FIBER MODIFICATION BY STEAM-EXPLOSION: MICROSCOPIC ANALYSIS OF CO-REFINED WOOD AND POLYPROPYLENE

Scott Renneckar¹, Audrey Zink-Sharp²* and Wolfgang G. Glasser²

SUMMARY

Wood chips were fiberized in the presence of isotactic polypropylene (iPP) by steam-explosion. A combination of microscopy methods was used to study the dispersion of iPP and wood fiber. In addition, a method for tagging a maleated polyolefin with a fluorescent label was developed to enhance the understanding of polyolefin association with cellulose fibers. Autofluorescence arising from cellulose and lignin, by excitation at 364 nm and 633 nm wavelengths, respectively, was used to reveal the redistribution of wood bio-polymer components during the steam-explosion processing. Melt viscosity was found to be a critical factor in maximizing wood and polyolefin interfacial contact for co-processed materials. Although optimum dispersion is limited by the molecular weight of iPP, co-steam-explosion processing can produce an iPP coated wood fiber bundle.

INTRODUCTION

Fiber from steam-explored wood has been used in products for almost a century (i.e. Masonite Board). Although the costs of producing the fiber are relatively low, c. $0.077/kg plus raw material cost (Avellar & Glasser 1998), the use of the fiber still appears as novel for many applications. Moreover, new opportunities have arisen for wood flour in the past 10 years with the advent of commercially available natural fiber-filled plastic composites. This material is used in applications ranging from decking to car interior paneling. Research has emphasized improved performance of the composites by addressing fiber matrix adhesion with coupling agents such as maleated polypropylene (Felix & Gatenholm 1991).

A number of studies have demonstrated that steam-explosion is practical for pretreatment of wood and natural fibers for thermoplastic composites. Fiber from steam-explored wood has higher crystalline cellulose content, hydrolyzed hemicellulose, increased surface roughness, and breakage of lignin carbohydrate complexes with reordering of lignin (Chomet & Overend 1991). Reduced amorphous carbohydrate content decreases the interaction between water vapor with fiber, while increased fiber surface roughness may serve as a nucleating agent for semicrystalline matrix materials.

1) Department of Wood Science and Forest Products, Virginia Tech, 1650 Ramble Road, Blacksburg, VA 24061, U.S.A. [E-mail: srenneck@vt.edu].
2) Department of Wood Science and Forest Products, Virginia Tech, 230 Cheatham Hall, Blacksburg, VA 24061, U.S.A.
* Corresponding author [E-mail: agzink@vt.edu].

Associate Editor: Laurence Schimleck
Takatani and co-workers compared steam-exploded fiber with wood flour for use as a filler in thermoplastic composites (Takatani et al. 2000). The authors reported that steam-exploded wood-filled thermoplastic composites had higher dimensional stability and greater fracture strength compared with the wood flour. Similar to wood flour, modification of steam-exploded wood with coupling agents improved thermoplastic composite performance. Angles et al. (1999) used steam-exploded softwood fibers that were treated with MAPP to reinforce polypropylene. Treated fibers were found effective for the improvement of mechanical properties of wood-filled polypropylene composites, while untreated steam-exploded fibers decreased the strength of the composite. In a similar manner, steam-exploded bast fibers were used as a filler for polypropylene composites (Vignon et al. 1996). The fibers increased the tensile modulus of all the composites; however, the percent elongation and strength at yield declined for the nontreated fiber composites that did not contain MAPP.

While coupling agents have been effective, reactive processing has been explored as a method to covalently link matrix and filler (cellulose-based oil palm fiber) during extrusion (Wirjosentono et al. 2004). This method follows the radical generation and polymerization during processing by means of thermal energy and additives such as peroxides. Along the same line, Brooks Associates recycled thermoplastic gas tanks and wooden pallets by co-refining the materials by steam-explosion processing (Brooks et al. 2002). Upon observation of the material with the unaided eye, the thermoplastic was no longer visibly distinguishable from the wood fiber.

