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## Results of experimental studies on the strength of adhesive-bonded joints of thermally modified ash wood

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**Abstract.** It is necessary to ensure the protection of the adhesive-bonded wood structures from the effects of temperature and humidity loads to improve their performance characteristics and increase the service life – this determines the relevance of this study. The purpose of the study is to examine, using a long-term method, the strength of thermoplastic adhesive-bonded joints of thermally modified ash wood and unmodified pine wood. The data were subjected to statistical processing and analysis. The experimental samples for the studies were prepared according to the proposed methodology in production conditions according to the existing technological process. The studies continued for two years, with periodic inspection of the samples and recording of the changes that occurred during the experiments. Regularly, every three months of the study, part of the samples was removed from the stand and tested to determine the change in strength. Methods were selected and described for conducting long-term experimental studies to determine the strength of adhesive joints of thermally modified ash wood and unmodified pine wood glued with polyvinyl acetate-based adhesives with durability class D4. Based on the study results, it was identified that the average strength of control samples of the thermally modified ash wood and unmodified pine wood was 7.12 MPa, and after two years of long-term testing, it decreased to 5.13 MPa (27.9%). In addition, it was identified that the operation of such adhesive-bonded joints in natural conditions depends on the cyclic temperature-and-humidity load. Namely, in

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the summer-autumn period, the strength decreases, while in the winter-spring period, it remains unchanged, and in some cases, increases. The obtained results are of great practical importance, since such adhesive-bonded structures, can be used for the manufacture of joinery and building products, which will improve their performance characteristics and increase their service life

**Keywords:** long-term method; pine; atmospheric factors; temperature-and-humidity loads; service life; adhesion

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## Introduction

Wood is an anisotropic, fibrous structural material that has certain advantages over other materials. L. Kristak *et al.* (2021), Y. Li *et al.* (2021), R. Afshar (2022) described that there are also disadvantages that limit its use in conditions with variable temperature and humidity loads. To eliminate these shortcomings and for rational use of wood, it is subject to modification (especially low-value and low-grade varieties). In the studies by Sandberg *et al.* (2017), D. Jones & D. Sandberg (2020), Repič *et al.* (2022), several methods of wood modification were examined, one of which is thermal modification. As a result of this modification, physical and mechanical properties of the wood change, including increased resistance to variable humidity and temperature conditions of operation compared to unmodified wood (Heat treatment..., 2017; Hill *et al.*, 2021; Giridhar & Pandey; 2022).

Therewith, there is a problem with gluing such wood because during the modification process, exposure to high temperatures changes the chemical composition, physical and mechanical properties of wood, including its adhesive properties (Candelier *et al.*, 2017; Kozakiewicz *et al.*, 2020; Zigon *et al.*, 2020). To date, several studies were conducted (Biazzon *et al.*, 2019; Chang *et al.*, 2019) regarding the bonding of thermally modified wood together. However, there are no studies on the bonding of thermally modified wood with unmodified wood. This problem cannot be solved without thorough research in this area.

P. Král *et al.* (2015) note that for bonding thermally modified and unmodified wood together, both thermosetting (phenol-formaldehyde, urea-formaldehyde, polyurethane, etc.) adhesives and thermoplastic adhesives (polyvinyl acetate) are used. Thermosetting adhesives better withstand variable humidity and temperature loads of the environment, cure quickly, form a mesh structure of the adhesive joint, and are relatively inexpensive. This ensures proper water, humidity, and heat resistance of the adhesive joints. However, they are environmentally dangerous both in the process of preparing glue and during the operation of finished products. In addition, such adhesives have a high percentage of moisture absorption and low adhesive properties to thermally modified wood, which does not allow the adhesive joints to provide proper strength under operating conditions. Kshyvetskyi *et al.* (2019) state that modern thermoplastic (polyvinyl acetate) adhesives have good adhesive properties to wood, provide proper strength of adhesive joints, and are eco-friendly and easy to use, which makes them promising for use.

To solve these problems, the authors decided to conduct experimental studies to examine the adhesive strength of adhesive-bonded joints of thermally modified ash wood and unmodified pine wood glued with thermoplastic polyvinyl acetate adhesives with durability class D4 (Datskiv & Kshyvetskyi, 2020).

