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PREDICTION OF THE STRENGTH OF OAKWOOD ADHESIVE JOINTS BONDED WITH THERMOPLASTIC POLYVINYL ACETATE ADHESIVES

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Abstract. Among the several kinds of thermoplastic adhesives, structured and non-structured polyvinyl acetate (PVA) adhesives have a rather wide application and are used currently for forming adhesive joints from different wood species, especially oakwood. To ensure proper conditions of oakwood adhesive joints use, it is important to have fast and accurate methods of predicting their strength and durability. The strength changes of the oakwood adhesive joints bonded with structured and non-structured PVA adhesives have been investigated by conducting long-term experiments. Based on the generalization of experimental data and theoretical predictions regarding the mechanism of the adhesive seam formation, equations that allow calculating theoretically the strength of oakwood adhesive joints bonded with non-structured and structured PVA adhesives have been proposed. The proposed equations reproduce experimental data with sufficient accuracy of ±3.5 % within the temperature range from 251 K to 306 K and humidity range from 40 % to 100 %, and therefore, are recommended for practical use.

Keywords: polyvinyl acetate adhesives, adhesive wooden joint, strength, durability, adhesive seam.

1. Introduction

Thermosetting and thermoplastic adhesives are polymeric materials that are widely used in the wood processing and furniture-manufacturing industries. Thermosetting adhesives (resins) such as phenol-formaldehyde, urea-formaldehyde, melamine-formaldehyde, melamine-urea-formaldehyde, resorcinol-formaldehyde as well as phenol-resorcinol formaldehyde, which forms adhesive joints with increased water-, moisture- and heat resistance, are the main adhesives presently used for bonding wood.[1-3] However, such adhesives are toxic and detrimental to human health, they can sorb water, and the adhesive seam is brittle.[4] Nowadays, thermoplastic adhesives, such as polyvinyl acetate, polyvinylidene chloride, and others, which have good adhesive properties to wood and possess themselves as environmentally friendly materials, are widely used.[5-6] However, their moisture and humidity resistance is inferior to thermosetting adhesives.[7]

Among thermoplastic adhesives used currently in wood processing and furniture-manufacturing industries, thermoplastic polyvinyl acetate (PVA) adhesives, which are mostly dispersed in the form of emulsions, have a rather wide application due to the advantages they possess, including water solubility, ease of application, low-curing temperature, satisfactory adhesion properties regarding wood and wood-based materials, satisfactory water resistance and thermal stability, non-toxic and nonflammable properties, competitive cost.[8-10] The main film-forming component of the PVA adhesives is polyvinyl acetate – an amorphous linear or branched polymer, the physical and mechanical properties of which depend on the degree of the polymer branching: the glass transition temperature of the polymer is 301–315 K, the tensile strength is 20–50 MPa, the elongation is 10–20 %, the density is 1.19 kg/m^3, and the softening temperature is 303–323 K.[11-12] Curing occurs due to the adsorption of the solvent by materials that are being bonded together and solvent evaporation, or due to the transition of the polymer during cooling from a viscous fluid to a glassy state. PVA adhesives are one- and two-component substances and are capable to form a linear or thin mesh structure of the adhesive seam. Non-structured PVA adhesives are one-component adhesives that form a linear structure of the adhesive seam and the adhesive wooden joint meets the requirements of water resistance class D2 according to EN 204. Structured PVA adhesives are one- and two-component adhesives that form a thin mesh structure of the adhesive seam and the adhesive wooden joint meets the requirements of water resistance class D4 according to EN 204.

Wooden joints, bonded with PVA adhesives, are used indoors and outdoors – in natural conditions under

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the direct influence of air humidity and ambient temperature. The requirements for such adhesives and adhesive joints have become increasingly stringent in recent years. They should provide adequate strength (σ) and durability (τ) during the long-term service of the structure in variable environmental conditions, and be environmentally friendly.[13]