A number of microscopy methods have been developed to aid in identification of additives applied to wood and wood fiber (Johnson & Kamke 1992; Grigsby et al. 2005). Contrast of additives relative to wood can be enhanced by a combination of fluorescence microscopy and additive-specific dye. If a confocal laser scanning microscope is utilized, out-of-focus fluorescence can be blocked by an aperture restricting the detection of light to the focal plane of interest. Samples can be optically sliced by adjusting the focal plane and then raster scanning the section. Moss et al. reported that the instrument could be used to determine fiber morphology, hand sheet structure, and surface roughness (Moss et al. 1993). Determining cell collapse was another application of CLSM related to fiber examination (Jang & Seth 1998).

In the following study, microscopy techniques are applied to study thermoplastic wood material produced from a new processing method of co-refining wood and polyolefins by steam-explosion. Previous examination using thermogravimetric analysis revealed that low molecular weight polyolefin is distributed upon the milligram scale with wood fiber that was co-processed by steam explosion (Renneckar et al. 2004). Sorption properties, as well as the chemistry of the materials were modified by co-steam-explosion of wood and polyolefins, demonstrating that this is a reactive technique and controlled by the amount of oxygen gas in the reactor (Renneckar 2004; Renneckar et al. 2006). The aim of the study is to determine the relationship between iPP parameters (molecular weight and initial form) and the resulting polypropylene domain size within the co-processed steam-exploded material. To assist in the investigation, a model cellulose fiber is steam-exploded with a fluorescently tagged polyolefin to identify the polyolefin on the fiber surface.
Table 1. Properties of polypropylene co-steam-exploded with wood chips.

<table>
<thead>
<tr>
<th>Description</th>
<th>Form</th>
<th>Source</th>
<th>M_n</th>
<th>M_w</th>
<th>Intrinsic viscosity</th>
<th>Melt flow index</th>
<th>Viscosity* (Pa·s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-iPP</td>
<td>coarse</td>
<td>SP²</td>
<td>–</td>
<td>–</td>
<td>2.2–2.5</td>
<td>4 g/10 min</td>
<td>3777</td>
</tr>
<tr>
<td>2-iPP</td>
<td>pellet</td>
<td>Aldrich</td>
<td>9,502</td>
<td>35,843</td>
<td>–</td>
<td>1000 g/10 min</td>
<td>23</td>
</tr>
<tr>
<td>3-iPP</td>
<td>pellet</td>
<td>Aldrich</td>
<td>5,000</td>
<td>12,000</td>
<td>–</td>
<td>–</td>
<td>0.4</td>
</tr>
<tr>
<td>4-iPP</td>
<td>pellet</td>
<td>Aldrich</td>
<td>50,000</td>
<td>190,000</td>
<td>–</td>
<td>35 g/10 min</td>
<td>2016</td>
</tr>
<tr>
<td>5-iPP</td>
<td>fiber</td>
<td>Fiber Visions</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>40 g/10 min</td>
<td>–</td>
</tr>
</tbody>
</table>

* Polypropylene viscosity values are reported for 1 Hz shear rate at 195 °C.

METHODS AND MATERIALS

**Steam-explosion**

A 1-gallon Parr reactor was converted into a steam-explosion vessel by modifying the lid with a steam inlet port and the base of the reactor with a 3/8” ball valve. *Quercus rubra* (red oak) chips were obtained from a local sawmill and isotactic polypropylenes (iPP) were obtained from a number of laboratory suppliers and one commercial source as indicated in Table 1. In addition, molecular weights and melt viscosities for the iPPs used in these experiments are also listed in Table 1. 90.9 g of red oak chips at 12% MC (that were screened by a 16 mm diameter hole and retained by a 10 mm diameter hole) and polypropylene (80 g) were placed in the reactor that was preheated to 230 °C and the reactor lid was bolted in-place. After the materials were placed in the reactor, a ball valve was opened, then steam with a temperature of 230 °C was introduced into the reaction vessel, and the auger-type stirrer turned at 100 rpm. After a residence time of five minutes, the exit ball valve was opened ejecting the material through a cyclone (steam-solids separator) into a collection container. The resulting material was washed with warm water (60 °C) in an Erlenmeyer flask and recovered by filtering in a Buchner funnel. Next, steam-exploded material was rinsed with twice the volume of water that was in the Erlenmeyer flask. The material was dried in a desiccator and stored until further analysis. This procedure was repeated for each wood and iPP combination listed in Table 1. Additionally, the procedure was used with regenerated cellulose fiber (lyocell fiber, a product of Acordis, diameter of 10 μm and length of 10 mm) and maleated polyethylene with a fluorescent label.

