BIOCOMPOSITE WITH POLYPROPYLENE AND ALFA FIBERS: STRUCTURE/PROPERTIES RELATIONSHIP

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ABSTRACT

We present a study on the development of thermoplastic bio-composites reinforced by Alfa fibers (stepa tenassicma). We applied different surface treatments on obtained fibers, to promote their adherence to the polypropylene matrix. The mixture of the fibers and the matrix was extruded and pelletized according to a defined operating mode.

We conducted a detailed study for the definition and optimization of injection molding process's industrial conditions of the composites. The tests carried out on the specimens showed that the mechanical and viscoelastic properties of these composites display a marked improvement depending on surface treatments and the rate of Alfa fibers and injection parameters. Other microscopic features were used to analyze the flowing behavior of Alfa fibers along a injected part, for fiber lengths greater than 4.5 mm, we highlighted the phenomena of deformation and coalescence and aggregation of the fibers according to the injection process parameters.

Key words: Bio Composite, Polypropylene/Alfa short fibers, alkali treatment, injection molding, morphology and mechanical behavior.


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1. INTRODUCTION

The use of bio composites based on plant fibers is growing rapidly with many industrial applications. Vegetable fibers are sought for the many environmental and economic and technological and health advantages whose industries could benefit of: renewable
material - biodegradable - neutral carbon balance - low density and energy saving - flexibility - low cost and less or no health problem of operators on production lines [1-5].

Bio-composite based on Alfa fibers may find their application in the health field such as dentistry or auto industry for applications such as interior door trim and the coffers of roofs and bumper.

However, technical brakes exist when processing these fibers included a specific technical developments. Today, the main lines of technical development of associated fibers and bio materials lie in improving fiber properties by the selection in optimizing industrial shaping processes [4-8].

The Alfa fibers are biological structures primarily composed of cellulose and hemicellulose and lignin. Unlike the other components of fibers having an amorphous structure, the cellulose has a large crystalline structure portion. Inside the fiber, the cellulose chains are joined as microfibrils that agglomerate form fibrils, the angle between these highly structured elements and the axis of the fiber determines the stiffness of the fiber [10].

The properties of the fibers (flexibility, smoothness, cellulose fiber content) make this plant a valuable source of raw material for the paper industry. "A clump of Alfa can withstand successive operations for 25 to 30 years, under the condition of a resting 2 to 3 years after a number of holdings [11-12].

The Alfa fibers have the following composition: 45% cellulose, 25% hemicellulose, 23% lignin, 5% of wax and 2% ash [13-16]. In terms of structure, observation by scanning electron microscopy shows that a Alfa rod consists of fiber bundles with lignin. Their form is a kind of tube whose section is not circular. The largest dimension of the tube section is approximately 200µm.

The treatment of the fibers with acetic anhydride is an effective method to reduce the hydrophilic nature of cellulose fibers, and improve the dispersion of fibers in a thermoplastic composite. Acetic anhydride reduces the fiber surface energy to make it non-polar and more similar to the thermoplastic matrix [18-19]. Acetate bonds are formed by reaction of acetic anhydride. The OH groups of the fiber surface are therefore no longer available to react with other groups, such as hydroxyl groups of water.

The objective of this work is to develop a method of extracting Alfa fiber and make thermoplastic composite’s formulations of polypropylene (PP) reinforced with short Alfa fibers with particular attention to the influence of fiber processing conditions and the conditions of implementation. The various observations will allow us to link the microstructure and behavior to the mechanical strength of our composites.

2. MATERIALS AND TECHNIQUES

2.1. Preparation of composite PP / Alfa short fibers

We carried on Alfa plants operations of drying and retting in a moist environment in the presence of salt. Thereafter, the fibers were combed and untangled and subjected a crushing operation, to prepare the mechanical carding, then grinding for optimal average length allowing the fiber to fully play its role of reinforcement without causing phenomena agglomeration.

Dispersing the fibers in a composite material is an important factor for achieving good mechanical properties. Bad distribution results in the presence of fiber agglomerations. Thus, areas rich in fiber or matrix appear randomly distributed and have bad properties. To ensure good dispersion of the fibers in the composite, it is essential to eliminate or limit the aggregation. Good wetting is achieved when each fiber is totally wrapped by the matrix.
The main problem with the use of Alfa fibers as a reinforcing material is cohesion and bonding with the matrix. Alfa fibers are hydrophilic, however the polymers used as matrices such as polypropylene, are often hydrophobic. Therefore, the functional groups do not bind easily. Thus, the composite loses its mechanical properties and is more easily broken at the connection.

