

DYNAMIC MECHANICAL ANALYSIS OF FPU BONDED BEECH WOOD AT VARIOUS TEMPERATURES

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ABSTRACT: This study investigates the dynamic mechanical properties of flexible polyurethane (FPU) adhesives bonded to European beech wood (*Fagus sylvatica* L.) using Dynamic Mechanical Analysis (DMA) over a range of temperatures and frequencies. Samples included clear beech wood and three-layered composite (beech:FPU:beech). DMA tests were performed using a three-point bending setup at 1 Hz and 10 Hz in the temperature range from -120°C to 140°C. The results demonstrated high thermal stability and strong bonding performance of FPU adhesives on beech wood. Frequency-dependent responses varied among FPU types but resembled the behavior of solid wood at lower temperatures. This confirms the suitability of DMA for evaluating adhesive-wood composite behavior under dynamic conditions. The combination of FPU adhesives and beech wood showed potential for thermally stable and energy-dissipating bonded assemblies.

KEYWORDS: dynamic mechanical analysis, FPU, beech wood

1 – INTRODUCTION

Due to the natural tendency of wood to shrink and swell, it is important that the joints in glued timber structures remain intact when exposed to different climatic conditions. The viscoelastic nature of wood adhesives can generate hysteretic energy under cyclic loading. Moisture-curing one-component polyurethane adhesives, which are known for their lower stiffness and hardness, can absorb more energy during deformation. This property leads to greater plastic deformation, which often results in improved dynamic properties (e.g. fatigue performance). Consequently, energy dissipation prevents cracking and reduces joint failure [1]. To characterize selected performance parameters of wood, adhesives and wood-adhesive composites, DMA provides a suitable method to measure the dynamic properties of these materials under the influence of forced, sinusoidally variable dynamic loads. DMA analyzes the viscoelastic properties of polymeric materials. It can describe various phase transitions at the molecular level during temperature exposure. For this reason, this method is not only used to

study the curing of adhesives [2], [3] but also to analyze composite materials [4]. Studies have been reported in the literature in which this method was used to follow material changes in water-saturated wood at different temperatures [5] or the effects of wood impregnation on mechanical properties [6], [7]. Due to its high sensitivity to structural relaxation phenomena, DMA also enables the quantification of relaxation times and activation energies. This provides insights into the molecular mobility within the adhesive matrix and the wood-adhesive interphases. Finally, this enables a more accurate prediction of long-term mechanical stability under different environmental conditions, which is crucial in the context of assessing the durability of bonded wood structures.

2 – BACKGROUND

In current timber construction practice, softwood species dominate, especially in glued laminated timber. However, hardwoods such as ash, oak and beech, traditionally used for non-structural applications, are increasingly being recognized for building structures. It is well known that

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these hardwood species are more difficult to bond than the traditionally used softwoods due to their higher density, porosity, chemical composition, etc. In order to utilize these wood species, it is therefore important to investigate how durable and long-lasting composite materials can be developed. In the search for environmentally friendly materials, there has been increasing interest in adhesives with low or no formaldehyde emissions. This can be achieved by replacing some of the traditional formaldehyde-based adhesive systems with polyurethanes [8]. These unique materials are known for their strong adhesion to a variety of substrates due to the presence of polar HNCOO groups with high cohesive energy. They exhibit elastic behavior, especially when combined with various additives, and are known for their chemical resistance as well as their resistance to moisture. In addition, polyurethane adhesives can withstand prolonged vibration and significant impact loads before failing [9]. Another important feature of polyurethanes is the ability to extensively customize their properties by modifying the molecular chain structure of both the soft segments (consisting of long polyols) and the hard segments (formed by short diols and isocyanates) that make up the PUR backbone [10]. This adaptation is primarily important for the process of gluing, followed by the properties of the glued wood products, where the adhesive must closely match the properties of the wood component, especially for the application of the product. PUR adhesives represent a

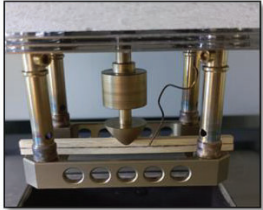
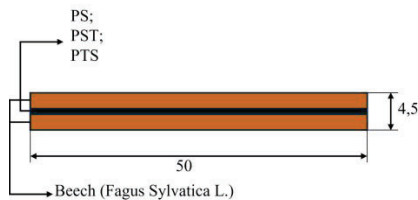
microphase separation between soft and hard segments due to their phase-separated morphology; this morphology has a decisive influence on the mechanical, thermal and viscoelastic properties of the finished adhesive, including its ability to dissipate energy under dynamic loading. The FPU adhesives used in this study are not typical adhesives for wood bonding, as they were primarily developed for reinforcing concrete and masonry to improve the seismic performance of buildings. As many glued wood products are also suitable for outdoor or construction use, the large daily temperature fluctuations typical of Central Europe should be considered. For this reason, the main objective of this study is to analyse the thermal stability of selected novel FPU adhesives investigated in [11] in combination with wood to ensure that their properties can withstand the different conditions in these applications.