The purpose of the study was investigating changes in the strength of thermoplastic adhesive joints of thermally modified ash wood and unmodified pine wood by the long-term method.

The object of the study is adhesive joints of thermally modified ash wood and unmodified pine wood. The subject of the study is the strength of thermally modified ash wood and unmodified pine wood glued together with thermoplastic adhesives.

The following tasks were set to achieve this goal:

1. Describe the method of conducting long-term experimental studies on the strength of adhesive joints of thermally modified ash wood with unmodified pine wood.

2. Conduct experimental studies to determine the strength of adhesive joints of thermally modified ash wood and unmodified pine wood by applying the long-term method.

3. Analyse the obtained results on changes in the strength of adhesive joints of thermally modified ash wood and unmodified pine wood.

Bonding of thermally modified wood and unmodified wood is promising for the joinery and construction industry, mainly in the manufacture of window blocks, since there is a problem with their durability during operation, especially outdoor operating conditions. Modern paint and varnish materials are not able to provide proper operating conditions for windows with prolonged negative effects of external factors. This problem can be solved by using an adhesively-bonded structure that combines thermally modified wood and unmodified wood.

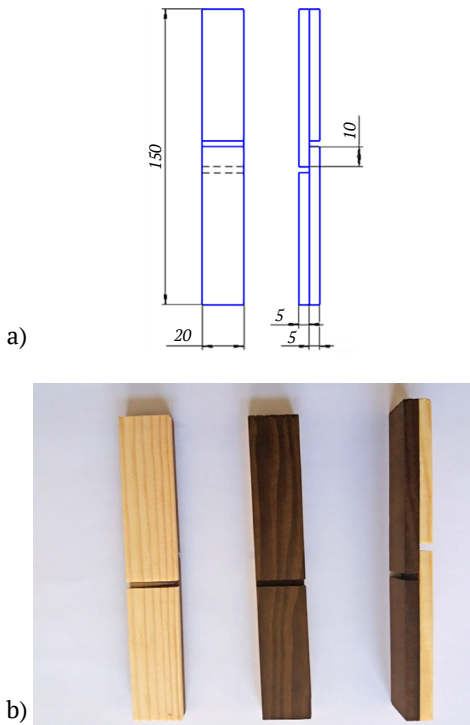
## Materials and Methods

The experimental studies determining the strength of adhesive-bonded joints of thermally modified ash wood and unmodified pine

wood were conducted in accordance with the requirements of tests in natural conditions (from November 7, 2019, to November 7, 2021), and the samples were prepared in accordance with the DSTU EN 205:2014 standard (2014). For bonding, a thermoplastic adhesive based on polyvinyl acetate RAKOLL ECO 4 (Germany) with durability class D4 was used.

The formation of adhesive joints was conducted on the basis of the Long Life Wood enterprise (Kyiv, Ukraine) under production conditions using the existing technological equipments. Thermal modification of ash wood was conducted in an autoclave for 12 hours at a temperature of 195°C. After the thermal modification, the samples were conditioned (18 hours).

The first stage was the manufacture of blanks (lamela), the dimensions of which were 600x130x5 mm. They were calibrated to a thickness of  $5\pm 0.1$  mm before bonding. The moisture content of pine wood slats was  $12\pm 1.5\%$  and that of thermally modified ash wood –  $6\pm 0.5\%$ . A laboratory moisture analyzer RAD WAG WPS 110 S (Poland) was used to determine the moisture content of the workpieces. The glue was applied manually with a brush. The adhesive was thoroughly stirred up before application until a homogeneous mass was obtained. The glue consumption was  $160\text{ g/m}^2$ . The viscosity was measured using a VZ-4 viscometer and adjusted to the requirements of the bonding mode parameters (70 seconds). After applying the glue, a workpiece was being pressed for 30 minutes at a temperature of +20°C using a pneumatic clamping machine. Technological exposure after bonding was 7 days. The next stage was the formation of samples of the appropriate shape and size according to the DSTU EN 205:2014 standard (2014). The dimensions of the experimental sample were 150x20x10 mm (Fig. 1).



