RESIN DISTRIBUTION IN MEDIUM DENSITY FIBERBOARD. QUANTIFICATION OF UF RESIN DISTRIBUTION ON BLOWLINE- AND DRY-BLENDED MDF FIBER AND PANELS

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ABSTRACT

A novel technique has been developed for visualizing urea formaldehyde (UF) resin distribution on fibers and within MDF panels. A fluorescent label was chemically bound to the resin, and digital images of resinated fiber, generated via confocal laser scanning microscopy (CLSM), were analyzed. Results indicate that this technique can be used to quantify UF resin coverage and distribution as well as provide information on resin film thickness on MDF fiber before pressing and in panels. The technique can distinguish between different methods of resination and was employed to determine that these processes can result in different surface coverages of UF resin on MDF fiber. Resin injected at the end of the blowline gave significantly less resin coverage of fiber than that which was injected at the start of the blowline. UF resin droplets were also relatively thicker and less dispersed when injected at the end of the blowline. Visualization of UF resin also illustrated resin distribution changes upon pressing of fiber particularly in the presence of wax. This result has important implications for future studies targeting optimization of resin deposition, since the droplet size distribution, as applied to the fiber, may not correspond to the droplet size distribution of resin in the panel.

Keywords: Medium density fiberboard, urea formaldehyde resin, confocal microscopy, resin visualization, resin distribution.

INTRODUCTION

Product performance in composite panels (particleboard, oriented strandboard (OSB), medium density fiberboard (MDF)), has been attributed to the uniformity of resin deposition throughout the product. ‘‘It is generally believed that a uniform distribution of small resin spots will produce particleboard with the best properties for a given resin content’’ (Kamke et al. 1996). Currently the results of

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the destructive internal bond strength test are, among other factors, taken to be an indication of resin distribution within a board (Younquist et al. 1987). While some work has been carried out on resin distribution in phenol-formaldehyde (PF) bonded particle- and flakeboard (Kamke et al. 1996), little is known about urea formaldehyde (UF) resin distribution in MDF. Development of a method to measure resin distribution on fibers and within MDF panels will help towards a better understanding of the factors involved in fiber-fiber bond development, and consequently panel properties. Ultimately, the aim should be to improve (or maintain) product performance while minimizing resin consumption.

Determination of UF resin distribution in wood panels is difficult due to the opaque nature of the resin when cured. This makes it very difficult to visualize using light microscopy techniques (Donaldson and Lomax 1989). Attempts at using scanning electron microscopy (SEM) to observe UF resin in MDF have been made but were not overly successful (Butterfield et al. 1992). Similarly, XPS has been tried (Grigsby et al. 2003) and although resin coverage could be quantified, imaging UF resin on fiber was unsuccessful (Grigsby and Thumm 2003). Microscopic techniques that make the resin distinguishable from the wood furnish are therefore required (Kamke et al. 2000a). Inclusion of dye (Albritton et al. 1978) or a fluorescent marker combined with fluorescence microscopy has been used to visualize UF resin in various panel products (Kamke et al. 2000a). Related here is the use of SEM coupled with a cathodoluminescent dye to visualize resin (Thumm et al. 2001) or energy dispersive analysis of X-rays using UF resin doped with water-soluble copper(II) salts (Feng and Hutter 2001). Use of such an approach, however, has limitations particularly if the dye can leach from the resin, which is a high risk given that resination is usually via the blowline (Feng and Hutter 2001) and resin can move within the fiber on pressing (Thumm and Grigsby 2003). Another approach is to stain the wood after it has been resinated. Kamke et al. (1996) have used this last approach, followed by digital imaging to quantify the results, to determine wax distribution in flakeboard. More recently, this technique has been attempted for distinguishing UF resin on MDF fiber (Kamke et al. 2000b). However, this approach was limited to unpressed fiber and cannot be used to distinguish resin in panels.

Phenolic resin distribution in hardboard has been investigated using fluorescent microscopic techniques (Murmanis et al. 1986) in wet- and dry-formed panels at high and medium densities. The results showed that for dry-formed panels, an uneven resin distribution was achieved compared to the wet-formed panels. Younquist et al. (1987) observed that altering resin application methods (solids content, application rate, and amount of blending) caused changes to the resin distribution in hardboard. They also showed that uniform distribution of resin throughout the hardboard produced boards with the highest IB strengths.

