

Mechanical properties of abaca fibre reinforced with recycled polypropylene composite

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This study aims to develop sustainable composite materials using recycled polypropylene and abaca natural fibres following the compression molding technique. The chopped abaca fibres have been treated with different alkaline (NaOH) concentrations (4, 8 & 12 wt %) to eliminate the lignin, hemicellulose, wax and pectin components and then subjected to FTIR analysis to verify the presence of their functional elements in the structure. Likewise, XRD analysis is performed to verify the crystalline phase transition in recycled polypropylene and abaca fibres. The SEM study reveals that the treated abaca fibres have a rougher surface than the untreated ones. TGA analysis confirms the good thermal stability of recycled polypropylene and abaca fibres. The mechanical parameters of the developed composite, including its tensile strength, flexural strength, and impact strength, are studied. It is found that the composites with greater abaca fibres loading and 12% NaOH treatment exhibit good mechanical strength. Also, the composite with 30% abaca fibre loading has the lowest water absorption levels as compared to other composites. This study also analyzes the failure mode of composites during mechanical testing. The developed composite can be used in interior car components.

Keywords: Abaca fibre, Composites, Alkaline treatment, Fibre-matrix interface, Flexural strength, Impact strength, Recycled polypropylene

1 Introduction

Natural fibres, such as hemp, coir, and abaca, are mainly used for composite development due to their merit properties, including low weight, low cost, less energy consumption, biodegradability, good mechanical properties and ecological sustainability¹⁻³. The incorporation of recycled polypropylene provides the desired performance from a sustainable point of view that would otherwise pollute the environment, if thrown away. Similarly, abaca fibres are sustainable and eco-friendly due to their raw materials, production and biodegradability⁴. The properties of natural fibre composites are comparable to those of synthetic fibre composites⁵. Nevertheless, low moisture resistance and fibre incompatibility with matrix restrict the manufacture of these composites⁶. To address these challenges and develop a good natural fibre composite, various polymers, including polypropylene, polylactic acid (PLA), polyhydroxyalkanoates (PHA), and other materials, have been employed as matrix materials. There are several benefits of using thermoplastic polymer matrix material for composite development. The primary benefit is that the thermoplastic material used

in the composite is recyclable. As observed in the literature⁷⁻¹⁰, polypropylene fibres exhibit good physical as well as thermal properties. To enhance the composite sustainability, recycled polypropylene (r-PP) is used as a matrix material in this study, boasting advantages, such as low 'Green House Gas' (GHG) emissions and high environmental benefits¹¹. Hence, recycled polypropylene is an important contributor for promoting sustainable waste management practices¹². Table 1 presents a comparison of the physical properties of recycled and virgin polypropylene.

As observed in Table 1, the physical properties of recycled polypropylene are comparable to virgin polypropylene, although somewhat reduced due to the recycling process. However, it can be enhanced during composite manufacturing by incorporating reinforcing fibres suitably into the matrix. The main benefits of natural fibre composites are their light weight, sustainability and low energy consumption. By reinforcing suitable natural fibres, the mechanical, thermal and barrier properties of natural fibre composites can be improved.

Abaca fibres are one of the strongest fibres available in the domain of natural fibres. The Philippines produces around 87% of the world's abaca fibres¹³. The fibres are extracted from the leaves surrounding the trunk of the abaca plant. Because of

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Table 1 — Physical properties of recycled and virgin polypropylene

Properties	Polypropylene	
	Recycled	Virgin
Tensile strength, MPa	24	31
Density, g/m ³	0.90	0.91
Breaking elongation, %	750	720
Maximum strength, MPa	27	39
Melting temperature, °C	141	166
Young's modulus, MPa	619	868

their high mechanical properties, abaca fibre-reinforced composites are becoming popular¹⁴. The abaca fibres contain lignin and wax, which cause poor adhesion between the fibres and the matrix during composite fabrication. Therefore, surface treatment of the abaca fibres is necessary to enhance its adhesion behaviour. Due to the increased roughness and reduced diameter of the fibres, a strong interfacial bond between the matrix and the fibres is formed¹⁵.

In a study, Rahman *et al.*¹⁶ investigated the mechanical characteristics of polypropylene composites reinforced with alkaline treated abaca fibres. This study found that the higher the composite's fibre loading, the higher is its tensile strength. Because the treated abaca-PP composites had stronger interfacial adhesion between the matrix and the abaca fibre. They displayed higher Young's modulus, flexural strength and impact strength than raw abaca fibre composite. In another study, Vilaseca *et al.*¹⁷ examined the abaca fibre-reinforced polypropylene composites for their mechanical characteristics. The maleic anhydride coupling agent was used to improve the compatibility of the PP matrix with the abaca fibre. Using the injection molding process, the composite specimens were formed at various fibre loadings, such as 20, 30, 40 and 50wt%. Although the 40 wt% E-glass fibre reinforced composite had a higher tensile strength than abaca fibre composites, higher temperature resistance was observed for abaca reinforced composite.