**Preparation of the fluorescent-labeled maleated polyethylene**

Fluorescein (max λ<sub>ex</sub> 494) and polyethylene-graft maleic anhydride (0.05 % MA by weight) were obtained from Sigma-Aldrich. Both of these materials were dried in a desiccator under vacuum over phosphorus pentoxide. Techniques for tagging the maleated polyolefin were adopted from established methods (Li et al. 1997; Tong et al. 2001). Maleated polyethylene (20.07 g) was added to a triple neck round bottom flask that contained a magnetic stir bar and was fitted with a condenser and water collector. The polyolefin was heated to 180 °C for 2 hours in a non-reactive atmosphere under nitrogen gas. Anhydrous o-xylene (325 ml) was transferred to the flask and refluxed until the maleated polyethylene was dissolved. Next, fluorescein powder (0.706 g)
Figure 1. Reaction scheme for the fluorescent labeling of maleated polyethylene. – A: maleic anhydride-g-polyethylene (0.05%), and B: fluorescein.

was added to the flask and the flask continued to reflux for 24 hours (Fig. 1). After the reported reaction time, octyl amine (0.403 g) was added to the solution to react for an additional 8 hours with any unmodified anhydride. Subsequently, the solution was cooled and precipitated by pouring it into 650 ml of acetone. The solution was filtered across Whatman filter paper recovering the precipitated polyolefin. To remove excess or unreacted fluorescein the precipitated polymer was then dissolved in hot xylene, cooled, precipitated in acetone, and recovered by filtering. The dissolution process was repeated three times.

For the control CLSM experiment, the fluorescein-tagged maleated polyethylene was dissolved in xylene and then the control lyocell fiber was dipped into the solution. Next, the fiber with adsorbed polyolefin was stained with either Congo red (max $\lambda_{ex}$ 500) in a water solution or rhodamine B (max $\lambda_{ex}$ 540) in an acidic ethanol solution as counter stains. Both counter stains worked well and used interchangeably to highlight the cellulose surface. The success of tagging the maleated polyolefin was demonstrated by fluorescence of the polyolefin.

**Digital photography and light microscopy**

A digital camera was used to record images of the steam-exploded material. Digital images did not undergo any editing, filtering, or enhancement. Steam-exploded material was placed in distilled water and the resulting slurry was transferred to glass microscope slides by pipette. A Nikon microscope fitted with a polarizer, analyzer and a CCD camera was used to record images of the material. The analyzer was manually
rotated so it was perpendicular to the polarizer for polarized optical micrograph images to help differentiate wood cells through birefringence.

**Field emission scanning electron microscopy**

Material from steam-explosion was prepared for scanning electron microscopy by two procedures. The first technique incorporated freeze-dried fiber that was placed on double-sided carbon tape attached to a sample stub. The second method used a pipette to place droplets of a dilute water solution of the material directly on a clean metal sample stub and subsequently allowed to dry.

Stubs and fibers were then coated with two nanometers (nm) of gold-palladium in a Cressington 208HR sputter coater. The stubs were transferred to a Leo 1550 field emission scanning electron microscope. Accelerating voltages between 2–5 kV were used with a working distance that varied between 3 and 8 mm. Digital images were recorded with either the in-lens or secondary electron detectors.

**Confocal laser scanning microscopy**

A Zeiss 510 confocal laser scanning microscope with a 40x oil plan-apochromat objective lens (NA 1.0) was used to record fluorescent images of the material. All images were recorded in a multi track mode where the system collected data sequentially, on separate channels, by switching between excitation lasers. The same method outlined above in the light microscopy section was used to prepare the specimens for CLSM. Each image was recorded with an average of two to eight scans to eliminate background noise with the pinhole aperture opened to 84 μm. For the co-steam exploded wood and polyolefin experiments, excitation wavelengths of 364 and 633 nm were used with band pass (385–470 nm) and long pass (650 nm) emission filters, respectively. For the steam-exploded lyocell and fluorescent-tagged maleated polyethylene, excitation wavelengths of 543 and 488 nm were used with two emission filters, long pass (560 nm) and band pass (505–550 nm), respectively. Note that colors presented in the following CLSM images are controlled by the software and do not result from the true wavelengths of light.

