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The Effect of Different Ambient Temperatures on the Adhesion Performance of Black Pine (*Pinus nigra*) Wood

Orhan KELLECİ[1], Süheyla Esin KÖKSAL[2,*]

[1,2] Bolu Abant Izzet Baysal University, Vocational School of Mudurnu Süreyya Astarci, Bolu, Türkiye

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Research Article

Abstract − In this study, the adhesion performance of wood glues was evaluated depending on temperature. Polyvinyl acetate (PVA) and poly urethane (PU) glue were used to adhere to the Back pine (Pinus nigra) wood samples. The adhesion performance of the samples was determined under three different temperature effects: below room temperature (4 ° C), at room temperature (22 ° C), and above room temperature (55 ° C). After sticking, the samples were clamped with a hand vise and left to cure at three different temperatures. Lap sheer strength (LSS), modulus of rupture (MOR) and modulus of elasticity (MOE) analyses were performed for the mechanical characterization of the samples. According to the obtained results, the LSS (Load at Specified Strain) strength increased below and above room temperature. In contrast, the MOR (Modulus of Rupture) and MOE (Modulus of Elasticity) strengths decreased below and above room temperature. Thus, it was concluded that in addition to the adhesive strength varying with temperature, the direction of the force application also influences the strength. Especially in small workshops, the ambient temperature is greatly influenced by summer and winter conditions. This, in turn, affects the adhesive strength and the quality of the work for wood bonding. Supporting small and medium-sized enterprises (SMEs) with similar studies is essential to improve the quality of their work.

Keywords − *Glue, timber, adhesion, wood mechanic, temperature*

Farklı Ortam Sıcaklıklarının Karaçam (*Pinus nigra*) Odununun Yapışma Performansına Etkisi

[1,2] Bolu Abant İzzet Baysal Üniversitesi, Mudurnu Süreyya Astarcı Meslek Yüksekokulu, Bolu, Türkiye

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Araştırma Makalesi

Öz − Bu çalışmada, ahşap tutkallarının yapışma performansı, sıcaklığa bağlı olarak değerlendirilmiştir. Karaçam (*Pinus nigra*) odun örneklerinin yapıştırılmasında, polivinil asetat (PVA) ve poliüretan (PU) yapıştırıcıları kullanılmıştır. Numunelerin yapışma performansı, oda sıcaklığının altı (4 °C), oda sıcaklığı (22 °C) ve oda sıcaklığının üstü (55 °C) olmak üzere üç farklı sıcaklık etkisi altında belirlenmiştir. Yapıştırıldıktan sonra numuneler el mengenesi ile sıkıştırılarak üç farklı sıcaklıkta kürlenmeye bırakılmıştır. Numunelerin mekanik karakterizasyonu için makaslamada çekme direnci (LSS-Load at Specified Strain), eğilme direnci (MOR- Modulus of Rupture) ve eğilmede elastikiyet modülü (MOE- Modulus of Elasticity) analizleri yapılmıştır. Elde edilen sonuçlara göre, LSS direnci oda sıcaklığının altında ve üzerinde artış göstermiştir. Buna karşın, MOR ve MOE güçleri, oda sıcaklığının altında ve üzerinde azalmıştır. Böylece, sıcaklığa bağlı olarak değişen yapışma mukavemetinin yanı sıra kuvvet uygulama yönünün de mukavemeti etkilediği sonucuna varılmıştır. Özellikle küçük atölyelerde ortam sıcaklığı yaz ve kış koşullarından büyük ölçüde etkilenir. Bu da ahşabın yapıştırılması için yapılan işin yapışma gücünü ve kalitesini etkiler. Küçük ve orta ölçekli işletmelerin (KOBİ'ler) benzer çalışmalarla desteklenmesi, yaptıkları işin kalitesinin artırılması açısından önemlidir.

