CHAPTER 5

EFFECT OF PROCESSING CONDITIONS ON TENSILE STRENGTH PROPERTIES OF CFF AND ITS HYBRID COMPOSITES

5.1 INTRODUCTION

This chapter discusses the effect of moulding temperature, pressure and time on the final compression molded hybrid composites product for its tensile strength. The results of tests conducted to characterize the hybrid composites developed with different proportion of fibres and processing parameters are discussed in this chapter.

Tensile strength is defined as a ratio of the maximum load a material can sustain without fracture when being stretched to the original area of a cross section of the material. When stresses less than the tensile strength are uninvolved, a material completely or partially returns to its original size and shape. As the stress move toward that of the tensile strength, a material that has begun to flow, forms a narrow, contracted region that is easily fractured. Tensile strengths are measured in units of force per unit area.

Rahman (2009) reported that the tensile strength and bending strength were found to increase on enhancing the percentage of jute content in the composite. The highest jute content in the composite manufactured was taken as 60 %. Jute content above 60% could not be taken because mixing of jute and polypropylene was not good as the amount of polypropylene was not sufficient enough to diffuse.
5.2 MATERIALS AND METHODS

Composites were prepared at different conditions to study the effect of the process parameters on tensile strength of the sample. The materials and methods adopted are as discussed in chapter 3.

5.3 RESULTS AND DISCUSSION

5.3.1 Tensile Strength and Response Surface Regression Equations of CFF and its Hybrid Composites

The tensile strength of the CFF and its hybrid fibre reinforced polypropylene composites with different processing conditions under this investigation are presented in Table 5.1 and it is evident that, at run order 8 all the respective composites show better tensile strength properties as compared to other run orders. As seen from the results shown in Table 5.1, the addition of 12.50, 25, 37.50 and 50 weight% of jute fiber increases the tensile strength of the composite. The 100% CFF composites tensile strength values obtained from all the run orders are compared with other fibre loading wt% composites and presented herein. The 75:25 CFF/ Jute composite shows an increase in the tensile strength by 0% to 27%. The 50:50 CFF/Jute composite shows an increase in the tensile strength by 6% to 94%. The 25:75 CFF/Jute composite shows an increase in the tensile strength by 11% to 145%. The maximum gain in tensile strength was observed in 100% Jute composite. The increase in tensile strength is about 45% to 185%. From this investigation, it was clear that the addition of 50 weight% jute fiber in the manufacturing of composites gain maximum tensile strength properties over 50 wt% CFF composite.
Table 5.1 Tensile strength (Newton) of CFF and its Hybrid composites

<table>
<thead>
<tr>
<th>Run Order</th>
<th>100% CFF</th>
<th>75:25 CFF/Jute</th>
<th>50:50 CFF/Jute</th>
<th>25:75 CFF/Jute</th>
<th>100% Jute</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.9</td>
<td>1.9</td>
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<td>2.1</td>
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</tr>
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</tr>
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<td>2.9</td>
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<tr>
<td>15</td>
<td>1.8</td>
<td>1.9</td>
<td>1.9</td>
<td>2.0</td>
<td>2.3</td>
</tr>
</tbody>
</table>

The tensile strength of the composite material was obtained and the actual results were used to get the regression equations and it was obtained after giving a different temperature, pressure and time. The equations are shown in Table 5.2. Here ‘Y’ is the predicted response.
Table 5.2  Response surface regression equations for tensile strength of the CFF and its hybrid composites