**Rheology**

Steady-state viscosity experiments were performed on a TA Instruments AR 1000 Rheometer using parallel plate geometry. Using a temperature control unit, the experiment was conducted at 195 °C at shear rates that ranged from 0.025 to 10.0 s⁻¹. Repeatable results were derived from using a minimum amount of material that filled the plate opening of approximately 100 μm. The material was placed on 25 mm disposable plates at room temperature and conditioned for approximately 30 minutes at 195 °C before experimentation.

**RESULTS AND DISCUSSION**

Co-processing the highest molecular weight iPP (Mₙ 50,000), or lowest melt flow index materials, with wood chips by steam-explosion resulted in a clearly visible separation between wood fiber and iPP on the macroscopic scale (Fig. 2). A majority of the iPP
was agglomerated and could be separated from the wood fiber by washing in hot water. Agglomerations of iPP contain a network of strings with an average diameter of one millimeter. Responses were the same for all low melt flow index (MFI) iPP added to the reactor that varied in form: pellet form with MFI 35 g/10 min, (Fig. 2A), fiber form with MFI 40 g/10 min (Fig. 2B), or powder form with MFI 4 g/10 min (iPP-1, not shown). These forms of iPP, fiber and pellets, have different specific surface areas (iPP fibers = 400 mm²/g and iPP pellets = 2 mm²/g, calculated) and, hence, heat transfer to the interior of the material occurs with different thermal gradients. In other words, there is a larger thermal lag for the iPP pellets than for iPP fiber, but this is not a limiting factor for the dispersion of iPP with wood fiber. Although, the steam temperature is approximately 60 °C above the melting temperature of iPP, the low melt flow index iPP does not “fluidize” in the melt state for dispersion with the wood fiber.

Using an iPP with lower molecular weight (Mₙ 9,500) causes a change in the dispersion of materials in the co-processed wood chips and iPP (Fig. 3). On the macroscopic scale there is no phase separation of the iPP from the wood fiber, in contrast to the high molecular weight iPP. While the steam-exploded wood appears in fibrous form (Fig. 3A), the co-steam-exploded wood and iPP appears as fiber bundles (Fig. 3B). This is

Figure 3. Steam-exploded wood (left image set) and co-steam-exploded wood and iPP, Mₙ 9,500 (right image set). A & B: Digital photographs. – C–F: SEM micrographs. – G & H: CLSM images excited with 633 nm red, 364 nm blue, and composite image. Red fluorescence related to lignin and blue fluorescence related to cellulose (Renneckar 2004) Image B, D & F demonstrate coating of wood fiber bundles with iPP. – Scale bars of A & B = 10 mm; of C & D = 20 μm, of E & F = 2 μm; of G & H = 5 μm.
evident in the SEM images that show individual wood fibers for steam-exploded wood (Fig. 3C) and wood fibers coalesced together in bundles for co-processed materials (Fig. 3D). The surface of the steam-exploded wood appears very heterogeneous: microfibrils, fibrillation of the fiber surface layers, coalesced material on the fiber surface, and ridges and folds along the fiber surface (Fig. 3E). In contrast, many of the surface features are veiled for the co-steam-exploded wood and iPP fiber bundles (Fig. 3F). The distinct fibril pattern is no longer apparent; instead, a continuous layer of material is present that contains some submicron-sized texture (Fig. 3F).

Autofluorescence of the wood fiber was exploited to further enhance the understanding of the steam-exploded fiber surface. For the steam-exploded wood fiber, red fluorescence from $\lambda_{ex}$ 633 nm appears in discrete clusters, while there is uniform blue fluorescence from $\lambda_{ex}$ 364 nm (Fig. 3G). The red fluorescence has submicron to micron size dimensions throughout the length of the fiber. Unlike the red fluorescence, the blue fluorescence is continuous except for the interruption by some pit structures in the cell wall. Furthermore, the discrete and continuous nature of the fluorescence is conspicuous in the composite image (Fig. 3G). On the other hand, co-steam-exploded wood and iPP (Fig. 3H) has subtle differences in fluorescence along with reduced emission signal strength. Although there are some bright clusters of red fluorescence, the submicron-sized clusters have disappeared. Furthermore, the distribution of red fluorescence across the fiber bundle is approaching that of the blue fluorescence (Fig. 3H). The blue fluorescence is not as uniform as seen in the steam-exploded wood sample. A purple hue is seen across the fiber bundle in the composite image indicating the overlap of cellulose and lignin fluorescence (Fig. 3H).

