**ORIGINAL PAPER**



# **Effect of Alkali Treatment on Mechanical and Morphological Properties of Pineapple Leaf Fibre/Polyester Composites**

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## **Abstract**

In this study, our aim is to analyze the influence of fibre treatments and different fibre loading on mechanical, physical and chemical properties of pineapple leaf fibre reinforced polyester composites (PALF/PE). Fibre treatments were carried out with 1 N NaOH and KOH for 1 h. The untreated and treated PALF/PE composites were fabricated with 25 wt%, 35 wt% and 45 wt% fibre loadings by compression molding technique. Fourier Transform Infrared Spectroscopy (FTIR) was used to understand the effects of chemical treatment on PALF mechanical test results revealed that 45 wt% of PALF/PE composites treated with NaOH showed a 35% increase in tensile strength compared to untreated PALF/PE composites. The tensile modulus and the flexural module are also the highest at 45 wt% of KOH treated composites. The highest impact strength of 70 J/m was obtained for PALF/PE composites with NaOH treated fibres at 25% fibre loading. The results show that the fibre treatments in terms of the flexural and inter-laminar shear strength of composites were not effective. SEM of the tensile fractured specimen of PALF/PE composites revealed the changes in fibre characteristics due to the alkali treatment and less fibre pull-out at higher fibre loading. Overall we conclude that 1 N NaOH, 45 wt% treated PALF/PE composites satisfactorily and effectively improved both the mechanical and morphological properties. Obtained composites would be promising for construction materials, furniture and automotive components due to their superior strength and modulus at higher fibre loading.

**Keywords** Pineapple leaf fibre · Polyester composites · Alkali treatment · Mechanical properties · Morphological properties · Inter-laminar shear strength

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## **Introduction**

At present, man-made fibres such as carbon, glass and aramid are extensively used in polymer-based applications owing to their light weight, high stiffness, superior strength, corrosion resistance and high energy absorption capacity [[1,](#page-9-0) [2](#page-9-1)]. Nevertheless, the man-made fibres have serious drawbacks like biodegradability, recyclability, initial processing costs, health hazards and machine abrasion. So, the researchers have changed their attention from synthetic fibres to renewable fibres. Natural fibres become popularly employed as reinforcements in polymer matrix composites. However, the renewable/natural fibres are not an issue-free alternative and these fibres also have some specific shortages in properties especially poor bonding between the fibre and matrix. The poor bonding could be due to (i) moisture absorption by the structural compositions such as cellulose, hemicellulose, lignin, pectin and wax (ii) fibres and matrix are not at all like in chemical structure. This causes incapable stress transfer at the interface of the composites. Accordingly, certain

surface treatments are unquestionably required to improve their interfacial adhesion between the fibre and matrix. Thus, the researchers in academia and industries are need to be actively involved for the development of interfacial bonding properties in polymer matrix composites. In fact, many research works have been already reported with the chemical treatments of natural fibres [\[3](#page-9-2)[–6](#page-9-3)].

Usage of pineapple leaf fibres as reinforcement in the thermosetting and thermoplastic resins has received widespread attention due to their outstanding specific properties [\[7\]](#page-9-4). Since the PALF possess higher strength/modulus and sustainable, it is also suitable for use in the textile industry. Other advantages of using natural fibres include easy availa-bility, low density, low cost, etc [\[8–](#page-9-5)[10\]](#page-9-6). PALF is highly suitable for reinforcement in polymer composites on any scale from macro, micro to nano-scale due to their high cellulose content. The discontinuous or randomly distributed PALF based micro and nano-composites could be used in applications like car parts, construction, tertiary structures and biomedical devices. Many researchers studied the mechanical properties of PALF reinforced composites with different resins such as polyester [[11–](#page-9-7)[13\]](#page-9-8), bisphenol [\[14](#page-9-9)], vinyl ester [[15\]](#page-9-10), and epoxy [\[16–](#page-9-11)[18](#page-9-12)] with different fibre loading and fibre orientations. In general, different chemicals were used to improve the fibre/matrix adhesion by many researchers [\[19–](#page-9-13)[21\]](#page-9-14). Some of research findings where natural fibres are reinforced in PE and their observations are given in Table [1.](#page-1-0)

