FLUORESCENCE MICROSCOPY OF HARDBOARDS[1]

Lidija Murmanis, Gary C. Myers, and *John A. Youngquist*

Research Forest Products Technologist, Research Forest Products Technologist
and Supervisory Research General Engineer
Forest Products Laboratory,[2] Forest Service, U.S. Department of Agriculture
Madison, WI 53705

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ABSTRACT

We developed a microscopic technique and used it to explore the internal structure and resin distribution in hardboards. The technique will enable us better to understand the behavior of hardboards in use. Glycol methacrylate (JB-4 embedding medium) proved to be satisfactory for preparing 10- to 15-μm sections of hardboards with a steel knife on a sliding microtome. This thickness of sample, when viewed in transmitted near-ultraviolet light, allowed a clear visualization of hardboard internal structure and resin distribution through the board thickness. We examined wet-formed and dry-formed hardboard samples. Wet-formed high-density and medium-density boards usually showed fibers consolidated into a compact structure and a uniform resin distribution. Dry-formed high-density boards had a compact structure and medium-density boards a less compact structure; both characteristically showed uneven resin distribution.

Keywords: Hardboards, wet-formed, dry-formed, embedding, fluorescence microscopy, resin distribution.

INTRODUCTION

Resin distribution, surface wetting, and resin penetration into wood products play an important role in determining the quality of bonded wood products such as industrial plywood, hardboards, and flakeboards. The wetting and permeability of resin for plywood have been studied in an effort to predict bond performance. However, microscopic techniques have not been developed for use in such studies with flakeboards or hardboards. This study was initiated to develop a technique and use it to examine microscopically the internal structure and resin distribution in hardboards.

There is a considerable amount of information on using embedding media, soluble in water or organic solvents, for semithin sectioning of animal tissues for microscopic study (e.g., Bennett et al. 1976; Semba 1979). For fluorescence microscopy of flakeboards, Lehmann (1968) embedded $\frac{1}{16}$-inch (230-μm) cross sections in a solution of xylene and Hercules Lewisol 7 resin. He removed the xylene by heating the solution, and then ground the cross sections with abrasive papers to a 75- to 125-μm thickness. He then viewed the sections in transmitted ultraviolet light. These thick sections and the low magnifications used were excellent for studying resin distribution parameters, but were not adequate for studying board structural features or wood–resin interactions.