The objective of this study was to establish if a fluorescently labeled UF resin, in conjunction with a confocal laser scanning microscope (CLSM), could be used to measure resin distribution and fiber coverage differences. It was envisaged that this technique could be used to distinguish any apparent differences on either dry-blended and blowline resinated fiber, and quantify any differences arising from changes in resinating conditions such as: resin content, resin solids, wax addition, and the point of resin injection into the blowline.

MATERIALS AND METHODS

Materials

A standard E1 type urea-formaldehyde resin was synthesized containing a fluorescent label which was covalently bound to the developing UF polymer, based on a similar approach for fluorescently labeling wax (Ede et al. 1998). This Rhodamine label was incorporated into the resin at a loading of 0.19% (w/w oven-dry solids). For blowline resination, the labeled resin was diluted with commercial UF resin

(UF2043) and water to give a solids content of 57% and a concentration of the fluorescent label of 0.05% (based on resin solids). For dry-blending, the base labeled resin was similarly diluted with commercial UF resin (U753) to give a fluorescent label loading of 0.05%. UF2043 is a commercial MDF type resin (E2) supplied by Bladgen Chemicals Ltd. UK. U753, also a commercial UF MDF resin, was obtained from Orica Adhesives and Resins Ltd. NZ.

For the refiner trial, wood chips, predominantly spruce (*Picea* spp.), were supplied by the Shotton Paper Company Ltd., UK. The average moisture content was approximately 130% (oven-dry basis). A standard wax emulsion (E538) was supplied by the Mobil Oil Company, UK.

Refining and resin application

A pilot plant consisting of a 60 L digester, an Andritz Sprout Bauer 12-in. single disc continuous pressurized refiner, and a blowline connected to a continuous flash drier was used to generate blowline resinated fiber. Two resin injection positions were used in this trial, one at the beginning of the blowline, with the other at the end, immediately prior to the continuous flash drier. The wax emulsion was added into the refiner. Fiber was collected from the end of the drier over a period of time. The moisture content was determined and the dry throughput of fiber calculated. Throughputs of the resin and wax pumps were adjusted according to the calculated dry throughput of fiber, which was approximately 40 kg/h (dry weight basis).

A number of different resinating variables were investigated during the trial and are summarized in Table 1. For each condition, a single panel measuring 1000 × 1000 × 12 mm was manufactured. A section of panel was cut from the center of each board, and along with a corresponding sample of oven-dried, resinated fiber, was used for analysis.

Dry-blending resin application

MDF panels containing labeled resin were prepared using standard laboratory dry-blending techniques (McLaughlan 1996). MDF fiber was generated from radiata pine chips. Several panels (9 × 260 × 290 mm) were manufactured with a target resin loading of 8%. Wax emulsion was added to the resin prior to blending.

TABLE 1. *Resinating conditions.*

Condition	Parameters used in trials
Injection position	9% resin[†] injected at the start or at the end of the blowline.
Resin loading	9 or 14% resin injected at the beginning of the blowline.
Wax	9% resin injected at the beginning of the blowline with or without wax (wax addition 1.2%, injected into the refiner).
Solids content	14% resin injected at the beginning of the blowline at solids contents of 57 or 50%.

[†] Unless stated, the resin was applied at a solids content of 57%. Resin loadings are target loadings expressed as resin solids on oven dry fiber.

Confocal laser scanning microscopy (CLSM)

Methodology for quantifying resin coverage and distribution on MDF fiber has been developed in conjunction with a closely related technique for visualizing and quantifying wax distribution in MDF panels (Ede et al. 1998). For each condition, 2–3 cubes (10 × 10 mm) were cut from panels, from which microtomed sections (ca. 40 μm) were obtained from just below the surface and from the core of the panel. Samples of either fiber or panel were mounted in a glycerol/water mixture (70:30) and analyzed using a Leica TCS/NT confocal microscope. Filters were used to separate the natural fluorescence of the fiber and resin label components into green light and red light, respectively. Images were obtained using a 20 × 0.5 dry lens. The images were acquired at a size of 512 × 512 pixels and a total field of view of 500 × 500 μm, yielding a resolution of ~1 pixel/μm. Scans in the z-direction were recorded at 5-μm intervals to a depth of ca. 40 μm (panels) or ca. 100 μm (fibers). The images from the different depths were then combined to produce a 2D projection of a 3D

