Chapter 6

PC COATED NATURAL FABRIC
STERCULIA URENS
6. PC COATED NATURAL FABRIC STERCULIA URENS

6.1. INTRODUCTION

Increased environment awareness and the interest in long term sustainability of construction materials have led to the development of environmental friendly alternatives to synthetic oil based FRP (Fibre reinforced plastic) composites. In this regard, significant efforts were directed to investigate the use of natural fibres as reinforcement in thermoplastics. Natural fibres such as wood fibres, wheat fibres, straw fibres, jute fibres and bagasse fibres have several benefits viz., low cost, low density, high toughness, acceptable specific strength properties, enhanced energy, recovery and biodegradability [1-5].

The use of natural fibres in plastic matrix leads to many benefits such as low volumetric cost, increase of heat deflection temperature, increase of stiffness of thermoplastics and improvement of fibre surface appearance. However, the main drawback of natural fibres is their hydrophilic nature when compared to hydrophobic polymeric matrix. If no modification of fibre or compatibilization of the two materials is made, the weak interfacial adhesion between fibre and matrix usually results in poor mechanical properties of the composites. In the composite field, therefore, most of research is focused on improving interfacial properties between the polymer matrices and natural fibres in order to enhance the physical and mechanical properties of the end products [6, 7]. Demir et al. [8] studied the effect of fibre surface treatment on the tensile and water sorption properties of polypropylene-\textit{luffa} fibre composites. They reported improved interfacial bonding when coupling agents were used. There are various methods of promoting interfacial adhesion in
systems where fabric materials are used as reinforcements, such as graft co-polymerization, plasma treatment, coating, surface modification and chemical treatment [9]. Threepopnakul et al. [10] studied the effect of coupling agent on the performance of pineapple leaf fibre-polycarbonate composites and reported improved mechanical properties when the coupling agents were used. Khan and Hassan [11] studied the effect of aminobased silane coupling agent on the performance of jute-polycarbonate composites. Using FTIR spectra, they established the formation of Si-OH bonds between the fibres and the coupling agent. He et al. [12] studied the morphological differences in bamboo and some natural fibres using XRD, solid state NMR and second derivative FTIR techniques. Nejad et al. [13] treated wool fabric with some enzymes and studied the effect of surfactants on its physical properties. They reported loss of tensile strength on this treatment. Ismail et al. [14] studied the effect of filler loading and bonding agent on the dynamic properties and swelling behaviour of bamboo fibre filled natural rubber composites. They reported improved mechanical properties with increasing filler loading and addition of bonding agent. Hemmatinejad et al. [15] studied the effect of surfactants on enzymatic hydrolysis of cellulosic fabric. They measured the catalytic specificity of the cellulose on the hydrolytic reactions and reported that the behaviour was case sensitive. Sereshiti and Rovshandeh [16] modified Beech wood with alkylation and acetylation agents. They successfully dissolved the modified wood in some organic solvents and cast films with the solutions.

The mechanical properties of composites depend on the orientation of the reinforcement towards the stress direction. If the reinforcement is composed of uniaxial fibres, then their orientation can be achieved accurately with ease. Rarely, some natural fabrics exist with the fibres arranged in a uniaxial fashion. Hildlegardia populifolia [17],
Ridge gourd [18], Polyalthia cerasoides [19] are some of the examples for uniaxial fabrics. Recently, the author identified the new uniaxial fabric Sterculia urens and studied its properties [20]. In the present study, the author studied the effect of the coupling agent on the structural, morphological and tensile properties of the fabric under study. For this, the author used the FTIR, SEM and Tensile testing methods. Further, the author also coated the natural fabric Sterculia urens with polycarbonate. The tensile strength, modulus and % elongation at break of the uncoated and coated fabrics were determined. The effect of alkali treatment and coupling agent on the tensile properties of the fabric was also studied, to ascertain whether the polycarbonate and Sterculia urens fabric system could effectively be used for making green composites. The morphology of the untreated and alkali treated fabrics was studied using Scanning Electron Microscopy and that of coated fabric by Polarized Optical Microscopic techniques. The author selected the system of Sterculia urens/polycarbonate for the present study because Sterculia urens is a uniaxial natural fabric and polycarbonate is a tough polymer.

