Abstract - This paper investigates the possibility of use of pistachio shell flakes with flax fibres as reinforcements in polyester composites. Flax fibres were treated with 2% vol. NaOH to make it compatible with the polymer matrix. Physical and mechanical properties of the treated fibre at four different durations were determined and compared with the properties of untreated fibre. Fibre with high tensile strength and enhanced surface was selected for composite fabrication. Hand lay-up and compression moulding were used to fabricate the composites with 15% fibre weight fraction with pistachio shell flakes varying from 1 to 3 % by weight. Results indicate that addition of pistachio shell flakes reduced the tensile properties of the composites while their flexural and impact properties improved.

Keywords – Flax fibres, Pistachio shell flakes, Polyester resin, Hand lay-up, Compression moulding, Mechanical properties.

I. INTRODUCTION

Composite material is a class of engineered material system which involves fabricating by mixing or combining two or more constitutes at the macro level. Though different constituents are mixed during fabrication, they don’t dissolve into each other thereby making it a system where the constituents remain physically distinct and mechanically separable [1]. Composite material is gaining a lot of importance in today’s world and is being considered as a potential replacement for materials being traditionally used. Advantages such as greater strength to weight ratio, ease of fabrication to suit the requirement, greater durability, improved resistance to corrosion make
them ideal material for several engineering applications. High strength composites make use of reinforcements in the form of glass, carbon, and aramid fibres. For low strength engineering applications with short life span, composite materials fabricated using these reinforcements do not present themselves as an attractive option mainly because of the high cost of the fibres. Moreover, such reinforcements throw up challenges at the end of their life cycle in the form of recycling difficulties and waste material handling. On the other hand, natural fibres as reinforcements reduce the fabrication cost due to their lower costs, possess no health hazards, are easy to handle while fabricating, biodegradable in nature and available in abundance. However, a major drawback of natural fibres is its incompatibility with polymer matrices, leading to poor bonding between the two. To overcome this drawback, fibre surface are treated which involves use of chemicals. Nevertheless, research has shown that natural fibres have potential as an alternative to synthetic fibres which is evident from various products being proposed covering a large range of applications [2,3].

Beldzki et al. [4] analysed the effect of acetylation on structure and properties of flax fibres. Modified fibres were used as reinforcement in polypropylene composites. Tensile, flexural and impact properties of such composites were compared with composites prepared with unmodified flax fibres. Increased tensile and flexural strengths were observed in case of composites with modified fibres but with a decrease in impact strength. Heijenrath and Peijs [5] explored the potential of flax fibre as a reinforcement in polypropylene based composite. They compared the strength of the composites with epoxy/ flax and epoxy/ glass composites. They concluded that natural fibre reinforced composites can be considered for engineering application of low cost since they possessed reasonably high specific stiffness. Bairado M. et al. [6] observed that the fibre length had a significant influence on the strength of the composite material. In their work on flax-bionolle composites they reported a decrease in strength of the composite with increase in fibre loading mainly due to reduction in length of the fibres during fabrication of the composites. Bax B et al. [7] reported higher impact and tensile strength for PLA-Cordenka fibre composites over PLA-flax fibre composites but a higher modulus was observed for the later.

Agricultural waste products like shells of nuts like almond, groundnut, walnut, pistachio, coconut have very less economic value. They are presently used in composts, animal feed, as fertilizers, in insecticides and pesticides but have potential to be used as fillers in composites [8-13]. Ghazanfari A et al. [14] prepared plates which were biodegradable out of high density polyethylene, date pits and pistachio shells. They observed that plates with pistachio shells had greater stiffness than the one’s made from date pits. Solace Sam-Brew and Gregory D. Smith [15] fabricated particle board reinforced with flax and hemp fibre and compared the mechanical properties with conventional particleboard. They reported improved mechanical properties of composite particleboard over the conventional one.

II. EXPERIMENTAL PROCEDURE

Polyester (unsaturated) with the required catalyst and accelerating agent were obtained from M/s. Mookambika Poly Products, Udupi. Water retted flax fibres were procured from Andhra Pradesh. Flax fibres are like cotton in appearance except for its colour and strength. Flax fibres are stronger than cotton and are more suitable as reinforcements in composite materials. Pistachio shells were obtained
from Jammu. To avoid common salt getting added as a constituent and to avoid hassles of washing them, unsalted pistachio shells were used.