Haque *et al.*¹⁸ examined the mechanical properties of polypropylene composites reinforced with abaca and coir fibres. The benzene diazonium salt was used to improve the fibre adhesion to the PP matrix. The tensile strength of the composites declined for higher fibre loading. On comparing the tensile strengths, the treated coir-reinforced PP composite and abaca fibre-reinforced composite showed nearly the same tensile behaviour. Similarly, for higher fibre loading levels (up to 30 wt%), the composite's hardness, flexural

strength, impact strength and Young's modulus were increased. Similarly, abaca fibre-reinforced high-density polyethylene (HDPE) composite was analyzed by Seculi *et al.*¹⁹. The coupling agent used in this study was maleic acid, which increased the interfacial strength of the PP matrix. Similarly, hexadecyltrimethoxysilane was also used to treat the surface of abaca fibre. The composite tensile strength and young modulus were increased with the addition of coupling agents to the polypropylene matrix. The 30wt% abaca fibre-reinforced composite with 8% maleic anhydride polypropylene exhibited higher tensile strength.

Similarly, other natural fibres have been studied as suitable materials for composite development for a wide range of applications²⁰⁻²³. It is reported that natural fibres have undergone a variety of surface treatments, including alkaline and MagPP treatments, to increase the composite's strength and adhesion. However, no study has been carried out on recycled polypropylene composite reinforced with abaca fibres. Hence, this study provides new insights into sustainable and ecofriendly composite material. Furthermore, there have been no reports on the water absorption behaviour and failure mechanism of abaca fibre/r-PP composite material, which is needed for automotive interior applications. Hence, this study also explores the effect of fibre loading, alkaline concentration and abaca fibre length on the mechanical and water absorption behaviours.

2 Materials and Methods

Abaca fibres purchased from Go Green Products, Chennai were used in three cut lengths (30,40 and 50 mm). Recycled polypropylene fibre from Kalyani Polymers Pvt Ltd, Bangalore was used to make fibre composite. Solubility and flammability tests were conducted to identify the supplied fibres. Acetic acid and sodium hydroxide were used to modify the surface of abaca fibres.

2.1 Surface Treatment of Abaca Fibres

The surface of the abaca fibres (fibre cut length 30, 40 & 50 mm) was treated with an alkaline solution (NaOH) of three different concentrations (4, 8 and 12 wt %) to improve the adhesion with the r-PP matrix for 24 h at 26 °C²⁴. Then, the fibres were taken out and neutralized using acetic acid and washed with fresh water to achieve a pH of 7. Finally, the abaca fibres were dried at 26°C for 24h. Material-to-liquor ratio of 1:20 was used.

2.2 Abaca Fibre/ r-PP Web Preparation

The treated abaca fibres were blended with recycled PP in different blend proportions (40/60, 30/70, and 20/80 abaca/rPP) using a miniature TRYTEX carding machine. The prepared fibre web was again passed into the carding machine to ensure uniform blending in the final card web.

2.3 Composite Preparation

The abaca/r-PP fibre web was used to prepare composites in a compression molding machine. The stacking of the abaca/ r-PP web was carried out to the required weight fractions, and the composite was made with a thickness of 2 mm. Throughout the composite manufacturing process in the compression molding machine, the consolidating pressure of 100 bar and temperature of 170°C were maintained for 45 min. The fibre weight fraction of 40% was maintained and various composites were prepared by reinforcing the different cut length fibres and NaOH treated fibres.

2.4 SEM Analysis

The surface morphology of untreated and alkali-treated abaca fibres was examined by scanning electron microscope (SEM). The magnification level used in the SEM analysis ranged from $\times 50$ to $\times 10000$. The surfaces of tensile fractured composite specimens were also examined by SEM.

2.5 XRD Analysis

X-ray diffraction (XRD) analysis was performed to determine any changes in crystallinity due to the alkaline treatment of fibres. The fibre sample was examined in a 2θ range extending from 10° to 50° . By observing the diffraction peaks of amorphous and crystalline areas in the resulting spectrum, the changes in fibres can be confirmed.

2.6 DSC and TGA Analyses

Differential scanning calorimetry (DSC) was utilized to measure the thermal behaviour of recycled PP fibre, following ASTM D3418 standards. DSC analysis identifies temperatures at which materials melt, crystallize, react, decompose, and undergo phase changes. Thermogravimetric analysis (TGA) was employed to assess the thermal stability of untreated abaca fibre and r-PP using a thermogravimetric analyzer (NETZSCH STA 449F3). Fibre sample (10 mg) was heated from 30°C to 550°C at a heating rate of 10°C/min. The test was carried out for 51 min.