3 – RESEARCH OVERVIEW

In this study, three two-component flexible polyurethane adhesives (FPU) were used to produce composite samples with beech wood. The description and density of the PS, PST and PTS adhesives used are listed in Table 1, while the exact material properties of these 3 FPUs can be found in [12]. In contrast to rigid PUR adhesives (modulus of elasticity > 1000 MPa), the tested FPUs exhibit lower stiffness (4–1000 MPa), lower tensile strength (1–22 MPa) and higher elongation at break (10–200%).

Table 1: Flexible FPU adhesive description [11].

| Adhesive | Density g/cm ³ | Description |
|----------|------------------------------|---|
| PS | 1.4 | Solvent-free, two-component, polyurethane-based adhesive. Flexible adhesive designed for making flexible joints and for coatings. A:B (100:11) |
| PST | 1.0 | Two-component adhesive material based on polyurethanes. Flexible adhesive designed for making flexible for making protective coatings. A:B (100:15) |
| PTS | 0,9 | Two-component adhesive material based on polyurethanes. Permanently elastic adhesive intended for making flexible joints and for making protective coatings. A:B (100:15) |



Gas flow: 50 mL/min
Temperature range:
- 120°C to +140°C
Heating rate: 3 °C /min
Frequency: 1 Hz and 10 Hz

Figure 1: Sample geometry and DMA 3-point bending test specifications.

4 – EXPERIMENTAL SETUP

Two series of samples were used for the tests (Fig. 1). Samples of clear beech wood and of beech:FPU:beech composites (FPU composites). Three samples per series were produced. The size of each sample was adapted to the requirements of the DMA 242 E Artemis instrument (NETZSCH). The measurements were performed under controlled conditions, including the fixture for 3-point bending, under a nitrogen atmosphere with a gas flow of 50 mL/min and a test frequency of 1 and 10 Hz. The test temperature was maintained from -120 to 140 °C with a heating rate of 3 °C/min, while the results are presented for temperatures between -100 and 100 °C. The average dimensions of the sample were 50 x 2.9 x 4.5 mm (L x W x H), assuming a nominal adhesive thickness of approx. 0.3-0.5 mm.

To produce the samples, six beech boards, each 2 mm thick and measuring 130 x 300 mm, were produced using a thickness planer. The average moisture content of the wood at the time of gluing was 10.8 %, measured with a Tanel WIP24 moisture meter (Fig. 2a). Two boards were then glued together using PS, PST and PTS adhesives. The adhesive components were mixed in the appropriate weight ratios (Table 1) and applied to the surface of the beech lamellas with a spatula (Fig. 2b). According to the technical data sheets of the FPU adhesives used, no minimum compressive force is required for the bonding process. In order to achieve the desired adhesive layer thickness, carpenter's clamps were therefore used during the bonding process. All test specimens were conditioned for 7 days in a controlled environment at a stable temperature and relative humidity (20 °C, 65 % ± 5 %). Seven days after bonding, the FPU composites were cut into samples.

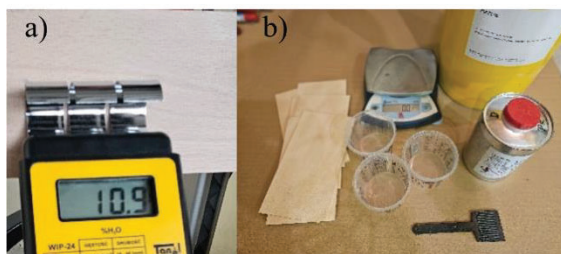


Figure 2: (a) Determination of the moisture content of beech boards prior to bonding using a Tanel WIP-24 moisture meter; (b) preparation of two-component polyurethane adhesive by weighing the components prior to mixing and application onto the beech boards using a spatula.