From Table [1](#page-1-0), it is clear that PE has been used along with different natural fibres. Various fibre treatments have been used to improve the fibre wetting with matrix. Studies on PALF/PE composites with the KOH treated fibres are rarely exist in the literature. Moreover, no literature is found that deals with the influence of KOH treatment on the PALF composites. In this experimental work NaOH and KOH treated PALF reinforced polyester composites with varying fibre weight  $(25 \text{ wt}\%), 35 \text{ wt}\%),$  and  $45 \text{ wt}\%$ ) are fabricated. The mechanical and morphological properties of the developed PALF/PE composites with the chemically treated fibres were examined and compared with that of untreated PALF/PE composites.

## **Experimental Procedure**

#### **Materials**

PALF used in this study was procured from Andhra Pradesh, India. Isophthalic unsaturated polyester resin and methyl ethyl ketone peroxide (MEKP) used as a catalyst and an accelerator were supplied by Vasivibala Resin (P) Ltd, Chennai. Mechanical and physical properties of PALF and PE used in this study are listed in Table [2](#page-1-1).

<span id="page-1-0"></span>**Table 1** Reported studies on mechanical properties of chemically treated natural fibre reinforced polyester composites



<span id="page-1-1"></span>

#### **Alkali Treatment of PALF**

Treatment of the PALF was performed with the NaOH and KOH independently. Prior to the treatment, the fibres were chopped to 3 mm lengths. The short fibres were drenched separately in a container with 1 N NaOH aqueous solution for 1 h. The chemically treated fibres were washed with distilled water several times to remove the residues.

During the KOH treatment, 1 N solution was prepared after addition of 40 g of KOH to 1000 ml of distilled water. The fibres were then soaked in the prepared solution for 1 h. After the interim time, the fibres were taken out from the solution and washed with dilute hydrochloric acid to remove residual KOH sticking to the fibre surfaces.

#### **Fabrication of Composites**

The treated PALF was mixed with PE and fabricated into composite by compression molding technique identical to the first phase of published work with the untreated fibres [\[28\]](#page-10-0).

## **Characterization**

#### **Fourier Transform Infrared Spectroscopy (FTIR)**

FTIR spectra of untreated, NaOH and KOH treated PALF fibres were characterized by using IR Tracer 100 spectroscope (Shimadzu, Maryland, USA) in the wave number ranging from 500 to 4000 cm<sup>-1</sup>.

#### **Tensile Testing**

Tensile test of PALF/PE composites were carried out according to ASTM D3039 standard with the INSTRON 3382 universal testing machine. Specimen dimension of  $120 \times 20 \times 3$ mm<sup>3</sup> and 50 mm gauge length was fitted to the grip and a crosshead speed of 5 mm/min was maintained. Average value of five specimens was reported.

#### **Flexural Testing**

Flexural testing of PALF/PE composites were carried out according to ASTM D790 standard. The dimension for the flexural test was  $120 \times 20 \times 3$  mm<sup>3</sup>. The gage length and crosshead speed was 50 mm and 1.27 mm/min respectively. Flexural stress–strain plot, strength and modulus of the composites were discussed. Average value of five specimens was reported.

#### **Impact Testing**

Impact strength was measured according to ASTM D256- 10. The dimensions for the composite samples were  $63 \times 13 \times 3$ mm<sup>3</sup> without using notch through 3.56 kg impact hammer. Five specimens were tested and the average value was reported.

#### **Short Beam Test**

Inter-laminar shear strength (ILSS) of the composites were studied by subjecting the specimen to 3-point bending. A support span of 24 mm and cross head displacement rate of 1.27 mm/min was used. The maximum load recorded from the test was used to calculate the ILSS as follows:

$$
\sigma = \frac{3P}{4bh}
$$

where  $\sigma$  is the inter-laminar shear strength, P is the maximum load in kN, b is the width of the specimen (mm) and h is the height of the specimen (mm).