Acetone-extracted lignin from the steam-exploded wood (in powder form) demonstrated “red” fluorescence with excitation of 633 nm light, while cellulose demonstrated “blue” fluorescence with excitation of 364 nm light (Renneckar 2004). These excitation wavelengths agree with reported values for alkali-extracted lignin and cellulose, respectively (Olmsted & Gray 1997). Assigning the red fluorescence to lignin and blue fluorescence to cellulose, the widely reported reordering of wood polymers caused by steam-explosion is illustrated in Figure 3G (Donaldson et al. 1988; Michalowicz et al. 1991; Kallavus & Gravitus 1995). Cleavage of lignin carbohydrate complex and lignin coalescence has been implicated for the discrete clusters within and on the cell wall. This was further verified with cross polarized $^{13}$C solid state nuclear magnetic experiments, in which the $T_{1p}$ relaxation times for the functional groups related to lignin were found to be distinct from the relaxation times for the cellulose (Renneckar et al. 2005). In solid wood, based on spin diffusion, the relaxation times are equivalent when there is a blend of components along the nanoscale (Laborie 2002). Although differences in CLSM data are subtle (Fig. 3G, H), NMR experiments concluded an alternative arrangement of wood polymers by co-steam-explosion processing (Renneckar et al. 2005). A lower $T_{1p}$ relaxation time for lignin was found within co-processed wood and iPP relative to the control samples when no iPP was present. The lower relaxation time was similar to the amorphous cellulose component relaxation time, which suggests, through spin diffusion, that lignin domains were not distinct relative to the amorphous cellulose domains.
The lowest molecular weight iPP (Mₙ 5,000) co-processed with wood chips by steam-explosion resulted in a mixture that appeared uniform on the macroscopic scale. Using polarized light microscopy, the wood fiber was differentiated from the polypropylene component (Fig. 4A). Co-steam-exploded material with iPP (Mₙ 5,000) contains dark coalesced clusters separated amongst the birefringent wood cells (Fig. 4A). There is a wide range of cluster size documented in the standard deviation. Average diameter of the clusters along their major axis is 52 μm with a standard deviation of 28 μm (n = 48). The distribution of iPP clusters among wood fibers (Fig. 4A) is distinctly different from co-steam-exploded material with higher molecular weight iPP (Fig. 2 & 3A, B). Additionally, steam-explosion with low molecular weight polyethylene (PE) (Mₙ 1,400) demonstrates similar behavior to the low molecular weight iPP-separation
of PE into micron sized droplets (Fig. 4B). However, the SEM image is inconclusive as to whether the texture on the wood fiber surface arises from polyolefin or adsorbed cell wall fragments.