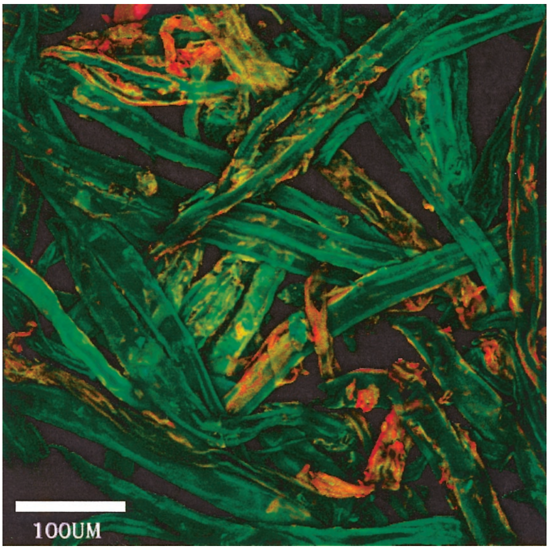

FIG. 1. CLSM image showing fibers (green) and resin (red) present within a section of MDF panel. Size 500 × 500 μm.

image. For each condition, typically 20 images were acquired using CLSM.

Image analysis methods: Quantification of resin distribution

The images obtained from CLSM were analyzed using the commercially available image analysis program V++ (Digital Optics). A custom written macro calculated the total area of fibers (green) covered by resin (red) in the images as well as determining the size of resin droplets (Fig. 1). Each image was analyzed using two different threshold settings to determine resin coverage and distribution. Distribution was determined by a high threshold setting, which only revealed resin droplets of a certain thickness. This was necessary because it was found that resin features often were connected through thin resin "bridges." If images had been analyzed with a low threshold, potentially all connected resin features could be interpreted as a single resin droplet. For the coverage analysis, a low threshold was used showing any resin present.

Image analysis determined the percent resin coverage by expressing the ratio of the area covered by resin to the total area of the image covered by fiber. The boundary of resin droplets at higher threshold were defined by the image analysis program, and then the area of these features was calculated and expressed as both a droplet size frequency and area-weighted distribution.

RESULTS AND DISCUSSION

Analysis of labeled resin

Covalently binding the fluorescent label, a sulfonamide derivative of Rhodamine, into UF resin polymer backbone during resin synthesis enabled certainty that the fluorescent label remained with the resin during both resin application and panel forming. Visual and HPLC analysis of uncured labeled UF resin precipitated into hot water or extracted from ground-up neat, cured resin confirmed no significant transfer of the highly colored Rhodamine-based label from the UF resin to either alcohol or aqueous solution. This indicates that the label is fully incorporated into the resin and, even after extended heating, does not readily hydrolyze from the resin. The approach of covalently binding the fluorescent label to the UF resin ensured that the label would remain with the resin, even in the extremes of the blowline and during pressing. Conversely, in the case of simply adding a dye to UF resin, there is potential for leaching from the resin throughout the MDF process (Feng and Hutter 2001).

Confocal laser scanning microscopy

For each resinating trial condition examined, confocal laser scanning microscopy (CLSM) revealed that every image of unpressed fiber or MDF had some resin present on the fiber. While data obtained from digital images of resinated fiber (Fig. 1) can be analyzed in a number of different ways (Kamke et al. 2000a), two methods specific to CLSM were developed during this study. Processing of the images using two differing threshold settings during image analysis enabled a distinction to be made between quantifying how

TABLE 2. *Resin coverage for fibers and panels.*

Condition	Measured resin content (%)	Fibers		Panels	
		Average (%)	Standard deviation (%)	Average (%)	Standard deviation (%)
9%[†] Start[‡]	9.7	56	14	69	13
9% End	8.3	62	14	42	8
9% with Wax	7.5	58	12	94	8
14% at 50% Solids	10.2	56	17	79	11
14% at 57% Solids	7.9	78	9	82	8
8% Dry-Blended	8.1	18	4	47	6

[†] Resin loading.
[‡] Injection point in blowline.

much of the fiber is covered by resin (coverage) and how the resin is distributed on the fiber as discrete droplets by measuring the sizes of individual resin droplets observed within each image (resin distribution).

