

# Effects of Surface Modifications on the Mechanical Properties of Reinforced Pineapple Leaf Fibre Polypropylene Composites

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## Abstract

Pineapple Leaf Fibre (PALF) is one of the natural fibres that have high potential in the industry. Natural fibres have become the main alternative source for reinforced polymer composites. The objective of this study is to observe the effect of chemical treatments using Sodium Hydroxide (NaOH) solution, Zinc chloride, Acetic Anhydride and Nitric acid on the mechanical properties of pineapple leaf fibre reinforced polypropylene composites. The tensile test was conducted by using the ASTM D638-10 to obtain the tensile strength (TS) and Young's modulus (YM), Flexural properties were conducted to determine the flexural strength (FS) and flexural modulus (FM) of the reinforced composites using the ASTM D256-10 method, and impact test was conducted to determine the impact strength (IS) of the reinforced composites using the Izod ASTM D790-17 method. From the results obtained, the composites with surface modified PALF fillers show enhanced mechanical properties over the untreated PALF fillers in this order; for TS untreated composite < modified with NaOH < modified with  $C_3H_6O_3$  < modified with ZnCl < modified with  $HNO_3$ . For YM untreated composite < modified with  $HNO_3$  < modified with  $C_3H_6O_3$  < modified with NaOH < modified with ZnCl. For FS, untreated PALF/PP composites < modified with NaOH < modified with ZnCl < modified with  $HNO_3$  < modified with  $C_3H_6O_3$ . For FM, untreated reinforced PALF/PP composites < modified with NaOH < modified with ZnCl < modified with  $C_3H_6O_3$  < modified with  $HNO_3$ . For IS, the untreated reinforced PALF/PP composites < modified with NaOH < modified with  $HNO_3$  < modified with ZnCl < modified with  $C_3H_6O_3$ . SEM analysis was carried out on the PALF before the compounding to analyze the effect of the surface modification agents.

## Keywords

Surface, Modifications, Properties

## 1. Introduction

Surface treatment or surface modification increases the surface roughness of natural fibres, it removes lignin, wax and oils of the fiber cell walls and disrupts hydrogen bonding in the network structure. In other words, increment of effective contact area in the fibers creates higher adhesion between the fiber and the polymer matrix, thus, improving the mechanical properties of the composites.

A number of studies have been carried out on applicable fibre surface modification agents, also, a number of researchers have been carried out using different methods to achieve better composites productions. However, no known research has been carried out in running a comparative study of a number of surface modification agents in this case, ZnCl, NaOH, HNO<sub>3</sub> and C<sub>3</sub>H<sub>6</sub>O<sub>3</sub>. This study tends to analyze which of the aforementioned surface modification agents gives PALF/PP reinforced composites with enhanced mechanical properties.

Among the various factors, the final performance of the composite materials depends to a large extent on the adhesion between the polymer matrix and the reinforcement and therefore, on the quality of the interface. To achieve optimum performance of the end product, sufficient interaction between the matrix resin and the cellulosic material is desired. This is often achieved by surface modification of the resin or the filler.

Fibres obtained from pineapple leaves have been previously studied by several authors, Arib *et al.* [1]; Asim *et al.* [2]; Chollakup *et al.* [3]; Devi *et al.* [4]; George *et al.* [5]; Hujuri *et al.* [6]; Shyamal *et al.* [7]; Smitthipong *et al.* [8]. Compared to other natural fibres, pineapple leaf fibres (PALF) exhibit superior mechanical properties due to its high cellulose content (around 70% - 82%) and low microfibrillar angle (14°) [9]. PALF are obtained from the leaves of pineapple plant *Ananas comosus*, which is a perennial herbaceous plant, widely cultivated in tropical regions of Asia, Central and South America [2]. After harvesting, the pineapple plant has to be removed, generating a large volume of wastes. Wastes consist of leaves of 30 to 90 cm length [10] [11] and they usually contain herbicides spreading into the air during incineration processes [10]. Moreover, postharvest utilization of pineapple waste would be an alternative and renewable source of natural fibers for industrial purposes since it can add value to pineapple cultivation and reduce negative environmental impacts in the field. Recently, PALF have been studied by several authors as a reinforcement in thermoplastic materials such as low-density polyethylene (LDPE), polypropylene (PP) and starch/poly (lactic acid) (PLA) as reported by [1] [3] [5] [8] [12] [13].

Daramola *et al.* [14] reported the variation of the ultimate tensile strength for neat Polyester and PALF/Polyester composites. The ultimate tensile strength of the neat polyester was 5.11 MPa. It was observed from the results, that, the strength of composites increases linearly from 10 wt% PALF/Polyester composite to 40 wt% PALF/Polyester composite where the optimum value of 29.19 MPa was observed. The incorporation of treated PALFs into polyester matrix at weight fraction of 40 wt% produced the increase in ultimate tensile strength by

about 471%, according to their results. The general improvement in the ultimate tensile strength of the treated PALF/Polyester composites is attributed to the enhancement of fibre-matrix interaction and more effective transfer of stress.

