Effects of Nanoclay on epoxy bonding of White Birch

Ava Schrenk
University of Minnesota – Twin Cities

Abstract: Samples of white birch were bonded with an epoxy resin designed for fast cure time and flexibility. Nanoclay was added based on percentage of the total mass of adhesive in 2.5%, 5%, and 10%. The lap joint specimens with the most nanoclay were the strongest and they survived soaking the longest without delamination. The wood-epoxy joints containing nanoclay also retained more strength after soaking. These specimens had more adhesive in the glueline, which contributed to greater water resistance and stronger joint strength.

Introduction

Epoxy resin has been used as an adhesive for wood products, but can it be improved by the addition of nanoclay? This experiment sought out to test the impact the addition of nanoclay has on the strength and water penetration of adhesive when applied to white birch.

Method

Model epoxy resin was prepared by mixing four compounds: the base resin (vinyl cyclohexene dioxide; ERL-4221), hardener (nonenyl succinic anhydride; NSA), flexibilizer (diglycidyl ether; DER-736), and accelerator (dimethylaminoethanol; DMAE). The mixing was carried out based on the following mass ratio.

NSA	ERL	DER	DMAE
0.524	0.202	0.242	0.0393

Nanoclay was added as a percentage of the total weight of the adhesive.

Lap-shear specimens of white birch were prepared with a spread rate of 0.37g/cm^2. Adhesive was applied equally to both bonded surfaces. The samples were pressed at 1000lbs for 30 minutes, and then heated to 100°C for 80 minutes. They were placed in a conditioning room (50% relative humidity and 20°C.) for 1 week, and subsequently tested at the same environmental condition

Soaking was done at 22°C. Specimens were spaced to have at least one inch between them. Soaking was done for durations of 5 minutes, 15 minutes, and 2 hour. A longer soaking (24h) was attempted, but all samples failed upon such exposure.

Dry Shear Strength

The strength of epoxy bonded lap-shear joints was measured with an Instron tensile tester. The dry tests conducted on samples conditioned at 50% RH and 20°C conclusively showed that the addition of nanoclay increases the shear strength of the bonded joints. Summarized data are in Table 1. As shown, even a small amount of nanoclay addition increases shear strength of the bond. The failures were intralaminal; they were not caused by failures of the wood or of the bonding to the substrate. More details on bondline failure will be provided later in this report.

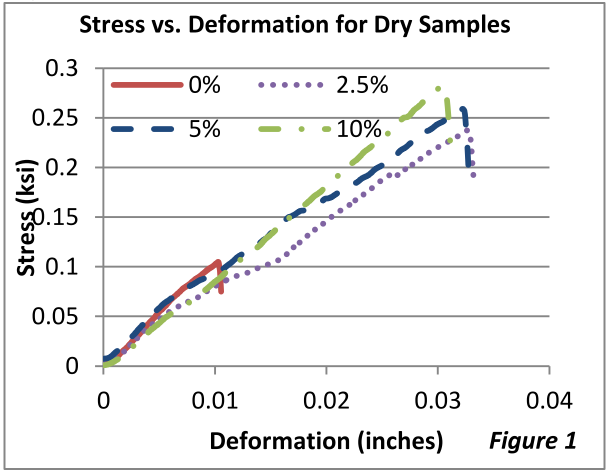
Stress vs. Deformation for Dry Samples

Figure 1

Table 1

Nanoclay	Avg Shear Strength (ksi)	Standard Deviation	Max (ksi)	Min (ksi)
0%	0.12	0.060	0.23	0.063
2.5%	0.25	0.069	0.34	0.12
5%	0.26	0.062	0.35	0.17
10%	0.29	0.035	0.37	0.25

Wet Shear Strength

Samples at 0% nanoclay and 10% nanoclay were subjected to water soaking. The non-delaminated lap specimens were tested for joint strength, and results are shown in Figure 2. Figure 2 shows that the lap joints decreased in strength significantly with soak time. Additionally, lap joints with 10% nanoclay were stronger than those without. This suggests that nanoclay helps retain joint strength when specimens were subjected to soaking.

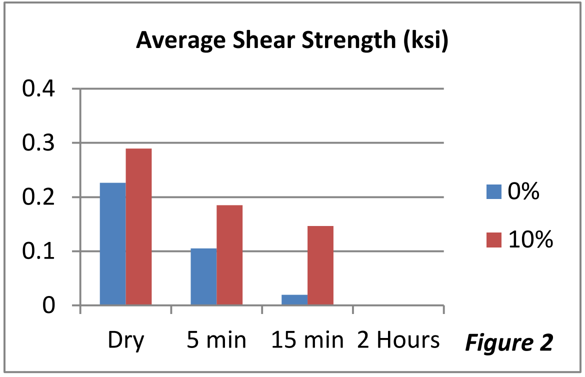

Figure 2

Figure 3 shows delamination rates of the two sample sets at various soaking periods. Delamination rate refers to the number of joints survived as a percentage of the total specimens tested in soaking. Results show that wood joints without nanoclay delaminated with less soaking and at a higher rate than those with 10% nanoclay. Upon 2-h soaking, delamination was observed for all lap joints without nanoclay.