<table>
<thead>
<tr>
<th>Sample</th>
<th>Regression Equation</th>
<th>R² %</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 % CFF</td>
<td>$Y = 1.70 + 0.25*(x_1) + 0.05*(x_2) + 0.07*(x_3) - 0.11*(x_1^2) - 0.08*(x_2^2) - 0.01*(x_3^2) - 0.02*(x_1x_2) - 0.12*(x_1x_3) - 0.02*(x_2x_3)$</td>
<td>95.97</td>
</tr>
<tr>
<td>75:25 CFF / Jute</td>
<td>$Y = 1.86 + 0.27*(x_1) + 0.01*(x_2) - 0.03*(x_3) + 0.06*(x_1^2) - 0.10*(x_2^2) - (x_3^2) + 0.02*(x_1x_2) - 0.12*(x_1x_3) + 0.15*(x_2x_3)$</td>
<td>82.39</td>
</tr>
<tr>
<td>50:50 CFF / Jute</td>
<td>$Y = 2.21 + 0.51*(x_1) + 0.52*(x_2) - 0.43*(x_3) + 0.20*(x_1^2) + 0.23*(x_2^2) - 0.04*(x_3^2) + 0.25*(x_1x_2) - 0.27*(x_1x_3) - 0.10*(x_2x_3)$</td>
<td>84.96</td>
</tr>
<tr>
<td>25:75 CFF / Jute</td>
<td>$Y = 2.86 + 0.38*(x_1) + 0.86*(x_2) - 0.62*(x_3) - 0.07*(x_1^2) + 0.17*(x_2^2) + 0.20*(x_3^2) + 0.32*(x_1x_2) + 0.10*(x_1x_3) + 0.10*(x_2x_3)$</td>
<td>89.46</td>
</tr>
<tr>
<td>100 % Jute</td>
<td>$Y = 3.19 + 0.20*(x_1) + 1.00*(x_2) - 0.66*(x_3) + 0.02*(x_1^2) + 0.62*(x_2^2) + 0.27*(x_3^2) + 0.49*(x_1x_2) - 0.12*(x_1x_3) + 0.24*(x_2x_3)$</td>
<td>95.60</td>
</tr>
</tbody>
</table>

(Refer Appendix 1 Table A 1.1 to Table A 1.5)

5.3.2  Effect of Processing Conditions on Tensile Strength of CFF and its Hybrid composites

The effects of process variables on mechanical properties can effectively be interpreted and explained by the contour plot shown in Figure 5.1. The information available from the contour plot diagram regarding the interactions of parameters on tensile strength properties is very much useful to manufacture CFF and its hybrid composites for various applications.
Figure 5.1 Contour plots of process conditions vs. tensile strength of CFF and its hybrid composites a) 100% CFF b) 75:25 CFF/Jute c) 50:50 CFF/Jute d) 25:75 CFF/Jute and e) 100% Jute
The influence of processing conditions on tensile strength of CFF and its hybrid composites samples by holding the middle value of time (6 Minutes.), temperature (175°C) and pressure (10 Bar) respectively is shown in Figure 5.1. Addition of jute fibres with CFF increased the tensile strength of the composites. If the proportion of the jute fibre was increased the tensile strength of the composites also increased.

The influence of processing conditions on tensile strength of 100% CFF composite is shown in Figure 5.1(a) and it confirms that, the increase in temperature directly increases the tensile strength of 100% CFF. But the maximum tensile strength (1.80N) was observed with increase in temperature at minimum pressure. It was observed that, when the pressure was increased the rate of increase in tensile strength is least and when the temperature increased the rate of increase in tensile strength show maximum value. It is due to the fact that at high temperature the polypropylene melts well and the interface between the fibre surface and resin became strong because of the homogeneous mixing of reinforced fibre and polypropylene in fibre stage during the carding process.

The influence between time and pressure for 100% CFF showed that the maximum tensile strength was achieved at moderate pressure with minimum time. Furthermore there was no drop or jump in the tensile strength with change in time. It is therefore clear that the only governing factor seems to be the amount of temperature applied on the composite.

The influence between time and temperature of 100% CFF composite samples showed the same trend as that in the previous case. As the temperature increased the tensile strength increased. The maximum tensile strength was observed at maximum temperature with minimum time. Melting happens when the polymer chains fall out of their crystal structures, and become a disordered liquid. Because of this, good binding will occur between
the reinforcement fibres, which in turn, increase the tensile strength. From the ANOVA results of 100% CFF composite (refer to Appendix 1 Table A1.6) it was observed that the F observed > F critical is 7.88, 89.76 (P< 0.05) for pressure, temperature respectively. F observed < F critical is 2 and 1.04 for pressure, time (P > 0.05) respectively. F observed > F critical is 8.41 (P<0.05), F observed < F critical is 1.16 (P>0.05) for temperature, time respectively. It was evident from the analysis that the temperature plays a significant role followed by pressure and insignificant influence by the time.

