Supporting Manufacturing Process Changes with an Innovative Microscopy Method

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Abstract

Bakelite Chemicals LLC has developed an innovative microscopy technique to view adhesives "in use." The cornerstone of the method is based on sample preparation. Samples of a composite wood product are sanded to a highly polished surface using a progression of sandpaper grits from lowest to highest. This preparation technique differs significantly from historical preparation techniques, which rely on soaking or embedding samples followed by thin-section creation using a microtome. The wet preparation technique causes wood cells compressed during production of the composite to swell and change how the adhesive is seen within those cells. The addition of water to the sample can also move uncured adhesive around the bond line. These changes can significantly alter the interpretation of how the adhesive performs during the manufacturing process. The new preparation technique combined with standard fluorescence microscopy has provided a groundbreaking and accessible ability to understand how an adhesive responds to process changes in manufacturing. Improving the understanding of the dynamic relationship between the adhesive and manufacturing processes will be crucial in helping the wood products industry take advantage of the rapid improvements in processing technology.

Introduction

The creation of a strong and durable bond is a cornerstone of the wood products industry. The bond between wood members is created in an extremely complex system consisting of the adhesive, a highly variable substrate, and multiple processing parameters. Understanding how an adhesive performs in this system is vital information used in the adhesive formulation process to ensure the adhesive performs in a specific manufacturing system. Bakelite Chemicals LLC (Bakelite) has developed a novel method of sample preparation and combined this with standard fluorescence microscopy and imaging. This new preparation method allows the researcher to see and understand how the adhesive responds in a wood product on a cellular level, where the strength of the bond is truly built.

Background

Multiple methods have been utilized to show the adhesive and wood interactions of a bond. A comprehensive review of glue-line imaging can be found in the literature (Kamke and Lee 2007, Shirmohammadi and Leggate 2021). The primary historical imaging method relies on saturating a bonded wood sample in water, preparing a thin section using a microtome, and staining the section for transmitted light microscopy. However, temperature, pressure, and moisture present during the manufacturing process cause compression changes of the cell structure along the bond line. It is widely known that wood swells in the presence of water and it can be hypothesized that this characteristic will cause compressed cells to swell and return toward a natural shape during the soaking preparation step. Water will also cause uncured adhesive in the bonded system to dilute and move around the bond line. These phenomena are discussed in Kamke and Lee (2007).

Recent research has focused on the use of advanced technology such as X-ray computed tomography, nanoindentation, and small-angle neutron scattering. Detailed descriptions of the use of these techniques can be found in the literature (Jakes et al 2018, Shirmohammadi and Leggate 2021). These methods have proven to provide excellent understanding of adhesive wood interactions but have limitations to frequent use and applicability to studying the adhesive in use for product manufacturing. These newer methods require highly specialized equipment that may not be readily available. The sample size can be limited due to instrumentation requirements, which in

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turn will limit the ability to understand the glue line in the context of a larger product such as plywood. These methods may also require the use of stains or additives to the adhesive, which could change both the cell structure and interactions of the adhesive.

Bakelite investigated a range of preparation techniques in the hopes of finding a method resulting in high-quality, detailed imaging of the wood and adhesive interface. Techniques such as embedding the bond line, cryo-fracturing, and diamond microtoming were evaluated in conjunction with standard optical microscopy and fluorescence microscopy. The combination of a highly polished surface and fluorescence microscopy was found to be the most effective methodology.

Materials and Methods

The primary component of the new microscopy method is the dry sample preparation. Samples are obtained from production wood composites and all relevant information on process conditions is recorded. The collected samples are then sanded on the cross-section of the sample thickness. Sanding is completed using a progression of sandpaper grits from lowest to highest: 80-grit, 100-grit, 150-grit, 180-grit, 220-grit, 320-grit, 400-grit, 600-grit, 1,000-grit, 2,000-grit, 3,000-grit. An electric palm sander is usually utilized to speed the process; however, this is not required. Special care is taken to ensure a flat surface during the sanding process. A flat surface is critical to producing high-quality images.

The polished surface of each sample is then viewed using a compound microscope set up to capture reflected light images using a digital camera. All images used in this document were captured under a ×10 objective combined with a ×1 mount tube to an inline Leica DMC5400 digital color camera. A high-energy light source is used in combination with light filter cubes to produce the fluorescent nature of the images.