To remove the impurities from the outer surface of the fibre as well as reduce the hemicellulose content, the flax fibres were subjected to alkali treatment. The fibres were treated with 2% vol. NaOH solution for various duration viz. 0.5, 1, 2 and 4 h. To neutralize the effect of base, the fibres were rinsed with 2% vol. HCl before washing them with distilled water. The fibres were first dried in sun and then later in a hot air oven at a temperature of 40°C for a duration of 5 hours.

To understand the effect of treatment on the physical and mechanical properties of the fibre, diameter and density and tensile strength were determined. Diameter was measured using a profile projector and verified using a digital micrometer having a least count of 0.001mm. Density of the fibres was determined using digital density balance by measuring the weight of the fibre in air (W_a) and then in methanol (W_l). The following equation was used to determine the density of the fibre, knowing the density of methanol (ρ_o).

\[ \rho = \frac{W_a}{W_a - W_l} \times \rho_o \]

Morphological studies using a Scanning Electron Microscope (SEM) was carried out to understand the effect of the treatment on the surface of the fibre. The fibres were coated with silver by sputtering technique. Fourier Transform Infrared Spectroscopy (FTIR) was conducted on the fibres to confirm the extent of removal of hemicellulose from the fibre surface during the treatment. Fibres were chopped to fine powder and mixed with potassium bromide to make pellets for the FTIR examination.

The tensile strength of the fibres were determined using a Universal Testing Machine (Zwick Roell make). The tests were conducted according to ASTM C1557 [16, 17]. Fibres having a uniform diameter over the gauge length were selected for tensile testing. The gauge length was fixed to 30 mm and the testing was done at a constant crosshead speed of 0.5 mm/min.

Composite panels were fabricated by hand lay-up technique. A mould of 300 mm x 300 mm was used. Release agent was applied prior to laying up process. Polyester resin, cobalt naphthenate (accelerator) and methyl ethyl ketone peroxide (catalyst) were mixed in the ratio of 100:1:1 by weight. Fibre weight fraction of 15% was maintained in all the panels. Fibres having the highest tensile strength were selected. Pistachio shell flakes were added in three different weight fractions. Pistachio shell was crushed using a pestle and mortar to get flakes. To ensure removal of voids and effective bonding between the various constituents, the panels after lay-up were placed in a compression moulding machine. The panels were cured under pressure at room temperature for a duration of 24h. The average thickness of the composite panels was about 4mm. Table I gives the designation and design of each composite panel. Fig. 1 shows a cured panel.

**TABLE I**

<table>
<thead>
<tr>
<th>Designation</th>
<th>Matrix (wt. %)</th>
<th>Flax Fibre (wt. %)</th>
<th>Shell Flakes (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PFP0</td>
<td>85</td>
<td>15</td>
<td>0</td>
</tr>
<tr>
<td>PFP1</td>
<td>84</td>
<td>15</td>
<td>1</td>
</tr>
<tr>
<td>PFP2</td>
<td>83</td>
<td>15</td>
<td>2</td>
</tr>
<tr>
<td>PFP3</td>
<td>82</td>
<td>15</td>
<td>3</td>
</tr>
</tbody>
</table>
The cured composite panels were subjected to various tests to ascertain its mechanical properties. Tensile tests were conducted on Zwick Roell UTM at a constant cross head speed of 2 mm/min. The dimensions of the test specimen were 250mm x 25mm x 4mm. Gauge length was fixed to 150mm. ASTM D3039 [18] was followed for the tensile testing. Flexural tests were conducted on the same UTM according to ASTM D7264 [19]. Three point bending procedure was followed. Cross head speed was set to 1 mm/min. The dimensions of the specimen were 96mm x 13mm x 4mm. Span length was fixed to 80mm. Impact tests were conducted on a Zwick Roell pendulum impact tester. The dimensions of the specimen were 63.5mm x 12.7mm x 4mm. The specimens were subjected to an energy of 5.5 J at a theoretical velocity of 3.5 m/s following ASTM D256 [20].

### III. RESULTS AND DISCUSSION

Table II presents the variation in diameter and densities of fibres with variation in treating conditions. It is seen that the diameter of the fibre decreases with increase in treating duration which proves that the treating agent is effective in removal of impurities from the outer surface. The decrease in diameter is gradual when duration is increased to 1h and 2h. When the duration is increased to 4h, the decrease in diameter is drastic, an indication that the effect of treatment is strong on the fibre surface. Alkali treatment leads to increase in density of the fibre. The increase is minimal when treatment duration is increased from 0.5h to 1h, but when the duration is increased further to 2h and 4h, the increase in density is abrupt. Elimination of elements like hemicellulose and pectin during treatment led to increase in the density of the fibres.