The influence of processing conditions on tensile strength of 75:25 CFF/Jute composite is shown in Figure 5.1 (b). The tensile strength of 75:25 CFF/Jute was observed. It gradually increased with increase in temperature. The influence of pressure on the tensile strength was not significant. Maximum tensile strength observed at maximum temperature with minimum pressure.

The influence of time and pressure was not significant on the tensile strength of 75:25 CFF/Jute composite. Time and the pressure shows a negative impact on the tensile strength ie: it shows decreasing trend with an increase in time and pressure. Higher processing pressure may cause damage to the fibre as well as leading to matrix starvation.

The impact of temperature on tensile strength is significant when compared with time for 75:25 CFF/Jute composite. Maximum tensile strength was observed at higher temperature and minimum time.

From the ANOVA results of 75:25 CFF/Jute composite (refer to Appendix 1 Table A1.7) it was observed that the F observed < F critical is 0.66 (P > 0.05) for pressure, F observed > F critical is 6.97 (P< 0.05) for temperature respectively. F observed < F critical is 0.23 and 0.28 (P > 0.05) for pressure, time respectively. F observed < F critical is 0.17 and 0.48 (P>0.05) for temperature and time respectively. It was observed that the
processing temperature had greater influence than the remaining conditions, since this showed the highest F value.

The influence of processing conditions on tensile strength of 50:50 CFF/Jute composite is shown in Figure 5.1 (c). It was apparent that the tensile strength of 50:50 CFF/Jute samples increased directly with increase in temperature and pressure. Maximum tensile strength was observed at the range of 12.5 – 15 Bar pressure and 180 -185°C temperature.

The influence between time and pressure, time and temperature for 50:50 CFF/Jute composite showed the same trend observed as in the previous case. Maximum tensile strength was observed at maximum pressure, temperature and minimum time. The time showed the negative impact on the tensile strength (i.e.) the tensile strength decreased with time even though there is an increase in pressure and temperature.

From the ANOVA results of 50:50 CFF/Jute composite (refer to Appendix 1 Table A1.8) it was observed that the F observed < F critical was 5.09 and 3.56 for pressure, temperature respectively. F observed < F critical was 2.17 and 6.18 for pressure, time (P=0.05) respectively. F observed < F critical was 5.19 and 3.30 for temperature, time respectively. The P > 0.05 was observed for all the remaining processing conditions. It was practical that statistically except the time the other processing conditions have no influence on the tensile strength of 50:50 CFF/Jute composite.

The influence of processing conditions on tensile strength of 25:75 CFF/Jute composite is shown in Figure 5.1 (d). The tensile strength of 25:75 CFF/ Jute samples maintained at the same level up to the moderate level of pressure even though there was increase in temperature. Tensile strength increased at high pressure level and showed maximum value when temperature and pressure were at a maximum.
The increase in pressure showed gradual increase in tensile strength whereas the increase in time showed decrease in tensile strength. Length of heating time influenced the wetting between fibre and matrix and also resulted in better impregnation of fibre and matrix. Contrary to the expectations, tensile strength decreased with an increase in processing time. This might be due to thermal degradation of the jute fibre, when exposed to higher processing time. The maximum tensile strength was observed at maximum pressure with minimum time. As time increased it was observed that, there was a gradual decrease in tensile strength of 25:75 CFF/Jute composite. Maximum tensile strength was observed at minimum time and maximum temperature.

From the ANOVA results of 25:75 CFF/Jute composite (refer to Appendix 1 Table A1.9) it was observed that the F observed < F critical was 4.12 and 0.51 for pressure, temperature (P> 0.05) respectively. F observed > F critical was 18.1 and 7.53 (P < 0.05) for pressure, time respectively. F observed < F critical was 1.12 (P>0.05) and 6.56 (P<0.05) for temperature, time respectively. It is evident from the results that, the pressure played a significant role and time showed a negative influence. It was observed that the processing parameter temperature had no influence.