Resin coverage

Average resin coverage values found for both unpressed fiber and panels for each trial condition are shown in Table 2. For unpressed fiber, the coverage ranges between 56 and 78%, while in panels values for coverage are between 42% and 94%. An average coverage value of 56% for unpressed fiber (9% resin at start of the blowline) means 56% of the fiber area within the images is covered in resin. Higher standard deviations of the unpressed fiber data arise because the images of panels contain ca. 90% fibers whereas the concentration of fibers in fiber-only images was ca. 30% (5–8 fibers). Additionally, these average coverage values may appear to be comparatively higher than observed by other methods (Kamke et al. 1996, 2000a) and are likely to be specific to this CLSM technique. The compression of a 3D image stacked to a 2D image led to more resin features being included and proportionately less fiber area due to fiber-fiber overlaps within the image (Fig. 1).

Results from panel data from the blowline trial indicate that coverage values increased with resin loading, and that greater coverage is obtained by injecting resin at the start of the blowline (69%) than at the end (42%, Table 2). Furthermore, the addition of emulsion wax led to a strong increase in resin coverage (94%) compared to panels without wax. Presumably the presence of wax allows greater movement of resin on the fiber surface during pressing than observed for unwaxed samples. Comparison of fiber and panel coverage data generally shows that resin coverage increases upon pressing of the fiber mat (Table 2).

In the case of dry-blended fiber, an average resin coverage of 47% is observed at 8% resin loading (Table 2). Generally, the coverage for this dry-blended panel, which contains wax mixed in with the resin, is relatively lower than the coverage observed for blowline addition, though comparable to the panel formed by resin injection at the end of the blowline (9% resin loading). It is also worth noting, analysis of coverage of high and low density MDF sections reveal, as for blowline resinated panels, no difference in resin coverage between the panel surface and core regions.

Resin droplet size distribution

Analyzing images with a higher threshold than that used for coverage analysis enabled discrimination of single resin droplets. The range of resin droplet sizes determined by image analysis for each trial condition is expressed as a frequency distribution with size classes ranging from 40 to over 3000 μm^2 in area (Fig. 2). Fiber width tended to be ca. 50 μm; therefore a fiber section with similar length as its width in an image would give an area in the range of the very large resin droplets. The resin droplet size distributions are

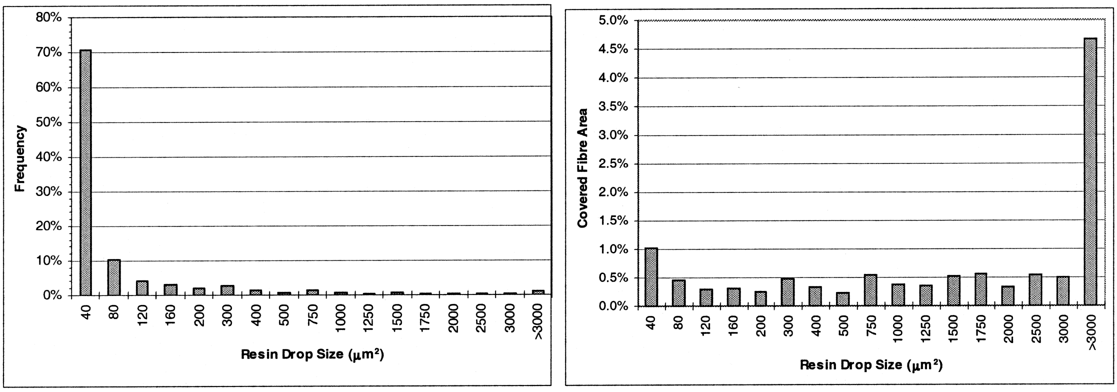

FIG. 2. Comparison of resin distribution displayed as (a) Frequency distribution (left) and (b) Area distribution (right) of resin injected at the start of the blowline.

presented in a way that shows how much coverage is contributed to the total coverage by each droplet size class (Fig. 2b). This enables some distinction in resin distributions between conditions which is not readily apparent in frequency distributions (Fig. 2a). For example, analysis of the resin distribution found on blowline-blended fiber shows high proportions of smaller resin droplets with a tendency to fewer droplets as the size of the droplets increases (Fig. 2a). However, if expressed as average % area covered, then it is apparent that these fewer, larger resin droplets contribute significantly to resin coverage of fiber (Fig. 2b). It is worth noting that the droplet size classes in this study have not been graphed linearly between 40 and >3000 μm^2, but vary in μm^2 bin size step. This was necessary due to the inherent variable contributions of resin droplet sizes. Additionally, the accumulated coverage (% covered area of fiber) of all the droplet size classes for a given condition (Fig. 2) will be considerably less than the coverage value given in Table 2 due to the differing thresholding (higher) level used to determine resin distribution than coverage.

