**EXPERIMENTAL AND THEORETICAL STUDY OF THE TENSILE MODULUS OF NEEDLE PUNCHED HEMP FIBER MAT COMPOSITES**

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1 Introduction

Despite its potential, current application of natural fibers in thermoset composites manufactured through VARTM (Vacuum Assisted Resin Transfer Molding) for semi-structural / structural applications is very limited due to lack of market pull, lack of commercial availability of natural fiber mat, and large scatter in properties of bio-composite [1-10]. A proper design of non-woven hemp mat is required to meet the functional and the manufacturing requirements of a composite part.

Fibers in the mat can be bound together by using either a binder (as in the case of currently used glass fiber mats) or needle punching. The latter (the focus of this study) uses a needle with a hook, which when driven into a mat pulls some fibers in the in-plane direction and reorients them in the thickness direction. These reoriented fibers along the thickness direction bind the fibers in the in-plane direction. Number of punches per unit area (i.e. punch density) and punch depth determine the level of binding, the structure and the thickness of the mat, which in turn influence the orientation and the volume fraction of fibers. The latter influence the tensile properties of the composite.

It can be inferred from Table 1 that needle punched improved the properties of natural fiber mat composites than glass fiber composites.

<table>
<thead>
<tr>
<th>Fiber</th>
<th>(w/w)%</th>
<th>Tensile modulus (GPa)</th>
<th>Tensile strength (MPa)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass</td>
<td>26.6</td>
<td>3.7</td>
<td>68</td>
<td>[11]</td>
</tr>
<tr>
<td>Glass</td>
<td>26.3</td>
<td>3.24</td>
<td>55.7</td>
<td>[11]</td>
</tr>
<tr>
<td>Flax</td>
<td>20</td>
<td>1.5</td>
<td>17.2</td>
<td>[12]</td>
</tr>
<tr>
<td>Flax</td>
<td>30</td>
<td>7</td>
<td>70</td>
<td>[13]</td>
</tr>
<tr>
<td>Hemp</td>
<td>30</td>
<td>2.6</td>
<td>32.9</td>
<td>[14]</td>
</tr>
<tr>
<td>Hemp</td>
<td>30</td>
<td>4.5</td>
<td>50</td>
<td>[2]</td>
</tr>
</tbody>
</table>

NP: Needle Punched

Table 1. Comparison between properties of needle punched natural fiber composites with that of glass fiber composites, with same polypropylene matrix

The modulus of hemp fiber mat composites, reported in the literature [2-10] vary widely in the range of 1.4 – 9.8 GPa, without any correlation to the fiber volume fraction. No additional information on mats, that could have explained this difference, could be found in these publications.

Hence, one objective of this study is to understand the effect of mat manufacturing parameters, specifically needle punch density, on the modulus of needle punched hemp mat composites.
While there are few published studies on applying micromechanical models to predict the modulus of natural fiber composite [15-19], a large difference between the predicted and experimental results are observed. While ignoring the variation of natural fiber’s modulus with diameter, observed in References 8, 20, and 21, is one possible reason, lack of consideration the effect of mat manufacturing parameters is believed to be another reason.

Hence, a second objective of this study is to develop a model to predict the modulus of needle punched hemp fiber mat composites that takes into account the scatter in natural fiber properties and the effect of mat manufacturing parameters.

2 Experimental Details

Unsaturated polyester (Stypol 8086 from Cook Composites and Polymers, Kansas, MO, USA) and Luperox 224 initiator from Sigma Aldrich (Oakville, Ontario, Canada) were used to manufacture the hemp fiber – unsaturated polyester composite. 1.25 % (w/w) of Luperox 224 was used. Hemp fibers were supplied by Stemergy Renewable Fiber Technology. Non-woven mats were manufactured by air-laying of the fibers to form a web and needle punching it. Needle punch density, in the range of 2.6-150 Punch/cm², and a needle punch depth of 8 mm were used. The areal weight of the mats was in the range of 900 – 1300 g/m². Composites were manufactured using VARTM. In order to vary volume fraction (Vf), panels were also cured under pressure (260 and 560 kPa) between the platens of the hydraulic press after resin impregnation under vacuum. The panels were cured under pressure, at room temperature, for 3 hours. After removal of the pressure, the panels were post-cured for six days at room temperature before testing. Tensile properties of composite were measured as per ASTM D4762-04 using INSTRON’s 5500R screw driven test frame. Volume of composite, resin, and fiber samples were measured using AccuPyc’s 1330 helium pycnometer as per ASTM D4892-89 and used with the mass of the samples determined using a precision balance, to determine the volume fraction of fibers (Vf ),