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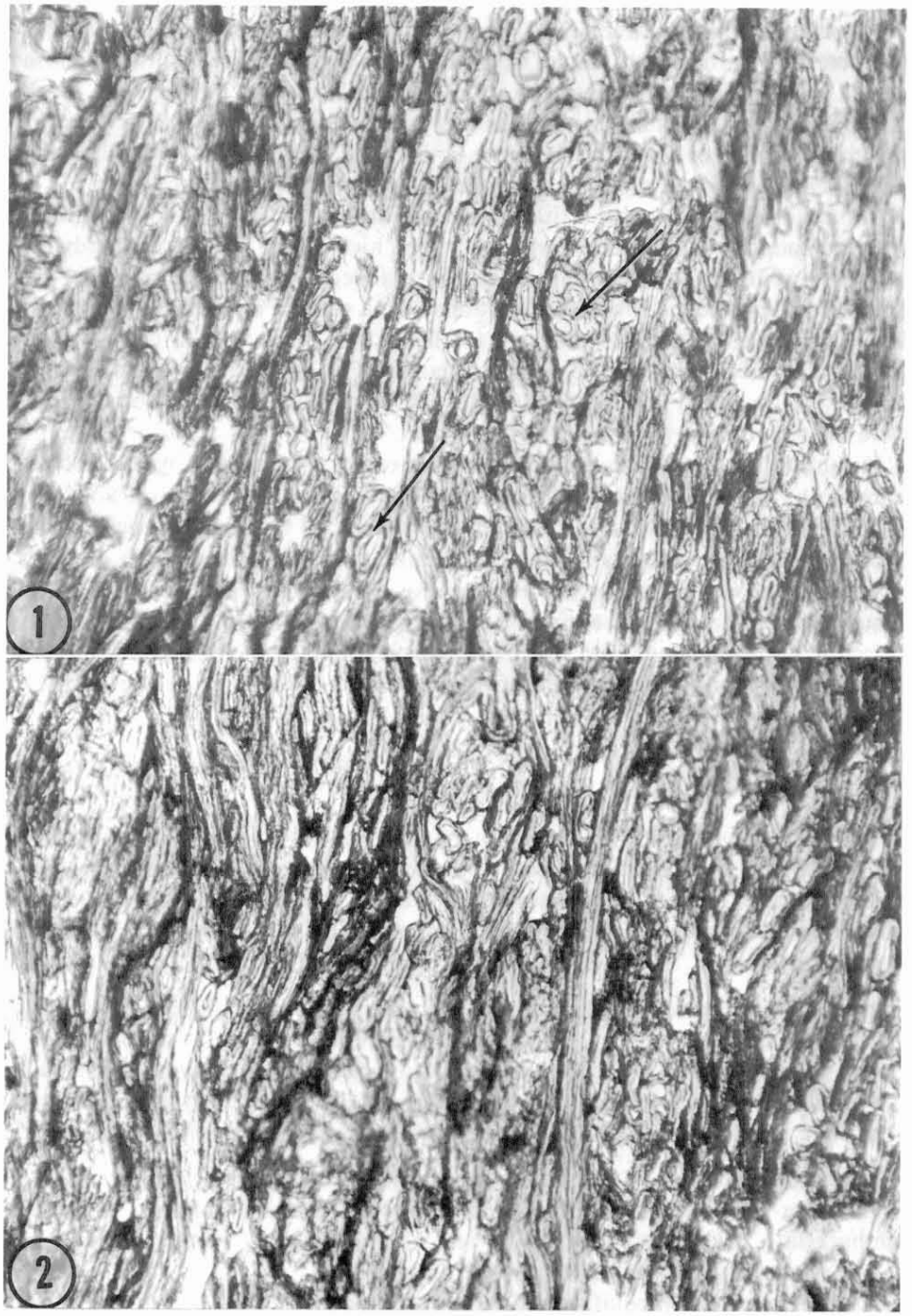


FIGS. 1, 2. (1) Wet-formed medium-density hardboard (2% PF). Fiber lumina free of resin (arrows); resin evenly distributed. ×200. (2) Wet-formed medium-density hardboard (4% PF). Resin deposition more intense because of higher resin concentration in the board. ×200.

Kruse and Parameswaran (1978) obtained 10- to 15-μm sections of bark particleboards by embedding samples in polyethylene glycol 1500 and cutting sections with a steel knife on a sliding microtome. Before each cut, they pasted "Tesa" film on the specimen's cutting surface and then fastened the section and film on the glass slide. They then dissolved the embedding medium and Tesa film off in water. They examined sections in transmitted near-ultraviolet light.

Furuno et al. (1984) used a microscopic technique in an effort to explain differences found in flakeboard durability when high- and low-density hardwood species were used. They cut flakeboards with a wedge microtome using K1 and K2 knives. To produce continuous sections with a smooth cross-sectional surface, they applied cyanoacrylate adhesive to the block's surface. The adhesive was removed by acetone after the section was cut. The cut surfaces of flakeboards were then immersed in a 0.2% acridine yellow solution to stain the wood and were viewed in reflected near-ultraviolet light.

Furuno et al. (1984) examined identical areas of flakeboards by fluorescence microscopy (reflected near-ultraviolet light) and secondary electron/SEM and cathodoluminescence/SEM microscopy and concluded that fluorescence microscopy is superior in investigating the distribution of resin on the surfaces of flakes.