Pretreatment can decrease the hydrophilicity of Alfa fibers and increase that of polymers for a better bonding is possible.

We have applied various surface treatments to the fibers obtained, for sodium hydroxide and maleic anhydride in order to promote their adhesion to the thermoplastic matrix.

The thermoplastic used in our composite is a polypropylene market density 0.905 g/cm³, and melt flow index of 12g/10min at 230°C and 2.16kg (according to ISO 1133 standard).

Polypropylene modified with maleic anhydride (MA) is a coupling agent composed of long polymer chains with a functional group MA grafted on one extremity. MAPP acts as a bridge between the non-polar polypropylene and polar cellulose fibers. MA group binds to the surface of the cellulose to form covalent or hydrogen bonds with the reactive OH groups on the surface of the cellulose and lignin. The rest of the chain remains free and forms tangles with the chains of the polymer PP.

The mixture of fibers/thermoplastic polymer and coupling agent and additives were extruded and granulated, in our laboratory by using an extruder whose screw profile is adapted to the composite compounding using a defined operating mode. Using an experimental design, we conducted an extensive study for the definition and optimization of injection molding conditions of the manufactured composites.

Figure 1 The main stages of development of bio composite material with polypropylene/short Alfa fibers.

A: Harvesting Alfa plants B: Retting and treatment
C: Grinding and final treatment of fibers D: Mixing and granulation and injection molding of the composite

2.2. Morphological and mechanical characterization of bio-composites with PP/Alfa Fibers

We determined the optimum processing parameters of our materials and we injected rectangular plates (dimensions 180 x 3.5 x 98 mm), equipped with variable angle of injection threshold over which we cut out the specimens used for morphological and mechanical tests.
The main injection parameters used for this study are summarized in the Table 1:

<table>
<thead>
<tr>
<th>Injection parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Injection temperature</td>
<td>200°C</td>
</tr>
<tr>
<td>Mold temperature</td>
<td>25°C - 80°C</td>
</tr>
<tr>
<td>Injection pressure</td>
<td>45 MPa</td>
</tr>
<tr>
<td>Clamping pressure</td>
<td>40 MPa</td>
</tr>
<tr>
<td>Clamping time</td>
<td>08 s</td>
</tr>
<tr>
<td>Screw speed rotation</td>
<td>142 tours/min</td>
</tr>
<tr>
<td>Injection speed</td>
<td>20, 35, 58 mm/s</td>
</tr>
</tbody>
</table>

3. RESULTS AND DISCUSSION

3.1 Morphological analysis

Morphological observations have allowed us to assess the phenomenon of fiber orientations in the polypropylene matrix, observe porosity defects on injected composite and evaluate the influence of fiber lengths on their agglomeration and on molding skin thickness.

We observed the flowing behavior of Alfa fiber along the injected part. For short fibers whose average length is less than or equal to 2.3 mm, the distribution is homogeneous on surface. The fibers orientation is controllable to a 15% rate of reinforcement, beyond this level, the orientation tends to be random in the vicinity of the mold walls, with better orientation heart.

When the composite flow in the mold cavity, each fiber moves and orients depending on the constraints imposed by its environment: the matrix and the other fibers and the walls of the cavity. After solidification, we observe a complex distribution of orientation which varies from one location to another of the part.

Then the overall observation of a sample cut in the plate's thickness at a distance of 90 mm from the injection gate, leads us to the following interpretation.

- Skin: the fibers are randomly oriented in the plane of the plate
- Under the skin: the fibers are mainly oriented in the flow direction
- Core: the fibers are oriented perpendicular to the direction of flow.

When we exceed a critical fiber length, it becomes very difficult to control their orientation by acting on the molding parameters [20]. When we used fiber lengths greater than 4, 5 mm, We observed phenomena of deformation and coalescence and aggregation of the fibers according to the processing parameters, Figure 4.
3.2. Influence of injection speed

The injection speed is a critical parameter. It is able to varying the fibers orientation in the mold because it occurs during the dynamic phase of filling of the mold cavity. Accordingly, the fibers conformation after cooling in a specific state will be strongly governed by the applied injection speed.

We tried to highlight the variation of the heart layer in molded parts, depending on the injection speed (Table 2).