6.2. EXPERIMENTAL

6.2.1. EXTRACTION OF THE FABRIC FROM THE TREE

Samples of the fabric were extracted from the branches of the tree Sterculia urens. They were kept in agitated water to remove the dirt and other foreign materials. They were then thoroughly washed and dried in the sun for a week.
6.2.2. SAMPLE PREPARATION

Some fabric samples were treated with 5% aq NaOH salutation for half an hour to remove the hemicelllose and other greasy materials. Some fabrics were sprayed with silane coupling agent - 1% triethoxymethylsilane in acetone and dried. The fabrics were then coated with 10% Polycarbonate solution prepared with dichloromethane as the solvent using a thin layer chromatographic spreader. The average thickness of the coating was found to be 0.15mm. The coating on the fabric was allowed to dry at room temperature. The above procedure was followed for both untreated and alkali-treated fabrics.

6.2.3. MICROSCOPIC ANALYSIS

The micrographs of the untreated and alkali-treated fabric were recorded using JEOL JSM 820 Scanning electron microscope. The samples were gold coated by an electro deposition technique to impart electrical conduction before recording the SEMs. The optical micrographs (both bright field and polarized) of the fabrics were recorded using a Leica DMLP Polarized optical microscope.

6.2.4. FTIR ANALYSIS

The FTIR spectra of the powders of the neat, alkali treated fabric samples with and without coupling agent were run on an ABB-BOMEM FLATA-2000 model spectrophotometer using KBr pellets. The concentration of the fabric powder was maintained at 1% in KBr.
6.2.5. TENSILE PROPERTIES

The ultimate tensile strength, % of elongation at break and the modulus were determined using a Universal Testing Machine (INSTRON 3369). The fabric specimens with dimensions of length 100mm and width 10mm were cut. A gauge length of 50mm was maintained for all samples. The test was conducted at a crosshead speed of 5mm/min using 10kg load cell. In each case, ten samples were used and the average values are reported.

6.2.6. CHEMICAL RESISTANCE

The chemical resistance of uncoated and PC coated Sterculia urens fabrics to chemicals was studied by using ASTM D 543-87 method. In each case, ten pre weighed samples were dipped in the respective chemical for 24 hours, removed immediately, washed with distilled water and dried by pressing them between the filter papers. The samples were then re-weighed and the % of weight loss/gain was determined.

6.3. RESULTS AND DISCUSSION

The scanning electron micrographs of fabric surface of the neat fabric, neat fabric treated with the coupling agent; alkali treated fabric and alkali and coupling agent treated fabric are shown in Figure 6.1 (a), (b), (c) and (d) respectively. Significant changes were observed in each case. For example, the content of white components belonging to the hemicellulose in untreated neat fabric [Figure 6.1. (a)] decreased on coupling agent treatment [Figure 6.1.(b)]. This indicates the elimination of some surface held hemicellulose by the acetone of the coupling agent solution. It can also be observed from Figure 6.1. (c)
Figure 6.1 Scanning electron micrographs of (a) untreated (neat); (b) untreated and coupling agent used; (c) alkali treated; (d) alkali treated and coupling agent used natural fabric Sterculia urens at same magnification.
and Figure 6.1. (d) that in the case of alkali treated fabric, many shallow grooves were formed on the surface when a coupling agent was used. This observation indicates that the coupling agent in acetone etched the surface of the fabrics in both the cases of untreated and alkali treated fabrics. Further, the coupling agent also made the surface of the fabric rough. When the micrographs in Figure 6.1. (a) and Figure 6.1.(c) are compared, it can be observed that the white layer (corresponding to hemicellulose) decreased considerably on alkali treatment. This is as expected since the hemicellulose is soluble in aq.NaOH solution.