Two specific filter cubes were utilized throughout production of the images in this publication. The first was an ultraviolet (UV) filter, which produces images showing the following colors. Natural wood shows up as a medium blue color of slightly different shades depending on species. A phenol formaldehyde (PF) adhesive will result in one of three colors depending on the state of cure: uncured adhesive of sufficient concentration appears as bright light blue, partially cured adhesive shows as a red/brown color within the cells, and fully cured adhesive is a deep brown towards black color. Research reported by Hunt et al. (2014) shows that the wood cell structure can act as a size-exclusion filter as the adhesive flows into the wood around the bond. The hypothesis is that the filtering results in the color differentiation seen within the images. Figure 1 shows these colors under the UV filter on a bond line between southern yellow pine (*Pinus spp*., SYP) veneers. The second filter is a green excitation cube. Natural wood shows up as a deep red color for each species that has been tested. A PF adhesive will result in two colors depending on the state of cure: uncured adhesive of sufficient concentration appears as bright red/orange, partially and fully cured adhesive shows up as deep brown or black. Figure 2 shows these colors under the green excitation filter cube on a bond line between SYP veneers. A more detailed description of the use of filters with a high-energy light source can be found in Kamke and Lee (2007). The comparisons given in the "Results" section below were taken under the green excitation filter as this tends to give the clearest view of adhesive penetration. Images taken with the UV filter also show adhesive penetration but give a better view of cell structure within the bond.

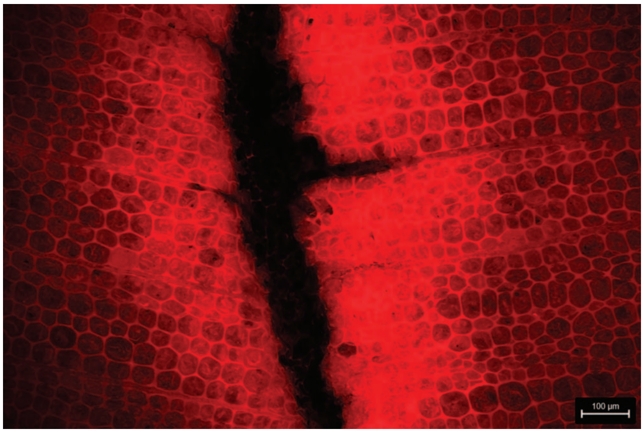

Figure 2.—A phenol formaldehyde (PF) bond line between southern yellow pine veneers and PF imaged using a green excitation filter cube. Image shows the color differences between wood and the adhesive under the filter.

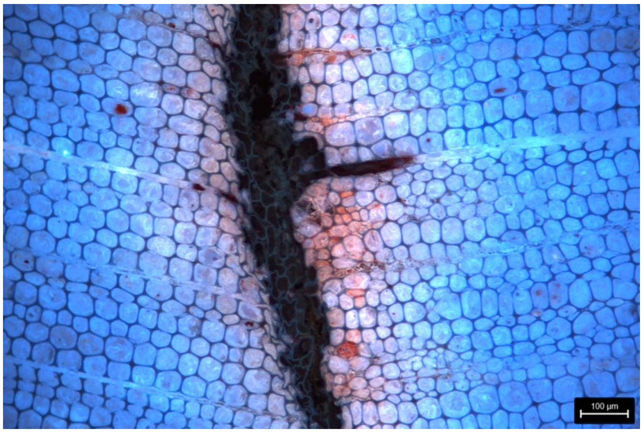

Figure 1.—A phenol formaldehyde bond line between southern yellow pine veneers imaged using an ultraviolet filter cube. Image shows the color differences between wood and the adhesive under the filter.

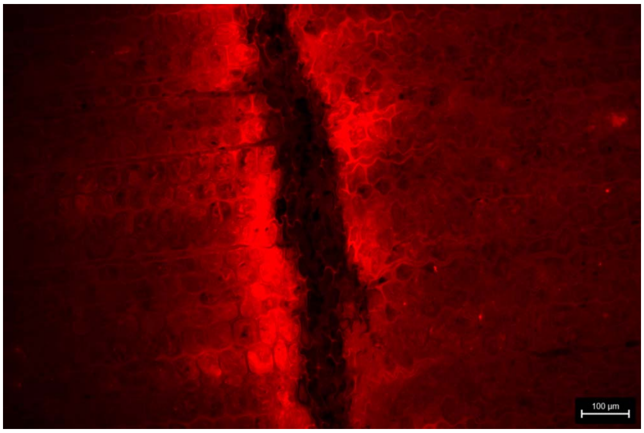

Figure 3.—The third bond line down in a 12-layer laminated veneer lumber specimen as produced. Image taken using the green excitation filter cube.

