## **ABSTRACT**

Natural fibre composites are being replaced in many applications today. Banana fibres have better properties compared with glass fibres in terms of specific strength and stiffness and can be used as effective reinforcement in composite manufacture. When Banana fibres are used as reinforcement in thermoplastic composites, they prove to be sustainable and cheap. Polypropylene is a cheap and easy processable matrix material. The adhesion between the banana fibre and thermoplastic polypropylene matrix is the major problem to attain the required mechanical properties. The various surface treatments of the banana fibres improve the interfacial adhesion between the fibre and matrix. The current investigation is to compare the low cost and eco-friendly alternative fibre treatment methods with the common alkali treatment. The laccase and xylanase enzyme treatments have been given to banana fibres for better interfacial adhesion with the polypropylene matrix.

Banana pseudo stems were collected from field and the middle layers of the pseudo stem were used for mechanical extraction in a banana fibre extraction machine. Banana fibres were treated with NaOH, laccase and xylanase enzymes separately at 10%, 15% and 20% concentration levels.

The untreated and all the treated fibres were tested for chemical composition, moisture regain, density, diameter, single fibre strength, colour, morphological analysis using SEM, identification of functional groups using FTIR, crystallinity using XRD and thermogravimetric analysis.

The chemical composition of the untreated banana fibre shows that it contains 17.8% lignin, 11.1% hemicellulose, 69.5% cellulose and other materials such as wax, pectin, ash and other impurities constitute 1.54%. The lignin removal was 20%, 37% and 28% for NaOH concentrations of 10% 15% and 20% respectively. Maximum hemicellulose removal of 54% was obtained using 15% NaOH concentration. The moisture regain for untreated fibre was 13.4% and on treatment, it decreased to 11.23% at 15% NaOH concentration and on further increase in NaOH concentration to 20%, the moisture regain increased to 14.2%. The hydrophilicity is a drawback when used as reinforcement in composite application since it reduces the wettability of the hydrophobic resin. The diameter of the fibres decreased gradually on treatment. The surface morphology reveals that there is a separation in the fibre bundles. There is an increase in density due to removal of the less dense non-cellulosic contents till 15% treatment concentration and finally at 20%, it starts to decrease. As a result of the removal of non-cellulosic materials, better packing of the cellulose chains takes place. The NaOH treatment showed increase in tenacity by 7% and 15% respectively for 10% and 15% NaOH concentration levels due to the removal of hemi-cellulose, lignin and other non-cellulosic materials, which are amorphous in nature and are of low strength. The FTIR results confirm the removal of lignin and hemicellulose. This improves the tensile strength and crystallinity of the fibres, which is an advantage in both textiles as well as in composite applications.

Surface analysis using SEM shows that after 15% alkali treatment, fibres show ridges between the cellulosic fibrils due to further removal of the non-cellulosic material. At 20% alkali treatment, clear ridges and rupture of the cellulosic substances on the surface is evident which, confirms with the density and tensile strength results. The colour has become darker and the hue has also increased with increase in NaOH concentration, which is a drawback from the aesthetic point of view. The crystallinity % for untreated fibre is 59%, whereas for the NaOH treated fibres of 10%, 15% and 20% concentrations it is 65%, 69% and 64% respectively. This confirms with the tensile strength results which is also the highest at 15% NaOH treatment level. The TGA analysis shows that the thermal stability is low for the untreated fibres compared to NaOH treated fibres. The highest thermal stability is at a treatment concentration of 15% NaOH and increase in treatment concentration to 20% has reduced the thermal stability.

The chemical composition analysis and FTIR studies revealed the removal of non-cellulosic contents, which resulted in the increase in the cellulose % and decrease in the hemicellulose and lignin content. The optical microscope and the SEM analysis revealed the removal of the non-cellulosic content as well as the roughening of the fibre surface. Also it was found that at 9%, there was slight degradation of cellulose and at 20% concentration of laccase and xylanase enzyme treatment resulted in the degradation of the cellulosic content was evident. This was confirmed by the single fibre strength analysis, which showed lower values for these higher concentrations. The density test of the fibre revealed that the density of the fibre has increased due to the removal of the non-cellulosic low dense lignin and hemicellulose contents.

The diameter of the fibre has decreased due to the treatments carried out. The colour value reveals the aesthetic appearance of the fibre when it is converted into a product. It is seen form visual observation as well as the colour test that the laccase enzyme as well as the xylanase enzyme treated fibres have no much change in colour, but the alkali treated fibres have become darker and aesthetically unappealing. Due to the removal of lignin and hemicelluloses, the moisture content of the fibres has been modified. There is decrease in the moisture content of the treated fibres is high for the xylanase enzyme treated fibres as it acts on the hemicellulose regions, which is major factor for water absorption than the lignin. It is to be noted that there is increase in water absorption of the alkali treated fibres. The TGA and DSC studies showed that the thermal stability of the alkali as well the enzyme treated fibres have improved.