Anahtar kelimeler − *Tutkal, kereste, yapıştırma, odun mekaniği, sıcaklık*

[1] orhankelleci@ibu.edu.tr
[2] esinkoksal@ibu.edu.tr
*Sorumlu Yazar / Corresponding Author

1. Introduction

After the trees are cut from the forest, they are converted into timber by cutting in the longitudinal and transverse directions (Lamb, 1998; Pirard et al., 2016; Ramage et al., 2017). The timber is reprocessed into small pieces for final use (Han et al., 2009; Susanty et al., 2020). These wooden parts are combined using various fasteners and techniques and transformed into finished products such as wooden structures, tools and furniture (Smardzewski, 2015; Ilgın and Karjalainen, 2021). Throughout history, various materials have been used to adhere the wood parts together such as rope and glues of animal or vegetable origin (Shields, 1985; Ülker, 2016).

Wooden material is preferred in furniture production due to its lightness, sound and heat insulation ability, and lack of corrosion (Kurtoğlu and Sofuoğlu, 2015). One of the most common forest trees in our country is the larch tree. In this study, black pine (*Pinus nigra* Arnold.) wood, which is frequently used in furniture and timber production, was preferred.

In recent years, synthetic wood glues have been developed to reduce costs, increase adhesion efficiency, and produce large quantities. In the wood industry, today, polyvinyl acetate (PVA) and poly urethane (PU) glues are the most used. Researchers frequently study these glues' performance. There are not many studies on the determination of the performance of wood glues under the effect of temperature. Some studies related to influence of temperature on the bonded wood reviewed below.

Kimeng et al. (2015) bonded *Funtumia africana* (Ire) wood using PVA and examined the adhesion performance at 0-30-40-50-70 and 100 °C. They reported that the compressive adhesion force decreased to 34.4 N/mm^2 at 0 °C, 41.2 N/mm^2 at 30 °C, and decreased above 30 °C. In another study, Clauss et al. (2011) investigated how the adhesion force was affected under thermal load by using PVA, PU and urea formaldehyde (UF) glues and *Fagus sylvatica* L. wood. They reported that LSS adhesion strength decreased as the temperature increased.

Obucina et al. (2015) studied *Fagus sylvatica* wood was glued under hot press using PVA glue and examined how the adhesion resistance was affected by temperature changes. As a result, they reported that the LSS strength increased with increasing pressing time and temperature. In another study, Tam et al. (2017) studied the adhesion performance of Carbon fiber-reinforced wood polymer (CFRP) at different temperatures using acoustic-laser technique and shear test analyses. As a result, they reported that adhesion was strong at low temperatures and that adhesion strength decreased as the temperature increased.

PVA emulsions are a type of thermoplastic polymer that has a glass transition temperature (Tg) close to room temperature. Even small changes in temperature near the Tg can significantly affect their mechanical properties such as modulus of rupture (MOR) and modulus of elasticity (MOE). This means that the quality of the bond is highly influenced by temperature (Motohashi et al., 1984). It is known that the physically hardened animal-based gluten glue and synthetic-based PVA emulsion glue soften again after the hardening of the temperature, and the adhesive bonds are dissolved (Altınok, 2002). It has been reported that when the temperature of the PVA emulsion glue wood joint is increased up to 60-80 °C, the hardened glue layer at the joint is dissolved (Corey et al., 1977). Altınok (2002) found that in the experimental samples glued with thermoplastic-based PVA and thermoset-based UF glues, the adhesion performance of wood samples decreases as the temperature of the holding environment increases.

At the interface between wood and glue, a certain amount of glue is required to penetrate into the microstructure of the wood (Frihart, 2009). Glue penetration partially affects bond strength. Insufficient penetration reduces the mechanical locking and performance of the glue. Excessive penetration creates a saturated interface and weakens adhesion (Hunt et al., 2018). Temperature is also an important variable that affects the adhesion performance of the glue. Temperatures of wood and ambient are the most important factors affecting production quality because temperature affects the glue adhesion quality.