2.7 FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) analyzes the presence of organic and inorganic compounds in the sample using the beams of infrared radiation. When infrared rays pass through the sample, they create vibrations according to the functional groups present, which are indicated as peaks. The frequency range used in this study for the measurement is from 4000 cm^{-1} to 6000 cm^{-1} .

2.8 Mechanical Testing

2.8.1 Tensile Test

The single abaca fibre strength was tested using a Zwickroell tensile tester according to the ASTM standard E3022-18. Single fibre strength was tested by applying longitudinal force to the fibre. Similarly, the tensile test for the developed composite was carried out in a Zwickroell tensile tester according to ASTM D3039 standard. The composite sample with dimensions of 250mm length, 25mm width and 2 mm thickness was prepared for the testing. The testing speed was 2mm/min, and the gauge length was 150 mm. The samples were placed in the Zwickroell universal testing machine, and the tensile load was applied until the breaking point under laboratory conditions.

2.8.2 Flexural Test

The flexural test was carried out using the four-point bending method in accordance with ASTM D6272- 02 standard on a Zwickroell universal tester. The composite sample having the dimensions of 76.2 mm length, 12.7 mm width, 50.8 mm span length, and 2 mm thickness was used for the test.

2.8.3 Impact Test

The impact test was carried out in an Izod impact tester in accordance with ISO 1598-1960 standard. The composite sample having the dimensions of 75 mm length, 10 mm width and 2 mm thickness was used for the test. A V-notch was created at a 45° angle as well as distance of 28 mm from the edge of the sample according to ISO 1598-1960 standard.

2.9 Water Absorption Test

The water absorption test for the developed composite sample was carried out in accordance with ASTM D570-98. The composite specimen with the dimensions of 50mm length and 50mm width was oven-dried for 24 h at 50°C. It was then immersed in a beaker of distilled water at 26 °C. After that, it was taken out and the weight gain was measured to calculate the moisture content of the composite.

3 Results and Discussion

3.1 Surface Analysis of Abaca Fibre using SEM Image

SEM image analysis is carried out to check the surface of alkaline-treated abaca fibres. Figure 1 shows the SEM images of surfaces of both untreated and treated abaca fibres (4%, 8% and 12 wt %). Due to the alkaline treatment of abaca fibres, the lignin is removed along with other substances, such as hemicelluloses, pectin, wax and other impurities as observed in Figs 1(a)-(d). As a result, the fibre surface becomes rough which can cause better adhesion with the matrix.

It is also evident that the surface of 12% NaOH-treated abaca fibre has a more uniform rough surface than other fibres treated with lower concentrations. Hence, better mechanical properties could be expected with a 12% alkaline treatment of fibres.

3.2 FTIR Analysis

As the surface of abaca fibres is modified by alkaline treatment, the change in functional groups of treated fibres is analyzed by FTIR. From the FTIR analysis, hydroxyl groups and O-H stretching

vibrations are confirmed by the peaks at around 2500 cm^{-1} . Similarly, the existence of the NO group is observed by the peak at around 1600 cm^{-1} , and AN, N-groups of cellulosic materials are observed at 1400 cm^{-1} . The characteristic of NO_2 symmetric deformation is obtained by the peaks at 1300 cm^{-1} . The removal of lignin in the treated fibre is confirmed by FTIR spectra.

3.3 TGA of Abaca and r-PP Fibres

TGA shows that the residual mass of abaca fibre decreases to 31.41% from the initial stage value (3.247 mg) at $548\text{ }^\circ\text{C}$. The fibre starts losing weight from $250\text{ }^\circ\text{C}$ onwards, indicating good thermal stability. Similarly, the TGA of r-PP shows decomposition starting at $270\text{ }^\circ\text{C}$ onwards. It suggests that r-PP fibre can have good thermal stability while processing in a compression molding machine for fabricating composite specimens.