5 – RESULTS

The viscoelastic properties of beech wood bonded with different flexible adhesives were analyzed using dynamic mechanical analysis (DMA), focusing on the storage modulus (E') as a function of temperature at different loading frequencies. Fig. 3 shows the storage modulus as a function of temperature for different composites with three different adhesive systems tested at 1 Hz and 10 Hz. The unbonded beech wood sample exhibited the highest initial E' values throughout the test, indicating the inherent stiffness of the wood. However, as the temperature increased, a slight decrease in E' was observed, which can be attributed to the thermal softening behavior of the wood structure. The samples bonded with FPU adhesives showed a significantly different behavior than the solid beech wood. The E' values of the PS- and PTS-bonded composites were initially similar to those of the solid wood, with all three exhibiting higher initial stiffness than the PST-bonded composite. During the test, the PST composite showed a moderate decrease in E' , indicating a balanced compromise between stiffness retention and flexibility with increasing temperature. In contrast, the PS and especially the PTS composites showed a more pronounced decrease in E' , with the PTS system showing the strongest decrease, indicating greater thermal softening. The frequency-dependent behavior was more pronounced for the bonded composite materials than for the solid wood. For all materials, the E' values at 10 Hz were consistently higher than those at 1 Hz, which is typical for viscoelastic materials — at higher frequencies, molecular mobility is more restricted, resulting in higher stiffness.

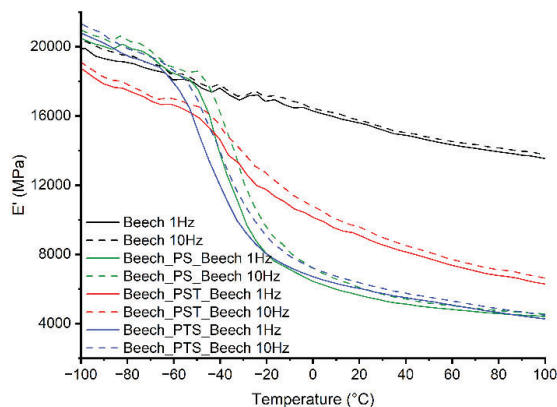


Figure 3: Comparison of storage modulus as a function of temperature for different composites with different adhesive systems tested at 1 Hz (solid) and 10 Hz (dashed).

However, as the temperature increased, the difference in E' between the two frequencies decreased, particularly beyond the glass transition region. This temperature and frequency sensitivity is primarily due to the flexible (soft) segments in the polyurethane adhesives being able to move more freely at higher temperatures, resulting in a decrease in stiffness. The greater decrease in E' observed in the PS and PTS composites probably indicates a higher proportion of these soft segments or a more compliant adhesive structure, so that they can absorb more energy at higher temperatures but are less stiff. This finding is crucial for the selection of suitable adhesives based on the desired balance between flexibility and stiffness in wood bonding.

In Fig. 4, the viscoelastic behavior is further investigated by decomposing the response into individual components: (a) storage modulus, (b) loss modulus (E'') and (c) $\tan \delta$. The trends observed in Fig. 4a) are consistent with those in Fig. 3 and confirm that PTS and PS composites exhibit the largest decrease in stiffness, while PST retains a higher stiffness. Fig. 4b) shows the E'' , which represents the energy dissipated by the sample due to softening of the adhesives. The largest area and E'' value were observed for the PS composite. The $\tan \delta$ values in Fig. 4 c) support this observation, as the peak values correspond to the temperature range at which the adhesives transition to their rubbery state, which is critical for damping performance. The shape and magnitude of the $\tan \delta$ peak for PST composites show that the transition occurs more slowly and over a wider temperature range. This usually means that the adhesive has a more mixed internal structure and contains many soft, flexible parts. On the other hand, the PS and PTS samples show a more distinct peak, indicating that their structure is more ordered. These results are consistent with those reported in a study on flexible polyurethane adhesives in wood joints [13], [14]. The results show that the flexibility of adhesives in combination with solid wood can be well studied, observing the effects of temperature.

The study has also shown that flexible adhesives provide increased deformation capacity while maintaining sufficient bond strength, making them suitable for applications where damping and flexibility are required. Furthermore, the pronounced viscoelastic signatures — such as the shift in the maxima of loss modulus and $\tan \delta$ to lower temperatures in PTS systems — indicate reduced glass transition temperatures of the adhesive phase, which is particularly relevant for energy dissipating applications under subambient thermal conditions

Overall, the results confirm that the choice of adhesive significantly influences the viscoelastic performance of bonded beech wood. The PST composite appears to provide an appropriate balance between stiffness and flexibility, while the PTS composite has the highest damping capacity, making it a potential candidate for applications requiring improved energy dissipation. Further investigation into long-term durability and cyclic loading behavior would be beneficial to fully assess the practical implications of these results.