Figure 3 shows the resin droplet size distributions as a result of injecting resin at either the start or the end of the blowline for both fibers and panels. Generally, the results for this blowline resinated fiber show that the majority of the resin coverage is caused by resin spots greater than 3000 μm^2 in area (Fig. 3). The number of objects in this category (>3000 μm^2) is, however, relatively small and the variation in their size high. When comparing the fiber distribution with the panel distribution in Fig. 3, it can be observed that overall, the bars are smaller for the fiber distributions. This means that unpressed fiber has less coverage than the panel that is made from this fiber, consistent with the observation of higher resin coverage of fiber upon densification (Table 2). Figure 3 also shows that for droplet sizes below 3000 μm^2, the difference in coverage caused by the point of resin injection is relatively small for both fibers and panels. For the very large resin droplets >3000 μm^2, analysis shows that resin injected at the start of the blowline results in more of these very large resin features, covering a greater area than resin injected at the end of the blowline. This effect is also more pronounced in panels than for unpressed fiber.

Shown in Fig. 4 are differences in resin droplet size distributions between waxed and unwaxed fiber and panels. As observed above, with the location of blowline resin injection (Fig. 3), the contributions to the overall coverage of the smaller resin droplet size classes are relatively similar between waxed and unwaxed fibers and panels. However, for resin features greater than 3000 μm^2, an increase can be found for both waxed unpressed fiber

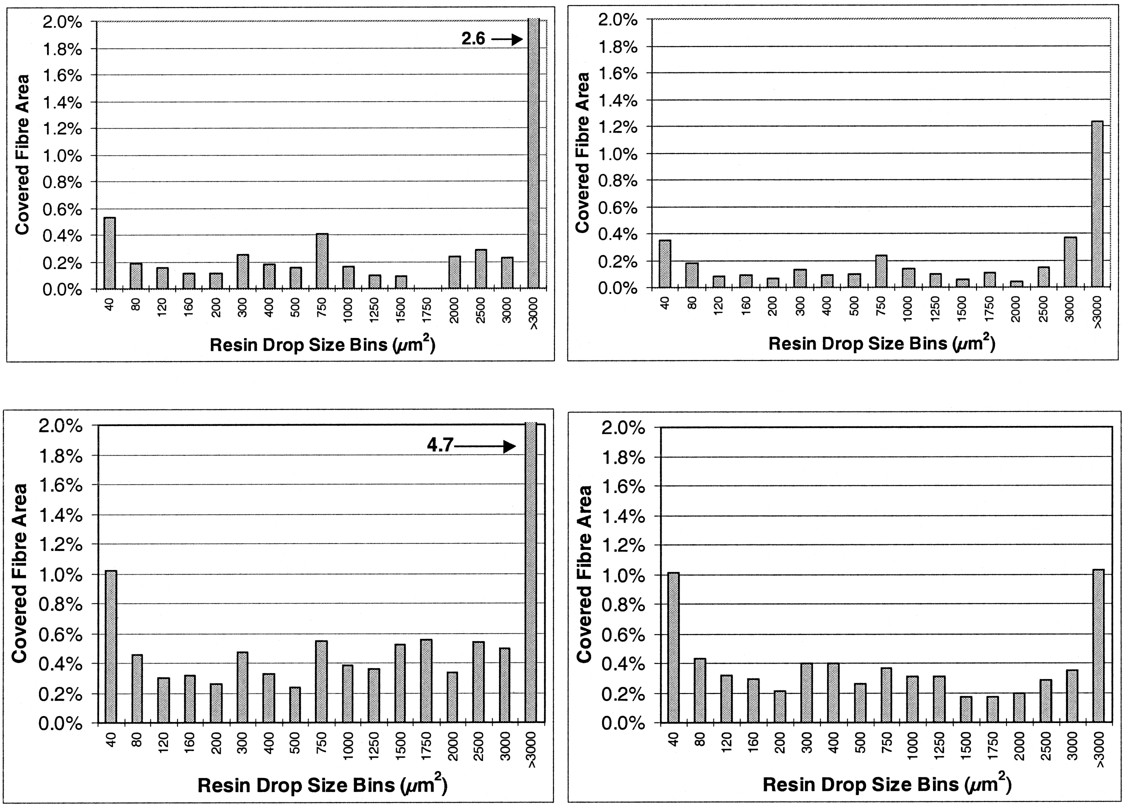

FIG. 3. Difference in area coverage distribution of resin injected at the start (left) and end of the blowline (right) for unpressed fiber (top) and panel (bottom).