**Figure 1.** Diagram and samples for determining the strength of the adhesive joint of thermally modified ash wood and unmodified pine wood (a) sample diagram; b) experimental samples)

**Source:** compiled by the authors

**Methodology for conducting long-term experimental studies.** A long-term (natural) method was used to examine the strength of adhesive joints of thermally modified ash wood and unmodified pine wood, which allows for assessing the change in the strength of adhesive joints of wood under the influence of atmospheric factors. This method of research is time- and labour-consuming, but more reliable. The results obtained using the long-term method allow considering the real impact of natural factors.

The shapes and sizes of the samples met the DSTU EN 205:2014 standard (2014). Before the study, a stand was made for testing samples, a glue

was prepared, samples were made, a stand was installed, and the samples were fixed on it (Fig. 2).



**Figure 2.** Stand for testing experimental samples in atmospheric conditions

**Source:** compiled by the authors

All the experimental samples that were used for long-term studies were divided into two parts. One part of the samples was placed on the test stand in atmospheric conditions according to the requirements described above, and the other part was used as controls that were not exposed to natural factors and were used to determine the initial strength of adhesive joints. The number of samples in one batch was determined by the conditions for conducting experimental studies.

The prepared samples were placed on a stand shelf at a distance of 20 mm from each other to ensure air circulation. The stand with the samples was fixed at a height of three meters from the ground level. The studies in atmospheric conditions were conducted for two years. Every three months from the start of the study, namely after 3, 6, 9, 12, 15, 18, 21, and 24 months, a batch of samples was removed from the stand and subjected to tests.

Changes in atmospheric factors (average daily humidity and ambient temperature) were recorded during the entire experiment. In addition, changes in the appearance of the samples, of their size and shape were periodically recorded.

The strength of adhesive joints of thermally modified ash wood and unmodified pine wood glued with thermoplastic glue was determined by stretching along fibres with a constant load (50 mm/s). The long-term testing of the samples was conducted on a break-

ing machine of the R-05 brand (Fig. 3). The results obtained after the long-term tests were recorded in the observation log. After that, statistical processing was performed on the basis of which graphical dependencies were constructed.



Figure 3. Breaking machine R-05

Source: compiled by the authors

### Results and Discussion

The obtained results of the experimental studies on the strength of adhesive joints of ther-

mally modified ash wood and unmodified pine wood by using the long-term method are presented in Table 1.

Table 1. Strength of thermally modified ash wood and unmodified pine wood glued with polyvinyl acetate adhesives

No.	Adhesive joint strength, MPa								
	Initial strength	Duration of experiment, months							
		3	6	9	12	15	18	21	24
1	7.05	5.95	6.00	4.80	5.00	5.55	4.75	6.75	5.50
2	6.55	6.90	6.15	6.25	5.50	4.80	6.80	5.75	4.75
3	8.25	5.63	6.25	4.75	7.63	6.75	5.50	6.65	4.50
4	6.75	5.85	8.00	5.00	6.65	5.80	6.75	4.80	6.00
5	6.00	6.38	6.75	6.25	5.50	5.30	5.50	5.00	5.50
6	6.85	6.25	6.85	7.37	5.50	5.60	5.75	6.25	4.35
7	7.25	6.85	5.40	4.74	7.00	7.00	6.50	5.00	4.74
8	7.00	6.25	6.75	7.75	7.00	6.05	5.40	5.45	6.25
9	7.65	6.55	5.60	5.50	6.50	5.75	5.75	5.45	5.00
10	7.65	7.20	6.75	4.75	4.75	5.95	5.25	6.25	5.25
11	6.75	6.25	5.50	5.25	7.00	5.75	7.25	5.50	5.75
12	6.00	6.15	7.35	7.00	4.00	4.70	5.75	4.50	4.45
13	8.00	5.00	6.00	4.50	5.50	5.65	7.00	5.75	6.00
14	7.25	6.50	7.75	6.75	6.50	6.25	5.35	5.15	5.00