In general, several different factors affect the strength and durability of adhesive wooden joints such as the properties of the adhesive in its application (*e.g.*, penetration behavior and surface free energy) and curing states (*e.g.*, creep and stiffness); the basic physical and structural properties of wood species (moisture content, density, porosity, properties of the cell wall, cellular and extracellular spaces); gluing conditions (curing method, curing time, ambient temperature and humidity).[14-15] These factors significantly impact the formation of the interfacial bond between the adhesive and wood species, thus determining the forming strength of the adhesive seam, and, therefore, the initial strength of the adhesive joint.[13] It should be noted, that the structure and chemical composition of wood species significantly affect the adhesion properties of adhesives and the initial and operational strengths. There is a direct correlation between the strength of the adhesive joints of different wood species made under the same gluing conditions and the density of wood, moisture content, the ratio of the earlywood and latewood zones in the annual rings, chemical composition, *etc.* If we consider wood as a substrate, then the cohesive properties of even one species are different, which is explained by the heterogeneity of the structure, porosity, hydrophilic behavior, moisture content, *etc.* The last category of influencing factors refers to the stresses to which the adhesive joints are subjected in the working environment, affecting the strength and durability losses, and therefore forming residual strength and durability. Humidity and temperature are the most important factors in determining the strength loss of the adhesive joint exposed to the working environment.[13]

Currently, oak (*Quercus*) belongs to the main forest-forming species in Ukraine (the forest cover is about 28 %) and it is commonly used in the wood processing and furniture-manufacturing industries. Oak is a hardwood broadleaved ring-porous species with unique characteristics and physical and mechanical properties, namely, an average density of 680 kg/m^3, average shear strength of 10.2 MPa, average radial hardness of about 67.5 MPa and it is resistant to environmental conditions. Therefore, this wood species is the most suitable material for being glued in woodworking.

For the widespread and varied use of oakwood adhesive joints, it is important to have fast and accurate methods of predicting their strength and durability to ensure proper conditions of their use. To date, there are no equations in the literature for predicting the strength of glued wooden joints, created based on the generalization of the results of long-term experimental studies. Instead, the strength prediction of adhesive wooden joints is proposed to be carried out based on empirical equations or with the help of complex mathematical calculations, which do not give reliable results and are inconvenient for use in industrial conditions. In addition, such mathematical calculation methods don't consider several factors that affect the adhesive wooden joints during their operation.

Therefore, this work aims to study the strength and durability of oakwood adhesive joints bonded with structured and unstructured PVA adhesives; and to propose equations for predicting the strength of the latter in a wide range of temperatures and humidity under operational conditions.

2. Experimental

2.1. Materials

Oakwood samples (a total of 1260 with dimensions of 50×20×20 mm and 50×20×10 mm and both radial and tangential areas for bonding), were cut from solid wood planks of oak according to the standard. Knots, cracks, or other damages and defects were not allowed in the wood samples. The oakwood samples were held for three months in a climatization room at a temperature of 293 ± 2 K and relative humidity of 65 ± 3 % until the constant weight was reached. The sample's average moisture content was around 10 ± 2 % (the optimal moisture content of wood for bonding by most adhesives). The surface of the samples was not specially treated before bonding, and its roughness of was about 63 μm. Thermoplastic PVA adhesives were used for bonding oakwood samples: non-structured with water resistance class D2 and structured with water resistance class D4.

2.2. Experimental Procedure

2.2.1. Bonding and testing procedure of oakwood samples

The oakwood samples were bonded with thermoplastic PVA adhesives according to the EN 204 standard. Bonding was performed in two stages using open exposure and pressing to promote good impregnation of the adhesive bonding. Technological parameters of the bonding mode: adhesive consumption 160 g/m^2 (normative); duration of open exposure 7–10 min; specific pressing pressure 0.6 MPa; pressing time 40 min; bonding temperature 293 ± 2 K; moisture content of oakwood samples 10 ± 2 %. Such joints form adhesive films with a thickness from 0.4 mm up to 0.8 mm. The shape and dimensions of bonded oakwood samples are shown in Fig. 1.

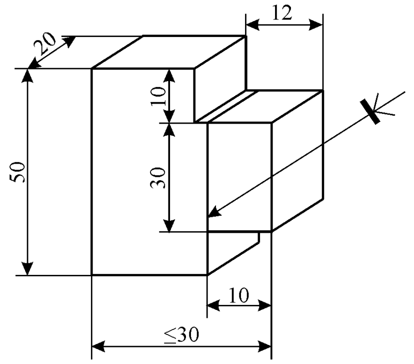


Fig. 1. Shape and dimensions of bonded oakwood samples

During long-term experimental studies, the changes in the strength of adhesive oakwood samples bonded with non-structured and structured polyvinyl acetate adhesives were determined depending on the effect of external factors, namely humidity, and temperature. The bonded oakwood samples (after 78 hours from the moment of bonding) were tested in the natural atmospheric conditions of the western region of Ukraine for three years, until the destruction of the last sample.