#### **Scanning Electron Microscopy (SEM)**

The microstructure of the tensile fractured specimens obtained from the scanning electron microscope (Hitachi S-3400N) were used for the investigation of failure behavior. The specimens were sputter coated with gold prior to the observation under SEM.

# **Results and Discussions**

#### **FTIR**

FTIR spectroscopy of the raw, NaOH treated and KOH treated PALF is presented in Fig. [1.](#page-3-0) The peak values that were observed in the FTIR spectra of the untreated and chemically treated fibres are listed in Table [3.](#page-3-1)

The peak values for the untreated fibre such as 2917 cm<sup>-1</sup>, 1736 cm<sup>-1</sup>, 1641 cm<sup>-1</sup>, 1251 cm<sup>-1</sup>, 1436 cm<sup>-1</sup> and 3100–3800  $cm^{-1}$  corresponds to the presence of C–H stretching vibration from the  $-CH<sub>2</sub>$  group of cellulose and hemicellulose, pectin, absorbed water molecules, lignin and cellulose respectively [\[29](#page-10-1)]. Characteristic peaks in the similar wavenumber range were also observed in this study. The peak at 2941 cm<sup>-1</sup> was attributed to the C–H stretching vibration from the  $-CH<sub>2</sub>$  group of cellulose and hemicellulose while the peak at 1739 cm−1 was from the C–O stretching of carbonyl groups in hemicellulose. The peaks observed at 1429 cm<sup>-1</sup> and 1254 cm<sup>-1</sup> was associated to the lignin and hemicellulose structure. The wider band that was observed between the wavenumber 3332–3836 cm−1 corresponds to

#### <span id="page-3-0"></span>**Fig. 1** FTIR spectra of untreated and treated PALF



<span id="page-3-1"></span>**Table 3** Peak values from the FTIR spectra of untreated and alkali treated fibres



the presence of cellulose in the untreated fibre. The characteristic peaks at 896 cm<sup>-1</sup>, 1155 cm<sup>-1</sup> 1202 cm<sup>-1</sup> and 1506 cm−1 were related to the Hemicellulose corresponds to the COC, CCO and CCH deformation and stretching from cellulose, C–O–C Hemicellulose and C–O–C asymmetrical stretching Cellulose symmetric stretching cellulose and lignin [\[30\]](#page-10-2).

The absence of individual peaks such as  $1739 \text{ cm}^{-1}$ from the FTIR spectra indicates that NaOH and KOH treatment resulted in the complete removal of hemicellulose. It implies that the alkali treatment was highly effective in removing hemicellulose from the fibre and this observation was also reported in the previous studies [[31–](#page-10-3)[33\]](#page-10-4). The peak 1425 cm−1 corresponding to lignin which was present in the case of untreated and NaOH treated fibres, was not observed in KOH treated fibres. This signifies the bleaching of lignin completely from the fibre due to the KOH treatment. Other than the disappearance of certain characteristic peaks, changes in peak intensity between the different wave numbers were noticed in the spectra (regions marked with red lines in Fig. [1\)](#page-3-0). For instance, fewer peak intensity and reduction in absorption band at wave numbers between 3300 and 3800 cm−1, 1900–2400 cm−1, 1200–1450 cm−1 and 550–700 cm−1 in case of the alkali treated fibres. This behavior highlights the interaction of alkali with PALF fibres which in turn leads to the modification of fibre composition.



<span id="page-4-0"></span>**Fig. 2** Tensile stress–strain plot of PALF/PE composites with untreated and alkali treated fibres under varying fibre wt%

The changes in characteristic peak intensities and absence of peaks were significant in case of KOH treated fibres than the NaOH treated fibres.

#### **Tensile Properties**

Tensile stress–strain plot of the PALF/PE composites at various fibre loadings is illustrated in Fig. [2.](#page-4-0) All the specimens showed almost linear increase in strength until final failure. The general observation was that load carrying capability of the composite was dominated by the fibre loading and fibre treatment. A slight inflection was observed in the stress–strain plot at the beginning of the test. This was supposed to be due to the machine slippage of the specimen [[34\]](#page-10-5).