Table 1, Continued

No.	Adhesive joint strength, MPa								
	Initial strength	Duration of experiment, months							
		3	6	9	12	15	18	21	24
15	8.35	5.80	7.10	6.75	4.50	6.50	5.25	6.00	6.00
16	6.90	5.25	7.10	4.75	6.00	5.50	6.50	5.15	4.45
17	6.55	6.25	8.25	7.25	5.25	6.25	6.25	5.25	4.50
18	7.25	7.11	6.60	7.75	4.21	6.05	6.50	5.75	5.75
19	6.40	5.70	5.00	4.75	6.00	6.65	5.50	5.25	5.50
20	8.00	7.11	6.50	5.75	6.25	5.25	6.30	6.35	4.50
21	7.55	5.50	6.20	7.00	6.25	5.80	5.75	6.00	4.10
22	6.95	6.20	5.75	6.00	4.75	6.15	6.25	5.35	4.30
23	6.78	6.80	7.75	7.50	7.25	6.65	5.75	5.75	5.50
24	6.95	7.55	6.50	6.25	5.45	6.10	4.75	5.35	4.50
25	7.25	7.50	6.25	6.75	5.25	5.05	6.25	6.10	6.00
<b>Avg.</b>	<b>7.12</b>	<b>6.34</b>	<b>6.56</b>	<b>6.05</b>	<b>5.81</b>	<b>5.87</b>	<b>5.93</b>	<b>5.62</b>	<b>5.13</b>

Source: compiled by the authors

Figure 4 shows a graphical interpretation of changes in the average strength of thermally modified ash wood and unmodified pine wood during two years of natural testing.

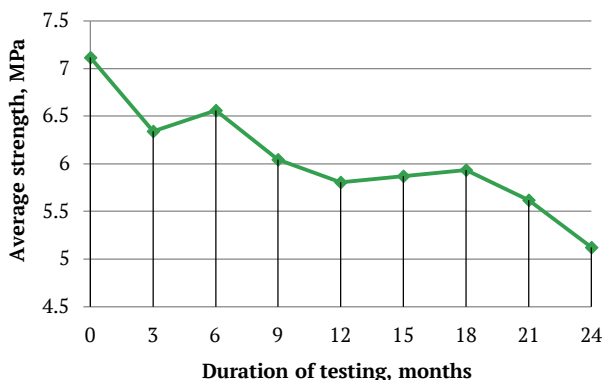


Figure 4. Change in the strength of adhesive joints during two years of testing

Source: compiled by the authors

The results of the long-term experimental studies—on the strength of adhesive joints of thermally modified ash wood and unmodified pine wood glued with thermoplastic polyvinyl acetate adhesives showed that the change in the

strength of such adhesive joints occurs along a sinusoidal curve. Namely, the average strength of the control samples is 7.12 MPa. The average strength after 24 months of testing decreased to 5.13 MPa, which is 27.9% as a percentage.

This reduction in strength is not critical for adhesively-bonded structures operating in a variable temperature and humidity environment.

The change in the strength of such adhesive joints will be further analysed step by step in more detail according to the methodology of experimental studies, namely every three months of operation in natural conditions. According to the experiment plan, the samples were put in a stand on November 7, 2019. The first batch of the samples was removed from the stand and destroyed three months later. Each subsequent batch was removed similarly every three months after cyclic temperature and humidity tests, namely after 6, 9, 12, 15, 18, 21, and 24 months. According to the tests, the average strength was: 6.34 MPa, 6.56 MPa, 6.05 MPa, 5.81 MPa, 5.87 MPa, 5.93 MPa, 5.62 MPa, 5.13 MPa. That is, the strength decreases gradually with increasing duration of the experiment.

Therewith, as can be seen from Figure 4, the strength of adhesive joints of thermally modified ash wood and unmodified pine wood glued with thermoplastic polyvinyl acetate adhesives varies depending on the time of year. Namely, in the summer, the strength decreases with increasing test duration, which is not observed in winter. As can be seen from the graph, after testing in winter, the strength of adhesive joints does not decrease, but on the contrary, increases slightly. For example, analysing the period from 3 to 6 months of testing, the strength of such a joint changes from 6.34 MPa to 6.56 MPa in three months of testing, which is 0.22 MPa more than the strength that was after 3 months of testing. The same pattern is observed after 15 and 18 months of operation. The strength changes from 5.87 MPa to 5.93 MPa, which is 0.06 MPa higher than the fifteen-month test period.