All samples used for testing were divided into two parts. The main number of samples was tested in natural atmospheric conditions within the changes of the temperature from 251 K to 306 K and the humidity from 40 % to 100 % by placing on a stand and displacing on the experimental site following the requirements. After a certain period, namely every six months of the atmospheric tests (6, 12, 18, 24, 30, and 36 months), the samples were subjected to destruction by their chipping along fibers, according to the requirements, with subsequent statistical and mathematical data processing. Control samples were kept indoors at a temperature of 293 ± 2 K, relative humidity of 65 ± 5 %, and were not exposed to atmospheric influence. After the removal from the stand, the samples were conditioned and subjected to destruction with subsequent statistical and mathematical data processing. The condensation process was carried out at a temperature of 293 ± 2 K, and relative humidity of 65 ± 5 % for 14 days.

Sample tests for chipping along the wood fibers were performed by using a bursting machine 2166 R-5, which allows recording the test results at a speed of 0.01 s, and with the help of a computer to perform necessary calculations, taking into account the bonding area.

3. Results and Discussion

3.1. Experimental Results

Experiments to determine the strength of oakwood joints bonded with non-structured and structured thermoplastic polyvinyl acetate adhesives were conducted according to the experimental procedure described in Subsection 2.2.1. Experimental strength data for oakwood adhesive joints is represented in Table 1.

The results of tests have shown that adhesive joints lose strength due to exposure to temperature and humidity changes. Changes in the strength of the adhesive oakwood joints under the influence of atmospheric factors during the tested period are presented in the form of experimental curves in Fig. 2.

Based on the obtained data (Table 1 and Fig. 2), the reduction in strength of all adhesive joints can be observed during the tested period. The research results showed more intensive strength decreases during periods of sudden changes in temperature and humidity of the environment. It was established experimentally, that the adhesive oakwood joint based on non-structured adhesives with water resistance class D2 withstood the strength for 18 months with a residual strength of 0.97 MPa, and the adhesive joint based on structured adhesives with water resistance class D4 withstood the strength for 36 months with a residual strength of 0.85 MPa.

Table 1. Strength of oakwood adhesive joints bonded with thermoplastic PVA adhesives

Wood species and wood moisture content	Adhesive, water resistance class	Strength, MPa												
		Initial strength, MPa	Duration of tests, months											
			6		12		18		24		30		36	
			Experimental	Calculated	Experimental	Calculated	Experimental	Calculated	Experimental	Calculated	Experimental	Calculated	Experimental	Calculated
Oak W=10±2 %	PVA, non-structured, D2	11.65	5.17	5.36	2.35	2.30	0.97	0.92	The samples were stratified					
Oak W=10±2 %	PVA, structured, D4	11.34	7.94	7.72	5.56	5.31	3.63	3.63	2.67	2.44	1.52	1.61	0.85	1.03

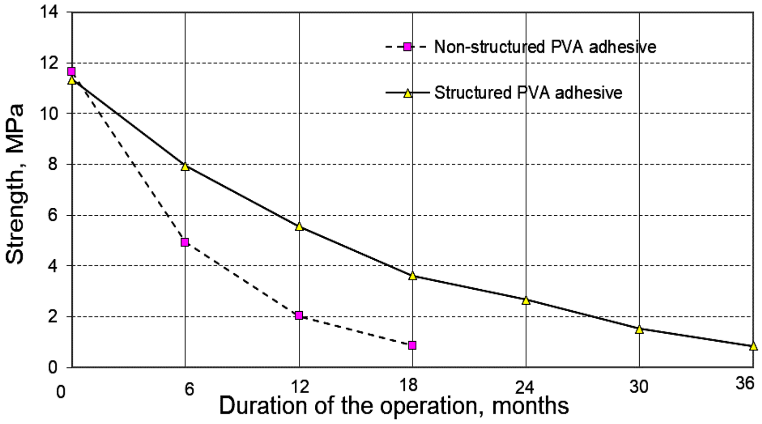


Fig. 2. Changes in the strength of the oakwood adhesive joints under the influence of atmospheric factors during the tested period

Taking into account that moisture has a significant and ambiguous effect on the strength and durability of adhesive wooden joints, the theoretical aspects of the formation of the adhesive seam strength and the influence of atmospheric factors on the strength of the adhesive joints during their operation were analyzed by the authors of this work.