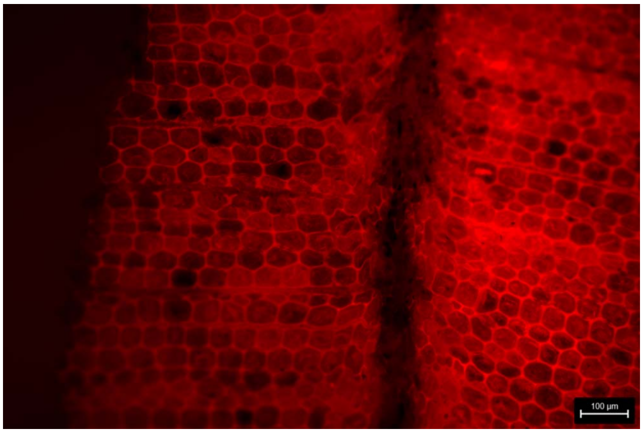

Figure 4.—The third bond line down in a 12-layer laminated veneer lumber specimen after undergoing vacuum pressure cycling in water. Image taken using the green excitation filter cube.

Following image collection, groups of images from each sample were compared to each other based on the process factors of interest. The comparisons were set up based on the production information recorded during sample collection.

Results

The theory that water used in the historical sample preparation step (for microscope imaging) will move uncured adhesive away from the bond line was introduced in the "Background" section of this publication. Bakelite has collected images that support this theory. Figure 3 shows a production sample prepared and imaged as is. Figure 4 shows a production sample, produced with the same conditions during the same run, that was subjected to cycling in water. A comparison of the two figures shows that Figure 4 indicates a significant increase in adhesive penetration within the wood. This is seen by comparing the bright red bands of adhesive on either side of the bond line in Figure 3 to the bright red adhesive appearing throughout the wood in Figure 4. The adhesive movement into the wood observed in Figure 4 can be attributed to the water cycling as this is the only difference documented between the samples. This is just one example of several obtained supporting the theories outlined in this document.

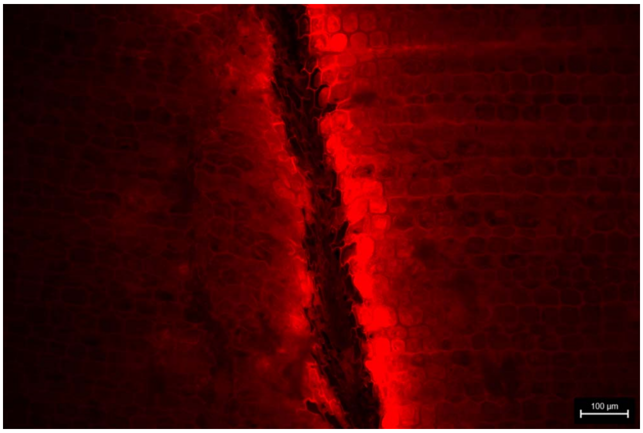

Figure 5.—A red pine bond-line image taken using the green excitation filter cube.

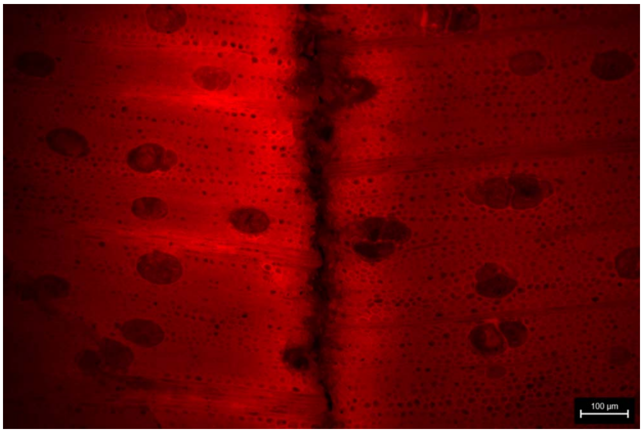

Figure 6.—A hard maple bond-line image taken using the green excitation filter cube.