In accordance with the requirements for wood adhesive joints, the bonding strength after experimental tests is considered satisfactory if

its numerical value decreases by less than 50%. In this case, this requirement is observed. Therefore, it can be assumed that the adhesive bonding of thermally modified ash wood and unmodified pine wood glued with thermoplastic polyvinyl acetate adhesives can be used for structures that will be operated in a variable temperature and humidity environment without any physical stress.

However, the strength of such adhesive joints is lower than that of unmodified ash wood with unmodified pine wood. The strength of such adhesive joints, according to the previous studies, should be within the strength limits of pine wood.

The behaviour of such an adhesive bonding can be explained by physical and chemical changes in thermally modified ash wood caused by the action of elevated temperatures. Namely, the action of temperature leads to the breakdown of hemicellulose and cellulose to form furfural, which affects the bonding surface of ash wood. Furfural leads to the formation of a smooth bonding surface, which negatively affects the adhesive properties of adhesives and thereby reduces the strength of the adhesive joint. Therewith, the increased temperature leads to a change in the mechanical properties of such wood (Zelinka *et al.*, 2022). That is, it makes it more brittle and changes its structure, which negatively affects the penetration of glue into the wood.

B. Kshyvetsky *et al.* (2019) note that glueing is one of the most common types of wood bonding used in the manufacture of joinery boards, furniture boards, facing panel materials, window and door blocks, floor coverings, etc. This method of joining is based on the phenomena of adhesion, that is, the ability to form various kinds of bonds between glue and wood.

Adhesives for gluing thermally modified wood, which would provide proper adhesive strength under operating conditions, currently practically do not exist. Therefore, there is a problem with gluing thermally modified wood and finding new adhesives.

A. Can *et al.* (2021) examined the strength of adhesive joints of thermally modified pine, which was first dried at a temperature of 103°C, and then modified in the temperature range from 190°C to 212°C for 2 hours. Polyvinyl acetate glue with a 195.54 hardener was used to form the adhesive joint. The strength of such adhesive joints decreased by 31-39% compared with the initial strength. H.R. Taghiyari (2013) concluded that this change in strength occurs due to a decrease in the wetting index, oxidation of the wood surface, changes in the chemical and physical properties of wood, and a substantial decrease in the number of polar groups.

M. Ilkiv *et al.* (2017) investigated the strength of adhesive joints of thermally modified ash wood glued with polyvinyl acetate glue. Thermal modification of wood was conducted for 4 hours at a temperature of 160°C and 220°C. The strength of the adhesive joints of the treated ash wood at a temperature of 160°C is 3.45 MPa, and at a temperature of 220°C – 6.34 MPa. The authors explain this change in strength through physical and chemical changes that occur in the wood when exposed to high temperatures. The increase in strength occurs due to an increase in the lignin content due to the crosslinking reaction. This creates more intermolecular bonds between the wood and the adhesive.

Z. Vidholdová *et al.* (2021) investigated the strength of adhesive joints of thermally modified spruce, which was dried at a temperature of 100 °C, and then subjected to thermal modification at temperatures of 160°C, 180°C, 200°C, and 220°C for 4 hours. Polyvinyl acetate and polyurethane adhesives were used for bonding. Forming adhesive joints, dried wood and thermally modified wood were combined. The bonded structures made only from thermally modified wood showed a substantial reduction in strength compared to the bonded structures that were formed from dried wood and thermally modified wood. The authors concluded that

this change in strength is associated with the degradation of amorphous polysaccharides under the influence of high temperatures, which leads to a decrease in the number of polar groups in the cell walls of the wood.

Similar results for bonding thermally modified wood are also presented in the papers (Uzun *et al.*, 2016; Andromachi & Ekaterini, 2018; Taghiyari *et al.*, 2020). The authors noted that the decrease in strength is affected by wetting ability, microcracks, density, surface of thermally modified wood, and the physical-chemical characteristics of the adhesive.