3.2. Theoretical Aspects of the Adhesive Seam Strength Formation and the Influence of Atmospheric Factors on the Strength of the Adhesive Joint During Operation

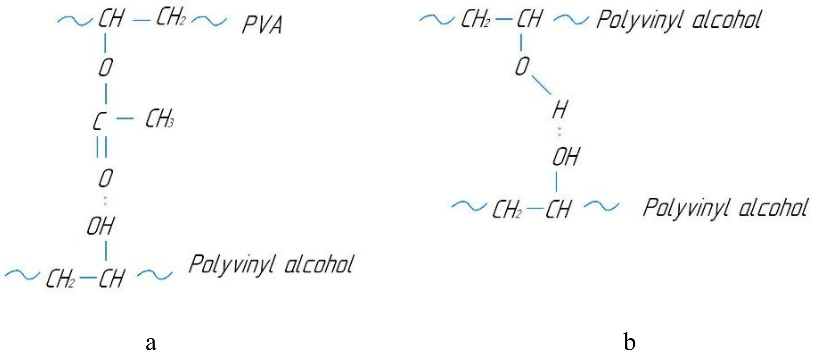
Thermoplastic non-structured (one-component) PVA adhesives can form linear, and structured (two-component) PVA adhesives – thin mesh structures of the adhesive seam. Such adhesive wooden joints should react differently to the influence of moisture as well as humidity, and environmental temperature, which ultimately affects their durability.

Non-structured PVA adhesives have high mobility of macromolecules, which causes low heat resistance of the adhesive joint and its creep under the atmospheric load. But the creep of the adhesive seam can be relaxed by internal stresses that arise under the influence of temperature and humidity, which makes the adhesive joint more durable. Hardeners are added to structured PVA adhesives during the formation of the adhesive seam, which causes the formation of the three-dimensional thin mesh structure and, thereby, slightly increases the water resistance of the adhesive joint, and somewhat reduces the elasticity of the adhesive seam.

The adhesive strength in the adhesive joint is formed by the physical contact of the adhesive and the substrate (by mechanical adhesion) as well as the intermolecular interaction and hydrogen bonds between the adhesive and wood components. The strength and durability of adhesive wooden joints will primarily depend on the adhesive and cohesive strength of the adhesive film and the processes associated with the mechanism of its formation. Therefore, in order for the adheforo be strong and durable, it is necessary to ensure the appropriate cohesive and adhesive strength. The adhesive and cohesive strength of thermoplastic adhesive oakwood joints will depend on intermolecular interaction, the energy of intermolecular bonds, electrostatic interaction, *etc*.

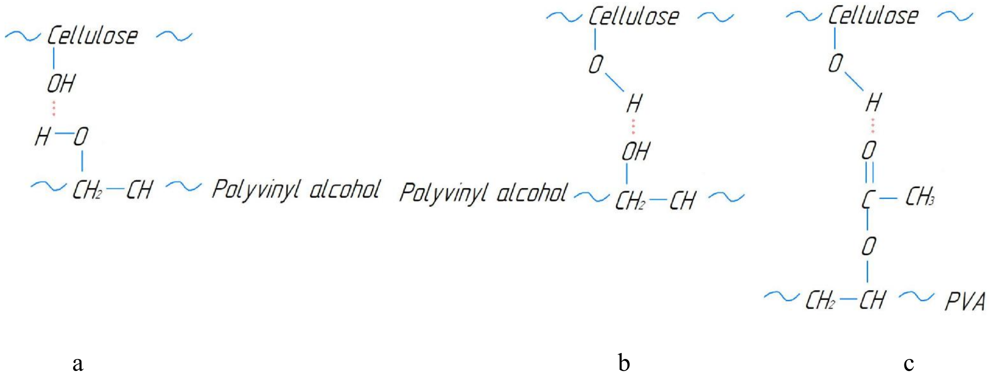
A hydrogen bond is a special type of three-centered chemical bond of the X–H–Y type, in which the H atom is connected by a covalent bond with the electronegative atom X (N, O, S), forming an additional bond with the atom Y (N, O, S), having an undivided pair of electrons. A hydrogen bond can be considered to be a separate type of coordination bond since the number of bonds formed by the central H atom is greater than its formal valence. The energy of hydrogen bonds is mainly in the range of 20–40 kJ/mol. Intermolecular hydrogen bonds cause strong cohesion for many polymers, in particular, cellulose. The higher the positive electrostatic potential near the H atom, the stronger the hydrogen bond. Therefore, the strongest hydrogen bonds are formed in those cases when the atom and the substituent groups in the molecule have the most negative charge, as well as when the molecule is capable of strong polarization. Hydrogen bonds are formed mainly in the liquid phase, and during crystallization, they are usually preserved.