Nayan *et al.* [15] studied the effect of mercerization process on the structural and morphological properties of Pineapple Leaf fibre (PALF) pulp and reported that then tensile strength of the un-mercerized PALF bundle corresponds well with previously reported works. Treatment with NaOH increases the tensile strength of mercerized PALF due to the removal of impurities and poor crystalline structure of hemicelluloses and lignin. Kasim *et al.* [16] reported that an alkaline treatment was conducted to enhance the PALF properties. The fabrication was made by compression molding technique with random orientation of PALF. From the experimental study, the results revealed that the voids percentage and interfacial bonding between the PALF and PP affected the mechanical properties of the PALF/PP composite. Kaewpirom and Worrarat [17] studied pineapple leaf fibre-reinforced poly (lactic acid) green composites by blending of poly (lactic acid) polymer and short-length-chopped Pattawia pineapple leaf fibres (1 - 3 mm) using a twin-screw extruder. Effects of the fibre content and the addition of a coupling agent on mechanical properties and morphology of the composites were investigated.

Flexural modulus is used as an indication of a material's stiffness when flexed. It is well known that the improvement in the modulus depends on the morphology of composites. From the findings of earlier researchers, the flexural modulus of the neat polyester matrix is 500.55 MPa. The flexural modulus of the composites follows the same trend with the flexural strength, there was gradual increase in the modulus from 10 wt% - 20 wt% fibre loading and begin to decrease as from 30 wt% - 40 wt%. The optimum value for PALF/Polyester composite is 704.59 MPa which is about 40.76% obtained at 20 wt% fibre loading. The reduction in the flexural modulus at higher fibre loading is a result the fibres touching each other which resulted into stress concentration at the tips of PALFs within the matrix.

Flexural strength is the ability of the material to withstand the bending forces applied perpendicular to its longitudinal axis. Generally, in the case of composites, the resistance to interlaminar failure controls the flexural properties. Therefore, high flexural strengths of composite are due to better interfacial adhesion of the fibre-matrix interface, which is a result of the chemical treatment of the PALF that enhanced the fibre-matrix interaction and thereby increased the interfacial bond strength and allowed strongest adhesion at the interface. The decrease in the flexural strength at higher fibre loading (40 wt% in this case) is as a result of non-uniform stress transfer due to PALFs touching each other within the matrix.

Gomze *et al.* [18] reported that PALF significantly improve the Izod impact energy of epoxy matrix composites. The incorporation of continuous and aligned PALF fabrics results in a marked change with respect to pure epoxy matrix (0% fibre) in which a totally transversal rupture occurs. The crack nucleated at the

notch will initially propagate transversally through the epoxy matrix, as expected in a polymer. However, when the crack front reaches a fibre, the rupture will proceed through the low strength interface. As a consequence, after the Izod hammer hit the specimen, some fibres will be pulled out from the matrix but, owing to their flexural compliance, the PALF fabric will not break.

Sri and Dadit [19], studied the effect of fibre surface modification on PALF loading and PALF fibre length on the mechanical properties of PALF/epoxy composites they also analyzed the compositions volume fraction of PALF/epoxy composites, which they fixed at 10/90%, 20/80%, 30/70% and 40/60%. The lengths of the pineapple leaf fibre were fixed to 20 mm, 30 mm and 40 mm. Before the fabrication, PALF underwent alkaline treatment to increase the strength of fibre. All samples underwent three different tests to determine the mechanical properties which are tensile test, impact and bending test. PALF loading of 30/70% with 40 mm in length shows the higher values of tensile stress, impact and bending which are 22.17 MPa, 27.63 J/m<sup>2</sup> and 35.53 N/mm<sup>2</sup> respectively.

## 2. Materials and Methods

### 2.1. PALF-PP Composite Preparation

The PALF particles were sieved to obtained uniform particle sizes of an average range of 2 - 6 mm. This particular size appears to be the most suitable processing size for melt mixing which gives effective reinforcement in Polypropylene as reported by George *et al.* [5] and Kalia *et al.* [10]. Melt mixing method was used to mix PALF and PP. Various compositions of PALF in PP were compounded in an injection molding machine as shown in **Table 1**.

### 2.2. Mechanical Properties Test of the PALF Composite

All the mechanical testing methods that were carried out were based on American Society for Testing and Materials (ASTM). There were three tests performed, namely Tensile Test (ASTM D638-10), Flexural Test (ASTM D256-10) and Impact Test (ASTM D790-17).

#### 2.2.1. Tensile Property

The ASTM-D638-10 method was adopted for this research work. The dumbbell shape (Type I) specimen is needed for reinforced composite testing. The testing was done in standard laboratory atmosphere of 37°C ± 2°C. A computerized Universal Testing Machine (TUE-C-100), manufactured by Fine Spavy Associates and Engineers PVT Miraj, India, was used at cross-head speed of 50 mm/minute. **Figure 1** shows the Universal Testing Machine used for PALF-PP tensile testing. The specimens were positioned vertically in the grips of the testing machine. The grips were then tightened evenly and firmly to prevent any slippage with gauge length kept at 50 mm. The precise five tested result were chosen for each fibre loading of PALF in PP matrix.