Bakelite has developed an extensive body of work using the methodology detailed here. Image comparisons are shared to illustrate the use of this method to support manufacturing process changes. Several examples of process changes that drive comparison are given here: adjusting adhesive spreads, change in process equipment type, change in wood moisture profile for the substrate, change of primary process equipment, and change in line speed. The list does not correlate to the examples given and is not exhaustive, as the interactions of the adhesive with the manufacturing processes are complex.

The first comparison illustrates the differences observed between samples where the only process variable difference is the wood veneer species. The product shown in Figure 5 was a laminated veneer lumber (LVL) sample constructed with red pine (*Pinus resinosa*) veneer. The product shown in Figure 6 was an LVL sample constructed with hard maple (*Acer* sp.) veneer. The adhesive formulation was also constant between the two samples. The images clearly show that the red pine veneer sample (Fig. 5) has greater cell compression along the bond line while the maple veneer sample (Fig. 6) has deeper adhesive penetration.

The second comparison illustrates the differences observed between samples where two different adhesive formulations were used. Production variables were similar between samples.

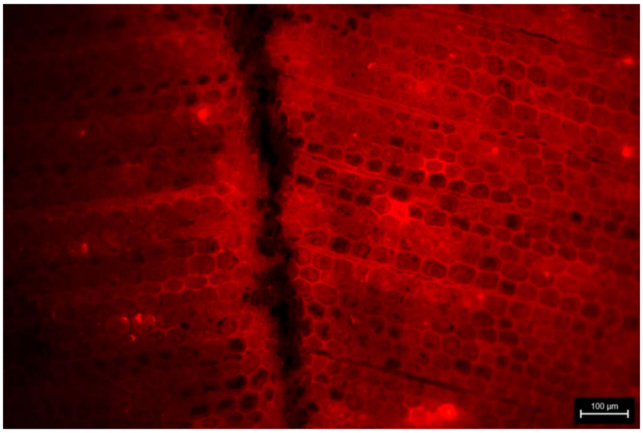

Figure 7.—The second bond line down in a four-layer plywood specimen made with adhesive A. Image taken using the green excitation filter cube.

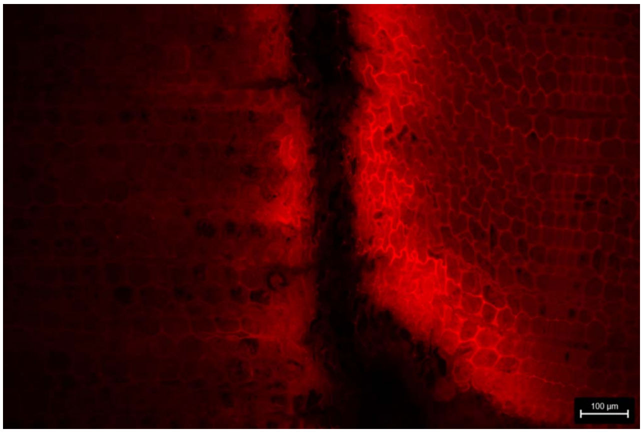

Figure 8.—The second bond line down in a four-layer plywood specimen made with adhesive B. Image taken using the green excitation filter cube.

Figure 7 shows an adhesive that penetrates deep into the wood structure and results in a thin bond line. Figure 8 shows an adhesive that stays in and just beside the bond line, resulting in a wide bond line. In this instance the researcher also took images from multiple samples with a variety of process variables to verify that the adhesive used in the Figure 7 sample truly has deeper penetration than the adhesive used in the Figure 8 sample.

The last comparison shows the differences observed when the adhesive spread rate is increased and all other process variables are held constant. Figure 9 is an image taken from a sample with normal adhesive application rates. Figure 10 is an image taken from a sample with elevated adhesive applications rates. Comparison of the two images shows that increasing the adhesive application rate increases both the bond-line thickness and the adhesive penetration depth.

The comparisons illustrated here show that this novel microscopy method is sensitive to changes in production variables. Furthermore, the results provide insight into the interaction of adhesives in a complicated system. Scientists using this method are provided an opportunity to understand which factors have the highest probability of impact on bond strength and durability.