We used the existing information on microscopy of wood products and animal tissues to develop our technique for examining hardboards by fluorescence microscopy. The relationship of the internal structure and the resin distribution to the mechanical properties of the hardboards examined here will be considered in a separate research report.

MATERIALS AND METHODS

The specimens we examined were prepared from wet-formed and dry-formed, high-density (0.8–1.2 specific gravity) and medium-density (0.5–0.8 specific gravity) hardboards made from pressure-refined aspen stemwood fibers bonded with phenol formaldehyde (PF) resin (Myers and Crist). Test materials were divided into two groups:

Group I: Wet-formed and dry-formed high-density and medium-density hardboards made with various concentrations of PF resin.

Group II: Wet-formed and dry-formed medium-density hardboards bonded with 4% and 6% PF resin, respectively. The resin had a fluorescent dye, 0.6% Rhodamine B, added to it before it was applied to the aspen stemwood fibers.

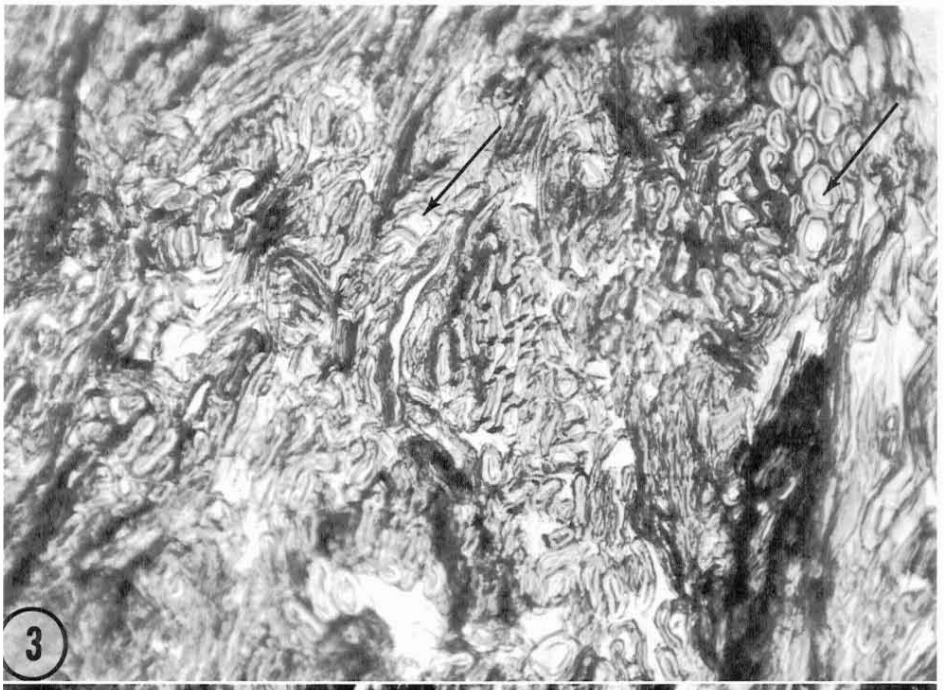
We tested several variables in developing the microscopic technique:

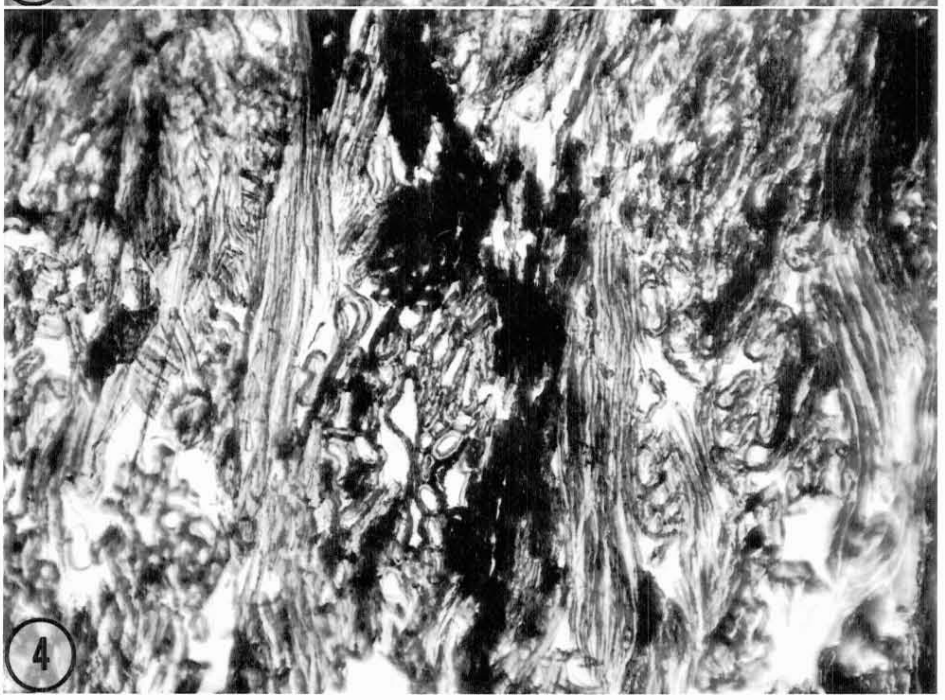
1. Water-, acetone-, or toluene-soluble embedding media (polystyrene, methyl methacrylate, glycol methacrylate) for sectioning 10- to 15-μm-thick sections.
2. Transmitted and reflected near-ultraviolet light.
3. Presence or absence of fluorescent dye (Rhodamine B).

The technique finally chosen involved the following steps:

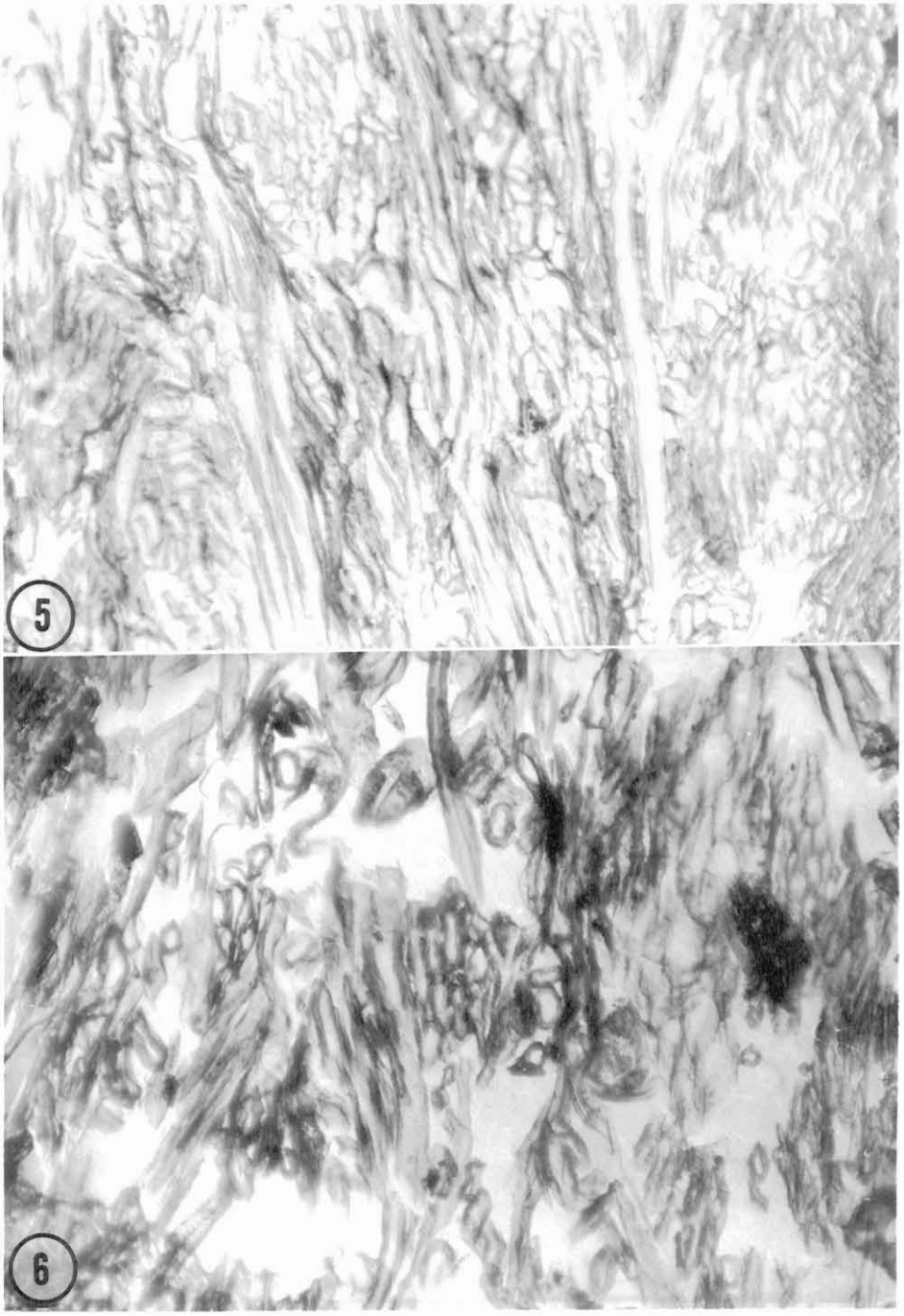
Samples were embedded in JB-4 embedding medium (JB-4 embedding kit by Polysciences, Inc.). The two stock solutions of this medium are Solution A (glycol methacrylate monomer) and Solution B (15 parts of polyethylene glycol 400 mixed with 1 part of N,N-dimethyl aniline).