The results were obtained using two methods, namely: long-term (natural) and accelerated (laboratory). As for the first long-term research method, it was conducted for the first time and deserves special attention, since it was conducted in the natural conditions of the Western region of Ukraine for two years. The obtained results reflect the factual behaviour of such adhesive joints under prolonged varying temperature and humidity loads. Therewith, the results of the studies of changes in the strength of adhesive joints of thermally modified ash wood and unmodified pine wood allow for building a mathematical model for predicting strength from the action of ambient temperature and humidity in the future. The accelerated method of experimental studies using various types of wood, especially thermally modified wood, is rare in adhesive structures. In the studies (Biazzon *et al.* 2019; Chang *et al.* 2019; Can *et al.* 2021;), the authors examined changes in the strength of adhesive joints of several-species of wood, using various adhesive materials, including polyvinyl acetate composition. Therefore, this experimental study cannot be compared with the results obtained by other authors, since they had different research conditions, namely the temperature of thermal modification of wood, test conditions, etc. In general, it can be noted that the dynamics of changes in the strength of adhesive

joints in this study and studies by other authors occur according to similar dependencies.

### Conclusions

Based on the results of long-term experimental studies of the strength of adhesive joints of thermally modified ash wood and unmodified pine wood glued with thermoplastic polyvinyl acetate adhesives, the following can be concluded:

1. A method for conducting long-term experimental studies in natural conditions was selected for thermoplastic polyvinyl acetate adhesive joints of thermally modified ash wood and unmodified pine wood based on Ukrainian and foreign standards. Samples were prepared for experimental studies.

2. It was found that the strength of adhesive joints of thermally modified ash wood and unmodified pine wood decreased by 27.9% after two years of testing and long-term experimental studies. Namely, it decreased from 7.12 MPa to 5.13 MPa.

3. It was found that the change in the strength of adhesive joints of thermally modified

ash wood and unmodified pine wood in different periods of operation does not change in the same way, namely, in summer, the strength decreases in a descending curve depending on the duration of operation, and in winter, this pattern is disrupted since the strength does not decrease, but on the contrary, increases. On average, it increases by 1%.

4. Based on the results of the long-term experimental studies, it was found that adhesive joints of thermally modified ash wood and unmodified pine provide operational characteristics in a variable temperature and humidity environment.

In the future, it is planned to conduct studies on the strength of adhesive joints of other species of thermally modified wood.

### Conflict of Interest

The authors declare no conflict of interest.

### Acknowledgements

None.

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## Результати експериментальних досліджень міцності клейових з'єднань термічно модифікованої деревини ясеня

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**Анотація.** Для покращення експлуатаційних властивостей та підвищення довговічності клейовим деревинним конструкціям необхідно забезпечити їх захист від дії впливу температурно-вологісних навантажень, що і зумовлює актуальність цього дослідження. Метою роботи було дослідження міцності термопластичних клейових з'єднань термічно модифікованої деревини ясеня і сосни немодифікованої за тривалим методом. Дані піддавались статистичній обробці та аналізу. Експериментальні зразки для дослідження були підготовлені згідно з запропонованою методикою у виробничих умовах відповідно до існуючого технологічного процесу. Дослідження тривали протягом двох років з періодичним оглядом зразків та фіксацією змін, які проходили під час випробувань. Регулярно після кожних трьох місяців досліджень частину зразків знімали зі стенду і проводили випробування для визначення зміни міцності. Підбрано та описано методику проведення тривалих експериментальних досліджень з визначення міцності клейових з'єднань термічно модифікованої деревини ясеня і сосни немодифікованої, клеями на основі полівінілацетату із класом довговічності D4. За результатами досліджень встановлено, що середня міцність контрольних зразків термічно модифікованої деревини ясеня і сосни немодифікованої становила 7,12 МПа, а після двох років тривалих випробувань зменшилась до 5,13 МПа (27,9 %). Крім того встановлено, що експлуатація таких клейових з'єднань у природних умовах залежить від циклічного температурно-вологісного навантаження. А саме, у літньо-осінній період міцність зменшується, а зимово-весняний залишається незмінною а в деяких випадках і підвищується. Отримані результати досліджень мають важливе практичне значення, оскільки такі клейові конструкції, можуть використовуватись для виготовлення столярно-будівельних виробів, що дозволить підвищити їх експлуатаційні характеристики та збільшити довговічність

**Ключові слова:** тривалий метод; сосна; атмосферні фактори; температурно-вологісні навантаження; довговічність, адгезія