Determination of polyolefin arrangement on/with the fiber was assisted with confocal laser scanning microscopy (CLSM) by identifying low melt viscosity (< 1.0 Pa-s) fluorescently-labeled polyolefin on a cellulose fiber surface (note: wood fiber was not used because the autofluorescence of the fiber could not be filtered from the label on the polyolefin). A control experiment demonstrated the utility of the method utilizing a model fiber, a regenerated cellulose (lyocell) fiber. This was done by dipping a lyocell fiber into a xylene solution containing fluorescent-labeled maleated polyethylene and counter staining the material with rhodamine B (Fig. 5). The counter stain was used to highlight cellulose-rich regions “purple” fluorescence relative to the polyolefin rich “green” fluorescence regions. In other words, the areas where there is no overlap of fluorescence are attributed to either cellulose or polyolefin. The once smooth fiber has a number of clusters visible in the transmitted light image (Fig. 5A). Fluorescence of the lyocell fiber (cellulose-rich component) with counter stain has a purple fluorescent intensity that varies across the fiber with both bright and muted areas (Fig. 5C). This may be explained from concentrated and depleted areas of counter stain. Fluorescence arising from the labeled polyolefin has two distinct green fluorescent patterns (Fig. 5E). There are large clusters that have micron dimensions and smaller clusters that have submicron dimensions. The composite fluorescence image shows a color gradient at the fiber surface with polyolefin-rich regions and cellulose-rich regions (Fig. 5G). The image illustrates that the muted purple fluorescent regions of cellulose have a correspondingly intense fluorescence from the tagged polyolefin identifying the polyolefin in the non-overlapping fluorescent regions. Co-steam-exploded lyocell and the tagged polyolefin were analyzed with the identical methodology. From the transmitted light image, the fiber edges appear uneven (Fig. 5B). Lyocell fiber has muted purple fluorescence in certain areas of the fiber surface (Fig. 5D). The dark clusters on the fiber surface, seen in the transmitted light image, are green relating to polyolefin fluorescence (Fig. 5F). Three distinct fluorescent regions are seen in the green fluorescence image: speckled fluorescence, a bright fluorescence streak in the center of the fiber, and areas of continuous fluorescence. Shown in the composite image is a lightened pinkish-green color resulting from the continuous polyolefin fluorescence overlapping with the cellulose fluorescence (Fig. 5H). Overlap of the fluorescence indicates that there is not polyolefin spreading on the fiber surface. Speckled fluorescence appears independent of the cellulose fluorescence in the composite image. Green fluorescent speckled patches in the composite image have submicron dimensions. These domains show corresponding reduced purple intensity in the composite image. Similar to what is seen with the control fiber (Fig. 5G) the stain cannot fully access the cellulose surface in these regions and therefore the speckled regions are attributed to tagged polyolefin. The use of the lyocell and fluorescent-labeled polyolefin demonstrates that there is polyolefin in submicron areas associated with the fiber surface. This result could not be exclusively verified by scanning electron microscopy because of the heterogeneous nature of the wood fiber surface (Fig. 4E).
Figure 5. Control experiment (left image set) and co-steam-exploded lyocell and fluorescent polyolefin (right image set). – A & B: CLSM transmitted light image. – C & D: CLSM fluorescence image arising from counter stain on lyocell surface. – E & F: CLSM fluorescence image arising from polyolefin. – G & H: Composite image of fluorescence. — Scale bars = 10 μm.
Figure 6. Polypropylene melt viscosity of varying molecular weights as a function of shear rate tested at 195°C.

The viscosity of polypropylene (in the melt state) was measured as a function of shear rate (Fig. 6). The three polypropylene samples used in the experiments above showed independent responses that varied in order of magnitude. The highest molecular weight polypropylene (Mn 50,000) had the most resistance to flow (2 x 10³ Pa-s for low shear rates). Additionally, the highest molecular weight iPP demonstrated decreased viscosity with increased shear rate (i.e. shear thinning). The Mn 9,500 molecular weight iPP had viscosities intermediate (1−3 x 10³ Pa-s) between the lowest and highest molecular weight iPP. This iPP also demonstrated a shear thinning response. In contrast, a shear thinning response was absent for the lowest molecular weight iPP (Mn 5,000) that had the lowest viscosities (0.2−0.4 Pa-s at 195°C). These values are comparable to the Brookfield Thermosel value supplied by the manufacture of 0.60 Pa-s at 190°C. The lowest MW iPP is lower than iPP’s critical entanglement molecular weight of 7,000 (Wool 1993), which means the majority of polymer chains in the melt are not affected by diffusion and entanglements that would increase the melt viscosity (and also cause the decrease in viscosity at higher shear rates for the shear thinning materials). It is evident that the magnitude of the melt viscosity sharply depends upon the molecular weight of iPP, which influences the dispersion of the iPP during co-processing.

Differences in the distributions of iPP among the steam-exploded wood fiber are summarized in Figure 7 as a function of iPP molecular weight. iPP with a Mn of 9,500 that is just above the critical chain entanglement molecular weight (MW 7,000) forms a coating on fiber bundles giving rise to the highest level of interfacial contact. iPP with a molecular weight below the critical entanglement molecular weight forms ag-
glomerations the size of tens of microns in diameter, with some residual iPP on the fiber surface. As the molecular weight increases above the critical chain entanglement molecular weight, the melt viscosity scales to a power of 3.4 of the degree of polymerization (DP). Cohesiveness of the iPP, reflected in the melt viscosity, quickly increases as the DP rises. As the molecular weight of the iPP is increased to a $M_n$ of 50,000, the cohesiveness of the melt is too great to achieve adequate dispersion. The result is that the high melt viscosity iPP cannot be dispersed easily with the fiber bundles and thus separates into macroscopic domains limiting interfacial contact. From these observations there appears to be a window of molecular weights that occurs right above the critical chain entanglement molecular weight that allows for a high interfacial area between wood fiber and iPP by forming film-like coating on fiber bundles. However, iPP of low molecular weights lack mechanical performance, but they have been used as dispersing agents for wood fillers (Bourland 1987). In this case, an iPP coated wood fiber bundle, produced by co-steam-explosion processing, may be an appropriate material for extrusion or injection molding processes.