Pieces of hardboards not exceeding the dimensions of 7 × 4 mm were cut from





FIGS. 3, 4. (3) Dry-formed high-density hardboard (2% PF). Fibers closely packed; resin unevenly distributed; fiber lumina free of resin (arrows). ×200. (4) Dry-formed medium-density hardboard (6% PF). Fibers less densely packed; resin densely aggregated. ×200.



FIGS. 5, 6. (5) Wet-formed medium-density hardboard (4% PF with 0.6% Rhodamine B). Fibers closely packed; resin evenly distributed. ×150. (6) Dry-formed medium-density hardboard (6% PF with 0.6% Rhodamine B). Fibers loosely packed; resin shows localized accumulations. ×150.

larger boards and, without dehydration, were infiltrated with catalyzed Solution A (100 ml Solution A plus 0.9 g benzoyl peroxide). The infiltrating solution was changed three times, each change lasting 24 hours at 4 C.

The infiltrated samples were embedded in a mixture of catalyzed Solution A (40 ml) and Solution B (1 ml) in gelatin or "Beem" capsules. The initial polymerization was allowed to proceed at room temperature for 2 hours, then continued in the oven at 45 C for 30 minutes.

Sections of 10- to 15-μm thickness were cut with a steel knife on a sliding microtome after the block cutting surface had been wetted with water or 70% alcohol.

The sections were floated in water on a slide that was covered with Haupt's adhesive. The sections were straightened out with dissecting needles under the dissecting microscope, if needed.

The slides were allowed to dry on a warm hot plate, and sections were mounted in a 3:1 glycerol–water mixture. From some sections the embedding medium was dissolved off with acetone or toluene.

The sections were examined in transmitted near-ultraviolet light with a transmission peak at 365 nm. Kodak 35 mm Plus-X pan film was used for black and white photomicrography, and Kodak Ectachrome 200 day-light film was used for color photomicrography. Color photomicrography was done on samples that had a fluorescent dye in the resin.