**Table 1.** PALF-PP composite weight ratio.

PALF Ratio (g)	Polypropylene Ratio (g)	Total Weight Composition (g)
40	360	400
80	320	400
120	280	400
160	240	400

**Figure 1.** The computerized universal testing machine used in this research.

### 2.2.2. Flexural Property

Flexural strength is the ability of the material to withstand bending forces applied perpendicular to its longitudinal axis. Sometime it is referred as cross-breaking strength where maximum stress developed when a bar-shaped test piece, acting as a simple beam, is subjected to a bending force perpendicular to the bar. The ASTM D790-10 method was adopted for flexural test in this research work. The three-point loading system applied on a supported beam was utilized.

The ASTM D790-10 Procedure A was adopted, where the width and depth of the specimen were measured to the nearest 0.03 mm at the center of the support span. The test samples were then placed on two supports and load will be applied. The distance of two supports span (L) was fixed at 100 mm. Flexural test was done by A computerized Universal Testing Machine (TUE-C-100), manufactured by Fine Spavy Associates and Engineers PVT Miraj, India at standard laboratory atmosphere of  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$ , as shown in **Figure 1**. This constant cross-head motion appeared to be 5 mm/min.

### 2.2.3. Impact Strength

The impact properties of the material are directly related to the overall toughness which is defined as the ability to absorb applied energy. The ASTM D256-17 test method A (Izod type) was used for testing. The apparatus involved was Cantilever Beam (Izod Type) Impact Machine and the specimens were notched. Notching was done because it provides a stress concentration area that promotes a brittle rather than a ductile failure. Furthermore, notching also drastically reduces the energy loss due to the deformation of the composite. In the testing, specimens were clamped vertically as a cantilever beam and then struck by a single swing of the pendulum released from a fixed distance from the spe-



cimen clamp. The line of initial contact is at a fixed distance from the specimen clamp and from the centerline of the notch and on the same face of the notch. This equipment is shown in **Figure 2**. There are a few parameters that are set according to the standard for instances, Hammer Velocity = 3.46 m/s and Hammer Weight = 0.905 kg. The equipment is manufactured by Samuel Denison Limited, England.