3.4 DSC Analysis of Abaca and r-PP Fibres

Figure 2 shows the DSC results of abaca and r-PP fibres. The degradation temperature of untreated abaca

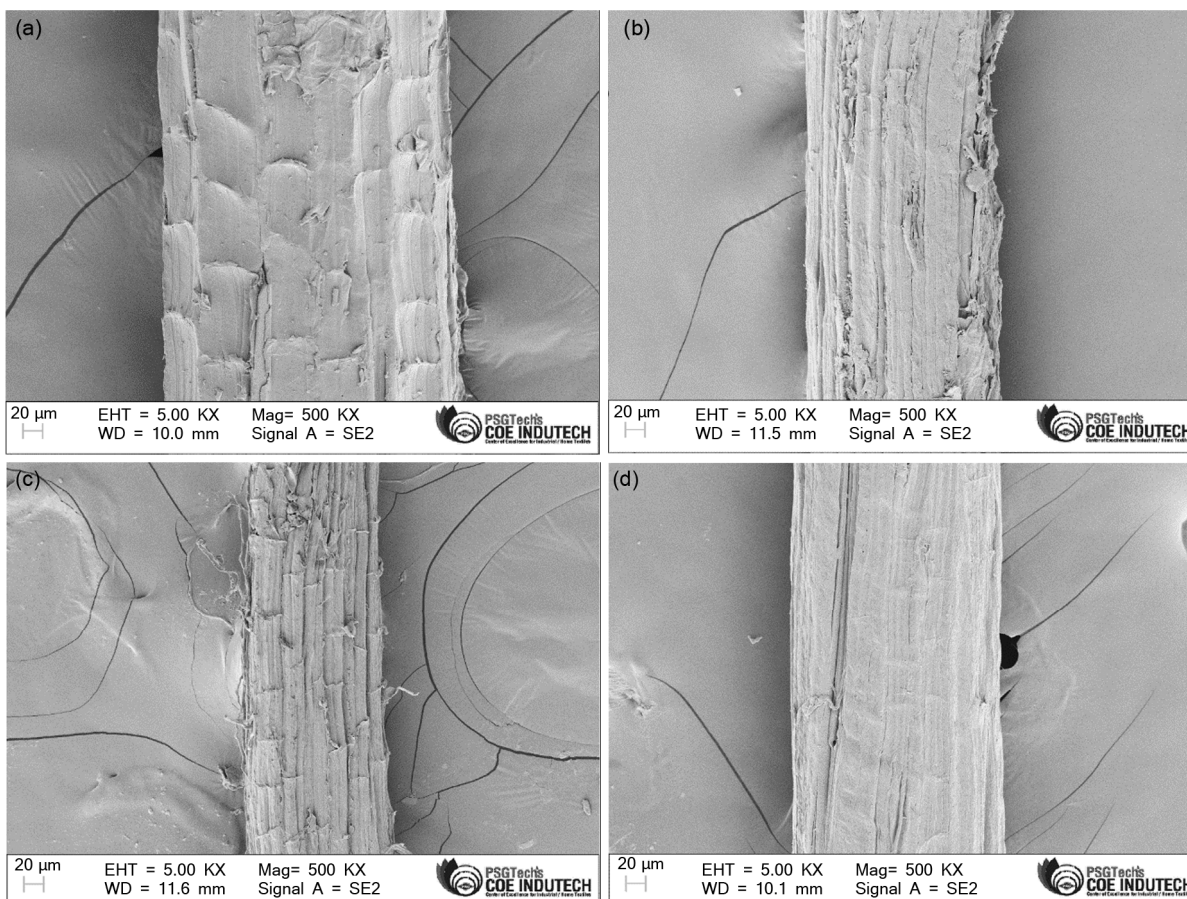


Fig. 1 — SEM images of (a) untreated, (b) 4% NaOH, (c) 8% NaOH and (d) 12% NaOH-treated abaca fibres

fibre is determined to be 347°C, indicating its ability to withstand temperatures up to 347 °C [Fig. 2 (a)]. Figure 2 (b) shows an endothermic peak at around 171.6- 179.9 °C in r-PP fibre, which refers to the exothermic reaction by melting (T_m). Hence, the melting temperature of r-PP is verified. The study reveals that both abaca and r-PP can withstand the processing temperature of 170 °C during composite preparation.

3.5 XRD Analysis of Treated Abaca and r-PP Fibre

The XRD results of untreated and 12% NaOH-treated abaca fibres are compared. Two peaks are observed in the XRD spectrum, one at 10°- 20° and the other at 20°- 30°. Due to NaOH treatment, there is

no crystallinity loss in the structure of abaca fibre. Similarly, r-PP fibre shows a good crystalline phase, making it a suitable matrix material for composite fabrication.