Examples of hydrogen bonds that can form components of the polyvinyl acetate adhesive composition between themselves are shown in Fig. 3 and examples of hydrogen bonds that can form components of the PVA adhesives with wood are shown in Fig. 4.



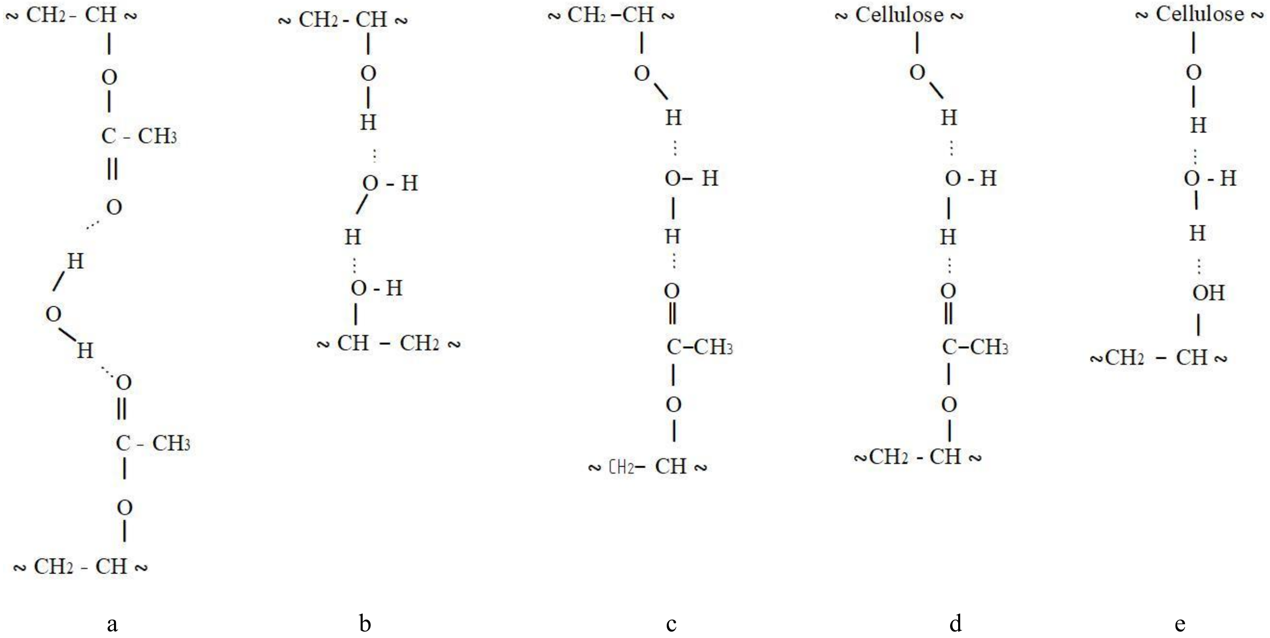
a b

Fig. 3. Examples of hydrogen bonds that can form components of the polyvinyl acetate adhesive composition between themselves (a, b)



a b c

Fig. 4. Examples of hydrogen bonds that can form components of the polyvinyl acetate adhesives with wood (a, b, c)



a b c d e

Fig. 5. Formation of additional hydrogen bonds between PVA components in the adhesive joint (a, b, c) and the adhesive joint and wood (d, e) through water molecules

The number of both intermolecular and hydrogen bonds between the macromolecules of the adhesive composition and, accordingly, the energy of cohesion will depend on the degree of order between the macromolecules in it. The formation of physical contact between the substrate (wood) and the adhesive is due to the wetting of a solid surface with a liquid. Contact formation is accelerated by the increase of pressure and time and the decrease of viscosity.

In addition, such adhesive wooden joints will be affected by environmental humidity during operation. Due to the sorption capacity and hygroscopicity of wood, moisture will enter the adhesive seam and affect the durability of the adhesive wood joint. Due to the large dipole moment of water, its hydrogen atoms can form hydrogen bonds with atoms O, N, F, Cl, S, and others that have free pairs of electrons.