The optimum conditions for lignin removal using NaOH would help in the further analysis as lignin is the major non-cellulosic material in banana fibre. Hence the aim of this study is to discover the effect of various experimental parameters such as alkali concentration, time and temperature on the removal of lignin from banana fibres. The effect of these experimental parameters and optimal experimental conditions were ascertained by Response Surface Methodology (RSM) using Box-Behnken design. According to the results of Box-Behnken method, the optimum conditions for higher lignin removal from banana fibres are NaOH concentration of 11g/L, treatment time of 2.5 hours and temperature of  $90^{\circ}$ C. There are clear changes in fibre composition due to alkali treatment under optimum conditions. The removal of lignin from mechanically extracted banana fibre was at least 40% in the optimum conditions. There was 20% increase in the cellulose content. The hemicellulose contents were reduced by half the actual content.

The characterization of the laccase and xylanase enzyme treated banana fibres revealed the following. The laccase as well as the xylanase enzymes change the chemical composition and improve the cellulose % of the treated fibres. The lignin removal efficiency was up to 29% for 15% laccase enzyme treatment and 27.7% for 15% xylanase enzyme treatment. Increase in concentration of enzymes up to 15% was beneficial and no further improvement in lignin removal was observed for both the enzyme treatments at 20% concentrations. The maximum hemicellulose removal efficiency of 61% was observed for xylanase enzyme at 15% treatment concentration, whereas for laccase enzyme, only a maximum of 10% removal could be achieved at 15% concentration. The change in chemical composition has resulted in the improvement of tensile strength since the cellulose is the major

load bearing component for both the laccase as well as the xylanase enzyme treated banana fibres. The improvement in the cellulose % has improved the tensile strength of the fibre up to 15% treatment and further increase in the concentration of enzymes to 20%, fibre degradation has occurred, which was confirmed in the SEM micrographs.

Also, the removal of the less dense hemicelluloses and lignin has increased the density of the fibre for both the enzyme treatments. The diameter has been reduced to a greater extent till 15% enzyme concentration and resulted in a finer fibre. For both the enzyme treatments there is no marked difference with respect to color and is a positive aspect when compared to NaOH treatment for banana fibres, which has darkened the fibre. The FTIR spectra confirm the lignin removal to a greater extent. The X-ray diffraction also shows a maximum crystallinity of 74% for the 15% laccase enzyme treated fibre and 70% for the 15% xylanase enzyme treated fibre. The TGA analysis shows that the 15% xylanase treated fibres have the highest thermal stability compared to all the treatments followed by 15% laccase enzyme treated fibres.

Needle punched nonwovens were produced from Banana and Polypropylene fibres. Composites have been produced using compression moulding technique and properties such as density, volume and void fractions were determined. Mechanical properties such as hardness, tensile strength, flexural strength, impact resistance, thermal conductivity and sound absorption were also determined for all the composite samples.

The actual densities of the composites were observed to be lower than the theoretical densities due to the presence of voids. The void contents ranged between 2.2% to 3.6% for treated banana fibres. The density of the alkali and enzyme treated composites were found to be higher than the composites developed from untreated banana fibres, due to higher density of the treated fibres as well as in the reduction in voids. The Rockwell hardness results indicate that the reinforcement has increased the hardness. The unreinforced PP had a hardness of HRB 46 and the reinforcement has increased the hardness values to HRB 61.5. The alkali treatment has increased the hardness to 70.8 for 15% treatment. The laccase and xylanase enzyme treatment gave a composite of hardness 71.7 and 74.5 for 15% and 20% treatment respectively.

The tensile strength results of the composites show that the banana fibre reinforcement has improved the tensile strength. The results show that among the alkali treatment, the 15% concentration was good. In the case of laccase and xylanase*,* 20% concentration was good and these treatments have increased the tensile strength of the Banana/Polypropylene composites in the range of 11% to 73%. The xylanase treatment at 20% concentration has the highest strength of 48MPa, followed by laccase enzyme and alkali treatments. The 100% polypropylene had a flexural strength of 25.43MPa and that for banana fibre reinforced composites, the improvements were in the range of 42 to 80%. The highest (3 point) flexural strength was found to be for 20% xylanase treatment with 65.23MPa. The impact strength results show that the reinforced composites have impact strength in the range of 12.54 to 26.9 kJ/mm<sup>2</sup>, whereas the 100% polypropylene had impact strength of 9.21  $kJ/mm^2$ . Impact strength also, followed the same trend as flexural and tensile strength.

The sound absorption studies were done at 500, 1000 and 2000Hz for these composites. The sound absorption studies revealed that the reinforcement of banana fibres have improved the sound absorption of the composites. The various treatments have improved the composites sound absorption but the voids have reduced, which has a greater influence on the sound absorption characteristics.

The thermal conductivity of the untreated and treated composites was very much lower than the other composite materials. The 100% PP had a thermal conductivity of 0.22 and the reinforcements have reduced the thermal conductivity in the range of 12 to 22% on reinforcements due to various treatments. They did not follow the same trend as the mechanical properties. The presence of voids and the surface treatments have influenced the thermal conductivity. The composites with higher voids and more surface roughness have lower thermal conductivity. The incorporation of banana fibre induced a decrease of thermal conductivity of the composites. The use of NaOH treated fibre caused an increase in the thermal conductivity properties of the composites. Of the various treatments given, the NaOH fibre composites showed the highest values of thermal conductivity and the xylanase treated have the lowest thermal conductivity.

The environmental analysis done objectively revealed that enzyme treatment is considered be eco-friendlier than that the NaOH treatment. Based on the evaluated properties, the developed composites from the treated fibres are suggested to be suitable alternative material for the door trims in automotives.