3.6 Tensile and Impact Strength of Abaca Fibre/r-PP Composites

The tensile testing of abaca fibre reveals that the fibre has the tensile strength of 8.04 N and elongation of 8%. Hence, the fibre has good tensile strength and can be used as reinforcement to develop the composite material. Figure 3 shows the tensile strength of composites reinforced with different abaca fibre lengths and various alkaline concentrations. To

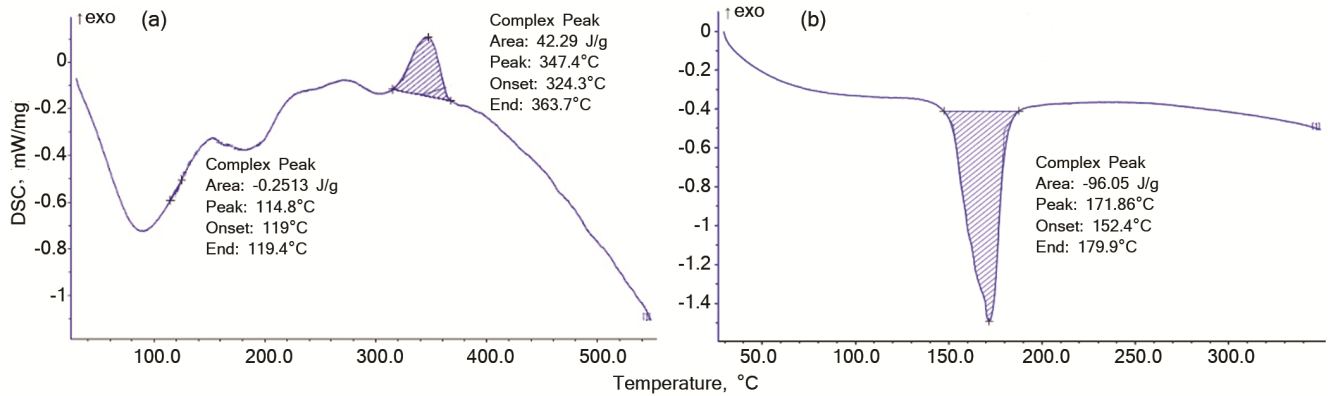


Fig. 2 — DSC of (a) abaca and (b) r-PP fibres

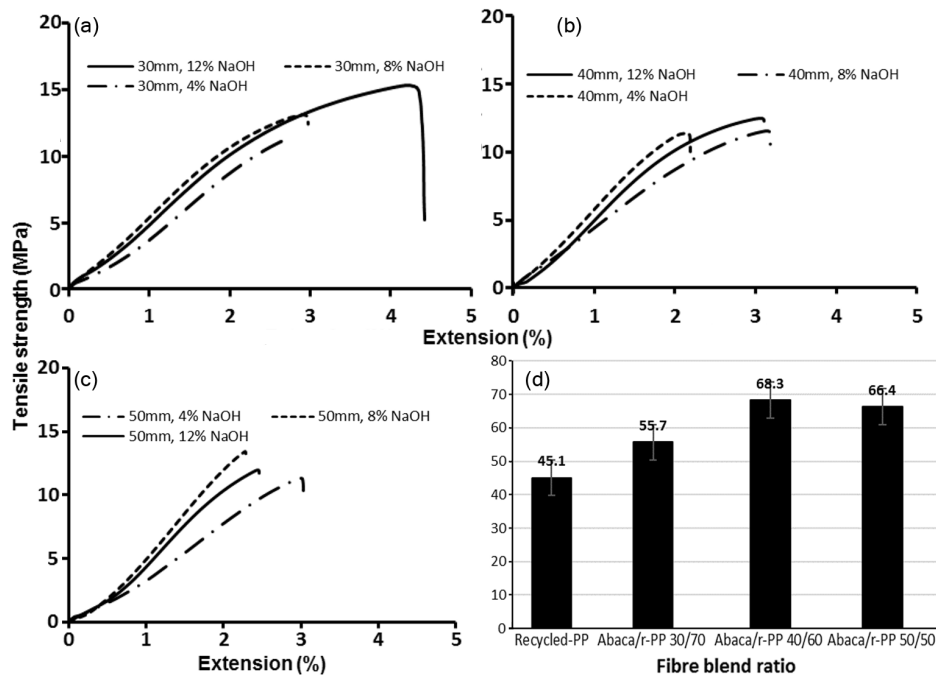


Fig. 3 — Tensile strength of abaca fibre composites reinforced with fibre lengths of (a) 30 mm (b) 40 mm and (c) 50 mm; and (d) tensile strength of composites at different fibre loadings (30, 40 and 50 wt %)

develop various composites, different cut lengths (30, 40, 50 mm) of abaca fibres are treated with 4 %, 8 % and 12 % NaOH. Nine different composites are developed with the same fibre loading level (40 wt %).