The influence of processing conditions on tensile strength of 100% Jute composite is shown in Figure 5.1 (e). It was observed that the tensile strength of 100% Jute composite increased with the increase in temperature and pressure. At the moderate pressure there was no significant rise in tensile strength, even if there was an increase in temperature.

While analyzing the influence of time and pressure on the tensile of the 100% Jute, the trend was same as above, as the pressure increased the tensile strength increased and showed decrease in trend when the time increased.
The influence between time and temperature on the tensile strength of 100% Jute composite showed that at minimum time and an increase in temperature the tensile strength increased. If the exposure of jute fibre in thermal condition increased during processing of composites, which might cause thermal degradation, it leads to the reduction of tensile strength. Jute fibres can basically be considered as having rigid, crystalline cellulose micro fibrils reinforcing an amorphous lignin and hemicelluloses matrix. The tensile strength of jute fibre was mainly influenced by its cellulose morphology. Highly cross-linking or branched lignin, pectin and hemicelluloses provide a degree of structural integrity and rigidity, contributing to its stiffness. Normally, natural fibre thermal degradation processes starts at temperatures as low as 120°C, resulting in the decomposition of waxes present in the fibres. Temperature around 180°C leads to a decomposition of pectin. Temperatures of approximately 230°C have the effect of decomposition of cellulose (Ochi 2008). Short, cellulose-based fibres will also tend to agglomerate.

From the ANOVA results of 100% Jute composite (refer to Appendix 1 Table A1.10) it was observed that the F observed > F critical was 7.38 and F observed < F critical was 0.72 for pressure (P < 0.05), temperature (P > 0.05) respectively. F observed > F critical was 30.01 and 10.72 for time, pressure (P < 0.05) respectively. F observed > F critical was 0.37 and 3.47 for temperature, for time (P > 0.05) respectively. It is evident from the ANOVA analysis that the pressure played a significant role followed by the time which showed negative influence and insignificant influence of temperature.

From the response optimization technique (refer Appendix 1 Figure A1.1 to A1.5) it can be observed that, to achieve the maximum tensile strength of the composites, from the available samples the global solution would be of Temperature (°C) = 185, Pressure (Bar) = 15 and Time (Minutes) = 3 for 100% CFF, 75:25 CFF/Jute, 50:50 CFF/Jute, 25:75 CFF/Jute and 100% Jute composite.
5.3.3 Tensile Fracture Surface Analysis of CFF and its Hybrid Composites

Interfacial properties of CFF and its hybrid fibre reinforced polypropylene matrix based composites were investigated by SEM and are shown in Figure 5.2.

![Figure 5.2 Tensile fracture surfaces of CFF and its hybrid composites](image)

(a) 100% CFF  b) 75:25 CFF/Jute  c) 50:50 CFF/Jute  d) 25:75 CFF/Jute  e) 100% Jute
The SEM picture of tensile fracture surface of 100% CFF composite is shown in Figure 5.2(a). SEM observations indicated that based on the processing variables there was a considerable difference in the fibre-matrix interaction between the composites. From the figure it was demonstrated that the interfacial bonding between the CFF and PP resin was not good, as indicated by the gap between them. This may be attributed to the low adhesion between the fibre surfaces and PP resin. Hence, the tensile strength of this composite was low. Fibre pull-out occurrence was observed. Gaps between CFF and matrix were clearly found for composite board manufactured with low pressure and temperature and this sample which are responsible for the low tensile strength properties.

The tensile fracture surface of 75:25 CFF/Jute composite is shown in Figure 5.2(b). Cracks and holes between reinforcement and matrix were clearly found for composite board manufactured with low temperature and this sample which are responsible for the low tensile strength properties. Weak interfacial adhesions easily lead to complete de-bonding from the matrix in the tensile fracture surface.

The tensile fracture surface of 50:50 CFF/Jute composite is shown in Figure 5.2(c). The presence of pulled out traces and many holes between reinforcement and matrix are clearly found for composite board manufactured with low pressure and temperature which indicated the weak interfacial adhesion at the interface mainly responsible for reduced tensile strength.