Table [4](#page-4-1) presents the tensile strength at maximum load and Young's modulus. The maximum tensile strength and Young's modulus for the PALF/PE with the untreated fibres and chemically modified fibres occurred at 45% fibre loading. The magnitude of tensile strength for PALF/PE with untreated fibres improved by 8% and 30% respectively as the fibre loading was increased to 35% and 45%. Similar results have been reported on the tensile properties of natural fibre reinforced composite, which are in agreement that increasing fibre loading in composites had a positive effect on tensile strength, e.g. short banana fibre reinforced with polyester composites with different fibre loads (30 wt%, 40 wt% and 50 wt%) [[35\]](#page-10-6). Another study from Ester Rojo et al. [\[36](#page-10-7)] on cellulosic fibres from eucalyptus wood reinforced Phenolic (PF) composites fabricated by varying different fibre loadings (1, 3, 5, and 7 wt%) also confirmed the enhancement in the mechanical strength of composites.

PALF/PE with KOH treated fibres exhibited 21% and 27% rise in strength due to the increase in fibre loading. Tensile strength for the PALF/PE composites with NaOH treated fibres were significantly higher than the other configurations at 25% and 45% fibre loading. At 35% fibre loading, the tensile strength dropped by 24%. Similar reduction in tensile strength at 35% fibre loading for natural fibre reinforced composites with the alkali treated fibres have also been reported by the previous researchers [[37,](#page-10-8) [38\]](#page-10-9). Young's modulus followed same pattern as that of the tensile strength. Highest stiffness value of 1.78 GPa was recorded for PALF/ PE composites with KOH treated fibres at 45% fibre loading. According to Herrera-Franco et al. [[39](#page-10-10)] usually improvement

<span id="page-4-1"></span>**Table 4** Tensile strength and modulus of PALF/PE composites with the untreated and treated fibres at different fibre weight%



in tensile modulus was not found in the composite with treated fibres. However, in this study, the tensile modulus increased by 2% and 15% for the NaOH and KOH treated PALF/PE composites at 45 wt% fibre weight in comparison to the counterpart on untreated PALF/PE composites.

Removal of hemicellulose and lignin from the fibre leads to a rougher fibre surface. This could provide larger contact surface area and more number of inter-locking sites for the fibre with the resin  $[40]$  $[40]$  $[40]$ . This improved compatibility between the fibre/matrix for the composites with alkali treated fibres led to enhancement in the tensile strength and modulus. The limitation associated with the alkali treatment is that fibrillation occurs at some concentrations/immersion time which can result in weak fibres. The weak fibre with fibrillation becomes ineffective in carrying the load and stress transfer and thus can be easily pulled out [[41](#page-10-12), [42](#page-10-13)]. This is the primary reason for lower strength and modulus at certain fibre loading for the composites with alkali treated fibres.

The optimum tensile strength and modulus at 45% fibre loading was inevitable because the fibres are primary load carriers in the composite and the presence of higher amount of fibres helped in effective stress transfer within the material. On the other hand, least tensile strength and modulus for the composites at 25% fibre loading gives a clear indication of the inability of the composite to withstand more load due to the lower fibre content. Haydaruzzaman et al. [[43\]](#page-10-14) highlighted that poor fibre dispersion in the composites at lower fibre loading does not allow the accumulated stresses in a matrix region to transfer to other regions. This can lead to highly localized strains and can cause failure at lower loads.

The microstructure of tensile fractured specimens with the untreated fibres and chemically modified fibres obtained from the SEM is given in Fig. [3](#page-5-0)a–f. The composites at 45% fibre loading (Fig. [3](#page-5-0)b, d, f) showed better fibre/matrix interfacial bonding and less fibre pull-out as compared to the composites shown in Fig. [3](#page-5-0)a, c, e. On the other hand, the

<span id="page-5-0"></span>**Fig. 3 a** SEM of tensile fractured PALF composites with untreated fibres at 25% fibre loading. **b** SEM of tensile fractured PALF composites with untreated fibres at 45% fibre loading. **c** SEM of tensile fractured PALF composites with NaOH treated fibres at 35% fibre loading. **d** SEM of tensile fractured PALF composites with NaOH treated fibres at 45% fibre loading. **e** SEM of tensile fractured PALF composites with KOH treated fibres at 25% fibre loading. **f** SEM of tensile fractured PALF composites with KOH treated fibres at 45% fibre loading



removal of greasy contents from the fibre surface due to the NaOH and KOH treatment led to rougher fibre surface and fibrillation as shown in Fig. [3d](#page-5-0)–f.