As observed in Fig. 3 (a-c), with the increase in the concentration of NaOH from 4% to 12 %, the tensile strength of the treated abaca fibre-reinforced composite is also increased. This is due to the improved bonding between the fibre and the matrix. Abaca fibre treated with 12% NaOH is taken for preparing the composite. While comparing the fibre length, the composite reinforced with 40 mm abaca fibre shows better tensile behaviour. Hence, composites with three different blend ratios of abaca fibre and r-PP (30/70, 40/60 and 50/50) are prepared. Figure 3 (d) shows the tensile strength of abaca fibre/r-PP composites with different fibre loadings of 30 wt %, 40 wt % and 50 wt %. All three developed abaca fibre/r-PP composites have fibre reinforcement of 40 mm length treated with 12% alkaline concentration. The tensile strength of fibre composite with 40 wt % fibre loading is higher than other composites. This is due to the presence of more fibres and better dispersion of fibre and matrix.

To assess the impact strength of developed fibre composite, Izod impact tester is used. The effect of fibre length and alkaline concentration on the impact strength of the developed composites is shown in Fig. 4 (a).

The impact strength of abaca fibre/ rPP composites reinforced with three fibre lengths (30, 40 and 50 mm) and fibre treatment concentrations (4, 8 and 12%) is compared. As seen in Fig. 4 (a), the composite reinforced with 50 mm fibre length and 12% NaOH treatment shows a higher impact strength of 6.9 J compared to other composites. To get effective composite fabrication, 40 mm fibre length is used. In order to investigate the effect of fibre loading on impact strength, various fibre proportions (30, 40 and 50 wt %) are analyzed [Fig. 4 (b)]. The composite with 40 wt% fibre loading shows higher impact strength than other composites. Beyond 40 wt % fibre loading, the impact strength of composite decreases.

3.7 Flexural Strength of Abaca / r-PP Composite

This study examines the flexural strength of untreated and alkaline-treated abaca fibre-reinforced r-PP. Figure 5 (a) shows the flexural strength of the developed abaca/r-PP composites, all having the fibre loading of 40 wt %.

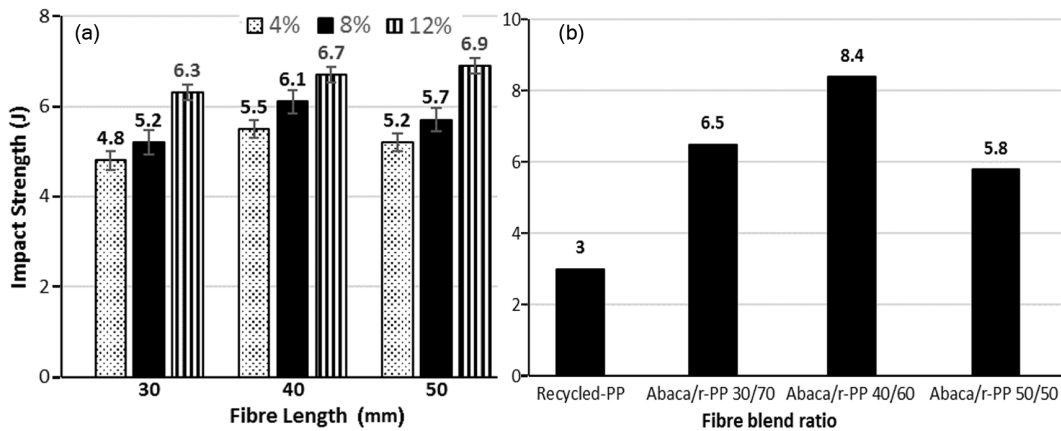


Fig. 4 — Impact strength of composites at different (a) fibre lengths and (b) fibre loadings

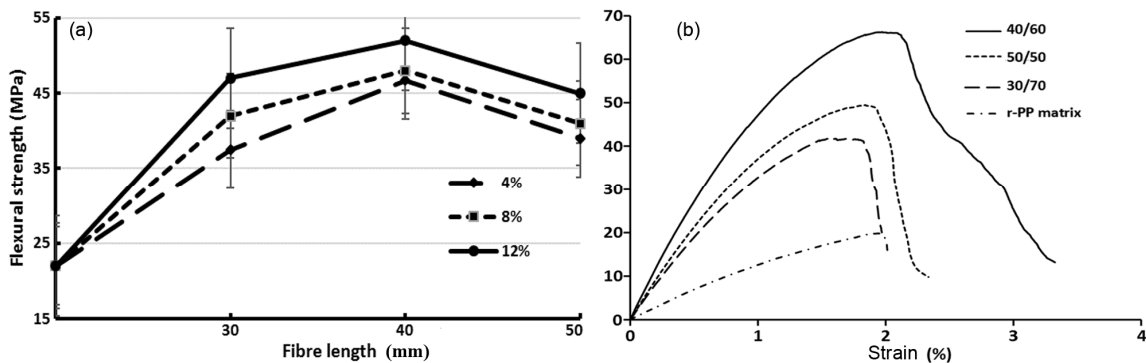


Fig. 5 — Flexural strength of developed abaca fibre composites reinforced with different (a) fibre lengths and (b) fibre loadings