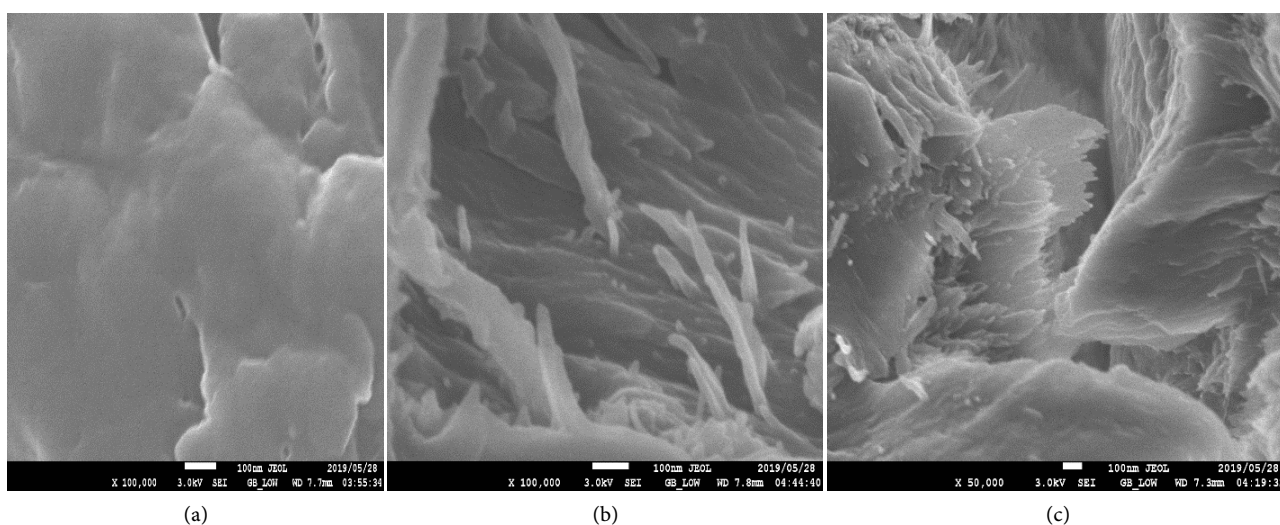


**Figure 2.** Danison universal pendulum impact system for Izod-Charpy tension and puncture.

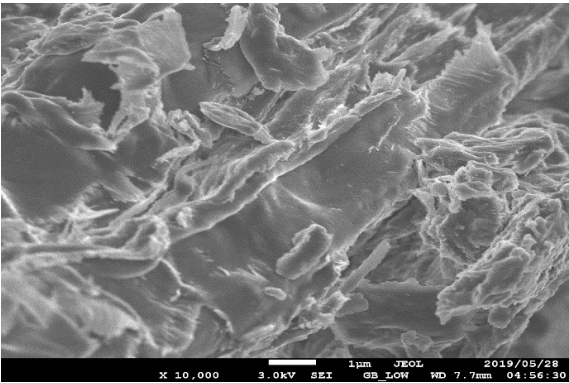
### 3. Results

#### 3.1. The Scanning Electron Microscopy (SEM) of the PALF

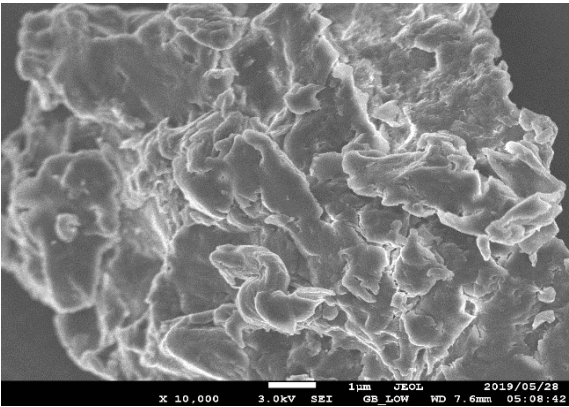
The Scanning electron micrographs of the pineapple leaf fibre (PALF) at different magnifications, showing the different behaviour of the PALF samples (**Figure 3** & **Figure 4**).



**Figure 3.** (a) SEM micrograph of the untreated (unmodified) PALF; (b) The SEM micrograph of the NaOH modified PALF; (c) The SEM micrograph of the ZnCl modified PALF.



(a)

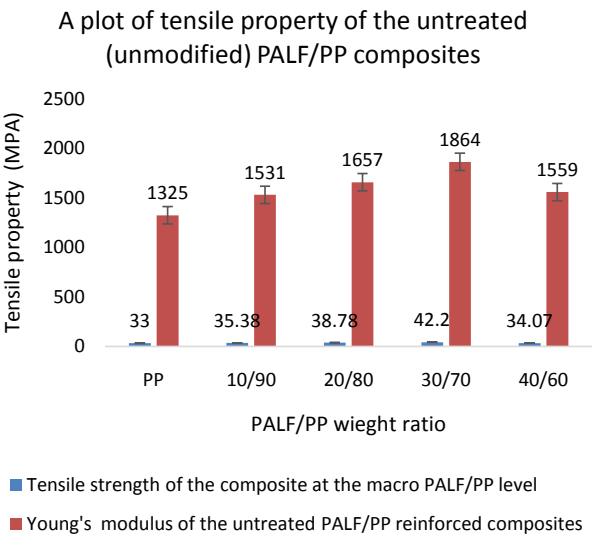


(b)

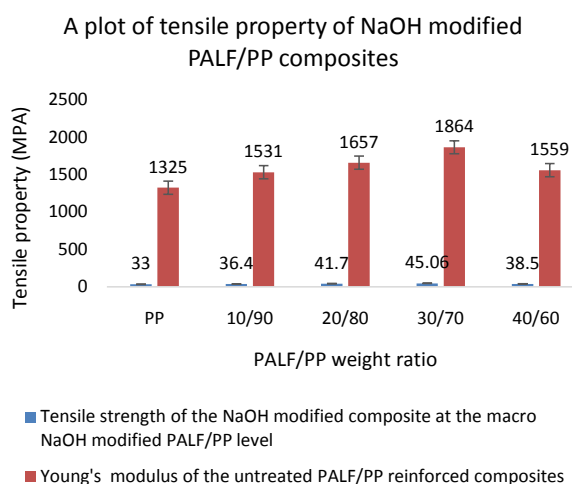
**Figure 4.** (a) SEM micrograph of the C<sub>3</sub>H<sub>6</sub>O<sub>3</sub> modified PALF; (b) The SEM micrograph of the HNO<sub>3</sub> modified PALF.

**3.2. Tensile Property of the Reinforced PALF Composite Produced**

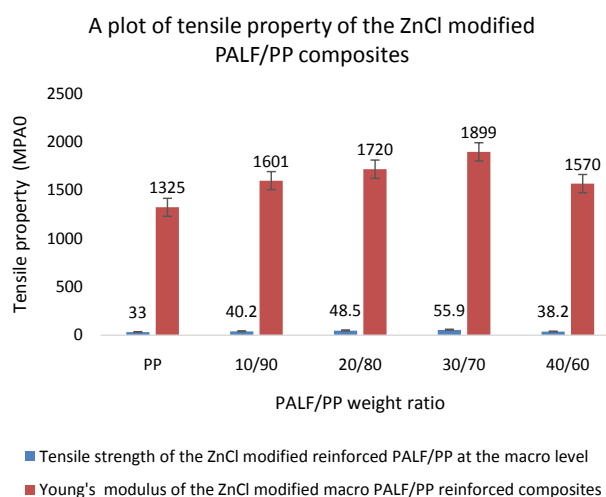
The results of the test carried out at room temperature and pressure are as given below in form of charts (**Figures 5-9**).



