based composites over most conventional materials as low thermal conductivity is a very critical determinant of material suitability for thermal insulation applications. This fascinating result should prompt research studies into suitability of fibres derived from other sections of oil palm like trunk, fruit bunch stalk and fruit mesocarp after oil extraction for thermal insulation polymer composite reinforcement. Incorporation of different coupling agents to optimize the interface reaction of fiber with matrix to raise mechanical and thermal properties of composites is recommended for further work. Optimization of fiber surface property by using other mercerization chemical reagents for better performance of oil palm fibre based composite may be considered for future study series.

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