As observed from Fig. 5 (a), increasing the NaOH concentration leads to an increase in the flexural strength of the composite. This is due to the formation of better bonding between fibre and matrix. The composite with 40 mm fibre length treated with 12% NaOH shows highest flexural strength of 49.4 MPa. The fibre length of 40 mm treated with 12% NaOH is selected to study the effect of fibre loading on the flexural strength of the composite. The abaca fibre composites prepared with three different fibre loadings (30%, 40% and 50%) are tested for flexural strength [Fig. 5 (b)]. Figure 5 (b) reveals the highest flexural strength for 40/60 fibre loading. This is due to the presence of more fibres and good dispersion of fibre and matrix.

3.8 Water Absorption Test of Abaca / r-PP Composite

The water absorption behaviour of composites reinforced with different fibre lengths (30, 40 and 50 mm) and different alkaline concentrations (4, 8 and 12%) is analyzed. Figure 6 shows the prepared composite board and water absorption % of all the developed abaca fibre composites.

As observed in Fig. 6 (b), the alkali treatment decreases the water absorption capacity of abaca fibre composites. Compared to treated fibre composite, a higher water absorption level is observed for untreated fibre composite (2.3%). This is due to the presence of hydroxyl group in the untreated fibre. As the alkaline concentration increases, the moisture absorption of composite reinforced with alkaline-treated fibre decreases. The composite reinforced with 30 mm abaca fibre length and 12% alkaline concentration shows the lowest water absorption level, i.e., 0.68% than other composites. This is due to the absence of lignin and wax content that reduces the

water absorption level. Hence, the water absorption value of composite is low after alkaline treatment. The water absorption behaviour of composites developed with fibre loadings of 30 %, 40 % and 50 % has also been compared. The composite with 30 % abaca fibre loading has the lowest water absorption levels as compared to other composites.

3.9 Fracture Analysis of Abaca Fibre/r-PP Composite

Fracture analysis helps to find the causes of the failure of composites during mechanical testing. This study analyses fractured surfaces of the tensile and flexural tested specimens using SEM image analysis. Figure 7 shows the cross-section of tensile fractured composite specimen having fibre reinforcement of 40 mm length and 12% alkaline treatments.

As seen in Fig. 7 (a), a crack is initiated due to the debonding of fibre from the embedded matrix during tensile testing. Similarly, the traces of fibre pullout from the embedded matrix are also observed in Fig. 7 (b). This may be due to matrix failure. Likewise, flexural fractured specimens are also analyzed using surface image analysis.

Figures 7 (c)-(d) show the SEM images of fractured specimens of flexural tested composites. The traces of fibre pull out and cracks formed in the composite after flexural failure. This may be due to the weakening of interfacial bonding after applying flexural load. As seen in Fig. 7 (d), good dispersion of fibres within the matrix is observed. As a result, good interfacial adhesion between the fibre and the matrix is identified, which helps to provide higher flexural strength. Hence, the interfacial bonding strength is better for 12% NaOH-treated abaca fibres.

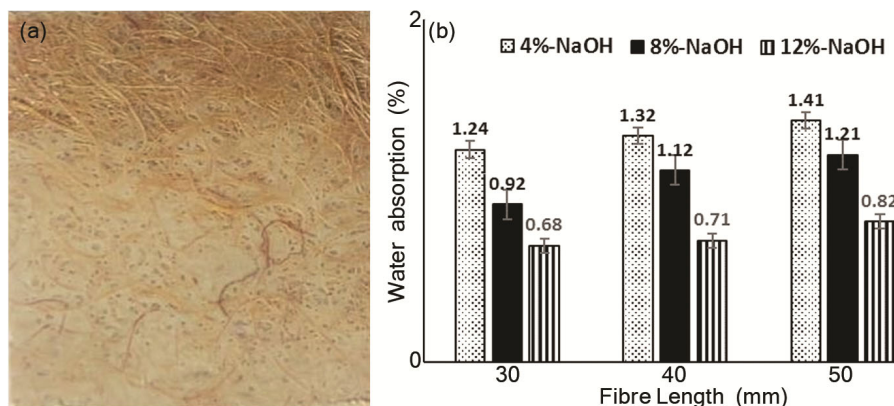


Fig. 6 — (a) Fabricated composite board and (b) water absorption % of composites reinforced with different fibre lengths and different alkali concentrations