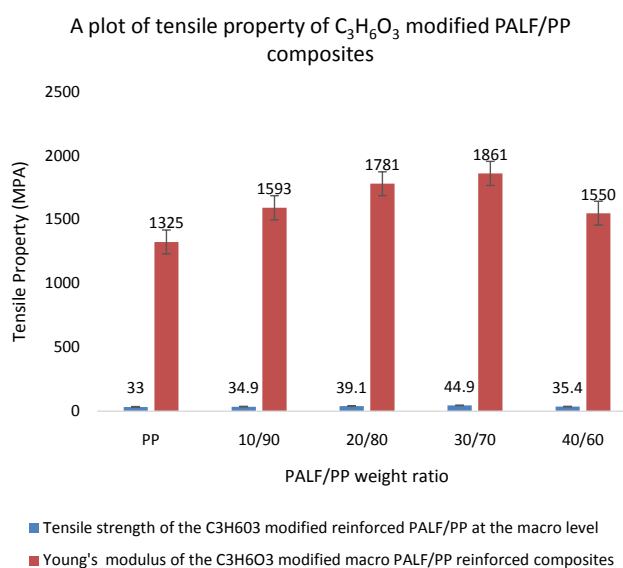
**Figure 5.** Tensile property of the untreated PALF/PP composites.



**Figure 6.** Tensile property of the PALF/PP composites treated with NaOH.

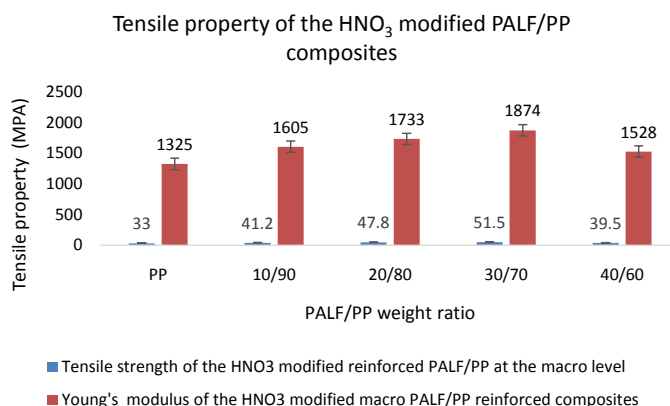


**Figure 7.** Tensile property of the PALF/PP composites treated with ZnCl.



**Figure 8.** Tensile property of the PALF/PP composites treated with  $C_3H_6O_3$ .

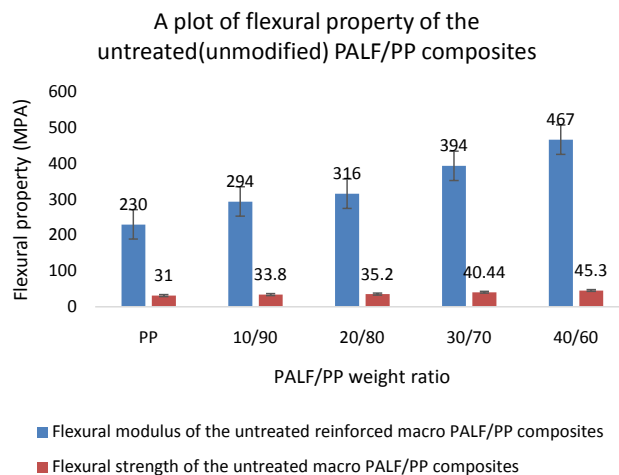




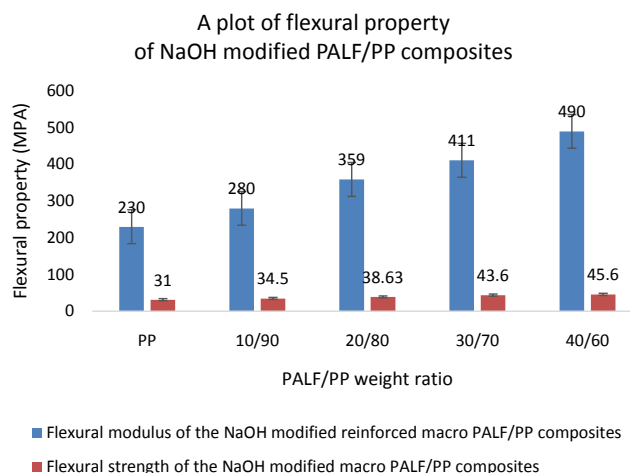
**Figure 9.** Tensile property of the PALF/PP composites treated with  $\text{HNO}_3$ .