Despite of the fibrillation, higher fibre content helped the composite to withstand slightly higher loads. This, in turn, resulted in better tensile strength and modulus than the other fibre loadings. This finding was in accordance with the results reported by various researchers on PALF reinforced composites with the short fibres [\[44–](#page-10-15)[48\]](#page-10-16).

#### **Flexural Properties**

The flexural stress–strain plot obtained from the test is given in Fig. [4](#page-6-0). All the specimens exhibited non-linear increase in load and drop in load on failure. Variation in flexural strength and modulus could be observed from the plot due to the fibre treatment and change in fibre loading.

Table [5](#page-6-1) provides the flexural strength at maximum load and flexural modulus of the composites. In case of composites with the untreated fibres, best flexural strength of approximately 83 MPa was obtained at 35% fibre loading and highest flexural modulus of roughly 6 GPa at 45% fibre loading. Composites with the NaOH treated fibres showed a decline in strength and modulus in contrast to the improvement shown in tensile properties while the trend for



<span id="page-6-0"></span>**Fig. 4** Flexural stress–strain plot of PALF/PE composites with varying fibre wt%

composites treated with KOH fibres were similar as well. Maximum strength and modulus were obtained for 45% fibre loading.

The lower flexural strength and modulus at certain fibre loadings for composites could be attributed to the fibre distribution and fibrillation effect due to the alkali treatment. The fibres cluster together as a result of alkali treatment. This could possibly lead to regions with the low fibre content and stress accumulation within the composite on application of the load. Moreover, the bending load is applied at specific points of the specimen unlike tensile load which is applied uniformly throughout the specimen. All these factors could be accounted for lower flexural strength and modulus of composites with the treated fibres. Instances of improvement in strength and modulus for composites with untreated fibres with the fibre loading [[38](#page-10-9), [49](#page-10-17)] and decrement in strength and modulus for composites with fibre treatment were also noticed in the previous studies [\[50](#page-10-18)].

In flexural test, the specimen subjected to 3-point bending undergoes compression and tension on the top and bottom surface [[51\]](#page-10-19). This leads to crack initiation in the matrix of tensile side or lower surface of the specimen below the loading nose. As the applied load continues, the crack propagates further until complete matrix breakage. At this point, the load drops and results in final failure as shown in Fig. [5](#page-6-2).

#### **Impact strength**

The impact strength of composites can be directly associated to its overall toughness [\[52](#page-10-20)]. Figure [6](#page-7-0) shows the variation of impact strength of the different composites. The factors like fibre properties, matrix type and interfacial



**Fig. 5** Tested flexural specimen

<span id="page-6-1"></span>**Table 5** Flexural strength and modulus of PALF/PE composites with untreated and treated fibres at different fibre weight%

<span id="page-6-2"></span>

<span id="page-7-0"></span>**Fig. 6** Impact strength of PALF/ PE composites with untreated and alkali treated fibres under varying fibre wt%



bonding strength between the fibre/matrix determines the impact strength of composite [\[51](#page-10-19)]. From the results shown in Fig. [6,](#page-7-0) it could be noticed that impact strength of untreated composites improved at higher fibre loading. Similar results reported by Senthil kumar et al. [[35\]](#page-10-6), Sultan [\[45\]](#page-10-21) and Uma devi et al. [[27\]](#page-9-20) on the increase of impact strength of natural fibre composites with higher fibre loading. Composites with the NaOH and KOH treated fibres exhibited highest tensile strength at 45% fibre loading while it had negative effect on the impact strength. The composites with NaOH treated fibres had better interfacial bonding between the fibre and matrix due to the removal of soluble greasy contents from the fibre as evident from the FTIR results. This improved interfacial bonding reduces the fibre pull-out which is not a favorable characteristic for composites requiring high impact strength. This factor was also highlighted in a previous study as reason for the lower impact strength in composites with improved adhesion between the fibre/matrix [[53](#page-10-22)]. Unlike tensile and flexural properties, KOH treatment was not effective in case of impact strength as no significant improvement was noticed due to the variation in fibre loading.