In order to investigate the structural changes in the fabric due to the alkali the FTIR spectra of untreated, alkali treated and polymer coated fabrics are presented in [Figure 6.2]. The spectra of neat [Figure 6.2.(a)] and alkali treated fabric [Figure 6.2.(c)] in the absence of coupling agent showed similar bands (corresponding to lignin and cellulose) except the disappearance of the band at 1730 cm$^{-1}$ when the fabric was treated with alkali. This band corresponds to the CO stretching of hemicellulose present in the untreated fabric. This indicates the elimination of hemicellulose on alkali treatment of the fabric. Further no appreciable changes in the spectra of the fabric in the absence and presence of coupling agent were noticed except the weak additional bands at around 860 cm$^{-1}$ when coupling agent was used for both neat [Figure 6.2.(b)] and alkali treated fabrics [Figure 6.2.(d)]. These bands around 860 cm$^{-1}$ correspond to the Si-OH bond which confirms the fact that –OH group of silanol reacts with cellulose or undergoes condensation reaction. Similar observation was made by Khan and Hassan et al. [11] in the case of jute fibres. The bands in the 860 cm$^{-1}$ appeared very weak due to the fact that the spectra were taken for bulk samples whereas the coupling agent used was only 1 % and it reacts only with surface groups of the fabric.
Figure 6.2 FTIR spectra of (a) untreated (neat); (b) untreated and coupling agent used; (c) alkali treated; (d) alkali treated and coupling agent used natural fabric *Sterculia urens*. 
The tensile properties, modulus, and % elongation at break of the uncoated and polycarbonate coated fabric with and without coupling agent are presented in Table 6.1. These values for the untreated and alkali treated fabric are also presented in the same table. The standard deviation in each case is also included in this table. In order to probe the effect of coupling agent on the properties of the neat and alkali treated fabrics, their tensile properties were also studied. From Table 6.1 it is evident that for both neat and alkali treated fabric, the coupling agent had little effect on the maximum (ultimate) stress (it increased only from 10 to 11 and 18.9 to 20.34 MPa respectively). This may be due to the fact that the changes were taking place only on the surface and not in the bulk. Further, for both the neat and alkali treated fabrics, the modulus was found to increase considerably whereas the %elongation at break decreased marginally when coupling agent was used. This may be due to the fact that as modulus is an initial property in the Stress-Strain behaviour, the slight increase in rigidity might have enhanced it.

From Table 6.1, it is further evident that the tensile strength of the untreated and alkali treated fabric increased from 10.0Mpa to 18.9Mpa. Similarly, the Young’s modulus increased from 640.7Mpa to 2018.6 Mpa on alkali treatment. This increment in tensile properties could be attributed to the elimination of the amorphous hemi-cellulose on alkali treatment. In the case of polycarbonate coated untreated and alkali treated fabrics also the tensile strength and the modulus increased appreciably on polymer coating. For the polycarbonate coated fabric, when the silane-coupling agent was used, the modulus increased remarkably. Khan et al. [21] made a similar observation in the case of jute-polycarbonate composites. They reported that the tensile strength and modulus of jute-
Table 6.1 Maximum stress, Young’s modulus and % elongation at break of untreated and alkali treated *Sterculia urens* natural fabric coated with polycarbonate in the absence and presence of coupling agent.