**CONCLUSIONS**

Molecular weight of the polyolefin plays an important role in the dispersion and coating of the wood fiber by the polyolefin during co-refining by steam-explosion processing. Although the range of molecular weights to achieve fiber bundle coating is not de-
fined in this study, it does give three examples that show three unique outcomes. Low molecular weight iPP (M<sub>n</sub> 5,000) does not offer significant coating of the wood fiber and forms distinct micron-sized clusters. iPP with a M<sub>n</sub> of 9,500 coats fiber bundles of steam-exploded wood. Finally, iPP with M<sub>n</sub> of 50,000 does not coat wood fibers and separates from the wood material in macroscopic agglomerations. Additionally, polyolefin form was shown to have a negligible effect on influencing the dispersion of the higher molecular weight iPP.

CLSM was used to confirm aggregation of lignin on the steam-exploded wood’s cell surface by use of autofluorescence that differentiated lignin and cellulose. Co-steam-explosion of wood and polypropylene (Mn of 9,500) material had a change in autofluorescence patterns with reduced emission signal intensity. The combination of CLSM and SEM microscopy revealed that polypropylene can veil the wood cell surface and that outcome can be controlled by the molecular weight.

ACKNOWLEDGEMENTS

This project was supported by the National Research Initiative of the USDA Cooperative State Research, Education and Extension Service, grant number 2001-35504-11138. Additionally, the Eastman Chemical Company provided financial support with a graduate fellowship. We would like to thank Kristi DeCourney of the Fralin Biotechnology Center and Stephen McCartney of the Macromolecules and Interfaces Institute for their useful discussions.

REFERENCES


Renneckar, S. 2004. Modification of wood fiber with thermoplastics by reactive steam-explosion processing. Dissertation, Virginia Polytechnic Institute and State University, Blacksburg, VA.


IAWA List of Microscopic Features for Hardwood Identification
by an IAWA Committee, edited by E.A. Wheeler, P. Baas and P.E. Gasson

Published for the International Association of Wood Anatomists
First printing 1989, Third printing 1999. 112 pp., 190 half-tone illustrations
Paperback — Price: USD or EUR 25.00*

This IAWA publication sets the standard for wood anatomical terminology and for the use of microscopic characters in wood identification. Altogether 163 wood anatomical descriptors are defined, annotated and illustrated with light micrographs. In addition, 57 non-anatomical descriptors are included and discussed. This IAWA List offers guidance to everyone who wants to interpret descriptive information from the vast wood anatomical literature or who needs to include descriptive wood anatomical information in original publications. Thanks to the concise definitions and high-quality illustrations, the IAWA Hardwood List is also very suitable for teaching purposes.

IAWA List of Microscopic Features for Softwood Identification

Published for the International Association of Wood Anatomists
2004. 70 pp., 76 half-tone illustrations [Reprinted from IAWA Journal 25: 1–70]
Paperback — Price: USD 30.00 or EUR 25.00*

This IAWA publication enumerates, defines, illustrates and annotates 124 characters that are relevant for the microscopic description and identification of the secondary xylem of trees and shrubs belonging to the Pinopsida (conifers) and Ginkgoopsida. It is a long-awaited companion of the highly successful IAWA List of Microscopic Features for Hardwood Identification, published in 1989, and now a much-used manual for teaching hardwood identification all over the world. The beautiful micrographs by Dietger Grosser (Munich) illustrate each feature and make the IAWA Softwood List a user-friendly introduction to the recognition of all subtle microscopic softwood features. The numerical coding adopted makes the list also suitable to model databases and interactive, computer-assisted identification keys on. Above all this List will be a useful tool for training students in the arts of softwood identification and description.

To be ordered from: IAWA Office
 c/o Nationaal Herbarium Nederland / Leiden branch
 PO Box 9514 – 2300 RA Leiden – The Netherlands
 Fax: +31 71 5273511 – E-mail: eevn@euronet.nl

*) Excluding handling and postage