In Northern Europe or Canada, it is recommended to perform wood adhesive applications at a standard temperature of 18 °C (EN 15497, 2014). Temperature-sensitive deformation is a distinct issue that holds significant importance in ensuring the safety of structural timber used beneath roofs in hot regions such as Mediterranean countries. These areas experience scorching under-roof temperatures during summer, which can soar up to 70 °C. Additionally, understanding the impact of temperature on structural behavior is crucial in the context of fire incidents. An increased amount of isocyanate in one-component polyurethane glues does give higher initial joint strength and lower creep at lower temperatures, namely up to 50 °C (Na et al., 2005).

Kollmann and Côté (1984) reported that cooled southern pine lumber wood improves MOR and MOE mechanical properties. According to Wang et al. (2015) concluded that temperature affects the performance of wood joints and shear strength decreases with temperature. Moussa et al. (2012) studied a low-temperature curing epoxy adhesive for carbon fiber-reinforced plastics for bridge constructions below 5 °C in the field. They concluded that the adhesive performance decreases with decreasing temperature. The curing temperature of adhesives significantly affects the mechanical properties (Moussa et al., 2012). LeBono et al. (2017) reported in their research that the adhesion performance decreases when the temperature decreases using acrylic adhesive. There is a European standard EN 15497 (2014) to ensure that the complete adhesive cure, which is a necessary safety condition in the production of wooden elements for bearing purposes, takes place within a certain period of time.

In this study, the mechanical characterizations of adhesives at temperatures below and above room temperature were investigated. Samples of PVA and PU adhesives were kept in a refrigerator at 4 °C for 24 hours to determine the adhesive strength in cold weather conditions. Additionally, samples were placed in an oven at 55 °C for 24 hours to determine the adhesive strength in hot weather conditions. Thus, it was aimed to determine the variations in adhesion strength of the adhesives at room temperature, below room temperature, and above room temperature.

2. Materials and Methods

2.1 Materials

2.1.1 Wood Materials

Black pine (*Pinus nigra*) wood samples were used to determine the adhesion performance of glues under the three different ambient temperatures. The woods were purchased from the local market. The wood was cut with a circular saw according to TSE 326-1 (1999) analysis standards after the thickness calibration.

2.1.2 Adhesives

PVA and PU glues were used to prepare the analysis samples. PVA glue was one-component D3 (TS EN 204, 2017) glue. PU was polyurethane-based D4 (TS EN 204, 2017) glue. The glues were purchased from the local market. PVA is produced by the polymerization of coal, lime, water, and vinegar acid. A mixture of coke and lime is heated in the oven to obtain carbide (CaC_2). By spraying water on top of the superheated carbide, acetylene gas (C_2H_2) is released, and vinyl ester is formed from the combination of acetylene gas and vinegar acid (CH_3CCOH). PVA is also obtained by polymerization of vinyl ester molecules (Baltaci, 2011). Polyurethane adhesives are synthesized through the reaction of a low molecular weight polymer possessing at least two -OH end groups with a diisocyanate. The polymers utilized in this process may include polyether, aliphatic polyesters, or polybutadiene. In the case of two-component polyurethane adhesives, the polymer and isocyanate components are blended and subsequently applied to the adherends, wherein hydroxyl groups on surfaces like paper, wood, or glass have the potential to engage in covalent bonding with the isocyanate, establishing a robust connection between the adhesive and substrate (Comyn, 2021).

2.1.3 Preparation of samples

Initially, wood samples were cut as large parts by 350 mm x 100 mm x 10 mm dimensions because the modulus of rupture (MOR) and modulus of elasticity (MOE) analysis samples were fairly small (300 mm x 20 mm x 20 mm). 200 g/m^2 glue was poured on the samples and spread over the surface by means of a sponge. Samples were compressed with a hand clamp. The samples were left to harden at 3 different ambient temperatures. The samples in Group 1 were kept at room temperature (22 °C), in Group 2 were kept in the refrigerator (4°C), and in Group 3 were kept in the oven (55 °C) for 24 hours to dry and harden. After the curing process, the analysis samples were cut again in the circular saw at the dimensions (300 mm x 20 mm x 20 mm) specified in the TSE and MOR samples were prepared. Lap sheer strength (LSS) analysis samples were produced with similar logic. Initially, large samples of adhesion were prepared. The samples were kept for 24 hours in 3 different ambient conditions at room temperature (22 °C), refrigerator (4 °C), and oven (55 °C). After the curing time, samples were cut as LSS analysis samples in a circular saw, in the dimensions specified in the TSE standard. Prepared samples are given in Table 1.