<table>
<thead>
<tr>
<th>Sterculia urens Fabric</th>
<th>Maximum Stress (MPa)</th>
<th>Young’s Modulus (MPa)</th>
<th>% Elongation at break</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>WOCA</td>
<td>WCA</td>
<td>WOCA</td>
</tr>
<tr>
<td>Untreated</td>
<td>10.0</td>
<td>1.0</td>
<td>640.7</td>
</tr>
<tr>
<td>(S.D.)</td>
<td>(0.8)</td>
<td>(1.01)</td>
<td>(29.3)</td>
</tr>
<tr>
<td>Alkali treated</td>
<td>18.9</td>
<td>20.34</td>
<td>2018.6</td>
</tr>
<tr>
<td>(S.D.)</td>
<td>(1.7)</td>
<td>(1.89)</td>
<td>(133.2)</td>
</tr>
<tr>
<td>Untreated and Polycarbonate coated</td>
<td>15.7</td>
<td>19.4</td>
<td>2113.4</td>
</tr>
<tr>
<td>(S.D.)</td>
<td>(3.1)</td>
<td>(2.1)</td>
<td>(140.2)</td>
</tr>
<tr>
<td>Alkali treated and Polycarbonate coated</td>
<td>27.1</td>
<td>32.3</td>
<td>2601.0</td>
</tr>
<tr>
<td>(S.D.)</td>
<td>(2.2)</td>
<td>(3.1)</td>
<td>(162.2)</td>
</tr>
</tbody>
</table>
polycarbonate composites increased by 28% and 70% respectively when amino based silane coupling agent was used. In the present case also we observed a similar enhancement of 19% and 53% when a silane coupling agent was used. However, no significant change in % elongation at break was observed for the polymer coated fabric. The improved bonding between the fabrics and polymer by the coupling agent might be responsible for this behaviour. Further, the polymer might have formed a film on the surface of the fabrics covering the void regions.

In order to probe the filling of the void regions of the fabric by the polymer, optical micrographs were recorded. The bright field and polarized optical micrographs are presented in Figure 6.3 & Figure 6.4 respectively for both untreated and alkali treated fabrics. The micrographs of the polycarbonate coated fabric both with and without coupling agent are also presented in the same figures.

From these micrographs, it is clearly evident that the polycarbonate film was formed on the fabric uniformly filling the void regions in it making the fabric a continuous one. Due to the formation of the continuum, the stress transfer is expected to be more uniform and effective and as a result, the tensile properties get enhanced. The film formation is visible clearly in bright field micrographs when compared to the POMs. As polycarbonate is amorphous in nature, its polarized micrographs yields only dark pattern and hence the filling up the voids by polymer could not be seen clearly in POMs. In order to probe the nature of interactions between cellulose and polycarbonate in the present system, their individual structures are presented Figure 6.5. and Figure 6.6.
Figure 6.3 Bright field micrographs of *Sterculia urens* natural fabric.

(a) Uncoated and untreated.
(b) Uncoated and alkali treated
(c) Untreated and polycarbonate coated in the absence of coupling agent
(d) Alkali treated and polycarbonate coated in the absence of coupling agent
(e) Untreated and polycarbonate coated in the presence of coupling agent
(f) Alkali treated and polycarbonate coated in the presence of coupling agent
Figure 6.4 Polarized Optical micrographs of *Sterculia urens* natural fabric

(a) Uncoated and untreated.
(b) Uncoated and alkali treated
(c) Untreated and polycarbonate coated in the absence of coupling agent
(d) Alkali treated and polycarbonate coated in the absence of coupling agent
(e) Untreated and polycarbonate coated in the presence of coupling agent
(f) Alkali treated and polycarbonate coated in the presence of coupling agent
Figure 6.5 Cellulose structure

Figure 6.6 Polycarbonate structure
PC Coated Natural Fabric Sterculia Urens

Chapter 6

From the above structures it is clearly evident that both cellulose and polycarbonate are having polar as well as hydrogen bonding attractive forces. As a result, better bonding due to these forces is expected between the polymer and the fabric under study. In order to quantify this effect, solubility parameter (which is a measure of inter molecular forces) are estimated using Hoy [22, 23] method. The estimated individual components - molar attraction ($\delta_T$), polar ($\delta_p$), hydrogen bonding ($\delta_h$) and dispersive force ($\delta_d$) are presented in Table 6.2.

From this table, it is clearly evident that for both the polymers, the molar attraction, polar and hydrogen bonding forces are higher than the weak dispersive force. Such higher forces facilitate better bonding between the two components.