The mechanical behavior of bonded beech wood at different temperatures has been studied in detail, and the results are consistent with our observations. A study [15] found that elevated temperatures negatively affect the tensile shear strength of various adhesives used for wood bonding. Thermoset adhesives such as MUF and PRF show higher strength at elevated temperatures than elastomeric adhesives such as one-component polyurethane and emulsion polymer isocyanate, while thermoplastic adhesives such as polyvinyl acetate show the lowest performance under thermal stress. Similar results for PUR adhesives were confirmed in [16], where it was found that these adhesives are least affected by elevated temperatures. As the adhesives used in this study show potential for structural applications, further tests need to be carried out, in particular shear tests to evaluate the adhesive properties and their resistance to temperature and water interaction, as suggested in the EN 302 standard.

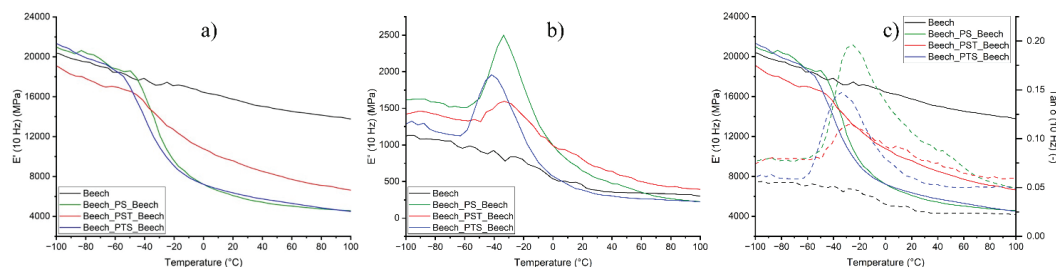


Figure 4: Viscoelastic behavior of beech wood bonded with different flexible adhesives: (a) storage modulus (E'), (b) loss modulus (E''), and (c) storage modulus (solid lines) and $\tan \delta$ (dashed lines), all as a function of temperature.

6 – CONCLUSION

The study confirms that flexible polyurethane adhesives significantly influence the viscoelastic behavior of bonded beech wood. More flexible systems (e.g. PTS) offer better damping but lower stiffness retention. PS adhesives offer a good balance of stiffness and damping. PST shows potential for applications in cold regions that require longer energy absorption. DMA proved effective method for evaluating adhesive properties and wood/adhesive interactions. The shift in the peaks of $\tan \delta$ and loss modulus to lower temperatures in PTS and PST reflects earlier softening, which is relevant for vibration damping applications. The heterogeneous nature of PST could offer advantages in wider service temperature ranges.

Future studies should investigate long-term and cyclic performance, different wood species and the effects of adhesive formulation on bond durability under different environmental conditions such as moisture, temperature cycling and mechanical fatigue, particularly in structural adhesive applications where long-bearing capacity and environmental resistance are critical.

7 – ACKNOWLEDGMENTS

This research was funded in whole or in part by the National Science Centre, Poland, under the OPUS call in the Weave programme (project No. 2021/43/I/ST8/00554). The DMA measurements were carried out at the Faculty Laboratory of Thermophysical Measurements at the Faculty of Material Science and Ceramics, AGH University of Krakow. The authors in Slovenia gratefully acknowledge the support of the Slovenian Research and Innovation Agency (ARIS) through research programs I0-0032, P2-0273, and P4-0430, as well as project grant N2-0280. The financial support from the National Science Centre, Poland, is also sincerely appreciated.

7 – REFERENCES

[1] Künniger, T., Clerc, G., Josset, S., Niemz, P., Pichelin, F., & Van De Kuilen, J.-W. G., "Influence of humidity and frequency on the energy dissipation in wood adhesives," *International Journal of Adhesion and Adhesives*, vol. 92, pp. 99–104, Jul. 2019, doi: 10.1016/j.ijadhadh.2019.05.003.

[2] Lei, H., & Frazier, C. E., "A dynamic mechanical analysis method for predicting the curing behavior of phenol-formaldehyde resin adhesive," *Journal of Adhesion Science and Technology*, vol. 29, no. 10, pp.