### 3.3. The Plots of the Flexural Properties of the PALF/PP Composites

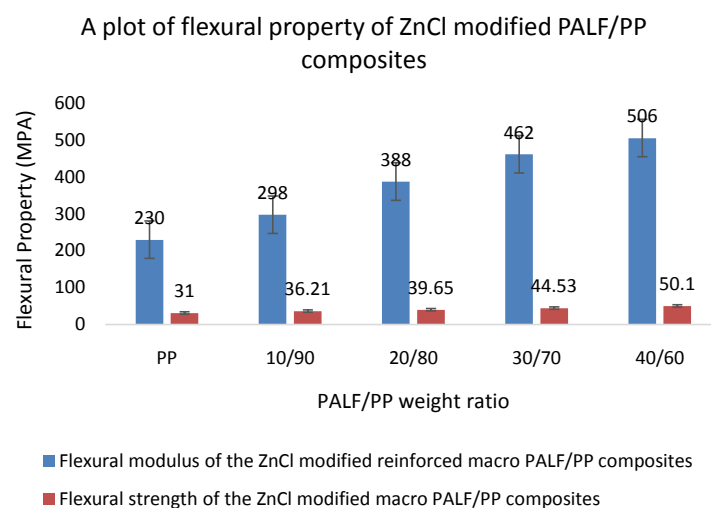
**Figures 10-14** show the flexural properties of the PALF/PP composites, depicting the flexural (FS) and flexural modulus (FM).



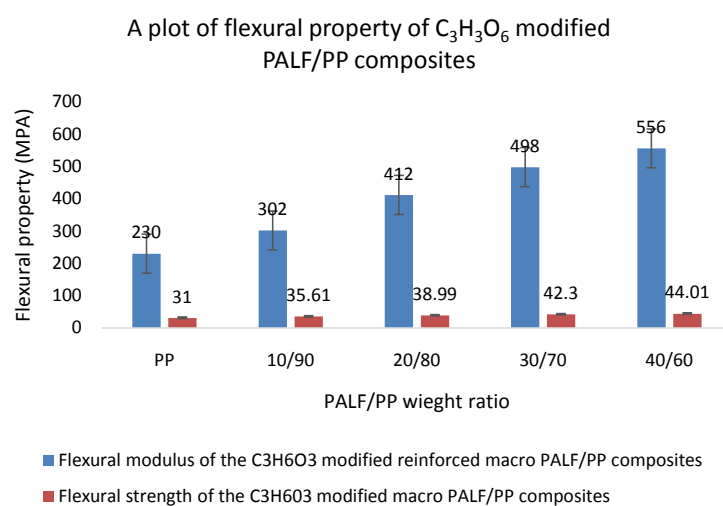
**Figure 10.** Flexural property of the untreated PALF/PP composites.



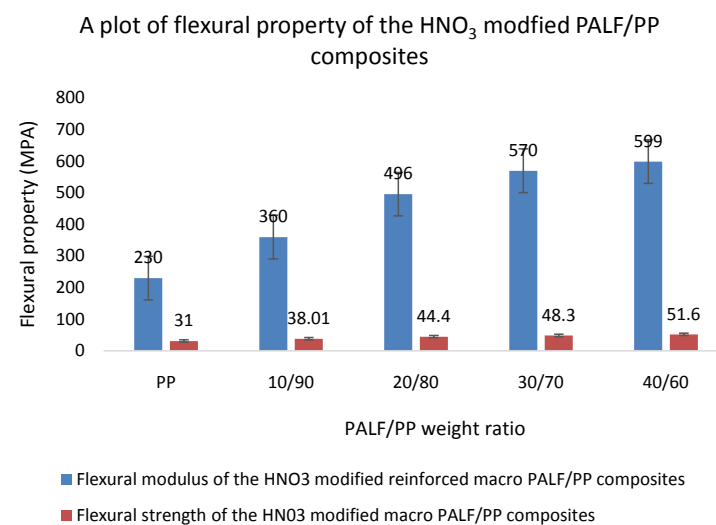
**Figure 11.** Flexural property of the PALF/PP composites treated with NaOH.



**Figure 12.** Flexural property of the PALF/PP composites treated with ZnCl.



**Figure 13.** Flexural property of the PALF/PP composite treated with  $C_3H_3O_6$ .



**Figure 14.** Flexural property of the PALF/PP composites treated with  $HNO_3$ .

### 3.4. The Impact Strength Plots of the PALF/PP Composites

Figures 15-19 show the impact strength properties of the PALF/PP composites, depicting the impact strength (IS) of the composites at compositional weight ratios.

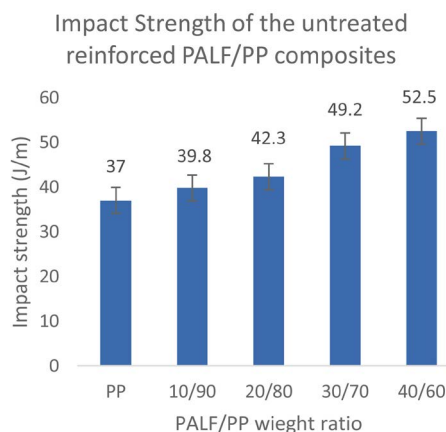


Figure 15. Impact strength of the untreated reinforced PALF/PP composites.