Table 1
Curing time and ambient temperature of the samples

Sample code	Description	Ambient temperature (°C)	Curing time (hour)
PVA-R	PVA glue at room temperature	22	24
PVA-F	PVA glue at fridge temperature	4	24
PVA-O	PVA glue at oven temperature	55	24
PU-R	PU glue at room temperature	22	24
PU-F	PU glue at fridge temperature	4	24
PU-O	PU glue at oven temperature	55	24

2.2 Methods

Adhesion characterization of the samples was carried out by modulus of rupture (MOR), modulus of elasticity (MOE), and lap shear strength (LSS) analyses according to TS EN 310 (1999) and, TS EN 310 (1999), TS EN 205 (2017) standards, respectively. Nine samples were prepared for each analysis. 9 repetitions were made to demonstrate the similarity (repeatability) of the analysis data obtained and to ensure the accuracy of the standard deviation and variance analyses. To ensure the accuracy of the analysis results, the analyzes are performed at least 5-6 times in the same laboratory, by the same researcher, on the same device (Merey, 2023). LSS test sample and diagram were given in Figure 1. LSS was calculated according to equation 1. Analyzes were performed without being acclimatized because the adhesion performances of the glues at different ambient temperatures were to be evaluated. The samples were placed in the Zwick tester as soon as the curing process was completed at the end of the 24 hours. The results were analyzed using the SPSS software package. One-way ANOVA was used to determine whether there were significant differences between the results (at the significance level of $p<0.05$). To find comparable groupings, Duncan's analysis was employed.

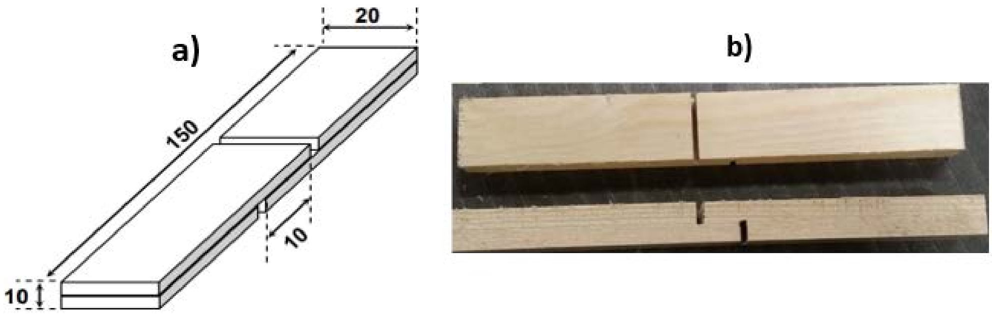

Figure 1. a) LSS diagram (dimension is mm), b) LSS test sample

$$LSS = \frac{Fmax}{A} = \frac{Fmax}{a\ x\ b} \qquad (1)$$

LSS: Single lap shear strength

A: Overlapped bonded surface

a: Length of bonded surface

b: Width of bonded surface

Fmax: Maximum force

3. Results and Discussion

3.1 Adhesive sticking properties

The mechanical strength analysis results are given in Table 2. When examining the MOR (Modulus of Rupture) strength, it was observed that the strength decreased at temperatures below and above room temperature. The highest MOR strength was obtained from the adhesive cured at room temperature. The lowest MOR strength was obtained from PVA-O and PU-F.