RESULTS AND DISCUSSION

We found that JB-4 embedding medium (glycol methacrylate) was the easiest to handle and gave the best results. Dissolution of the embedding medium (which in the case of JB-4 medium involved prolonged soaking of sections in acetone or toluene) did not improve visualization of the hardboard structure or the resin distribution.

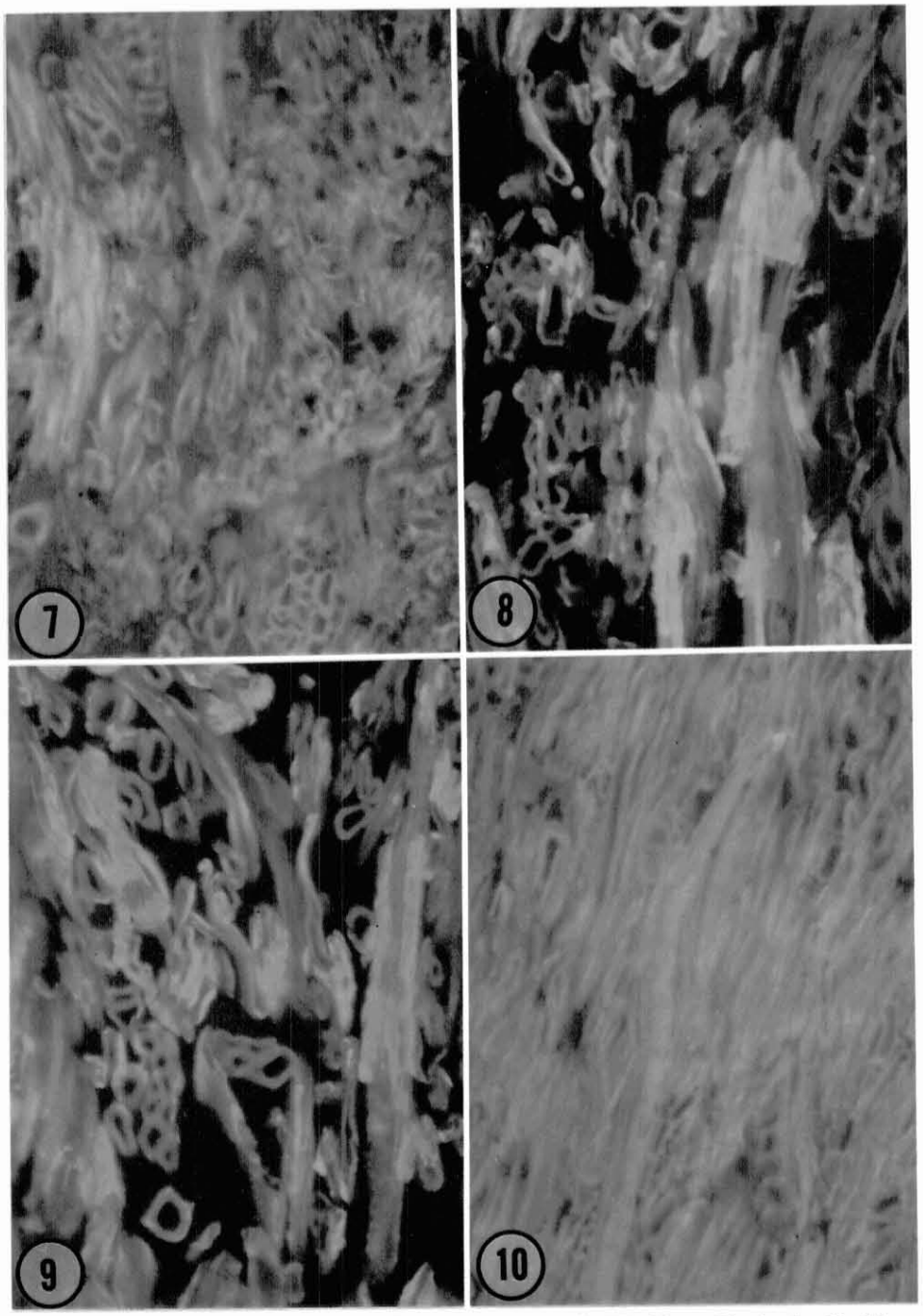
Transmitted near-ultraviolet light proved to be efficient for examining resin distribution in hardboards. Wood has a natural bluish auto-fluorescence at this wavelength, whereas PF has no fluorescence and appears reddish brown. Therefore, it is easily distinguishable from the fibers.