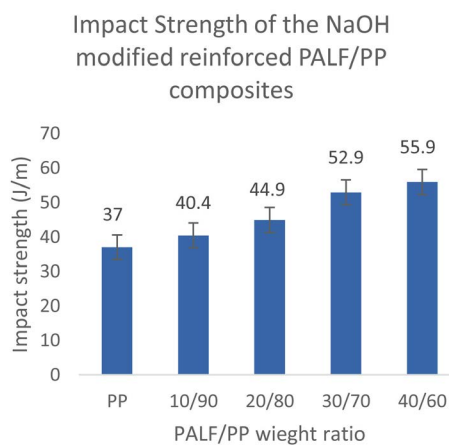


Figure 16. Impact strength of the reinforced PALF/PP composites treated with NaOH.

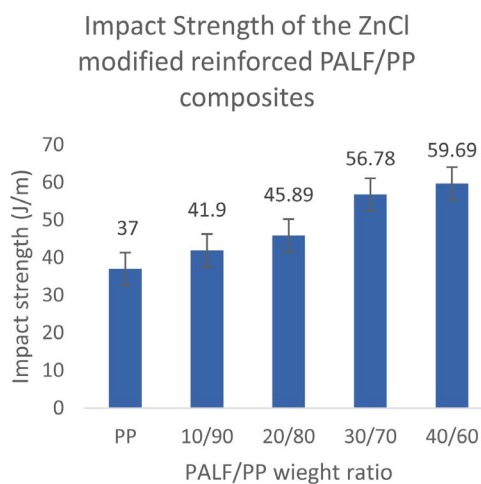
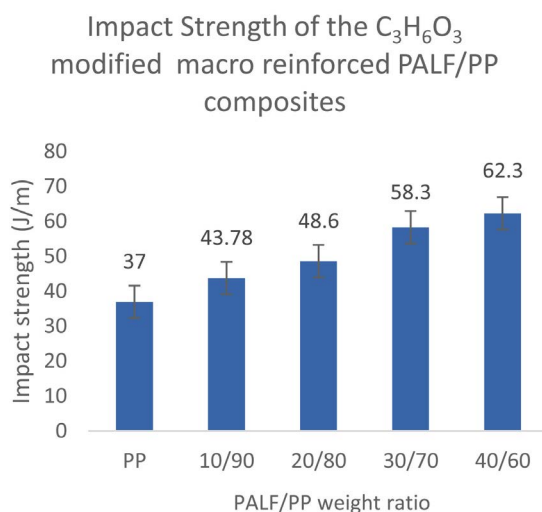
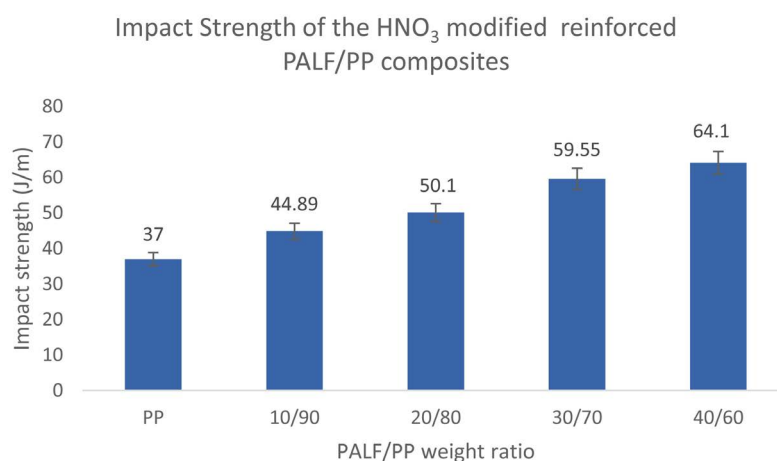


Figure 17. Impact strength of the reinforced PALF/PP composites treated with ZnCl.



**Figure 18.** Impact strength of the reinforced PALF/PP composites treated with  $C_3H_6O_3$ .



**Figure 19.** Impact strength of the reinforced PALF/PP composites treated with  $HNO_3$ .

## 4. Results Discussion

**Figures 3(a)-(c), Figure 4(a)** and **Figure 4(b)** show the SEM micrographs of the PALF. **Figure 3(a)** shows that the surface of the untreated PALF is clogged together, depicting that the PALF cannot readily mix with the matrix there by giving a composite with enhanced properties. **Figure 3(b)** shows the SEM of the NaOH modified PALF that is not as clogged as that of **Figure 3(a)**, the micrograph shows the surface of fibre particle that are separated and can readily serve as good reinforcement in the PP matrix. **Figure 3(c)** shows the SEM micrograph of the ZnCl modified PALF, here, the micrograph depicts a more dispersed fibre surface that is indicating a better PALF/PP composite. **Figure 4(a)** represents the SEM of the  $C_3H_6O_3$  modified PALF, the PALF samples here shows a surface that is more dispersed when compared to the samples modified with ZnCl and NaOH. **Figure 4(b)** shows the SEM micrograph of the  $HNO_3$  modified PALF, the samples modified show better separation, thus, depicting samples with expected behavior of composites that will exhibit better performance when com-

pared with the other samples.