and panels. This effect is particularly noticeable in the panel where the presence of wax contributed to a large increase in resin features >3000 μm^2. The differences in resin distributions between waxed and unwaxed fiber observed on pressing are also reflected in the average resin droplet size of waxed and unwaxed fibers and panels. Although the average droplet sizes for unwaxed and waxed fibers are 160 and 121 μm^2, respectively, in the unwaxed panels, the average droplet size is 163 μm^2 whereas this value is higher in the waxed panels (227 μm^2).

For dry-blended fiber, the resin distribution on unpressed fiber and panels differs greatly compared to that observed for blowline resination (Fig. 5). On unpressed fiber, there is a tendency for higher contributions of smaller droplets to the fiber coverage and likely reflects the type of resin application used. Resin is sprayed onto dry fiber with sufficient air pressure to avoid or minimize resin spotting in panels. On pressing, the resin distributions are found to have comparatively higher proportions of larger resin droplets (1000–>3000 μm^2). Given the role wax plays in resin coverage and distributions above, it is possible that the inclusion of emulsified wax with the resin has promoted the increase of these larger resin droplets in the dry-blended panel.

Resin droplet "thickness"

In addition to having gained information from the CLSM images regarding resin coverage of fiber and resin droplet size distribution, image processing is also able to provide information on the relative thickness of resin

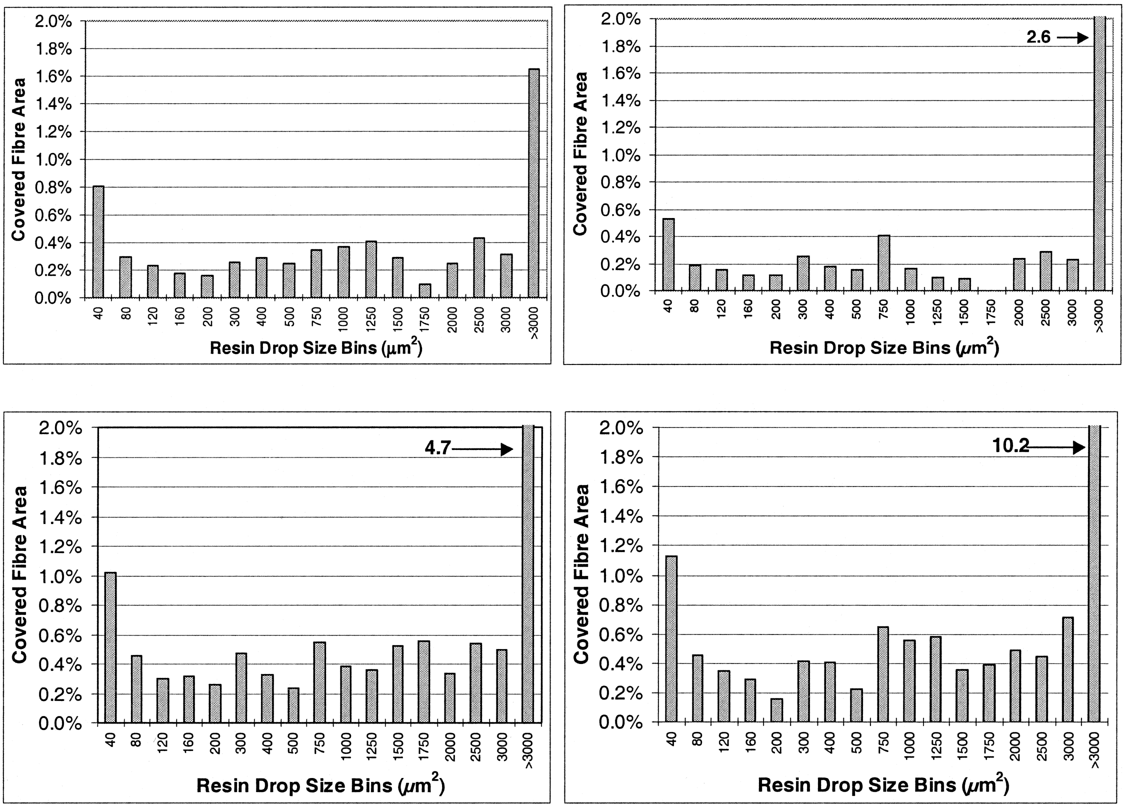

FIG. 4. Difference in area coverage distribution of resin between unwaxed (left) and waxed (right) conditions for unpressed fiber (top) and panels (bottom).