981–990, May 2015, doi: 10.1080/01694243.2015.1011735.

[3] Fitrianum, F., et al., "Adhesion and Cohesion Strength of Phenol-Formaldehyde Resin Mixed with Different Types and Levels of Catalyst for Wood Composites," *J. Compos. Sci.*, vol. 7, no. 8, p. 310, Jul. 2023, doi: 10.3390/jcs7080310.

[4] Adachi, K., Yamauchi, H., & Inoue, M., "Flexible LVL: Potential for Newly Material Design," 2010.

[5] Horiyama, H., Miyoshi, Y., Kojiro, K., & Furuta, Y., "Thermal softening properties of various wood species within an annual ring," *J. Wood Sci.*, vol. 69, no. 1, p. 30, Sep. 2023, doi: 10.1186/s10086-023-02104-2.

[6] Lemaire-Paul, M., & Foruzanmehr, M. R., "The study of physico-mechanical properties of SiO₂-impregnated wood under dry and saturated conditions," *Wood Sci. Technol.*, vol. 57, no. 5, pp. 1039–1059, Sep. 2023, doi: 10.1007/s00226-023-01492-4.

[7] Choi, H., Dalton, L. E., Peszlen, I., & Pourghaz, M., "The impacts of CaCO₃ deposition in natural wood on its viscoelastic properties," *Compos. Part B Eng.*, vol. 275, p. 111324, Apr. 2024, doi: 10.1016/j.compositesb.2024.111324.

[8] Bockel, S., et al., "Modifying elastic modulus of two-component polyurethane adhesive for structural hardwood bonding," *J. Wood Sci.*, vol. 66, no. 1, p. 69, Dec. 2020, doi: 10.1186/s10086-020-01917-9.

[9] Loginova, S. E., Averchenko, E. B., Kurilova, E. A., Nikonova, N. V., & Gladkikh, S. N., "Polyurethane Adhesives for Structural Parts of Means of Transport with Enhanced Processing Properties and Performance," *Polym. Sci. Ser. D*, vol. 12, no. 4, pp. 351–356, Oct. 2019, doi: 10.1134/S1995421219040087.

[10] Somdee, P., Lassú-Kuknyó, T., Kónya, C., Szabó, T., & Marossy, K., "Thermal analysis of polyurethane elastomers matrix with different chain extender contents for thermal conductive application," *J. Therm. Anal. Calorim.*, vol. 138, no. 2, pp. 1003–1010, Oct. 2019, doi: 10.1007/s10973-019-08183-y.

[11] Rutkowski, P., et al., "Thermal Stability and Heat Transfer of Polyurethanes for Joints Applications of Wooden Structures," *Molecules*, vol. 29, no. 14, p. 3337, Jul. 2024, doi: 10.3390/molecules29143337.

[12] Szeptyński, P., Pochopień, J., Jasińska, D., & Kwiecień, A., "The Influence of the Flexibility of a Polymeric Adhesive Layer on the Mechanical Response

of a Composite Reinforced Concrete Slab and a Reinforced Concrete Beam Girder,” *Polymers*, vol. 16, p. 444, 2024, doi: 10.3390/polym16030444.

[13] Pečnik, J. G., et al., “Mechanical performance of timber connections made of thick flexible polyurethane adhesives,” *Eng. Struct.*, vol. 247, p. 113125, Nov. 2021, doi: 10.1016/j.engstruct.2021.113125.

[14] La Scala, A., Śliwa-Wieczorek, K., Rizzo, F., Sabbà, M. F., & Zajac, B., “Flexible Polyurethane Adhesives: Predictive Numerical Model Calibration through Experimental Testing at Elevated Temperature,” *Appl. Sci.*, vol. 14, no. 5, p. 1943, Feb. 2024, doi: 10.3390/app14051943.

[15] Bernaczyk, A., Wagenführ, A., Terfloth, C., Lincke, J., Krystofiak, T., & Niemz, P., “Investigations into the Influence of Temperature on the Tensile Shear Strength of Various Adhesives,” *Materials*, vol. 16, no. 18, p. 6173, Sep. 2023, doi: 10.3390/ma16186173.

[16] Pečnik, J. G., Pondelak, A., Burnard, M. D., & Sebera, V., “Mode I fracture of beech-adhesive bondline at three different temperatures,” *Wood Mater. Sci. Eng.*, vol. 18, no. 4, pp. 1349–1359, Jul. 2023, doi: 10.1080/17480272.2022.2135135