Tensile strength (TS), tensile modulus (TM), flexural strength (FS), flexural modulus (FM), and impact strength (IS) of the prepared composites were studied and the data are given from **Figures 5-9**. The TS, TM, FS, FM and IS of the PALF/PP composites are as discussed. **Figures 5-9** give the results of the tensile property test (TS and TM) of the untreated (control) PALF/PP composites at the macro particle level. From **Figure 5**, there is a gradual increase in TS and TM as the percentage volume of the filler (PALF) increases from 10% to 30%, here we had a steady increase of up to 19.28% as the fibre content increases from 10% to 30%, there is a sharp decline from 40% fibre content, similarly, the TM increased in proportional dimension and gave an increase in TM of 22.76% between the fibre content of 10% to 30%, where there is an observed decline.

**Figure 6** gave a result in similar pattern with an increase in TS by 25.27% and TM of 35.40% for the sample treated with NaOH. **Figure 7** depicts an increase by 29.05% of TS and 43.00% for TM for samples treated with ZnCl, **Figure 8** depicts an increase by 48.60% of TS and 30.72% of TM for the sample treated with  $C_3H_6O_3$ . **Figure 9** depicts an increase by 49.27% of TS and 37.93% for TM for sample treated with  $HNO_3$ .

A similar pattern of percentage increase in the FS and FM of the PALF/PP composites is seen as the fibre percentage volume increases from 10% to 30%, however, a gradual decline is observed as the percentage fibre volume increases beyond 30%. **Figure 10** shows a composite of the untreated (control) PALF/PP, which depicts a steady increase of FS by 34.02% and FM increase by 74.84% at the macro particle level. **Figure 11** shows a composite treated with NaOH with an increase in FS and FM of 37.77% and 90.24% respectively at the macro particle level. **Figure 12** shows a composite treated with ZnCl with an increase in FS and FM of 38.36% and 92.52% respectively at the macro particle level. **Figure 13** shows a composite treated with  $C_3H_6O_3$  with an increase in FS and FM of 35.82% and 86.36% respectively at the macro particle level. **Figure 14** shows a composite treated with  $HNO_3$  with an increase in FS and FM of 35.76% and 83.33% respectively at the macro particle level.

**Figures 15-19** gave the results of the impact strength (IS) of the PALF/PP composites at the macro, micro and nano particle levels. It has been observed that the impact strength of the PALF/PP composite increases as the volume of fibre (PALF) increases in the matrix (PP).

From **Figure 15**, the IS of the untreated PALF/PP composite increased by a percentage of 37.62% between a fibre volume of 10% to 40%. From **Figure 16**, the IS of the PALF/PP composite treated with NaOH increased by a percentage of 59.31%. From **Figure 17**, for PALF/PP composite treated with ZnCl, the IS increased by a factor of 74.29%. From **Figure 18**, the IS for the PALF/PP composite treated with  $C_3H_6O_3$  increased by a factor of 78.54%. From **Figure 19**, the IS for the PALF/PP composite treated with  $HNO_3$ , there is a percentage increase of 70.82%.

Kasim *et al.* [16] investigated the mechanical properties of high impact poly-

propylene composite reinforced with pineapple leaf fibre from the Josapine cultivar as a function of fibre loading, the results they obtained are in tandem with the ones obtained in this report. From the results, it can be concluded that properties of fiber-reinforced composites depend on many factors *i.e.*, fiber-matrix adhesion, volume fraction of fiber, fiber aspect ratio, fiber orientation as well as stress transfer efficiency of the interface between the matrix and the reinforcement as reported by Chollakup *et al.* [3] and Arib *et al.* [1] who investigated the mechanical properties of pineapple-leaf fiber polypropylene composites as a function of volume fraction. They found that flexural and tensile strength increased slightly from 2.7% to 16.2%, respectively, when the fiber content increased.

The data above means that, with the increase of fibre content, all the mechanical properties increase. PALF composites gained huge mechanical properties over the matrix material and thus indicated good fibre matrix adhesion, more especially for the PALF with chemically enhanced surfaces.

## 5. Conclusions

The superiority of mechanical properties of pineapple leaf fibre can be related to the high content of alpha-cellulose content with low microfibrillar angle (14°). As reinforcing agent PALF has both qualities, that is, high content of alpha-cellulose content with low microfibrillar angle (14°), the results of PALF based polymer composites show excellent stiffness and strength compared to other cellulose based composite materials.

The results obtained are in close agreement with those obtained by earlier researchers. It has been observed that, when the fiber content is higher, properties of reinforced polymer composites tend to decrease due to the increase in fiber to fiber interactions, void content, dispersion and fiber alignment problems. On the other hand, other authors have associated the poor dispersion of the fibers and the void content in polymer composites with the hydrophilic character of natural fibers. Strongly polarized hydroxyl groups found inside natural fibers provide its hydrophilic character.

## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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