$$V_f = \frac{\rho_c - \rho_m}{\rho_f - \rho_m}$$

Figure 1. 3D image of a hemp mat using X-ray imaging

Distribution in the diameter of hemp fibers was determined using a sample batch of 0.5 gram of fibers randomly chosen from the fibers obtained from the supplier and Nikon’s Eclipse LV100 microscope. The cross section area of fibers was assumed to be circular and the diameter measurements were done for at least 10 positions along the fiber length and the average diameter is reported. The tensile modulus of these fibers using TA Instruments’ DMA Q800, and tension clamp as per ASTM D3822. Tests were run in ramp force mode at a rate of 1 N/min to 16 N. The modulus of fibers were calculated from the slope of the stress -strain curve within a strain range < 0.5%. These two rest results were used to determine the
distribution in the modulus of the fibers in the hemp mat.

X-ray tomography was used to determine the FOD of fibers in the composites. Due to the small difference in the densities of the polyester matrix and hemp fiber, imaging small diameter fibers was difficult. Hence, the FOD in dry mats subjected to same consolidation pressure as the composite was determined and input to the model. The 3D image of the mats was non-destructively imaged using X-Radia’s Micro X-ray CT and a representative image is shown in Fig.1. These images were subsequently analyzed using AVIZO’s Fire 7 software to obtain the FOD and diameter distribution in the mat.

3 Model Details

The needle punched composite was modeled as a laminated composite. The fiber orientation of a ply and the number of plies with that fiber orientation were determined using the experimentally determined Fiber Orientation Distribution (FOD).

The laminated analogy was introduced first by Halpin et al. [22]. They used a quasi-isotropic lay-up to predict the tensile modulus of short glass fiber composite and compared it with Halpin-Tsai predictions in Ref. 23. Later, they [24] improved the accuracy of predictions by assuming fiber orientations in 18 angles equally spaced between $0^\circ$ to $90^\circ$ and compared the predictions with experimental values reported in Ref. 25. Fu et al. [26] extended this approach to include a continuous orientation distribution from $0^\circ$ to $90^\circ$. They suggested a mathematical function for orientation distribution function and studied how different parameters in orientation distribution function can affect the modulus prediction. However, they did not have experimental orientation data to calculate the orientation function. This laminated analogy has not been applied in the past to needle punched mat composites.

In this study, the equivalent laminate is assumed to be balanced and symmetric $[\pm \theta_1, \pm \theta_2, ..., \pm \theta_n]_s$, and is made up of transversely isotropic lamina layers.

The number of plies and stacking sequence were calculated using the FOD, determined experimentally.

The normalized frequency of plies ($f(\theta_i)$) with orientations of $\pm \theta_k$ was calculated using

$$ f(\pm \theta_k) = \frac{[n(+\theta_k) + n(-\theta_k)]/2}{\Sigma n(\theta_p)} \quad (2) $$

where $f(\theta_i)$ is the normalized frequency of fibers in each orientation. It also represents the fraction of laminate thickness with fibers oriented at an angle of $\theta_k$. The number of plies in each orientation can be calculated using

$$ n_{\theta_i} = f(\theta_i) \times \frac{h}{t_{\text{lamina}}} \quad (3) $$

where $h$ is the total thickness of composite laminate and $t_{\text{lamina}}$ is the thickness of a ply. A value of 0.25 mm was found to satisfy all punch densities. The stiffness of the equivalent composite was calculated using 2D lamination theory as per

$$ A_{ij} = \Sigma_{k=1}^n n_{\theta_i} \times (\bar{Q}_{ij})_k \times t_{\text{lamina}} \quad (4) $$

where $n$ is total number of plies and is calculated by dividing $h$ by the thickness of each lamina. $(\bar{Q}_{ij})_k$ is the stiffness matrix for each lamina determined using engineering constants of the hemp fiber lamina. The effective longitudinal ($E_x$) and transverse ($E_y$) tensile moduli of the composite were determined using...
where $a_{ij}^*$ is the inverse of the $A_{ij}$ matrix.