In the reflected near-ultraviolet light, it appeared impossible to follow the continuity of resin distribution within the boards. Similarly, Lehmann (1968) reported that the apparent depth of gluelines gave misleading impressions as to their continuity in the reflected light.

The fluorescent dye (Rhodamine B) provides an orange color to the resin. In black and white photomicrographs, the PF resin appears black and the fibers are white. In color photomicrographs, the resin is orange and the fibers are blue.

In Group I wet-formed high-density and medium-density hardboard samples, the fibers were closely consolidated and the resin had an even distribution (Fig. 1, medium density, 2% PF). The resin was mainly observed in the spaces between fibers, as it rarely showed up in the fiber lumina (arrows). Occasionally relatively large voids were encountered in both the medium- and high-density boards. Wet-formed medium-density boards with 4% PF showed a higher concentration of resin with a uniform distribution pattern (Fig. 2).

In dry-formed high-density hardboards, the fibers were closely consolidated into a compact structure but the resin showed localized aggregations (Fig. 3, 2%



FIGS. 7–10. (7) Dry-formed medium-density hardboard (6% PF without Rhodamine B). Fibers (blue) loosely packed; resin (reddish brown) unevenly distributed. ×145. (8, 9) Dry-formed medium-density hard-boards (6% PF with 0.6% Rhodamine B). Fibers (blue) loosely packed; resin (orange) unevenly distributed. ×145. (10) Wet-formed medium-density hardboard (4% PF with 0.6% Rhodamine B). Fibers (blue) closely packed; resin (present as faint orange coloration) evenly distributed. ×145.

PF). The fiber lumina usually had no resin (arrows). Dry-formed medium-density hardboards had very uneven resin distribution and most often the resin was present as large, black clumps (Fig. 4, 6% PF).

In Group II wet-formed and dry-formed medium-density hardboard samples, a fluorescent dye added to the resin—as was done with flakeboard by Lehmann (1968) and with particleboard by Kruse and Parameswaran (1978)—proved unnecessary for the black and white photomicrography. For color photomicrography, it changed the color of resin from an indistinct reddish brown to orange.

In wet-formed medium-density hardboards, the fibers were closely packed and the resin distribution was so uniform that it was difficult to see it (Fig. 5, 4% PF). Dry-formed medium-density hardboards showed some voids and localized accumulations of the resin (Fig. 6, 6% PF).

In color photomicrographs of the Group II samples, the resin is reddish brown without the fluorescent dye (Fig. 7); whereas, the resin is orange and the fibers are blue with the dye (Figs. 8 and 9). The fiber lumina most often were free of resin. In wet-formed medium-density boards the resin was very evenly distributed and appeared as a faint coloration over the entire area of the board (Fig. 10).

CONCLUSIONS

The embedding technique and sectioning procedure described clearly show the location of phenolic resin and its distribution in high-density and medium-density hardboards. The use of a fluorescent dye to enhance the visualization of resin is not necessary for black and white photomicrography. The addition of 0.6% Rhodamine B to the resin made the resin stand out vividly for color photomicrography.

Wet-formed high-density and medium-density boards usually showed fibers consolidated into a compact structure and a uniform resin distribution. Dry-formed high-density boards had a compact structure and medium-density boards a less compact structure; both characteristically showed uneven resin distribution.

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