When LSS strength was examined, the highest strength in each sample was obtained from the samples kept in the refrigerator. The MOE strengths of the samples were also similar to the MOR strengths. They decreased below and above room temperature.

Table 2
Analysis results

Glue	Sample Code	MOR (N/mm^2)	MOE (N/mm^2)	LSS (N/mm^2)
PVA	PVA-R	74,35 (±7)* c	28852 (±152) ab	4,3 (±1,3) a
	PVA-F	72,34 (±8) a**	24895 (±125) a	8,3 (±1,4) b
	PVA-O	65,63 (±8) bc	23395 (±132) ab	5,5 (±1,6) a
PU	PU-R	77,55 (±6) bc	28311 (±175) ab	4,9 (±0,8) a
	PU-F	54,83 (±13) bc	21765 (±136) ab	8,9 (±3,3) b
	PU-O	69,38 (±18) b	23764 (±127) b	5,1 (±1,9) a

*: Standard deviation in parenthesis, **: Duncan analysis groups

When Figure 2 was examined, it is observed that decreasing temperatures increased the adhesive (LSS) strength. The LSS analyses were conducted at room temperature, and it is also considered that room temperature or other temperature could influence the LSS strength. In other words, the room temperature during the analysis is thought to affect the sample temperature and may change the LSS resistance.

The aim of this study was to determine how wood joints made with PVA and PU in a workshop were affected by ambient temperature. For this purpose, samples were kept at 4 °C in a refrigerator to assess the effects of cold ambient conditions on wood joints made in the workshop during the winter season. Subsequently, the analyses were conducted at room temperature to represent the in-use behavior of wood joints made with PVA and PU adhesives in a cold workshop.

In conclusion, this study sheds light on the behavior of wood joints made in a cold workshop environment, especially for workshops without adequate heating during the winter season.

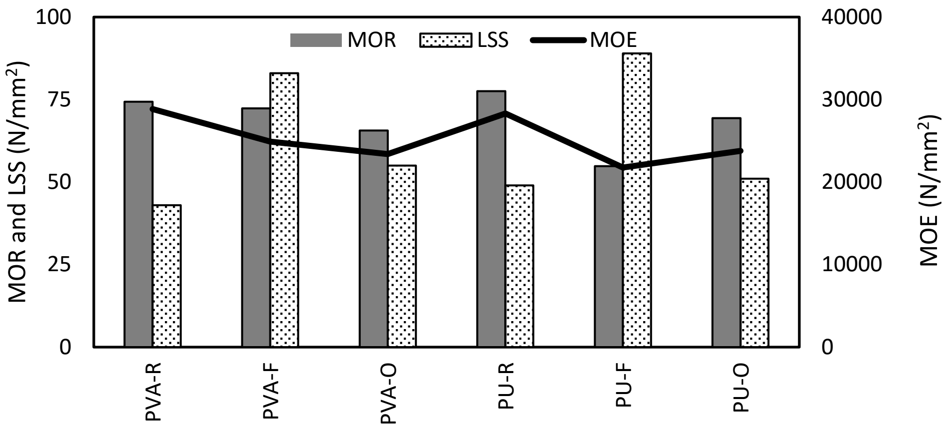

Figure 2. MOR, MOE, and LSS values of samples

Šedivka et al. (2015) examined the adhesion forces of 3 different PVA-based wood glues at different temperatures (between 20 and 140 degrees). After cooling the adhesion samples to the initial temperature, all tested samples complied with the minimum strength, i.e. 10 MPa. They reported that wood samples joined using PVA adhesives showed that the adhesion resistance increased over time. However, in our study, the adhesion strength of PVA glue to wood was examined at below room temperature, at room temperature and above room temperature.