**Table 2** Evolution of the core thickness depending on injection speed

<table>
<thead>
<tr>
<th>Test N°</th>
<th>Speed (mm/s)</th>
<th>Thickness (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20</td>
<td>15,4</td>
</tr>
<tr>
<td>2</td>
<td>35</td>
<td>17,8</td>
</tr>
<tr>
<td>3</td>
<td>58</td>
<td>23,5</td>
</tr>
</tbody>
</table>
We note that the thickness of core layer increases depending on injection speed. This difference is explained at first by the variation of the solidified layer and the speed profiles shape during the materials flow in the mold (fountain effect). The fluid is highly viscous at high injection speed and the type of flow is "plug" kind, and the shear at core is almost zero, which allows the fibers to maintain the initial orientation to the mold inlet.

3.3. Mechanical characterization

Tensile tests were done on samples cut from molded plaques according to the conditions identified in Table 1. Figure 5 shows the evolution of the tensile strength of polypropylene composite reinforced with different rate of treated short Alfa fibers. We observe a change in the tensile behavior of the composite depending on rate of fiber and particularly a clear decrease of the nonlinear distortion depending on the Alfa fiber content, with improved tensile strength.

![Figure 5](image-url)  
Figure 5: Evolution of tensile behavior of polypropylene reinforced with different rates of short Alfa fibers

Table 3 summarizes the results representing the average value performed on seven specimens tested in each formulation case and highlighting the influence of fibers chemical treatment on the mechanical properties of polypropylene bio-composite reinforced with 20% short Alfa fibers.

**Table 3** Influence of Alfa fibers treatment on tensile properties of the composite

<table>
<thead>
<tr>
<th>Specimens</th>
<th>σ Break (MPa)</th>
<th>A%</th>
<th>Standard deviation A%</th>
<th>Module (MPa)</th>
<th>Standard deviation Module (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP+ 20% treated Alfa fibers</td>
<td>48.3</td>
<td>4.69</td>
<td>0.21</td>
<td>2585</td>
<td>43</td>
</tr>
<tr>
<td>PP+20% untreated Alfa fibers</td>
<td>41.5</td>
<td>5.56</td>
<td>0.26</td>
<td>2267</td>
<td>41</td>
</tr>
</tbody>
</table>
3.4. Analysis of viscoelastic behavior

The thermomechanical tests were performed with a dynamic mechanical measuring device (DMTA). This measure gives the variation of the torsional shear modulus $G'$ and the bending elasticity modulus $E'$ as a function of temperature at a given frequency and mechanical damping coefficient $\tan \delta$.

Figure 6 shows the influence of the mold temperature on the mechanical damping of composite PP/short Alfa fibers. A mold temperature of 25°C markedly improves the damping of the materials thus its impact resistance.

![Figure 6](image_url)

**Figure 6** Composites viscous modulus (70/30), PP/short Alfa fibers at $T_m = 80^{\circ}C$

Similarly, Figure 7 shows the evolution of the elasticity modulus depending on the temperature at different mold temperatures. We note that the mold temperature has a positive effect on the elastic modulus.

![Figure 7](image_url)

**Figure 7** Composite's elastic modulus of the PP/short fiber Alfa (70/30), at different mold temperature
Figure 8 shows the evolution of the tangent of the mechanical loss angle versus temperature for different rate of short Alfa fibers. The transition peak decreases when the fiber content increases. This results in a decrease of the material damping and therefore its impact resistance.

![Figure 8](image_url)

**Figure 8** Evolution of the tangent of the mechanical loss angle of the composite PP/Alfa as a function of the temperature at different rates of short fibers Alfa

The study of the evolution of the thermomechanical properties of thermoplastic composites based on organic injection parameters, classifies the relative importance of these parameters.

The mold temperature is the parameter whose variation induced the greatest changes in the mechanical properties. When the mold temperature increases, the values of stress at break and the elasticity modulus increases significantly. However, the impact resistance and the tangent of the mechanical loss angle decreases. The variation of the filling speed also influences the mechanical properties.

4. CONCLUSION

After controlling the process of extracting Alfa fibers from the plant, we have developed several formulations of bio thermoplastic composites reinforced with Alfa fibers. We then conducted an extensive study for the definition and optimization of injection molding process's industrial conditions of the composites. We determined the optimal injection process parameters of our materials and manufactured rectangular plates.

Morphological observations were used to analyze the flowing behavior of Alfa fibers along an injected part. We have highlighted the distribution phenomena and fiber's orientation based on injection molding process parameters. This partly explains the variation of mechanical properties with the loading direction on the one hand and on the other hand we have highlighted the influence of fiber's chemical treatment and mold temperature on the mechanical properties of injected parts.

This study represents a very important step towards understanding the benefits of Alfa fiber forms and qualities and opportunities to be one of fibrous plants used in the composite industry.
REFERENCES


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