features by analysis of the intensity of fluorescence of the resin label. For blowline resinated panels, when resin coverage is compared with the resin droplet sizes of a given condition, higher coverage is usually associated with a higher proportion of large resin features. This is evident for resination at the start of the blowline, and also wax addition

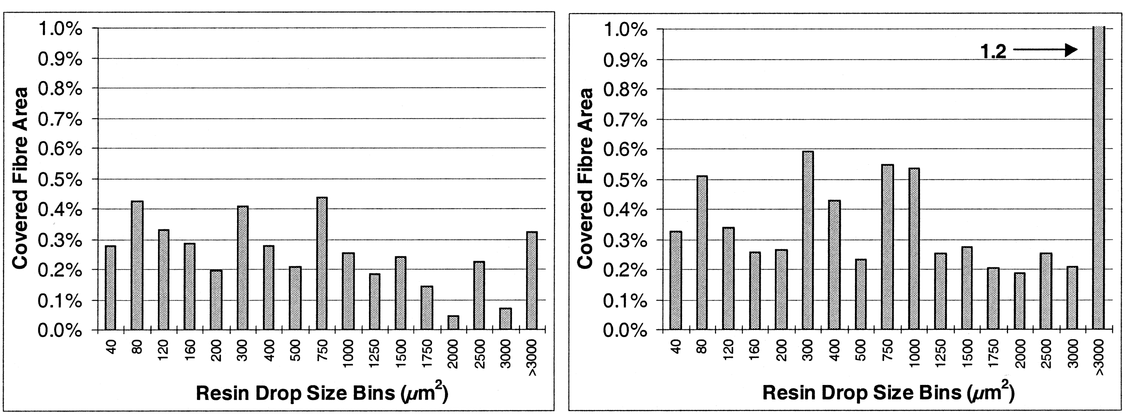

FIG. 5. Difference in area coverage distribution of dry-blended fiber (left) and panels (right).

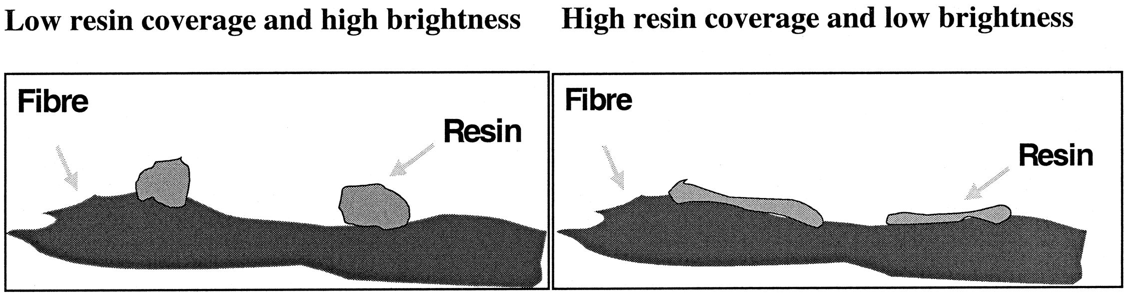

FIG. 6. Illustration of the relationship of resin brightness (fluorescent intensity) and coverage.

(Figs. 3 and 4, and Table 2). As the total amount of resin is constant, this observation suggests that treatments that show high coverage should also have a low resin layer thickness where the resin is likely to be spread out more thinly. It would be expected this thin resin thickness should also be associated with lower fluorescent intensity (Fig. 6). Shown in Fig. 6 are diagrams of resin droplet features having equal volume. Given equal amounts of resin, a thicker resin droplet would appear more intense with a lower coverage area (more fluorescent label giving a stronger fluorescence owing to the thickness of the droplet), while droplets that are thinner have a lower intensity and show a higher area coverage.

Evaluation of resin fluorescence intensity (Table 3) shows that average resin droplet brightness for resin injected at the end of the blowline is greater than resin injected at the start of the blowline. It was also found that resin coverage is lower for resin injected at the end of the blowline, implying that the average resin film thickness is greater for resin injected at the end of the blowline i.e., fewer, large thin droplets of resin are found in the panels when the resin is added at the end of the blowline. In the presence of wax, images of panels have lower brightness, indicating a lower resin film thickness, consistent with more, larger thin resin droplets than panels without wax.