Table 6.3, presents the weight loss/gain of the both coated and uncoated fabrics after immersion in different chemicals. The values are presented for the untreated and alkali treated fabrics in both the absence and presence of silane coupling agent. It is observed that coated fabrics showed weight gain when they were immersed in the acids and alkalis. As in the case of polycarbonate (PC) coated fabric, in the present case also the weight gain indicated the chemical resistance of PS coated fabrics for acids and alkalis. The coated fabrics show a weight loss on solvents treatment. This is due to fact that the polymer gets dissolved in the solvents. On the whole, it is observed that the Sterculia urens fabrics coated with PS showed better chemical resistance. Further, it is observed that the uncoated Sterculia urens fabrics absorb high quantity of water, while the PS coated fabrics are water resistant. Thus, Table 6.3 shows that the PS-coated fabrics gain chemical-resistance to acids alkalis whereas the resistance to solvents decreases.
Table 6.2 Estimation of solubility parameters of cellulose and polycarbonate by Hoy method.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Cellulose</th>
<th>Polycarbonate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total molar attraction component (δ_T)</td>
<td>29.56</td>
<td>19.97</td>
</tr>
<tr>
<td>Polar forces component (δ_p)</td>
<td>17.81</td>
<td>12.24</td>
</tr>
<tr>
<td>Hydrogen bonding component (δ_h)</td>
<td>18.25</td>
<td>11.55</td>
</tr>
<tr>
<td>Dispersive force component (δ_a)</td>
<td>14.95</td>
<td>10.75</td>
</tr>
</tbody>
</table>
Table 6.3 Effect of Chemicals on weight of *Sterculia urens* fabric coated with Polycarbonate.

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Untreated Blank</th>
<th>Treated Blank</th>
<th>UNTW OCA</th>
<th>TWOCA</th>
<th>UNTW CA</th>
<th>TWCA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetic acid (8%)</td>
<td>-3.69</td>
<td>15.67</td>
<td>11.25</td>
<td>14.58</td>
<td>11.98</td>
<td>16.07</td>
</tr>
<tr>
<td>Nitric acid (40%)</td>
<td>-19.52</td>
<td>-14.82</td>
<td>1.58</td>
<td>2.56</td>
<td>3.07</td>
<td>4.25</td>
</tr>
<tr>
<td>Hydrochloric acid (10%)</td>
<td>10.45</td>
<td>12.98</td>
<td>3.09</td>
<td>4.01</td>
<td>4.36</td>
<td>5.02</td>
</tr>
<tr>
<td>Sodium hydroxide (10%)</td>
<td>6.05</td>
<td>12.63</td>
<td>1.58</td>
<td>2.89</td>
<td>3.14</td>
<td>4.09</td>
</tr>
<tr>
<td>Sodium carbonates (20%)</td>
<td>12.85</td>
<td>17.11</td>
<td>2.41</td>
<td>3.25</td>
<td>4.05</td>
<td>5.63</td>
</tr>
<tr>
<td>Carbontetrachloride (250ml)</td>
<td>4.8</td>
<td>9.78</td>
<td>-4.25</td>
<td>-6.36</td>
<td>-5.48</td>
<td>-9.071</td>
</tr>
<tr>
<td>Water (250ml)</td>
<td>-10.25</td>
<td>-6.05</td>
<td>-3.08</td>
<td>-0.98</td>
<td>-4.39</td>
<td>-2.98</td>
</tr>
</tbody>
</table>
6.5. CONCLUSIONS

The effect of coupling agent on the neat and alkali treated uniaxial natural fabrics Sterculia urens. FTIR studies indicated the formation of Si-OH bonds on the surface of the coupling agent treated fabric. The SEM studies indicated marked surface modifications when coupling agent was used. Alkali treatment, polycarbonate coating and triethoxymethylsilane coupling agent increased the tensile properties of the Sterculia urens natural fabric. The elimination of amorphous hemicellulose by alkali treatment and filling up of the void regions of the fabric by polymer may be responsible for this behaviour. The optical micrographs confirmed the formation of the polymer film on the surface of the fabric and also filling its void regions. The presence of triethoxymethylsilane coupling agent further enhanced the tensile properties. The coated fabrics had better acid and alkali resistance. The improved bonding between the fabric and polycarbonate by the coupling agent may be the reason for this improvement.
REFERENCES