<table>
<thead>
<tr>
<th>Duration of Treatment</th>
<th>Diameter (mm)</th>
<th>Density (g/cc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated (UT)</td>
<td>0.021</td>
<td>1.458</td>
</tr>
<tr>
<td>0.5 h</td>
<td>0.018</td>
<td>1.465</td>
</tr>
<tr>
<td>1 h</td>
<td>0.017</td>
<td>1.470</td>
</tr>
<tr>
<td>2 h</td>
<td>0.016</td>
<td>1.492</td>
</tr>
<tr>
<td>4 h</td>
<td>0.012</td>
<td>1.510</td>
</tr>
</tbody>
</table>

Variation in tensile strength of flax fibre is presented in Fig. 2. It can be observed that there is an increase in the tensile strength of flax fibre due to the alkali treatment till duration of 1h. With further increase in duration of treatment, tensile strength decreases which suggests that duration longer than 1 h has adverse effects on the structure of the fibre.

![Fig. 1 Cured composite panel](image)

![Fig. 2 Variation in tensile strength of flax fibre](image)
Highest tensile strength of 3.25 GPa was obtained for the fibre which was immersed in the NaOH solution for 1 h while that of the untreated fibre, the strength was 2.45 GPa. Modification in hemicellulose content led to increase in the strength of the fibres. The tensile strength of the fibres treated for 4 h was much lesser than the untreated fibres, i.e. 1.95 GPa. Reduction in strength is due to degradation of the cellular structure due to over exposure of the fibre in alkali solution.

Fig. 3 SEM Micrograph of untreated flax fibre

Fig. 4 SEM Micrograph of 0.5 h treated flax fibre

Fig. 5 SEM Micrograph of 1h treated flax fibre

Fig. 6 SEM Micrograph of 2h treated flax fibre

Fig. 7 SEM Micrograph of 4h treated flax fibre

Figure 3 to 7 above show the SEM micrographs of untreated as well as treated flax fiber samples. It can be observed that alkali treatment has resulted in removal of waxy outer layer along with surface impurities. Unidirectional grooves start to appear more prominently on the surface as duration of treatment is increased making the surface appear rough when compared to surface of the untreated fibre.

Fig. 8 FTIR spectra of untreated and treated flax fibres

FTIR spectra of the fibres are shown in Fig. 8. A decrease in the stretching frequency can be seen from...
1743 cm\(^{-1}\) to 1683 cm\(^{-1}\) between untreated and fibre treated for 4 hours. This is a clear indication of reduction of hemicellulose content [21]. Based on the tensile test results and the surface appearance from SEM micrographs, flax fibres treated for duration of 2h was selected for composite fabrication.

Inclusion of pistachio shell flakes had negative effects on the tensile properties of the composite material. This is evident from Fig. 9 where tensile property reduces with increase in pistachio shell flakes in the composite mix. Flax fibre reinforced composites had the highest strength of 51.62 MPa while the lowest strength of 30.59 MPa was seen for flax fibre composites with 3% by weight pistachio shell flakes in it. Pistachio shell flakes are not flat, this may have led to reduction in tensile strength of the composites. Effective transfer of stress between the matrix and reinforcing phases is not possible when the geometry and shape of the reinforcing element is irregular.

Flexural test results (Fig. 10) improve with addition of pistachio shell flakes in the composites. For flax fibre reinforced composite the flexural strength was observed to be 63.71 MPa while that for the composites with 3% by weight pistachio shell and flax the flexural strength was 79.32 MPa. Improved flexural strength with addition of pistachio shell flakes is probably due to high stiffness of the shell flakes under bending load. Though the improvement in strength was marginal, with coupling agent and uniform geometry of the pistachio shell flakes further improvement in strength can be expected.

Fig. 11 presents the impact test results. Presence of pistachio shell flakes resulted in improved energy absorption when compared to composites with only flax fibres as reinforcement. Increased resistance to impact loading is mainly because of the resistance provided by the flakes to propagation of cracks.

IV. CONCLUSION

Treatment of fibres with NaOH led to decrease in the diameter of fibres with increase in its density as duration of treatment was increased.
micrographs showed removal of impurities and waxy layers from the surface of flax fibres. FTIR tests confirmed the removal of hemicellulose during the treatment. Tensile tests of single fibres showed improvement in their strength with increase in duration till 2h. Treating the fibres for duration of 4h resulted in deterioration of the fibres leading to reduced tensile strength.

No improvement was seen in tensile strength of composites with inclusion of pistachio shell flakes while flexural strength improved. Impact strength of the composites improved with addition of pistachio shell flakes but with increase in proportion of the flakes, impact strength of the composites reduced.

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