TABLE 3. *Average resin coverage and resin droplet brightness in panels.*

Condition	Coverage	Average resin droplet brightness	Brightness normalized to 'Start' condition brightness (=1.00)
9% Start	69	69	1.00
9% End	42	79	1.14
9% with Wax	94	63	0.91
14% at 50% Solids	79	80	1.16
14% at 57% Solids	82	76	1.10

Panel properties

The long-term goal of this resin visualization project is to be able to relate the distribution of UF resin in a panel to the performance of the panel. From this initial study, it is obvious that to obtain such a relationship, a more comprehensive investigation will be required, with production of a greater number of panels. Due to a fixed resin quantity, there was insufficient time available to stabilize the plant to allow generation of panels with consistent properties. Accordingly, the panel property data (Table 4) should be treated with caution as they are based on only one panel (1 m by

TABLE 4. *Summary of average panel properties.*

Condition	Measured resin content (%)	Density (kg/m^3)	24 hour thickness swell (%)	Internal bond strength (MPa)
9% Start	9.7	794	26.3	0.96
9% End	8.3	729	51.9	0.47
9% with Wax	7.5	756	12.5	0.87
14% at 50% Solids	10.2	746	36.8	0.38
14% at 57% Solids	7.9	774	40.4	0.44

NB. 10 samples were tested per resinating condition. Samples were tested according to EN317 and EN319.

1 m) per condition. Differences in the resin loadings also make it difficult to compare the properties of the panels.

Injection of resin at the end of the blowline led to a panel with low internal bond strength (IB) and high thickness swell compared to the panel manufactured from resin injected at the start of the blowline. However, it should be noted that this panel was determined to have a lower resin content and lower density compared to resin injected at the start of the blowline. Those panels made with a target resin loading of 14% have lower IB and higher thickness swell than would be expected. The reason for this is not known but may reflect that the panels were manufactured a few weeks after the original trial, and it is possible the resin properties may have deteriorated in this time and as a consequence, resin distribution data for these panels has not been considered.

Although panel performance could not be readily correlated, through visualizing UF resin, we have established that variation in processing conditions leads to measurable distinctions in resin distribution and coverage of fiber. Resin injected at the start of the blowline gives significantly more resin coverage of fiber than when injected at the end of the blowline, whereas injecting resin at the end of the blowline led to resin droplets that are thicker and less dispersed than is the case for resin injected at the start of the blowline. It can be hypothesized that the dynamics of the blowline with high turbulence and fiber-fiber contacts contribute to higher proportions of larger resin droplets through greater smearing of resin injected at the start of the blowline. High temperatures and moisture in the blowline, and the influence of adhesive viscosity will also contribute to the degree of resin smearing. For resin entering the end of the blowline, the potential of these fiber contacts and smearing are far less before drying. The panel properties for these panels would imply that the resulting resin distribution and greater coverage when injecting resin at the start of the blowline is associated with a higher panel IB value.

Generally, on pressing fiber, the resin coverage and distributions are observed to change. In particular, with wax addition, the average resin droplet size is smaller on the fibers than in panels, implying that the resin is being redistributed into larger resin objects during pressing. Presumably during pressing, the resin is more mobile on the fiber and able to spread, contributing to increases in fiber resin coverage and higher resin droplet sizes. Furthermore, the presence of wax may aid resin spread and the agglomeration of smaller droplets into larger resin objects on pressing. This result has important implications for future studies involving optimization of resin addition, since the droplet size distribution as applied to the fiber may not correspond to the droplet size distribution of resin in the panel.

CONCLUSIONS

The results clearly show that coupling CLSM with the labeled resin technique enabled the visualization and quantification of UF resin coverage and distribution on both blowline- and dry-blended MDF fiber and panels. It was found that resin distributions changed upon pressing of resinated fiber, implying that resin was being redistributed during pressing. The technique was also able to distinguish between different points of resin addition into the blowline and determined that this can cause different resin surface coverage of fiber. Resin injected at the start of the blowline or wax addition gave significantly more resin coverage, with the greatest differences in these treatments apparent in the distribution of large resin features with coverage areas over 3000 μm^2. Having established this resin visualization technique, relationships between resin distribution and panel performance can now be evaluated and employed to achieve optimization of UF resin usage in panel products.

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