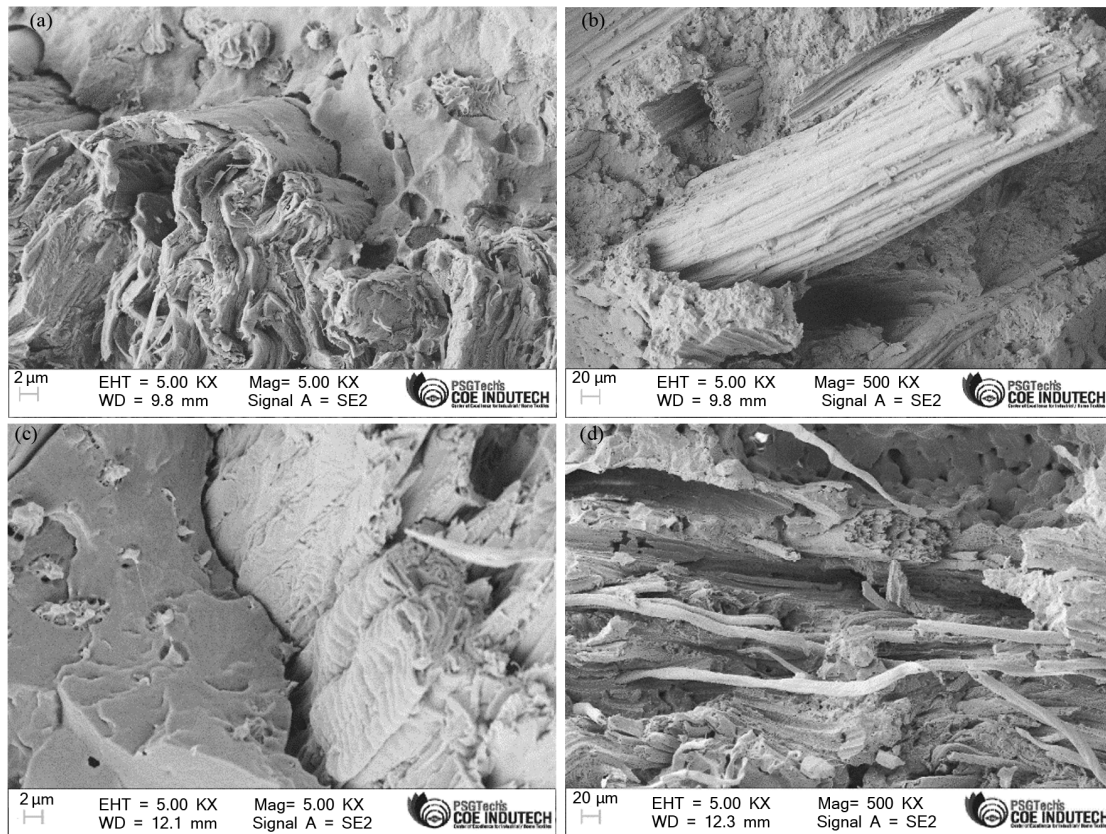


Fig. 7 — SEM images of tensile fractured composite reinforced with 40 mm length and 12% NaOH treated fibres (a) debonding and (b) fibre pullout, (c) crack formation and (d) fibre pull out

4 Conclusion

In this study, abaca fibre-reinforced recycled polypropylene composite is developed using the compression molding method and the effects of various parameters on the performance of composites are investigated. Various characterizations of abaca fibres have been carried out using TGA, DSC, FTIR and XRD analyses and following inferences are drawn:

4.1 FTIR analysis of alkaline-treated abaca fibre confirms the removal of lignin and wax substances with 12% NaOH.

4.2 XRD analysis confirms that the alkaline treatment does not affect the crystallinity of abaca fibre.

4.3 SEM analysis of treated fibre surface reveals roughening of fibre surface due to alkaline treatment.

4.4 Similarly, the thermal stability of abaca fibre and recycled polypropylene is confirmed by TGA and DSC analyses.

4.5 The mechanical characteristics of the composite, such as tensile strength, flexural strength and impact strength, are analyzed. The tensile strength is found higher for the composite with 40/60 abaca/r-PP

loading and 12% NaOH-treated fibres than other developed composites. Similarly, the flexural strength of composite having 40 mm fibre length and 12 % NaOH treated fibres shows a higher value of 49.4 MPa. The impact strength is also found the highest (8.4 J) for the composite, which has 40 % fibre loading and 12 % NaOH-treated fibres as compared to other composites.

4.6 The water absorption % of the developed composites is also tested. The composite reinforced with 12 % alkaline treated fibre shows low water absorption and better mechanical properties.

4.7 Fracture analysis of the tested composite specimens confirms that failure of the composite occurs due to debonding and crack formation at the fibre-matrix interface. The developed composite has potential applications in developing the automotive interior parts.

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