In a study it was reported that the adhesives cured by temperature show shrinkage in the adhesion zone when the temperature reaches room temperature (Watts et al., 2002). Shrinkage can be 5 % in epoxy adhesives and 15 % in acrylic adhesives (Yu et al., 2014). In our study, curing does not occur with temperature. No hot press or temperature effect was used in the study. MOR and MOE properties of wood bonded slightly above and below room temperature (15-20 degrees) were examined. PVA and PU adhesives do not have a shrinkage issue by volume. Also, Coppendale (1977) reported that the shrinkage amount of adhesives cured at room temperature is negligible.

The adhesion strength of can be increased by mixing glues used at high temperatures (HTA) and low temperatures (LTA), but attention should be paid to the geometry and ratios between LTA and HTA for behavior on the adhesion surface composed of HTA (Hart-Smith, 1973). In our study, it is also believed that a combination of PVA and PU adhesives can be used to produce an adhesive that is resistant to both cold and hot conditions.

The hardening in the glue takes place purely physically, and when the temperature increases, the hardening time decreases. At room temperature, the minimum pressing time is 30 minutes. In hot pressing, a maximum temperature of 80 °C and a pressing time of 8–10 minutes are applied. At temperatures above 80 °C, the glue dissolves and does not harden. After hot pressing, up to 50 °C, the work piece must remain tight in the press (Karaaslan, 2004; Ordu, 2007).

Thermal expansion affects the curing time of adhesives. Bal et al. (2012) studied the thermal expansion coefficients of solid wood and laminated wood materials made from beech, poplar, and eucalyptus. They found that poplar laminated wood had a higher thermal expansion coefficient in the tangential direction than solid wood, while there was no significant difference between solid wood and laminated wood materials in beech and eucalyptus. The study highlighted the importance of considering the specific wood species and construction materials when assessing thermal expansion properties.

Indeed, the prolonged durability of engineered wood flooring is crucial, especially considering the influence of heat and moisture fluctuations. Blanchet (2008) revealed that most adhesive, except for epoxy adhesive, demonstrated comparable performance. The utilization of epoxy adhesive resulted in notable inconsistencies and delamination in the structures.

4. Conclusions

This study investigated the relationship between ambient temperature variations and adhesive strength of PVA and PU adhesives using black pine wood samples. The mechanical characterization of the bonded samples was conducted through LSS (Lap shear strength), MOR (Modulus of Rupture), and MOE (Modulus of Elasticity) analyses.

According to the results obtained, it was found that even at temperatures below room temperature (4 °C), the LSS strength was close to the level at room temperature and above. Moreover, at temperatures above room temperature (55 °C), the LSS strength is higher. This trend is similar for both PVA and PU adhesives.

However, the MOR strengths of the samples differ from the LSS strengths. It was observed that there is a decrease in the MOR strengths of the adhesives at temperatures below and above room temperature. The change in MOR strength is consistent with the change in MOE strength.

In conclusion, the ambient temperature variations affected the adhesive strength, but the effect depend on the direction of the force. The adhesive strength against a force applied parallel to the surface (LSS) increases above and below room temperature, while the adhesive strength against a force applied perpendicular to the surface (MOR) decreases.

In summary, the study demonstrated that ambient temperature variations influence the adhesive strength of PVA and PU adhesives, but the direction of the force plays a role in determining how the adhesive strength changes with temperature. MOR and MOE are important parameters for wood products produced in small and medium-sized enterprises (SMEs). For this reason, the temperature of the production area should not be above or below room temperature. Therefore, necessary precautions should be taken to adjust the ambient temperature. Additionally, doors used for workshop entrances and exits should not be opposite each other. The fact that the doors are opposite each other causes air flow. This means that if the wooden material in the workshop is not stacked properly, it causes the wood to change size by shrinking and expanding. More research should be conducted in this area to improve the adhesive strengths of wood adhesives.

Authors contribution

Orhan Kelleci: Conceptualization, Methodology, Data curation, Formal analysis, Writing-original draft. Süheyla Esin Köksal: Conceptualization, Methodology, Data curation, Formal analysis, Writing-original draft.

Conflict interests

The authors have no conflicts of interest to declare that are relevant to the content of this article.

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