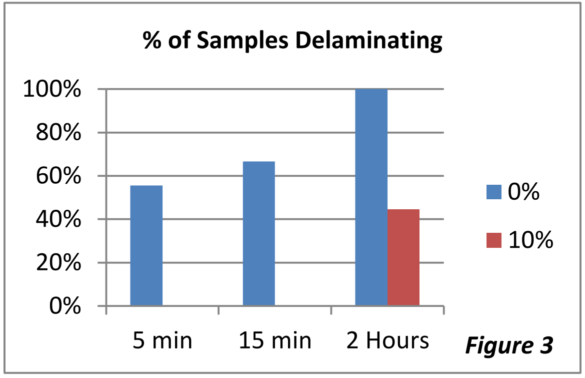

Figure 3

Figure 2 also shows that the shear strength is only a fraction of what it was when dry.

Penetration and Interfacial Adhesion

According to Washburn theory, the time required for penetration is:

$$l^2 = \frac{tr\gamma\cos(\theta)}{2\eta}$$

Where l is the depth of penetration, t is the elapsed time, r is the pore radius of the substrate, γ is the surface tension of the liquid, η is the liquid's viscosity, and θ is the contact angle. Therefore, the penetration on a porous substrate of an adhesive is indirectly correlated with its contact angle and viscosity for angles between 0 and 90 degrees.

On a non-porous substrate, paraffin wax, the contact angle is indistinguishable between the 0% nanoclay sample and the 10% nanoclay sample, as shown in Photo 1. The elapsed time between droplet formation and moment of imaging was 30 seconds.

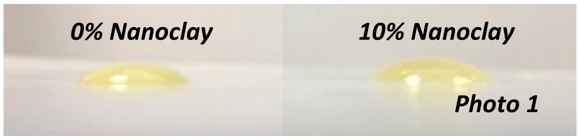

Photo 1

However, when the adhesive was applied to the porous substrate, white birch, there was a clear difference in the contact angle.

The 0% nanoclay samples had a smaller contact angle than the 10% nanoclay samples. The same specimen of white birch was used for each test, so that variation would be kept to a minimum. This can be seen in Photo 2. The elapsed time is 30 seconds.

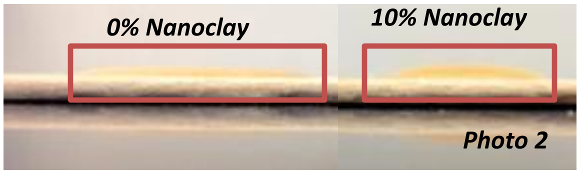

Photo 2

It has been shown in a previous study, published in the journal of the Materials Research Society by Asma Yasmin, et al., that the viscosity of the epoxy resin increases with increasing percentages of nanoclay. Since both a smaller contact angle and increasing viscosity are associated with less penetration in the Washburn equation, it can be concluded that the epoxy with 10% nanoclay did not penetrate as much in the same amount of time as the epoxy with no nanoclay.

Adhesive that penetrates the white birch is, by necessity, not available at the glueline. Therefore, it is suspected that the specimens with 10% nanoclay had more adhesive at the glueline than the samples without nanoclay.

This difference in amount of adhesive at the glueline may have contributed to the greater shear strength of the joints with 10% nanoclay. Where there is a defect of unevenness at the surface of the white birch, the adhesive with 10% nanoclay can fill in the "valleys" to provide a better contact at the glueline. For the adhesive without nanoclay, these "valleys" remain unfilled and results in a loss of joint strength, and hastens delamination when soaking.

Conclusions

The addition of nanoclay has a strong effect on the shear strength of lap joints of white birth bonded with epoxy adhesive. This addition also improves the water resistance of the lab joints when the substrate is soaked in water.

This increase in strength and decrease in delamination is due to the increased amount of adhesive in the glueline. The adhesive with no nanoclay penetrates white birch more quickly and further than the adhesive with added nanoclay. There is more adhesive available to fill in defects at the surface of the substrate when nanoclay is added, creating a stronger bond that has a higher resistance to water penetration.

References

Arthur R. Spurr, A low-viscosity epoxy resin embedding medium for electron microscopy, Journal of Ultrastructure Research, Volume 26, Issues 1–2, January 1969, Pages 31-43, ISSN 0022-5320, 10.1016/S0022-5320(69)90033-1.

Asma Yasmin, Jandro L. Abot and Isaac M. Daniel (2002). Processing of Clay/Epoxy Nanocomposites with A Three-Roll Mill Machine. MRS Proceedings, 740, I3.7 doi:10.1557/PROC-740-I3.7.

Czachor, H. (2007), Applicability of the Washburn theory for determining the wetting angle of soils. Hydrol. Process., 21: 2239–2247. doi: 10.1002/hyp.6753

Acknowledgements

The author thanks the Undergraduate Research Opportunity Program for the funding to do this research, William Tze of the University of Minnesota, Twin Cities, for guidance and direction on the project, Han Seung Yang of Tze Research Group for assistance with equipment and training, and Jonathan Schilling for access to his equipment.