The engineering constants of the unidirectional lamina were calculated using the well-known micromechanical models [27-30]. The tensile modulus of the hemp fibers and the polyester resin matrix were determined experimentally in this study. The remaining properties of the fiber, taken from the literature, are tabulated in Table 2 along with those of the resin.

<table>
<thead>
<tr>
<th>$E_m$ (GPa)</th>
<th>$G_t$ (GPa)</th>
<th>$G_m$ (GPa)</th>
<th>$\nu_m$</th>
<th>$\nu_f$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.869</td>
<td>6</td>
<td>1.2</td>
<td>0.32</td>
<td>0.24</td>
</tr>
</tbody>
</table>

Table 2. Elastic constants of Hemp fibers and Stypol 8086 resin

### 4 Results and Discussion

Experimentally determined longitudinal tensile modulus of the hemp fibers in the supplied fibers is plotted in Fig. 2 as a function of fiber diameter. The modulus increased exponentially with decrease in hemp fiber diameter.

The distribution in the diameter of the fibers in the hemp mat was different from that of the as received fibers. Hence, the diameter distribution in each mat was determined using the X-ray image. Using this and the data in Fig. 2, the distribution in the modulus of hemp fibers in the mat was determined. A representative result for 0-P mat is plotted in Fig. 3. It can be inferred that the tensile modulus of hemp fibers in 0-P varied from 2 GPa to 20 GPa. The average modulus in a mat was calculated using equation (7).
Table 3. Experimentally measured \( V_f \) and modulus of hemp fiber mat composites manufactured at VARTM pressure (101 kPa)

\[
E_f\text{avg} = \frac{n_f E_f}{\sum E_f} \tag{7}
\]

where \( n_f \) is the frequency of a certain tensile modulus. \( E_{f\text{avg}} \) was used in determining the moduli of the lamina.

The transverse modulus was higher than longitudinal modulus for punch densities. The moduli increased with punch density due to increase in \( V_f \). Despite higher \( V_f \), the modulus of 150-P composite is lower than that of 30-P and the modulus of 70-P is lower than that of 20-P composite. This is due to change in FOD, observed in Fig. 4.

Since the interpretation of the FOD is difficult, it was used to determine a single planar orientation factor \( (f_p) \) defined as

\[
f_p = 2 < \cos^2(\theta) > -1 \tag{8}
\]

where \( < \cos^2(\theta) > = \int_{0}^{\pi/2} f(\theta) \cos^2(\theta) d\theta \)

The values of -1 and +1 for this factor correspond to orientation of all fibers along the transverse direction (\( \theta=90^\circ \)) and the longitudinal direction (\( \theta=0^\circ \)) respectively. A value between -1 and 0 means more fibers are oriented at angles closer to 90\(^\circ\) than 0\(^\circ\). A value between 0 and +1 means more fibers are oriented at angles closer to 0\(^\circ\) than 90\(^\circ\). The negative values for all punch densities correlate well with the higher transverse modulus (than longitudinal modulus) observed in Table 3.

The modulus is a function of both \( V_f \) and \( f_p \). Difference in \( f_p \) is the reason for lower moduli of 70-P and 150-P, despite the higher \( V_f \) than 20-P and 30-P, respectively.

The effect of consolidation pressure on composite properties is shown in Figures 5 and 6 for longitudinal and transverse modulus, respectively.
The $V_f$ increased with pressure as expected. However, the moduli did not increase proportionately. This is due to the complex change in the FOD with pressure, as observed in Fig. 4. Similar trend was observed at other punch densities. At a given $V_f$, there is no correlation between the punch density and moduli due to difference in the FOD.

The moduli, predicted using the model presented in Section 3 are compared with experimental results in Figures 5 and 6. The model predicted the non-linear change in moduli with $V_f$ correctly. The error in the predictions was less than 37%. Similar results were obtained for other punch densities.

Predictions using well known micromechanical models, simple rule of mixture (ROM) [24], inverse rule of mixture modified by Morais (IROM) [28], and Halpin-Tsai model [33] are also plotted in Figures 5 and 6. These models predicted a linear increase in moduli with $V_f$ and did not predict the observed trend.

5 Conclusion
The modulus of needle punched hemp fiber composites vary significantly with punch density and consolidation pressure applied during manufacturing. This variation is due to change in $V_f$ and FOD with punch density and pressure. An equivalent laminate, with its lay-up defined by experimental FOD, combined with lamination theory has been shown to predict the change in modulus with pressure correctly for all punch densities.

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References


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