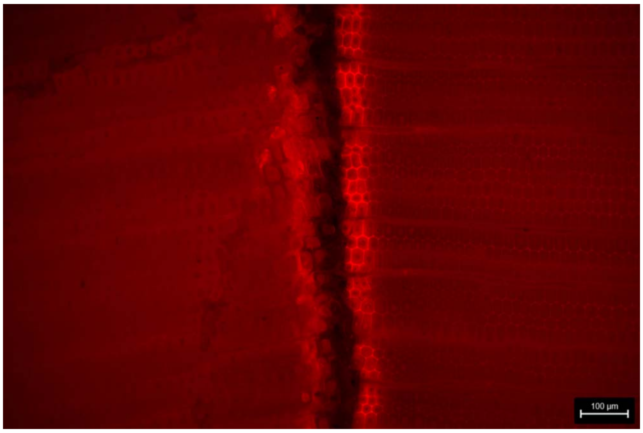

Figure 9.—The seventh bond line down in a 13-layer laminated veneer lumber specimen made with normal adhesive application rates. Image taken using the green excitation filter cube.

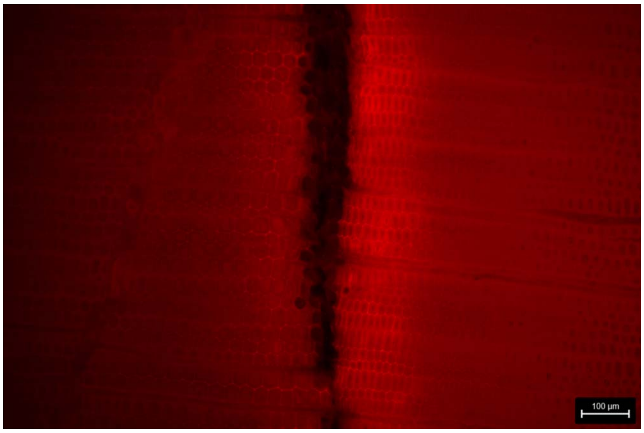

Figure 10.—The eighth bond line down in a 13-layer laminated veneer lumber specimen made with increased adhesive application rates. Image taken using the green excitation filter cube.

Discussion

There are two discussion points critical to understanding the use of this new methodology. The first point is a discussion on interpreting the images. The second point is on the utilization of the method for adhesives other than PF chemistry.

The comparisons given in the "Results" section here use one image of one bond line per sample. The researcher should note that a valid comparison uses multiple images across all bond lines using both microscope filters. Imaging should also focus on differences in standard wood anatomy, such as earlywood and latewood cell structure. Utilizing multiple images to develop an interpretation ensures that the observer accounts for variation inherent in the bonded system. Bakelite has found that the multiple-image analysis approach ensures the interpretation will be found valid even in a blind sample study scenario.

The images provided above all show bonds of wood with PF adhesives. The primary focus of the work completed at Bakelite has been on bonds with this adhesive type. However, there are indications that the method can also be utilized with alternate chemistries. Figure 11 shows an adhesive bond formed with an urea formaldehyde adhesive. This image was taken with the methods described above and the adhesive can

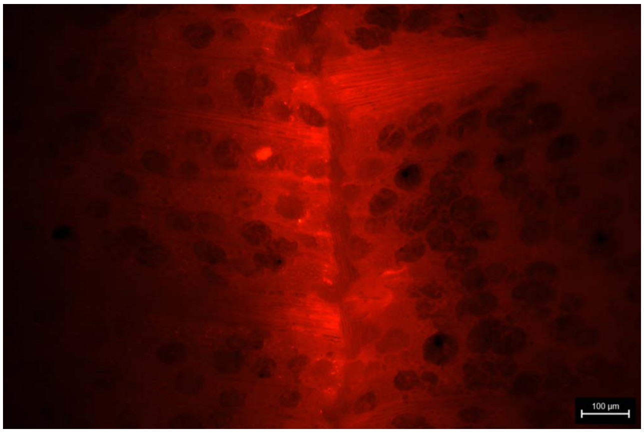

Figure 11.—An adhesive bond line between maple veneers and urea formaldehyde adhesive. Image taken using the green excitation filter cube.

clearly be seen within the wood structure. One excellent area of further research would be to study alternate filters available for the microscope system with common wood adhesive chemistries.

Conclusions

The innovative microscopy method detailed here has shown clear potential to support adhesive formulation adjustments in response to changes in the manufacturing process. The ability to respond to shifts in manufacturing will become increasingly important as rapid technology advancements drive significant improvements in processing.

Acknowledgments

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