### **Inter Laminar Shear Strength (ILSS)**

The apparent inter-laminar shear strength obtained from the short beam test is a measure of the bonding strength between the fibre/matrix in composites. ILSS value for the composites with untreated and chemically modified fibres is shown in Fig. [7.](#page-8-0)

The magnitude of ILSS for composites with untreated fibres was found to increase by onefold and twofolds respectively as the fibre loading was increased from 25 to 35% and 35–45% respectively. Since the magnitude is a direct indication of the bonding strength, the higher magnitude implies better performance. The superior tensile and flexural properties observed with the increase in fibre loading also goes well in agreement with the observation on ILSS. A general observation from the previous studies is that fibre treatment with alkali resulted in higher magnitude of ILSS in composites with treated fibres [\[34](#page-10-5), [51](#page-10-19)]. Similar trend was noticed for composite with the chemically treated fibres only at 25% fibre loading. At higher fibre loading, a declining trend was observed for composites with the KOH and NaOH treated fibres. The values were considerably lower than their respective counterparts compared to the composite with untreated fibres at 35% and 45% fibre loading. This leads to a conclusion that fibre treatments with NaOH and KOH actually weakened the fibres due to its concentration used in the study. The presence of more fibres at higher fibre loading allowed the specimens with chemically modified fibres to withstand more load, thereby superior strength and modulus at such fibre loadings. Among the treatments, composites with the KOH treated fibres exhibited the least ILSS values. This indicates the severity of 1 N KOH concentration on the fibres. At such concentration, more damage to <span id="page-8-0"></span>**Fig. 7** ILSS of composites with the untreated and chemically

modified fibres



the fibres in the form of fibrillation could have damaged the fibres which in turn led to low ILSS values. The disappearance of many characteristic peaks in the FTIR spectra for KOH treated fibres also supports the fact that treatment was severe on fibre.

The failure behavior of specimens from the short beam test was analogous to the flexural test with fibre/matrix debonding and matrix breakage (Fig. [8](#page-8-1)a, b). Since the shear stresses dominate over the normal stress in case of the short beam test, fibre pull-out could also be observed.

# **Conclusion**

In this work a successful attempt were made to treat the PALF with NaOH and KOH and to fabricate PALF/PE composites at different fibre loadings. Developed PALF/PE composites are characterized and compared with untreated fibres in terms of chemical, mechanical and morphological properties. Following conclusions were derived from the study:

- The physical changes to the fibre surface and chemical composition after treatment were evident from the microstructure of the fractured specimens and FTIR spectra. A substantial removal of hemicellulose and lignin resulted in disappearance of their respective characteristic peaks and reduced peak intensities.
- The optimum strength and modulus for composites with the untreated and treated fibres were obtained at different fibre loading.
- For composites with the untreated fibres, increasing the fibre loading was favorable for tensile, flexural and shear properties while it was not beneficial in terms of impact strength. The fibre treatments were highly benefi-



<span id="page-8-1"></span>**Fig. 8 a** PALF/PE composites with KOH treated fibres from the short beam test at 35% fibre loading. **b** PALF/PE composites with NaOH treated fibres from the short beam test at 45% fibre loading

cial for tensile properties, as considerable improvement was noticed in comparison to the composites with the untreated fibres.

• PALF/PE composites with the NaOH treated fibres outperformed composites with the KOH treated fibres for the same concentration and immersion time.

Based on the encouraging results, the newly developed PALF/PE composites could be employed in the development of renewable materials in buildings, construction and house hold applications.

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