The tensile fracture surface of 25:75 CFF/Jute composite is shown in Figure 5.2(d). From the SEM picture more fibres could be seen rather than polymer matrix and the large amount of poor bonded interface areas between reinforcement and matrix were responsible for the deformation of the composite board manufactured with low pressure and temperature.
The tensile fracture surface of 100% Jute composite board is shown in Figure 5.2(e). Fibre break and pull-out occurrence was observed. Some gaps between jute fibre and matrix were clearly found for composite board manufactured with low pressure and high temperature and these samples which are responsible for the low tensile strength properties. This may be either due to matrix fracture or due to fibre pullout.

5.3.4 Water Absorption Character of CFF and its Hybrid Composites

Water absorption characteristics of the manufactured composites against run order (processing conditions) is shown in Figure 5.3. Since water absorption of fibres plays a very important role in the reinforcement, the hydrophilic character of CFF and its hybrid fibre composite was investigated. Water absorption (%) increased with the increase in jute fibre loading. This may be due to the effect of the functional group polarity. With the water absorption, the fibres swell laterally, and apparently the water molecules which mediate between the chains comprise the fibre so that the secondary valence forces are weakened, thus assisting the slippage of the chain. That leads to changes in the physical properties of the fibre as well as the composites (Gassan and Bledzki 1999).

Figure 5.3 Water Absorption % of CFF and its Hybrid composites
It has been shown that water absorption of a fibre bunch takes place in three phases: during the first phase there is diffusion through the air from the water vapour to the fibre surface, the second phase involves diffusion through the air in the spaces between fibres from the surface of the bundle to the surface of a single fibre and the third phase involves diffusion from the surface of a fibre to its interior (Stamboulis et al 2001). As expected, the water absorption of composites increases if the jute wt% is increased. This is because of the hydrophilic nature of jute fibre. The water absorption of CFF composite is less when compared to jute composite, since amino acid content test of CFF (refer chapter 4) already revealed that the fibre possesses both hydrophilic and hydrophobic nature. High water absorption may cause application issues and requires further research work. For application, a low absorption is ideal; this may be reached either by fibre surface modification or improved interface.

5.4 CONCLUSION

CFF and hybrid fibre reinforced polypropylene matrix based composite boards were prepared by using compression moulding machine. The tensile strength of the composites produced with varying the process conditions has been analyzed and the following conclusions are drawn.

1. When comparing the overall results on the tensile strength of the composites, 100% Jute fibre composite showed highest tensile strength by keeping the process variables at 185°C temperature, 15 Bar pressure and 6 Minutes time. The lowest tensile strength was observed on 100% CFF composite by keeping the process variables at 165°C temperature, 10 Bar pressure and 3 Minutes time.
2. The influence of temperature and pressure were significant on the tensile strength of 100% CFF samples and the effect of time was insignificant. Maximum tensile strength of 2 N observed at temperature ranges from 175° to 185°C.

3. The maximum tensile strength of 2.4N was observed at maximum temperature and moderate pressure. The influence of pressure and time on the tensile strength is insignificant for 75:25 CFF/Jute samples. The temperature plays a major role in increasing the tensile strength.

4. The influence of temperature, pressure and time was not significant on the tensile strength of 50:50 CFF/Jute samples. Time has negative impact on the tensile strength. Maximum tensile strength (4.4N) was observed at maximum temperature and pressure.

5. The influence of pressure and time are significant on the tensile of 25:75 CFF/Jute and 100% Jute samples, whereas the time has a negative effect on the tensile strength. The role of temperature on tensile strength was insignificant. Maximum tensile strength of 4.4N and 5.7N was observed respectively.

6. The tensile fracture analysis by SEM reveals that presence of pulled out traces; cracks and many holes between reinforcement and matrix were clearly found for composite boards manufactured with low pressure and temperature.

7. The optimized processing conditions to achieve the maximum tensile strength of the composites, would be of Temperature (°C) = 185, Pressure (Bar) = 15 and Time (Minutes) = 3.