Cellulose, as the main wood component, is a linear polymer that consists of β-D-glucopyranose residues and has a large number of -OH groups.[16] The regular structure of the cellulose macromolecule makes it possible to form intermolecular hydrogen bonds that tightly connect polymer chains, forming micro- and macrofibrils. -OH groups, located on the surface of micro- and macrofibrils, can form hydrogen bonds with water molecules and thus form a molecular layer of water on their surface.

The adhesive seam formed by PVA-based thermoplastic adhesives to some extent is similar to wood in its structure. The main components of PVA-based adhesives are polyvinyl acetate and polyvinyl alcohol, which can change their physicochemical and physicomechanical properties under the influence of moisture.

If there is a slight water content in the adhesive joint, the number of hydrogen bonds should increase, since additional hydrogen bonds can be also formed between PVA ester groups through water molecules (Fig. 5a), as well as between –OH groups of polyvinyl alcohol (Fig. 5b), –OH groups of polyvinyl alcohol and distant from each other ester groups of PVA (Fig. 5c), between –OH groups of wood components (cellulose) and ester groups of PVA (Fig. 5d), or –OH groups of polyvinyl alcohol (Fig. 5e).

Thus, according to theoretical predictions, the formation of hydrogen bonds between the components of the adhesive as well as the adhesive composition and wood can occur due to the presence of water in the adhesive joint (at the moment of wood samples bonding) and during the adhesive joint operation. Therefore, a small content of moisture in the adhesive joint during the oakwood samples bonding and a small content of moisture adsorbed from the environment at the initial stages of operation of the wooden adhesive joints will have a positive effect on the strength of the oakwood adhesive joints bonded with thermoplastic polyvinyl acetate adhesives.

However, as confirmed by the results of experimental studies, the long-term effect of water (atmospheric precipitation and increased environmental humidity) leads to a gradual decrease in the strength of the adhesive joints due to the swelling of the wood and adhesive seam. The medium temperature fluctuation also negatively affects the strength of the adhesive joint, increasing its creep. As a final result, the combined effect of these factors leads to the destruction of the adhesive joint.

Given the foregoing, the equation was proposed for predicting the strength of adhesive wooden joints bonded with PVA thermoplastic adhesives, which takes into account the influence of temperature, humidity, as well as the duration of adhesive joints operation, and which can be represented as:[17]

$$\sigma = k_{sp.} \cdot (A^{(i)} \cdot \Delta T^{(i)} + B^{(i)} \cdot \Delta W^{(i)} \exp(C^{(i)} \cdot \tau^{(i)})) \quad (1)$$

where σ is the strength of the adhesive wooden joint, MPa; $\Delta T^{(i)}$ is the weighted average temperature of the environment, ^{0}C; $\Delta W^{(i)}$ is the weighted average humidity of the environment, %; $\tau^{(i)}$ is time, days; $A^{(i)}{}_, B^{(i)}$, $C^{(i)}$ are coefficients, which values depend on the range of changes in ambient temperature, humidity, and the degree of atmospheric loading of the adhesive wooden joint, respectively; k_{sp} is a coefficient that takes into account the physical and mechanical properties of the wood species.

Equation (1) makes it possible to predict the strength of adhesive joints for certain types of wood species at a known operation time of the adhesive joint and certain ranges of changes in temperature and humidity of the environment. To use Eq. (1) for the prediction of the strength of oakwood adhesive joints, bonded with structured and non-structured thermoplastic PVA adhesives, in a wide range of temperature and humidity changes in the environment, it is advisable to generalize the theoretical and experimental research results to determine unknown coefficients k_{sp}, $A^{(i)}{}_, B^{(i)}$, $C^{(i)}$.

3.4. Generalisation of the Results

Thus, based on the generalization of experimental data and theoretical predictions regarding the mechanism of the adhesive seam formation, the coefficients of Eq. (1) were determined: $A^{(i)} = -0.0006; B^{(i)} = 0.1372; C^{(i)} = -0.0082$ – for oakwood adhesive joints bonded with non-structured PVA adhesives and $A^{(i)} = -0.024; B^{(i)} = 0.1151; C^{(i)} = -0.00205$ – for oakwood adhesive joints bonded with structured PVA adhesives. For the adhesive joints of oakwood, the value of the coefficient $k_{sp.}$ is equal to 1. Therefore, taking into account the values of coefficients $A^{(i)}, B^{(i)}, C^{(i)}$ and $k_{sp.}$, Eq. (1) can be represented in the form of Eq. (2) and Eq. (3), which make it possible to predict the strength of the oakwood adhesive joints bonded with non-structured and structured PVA adhesives, respectively:

$$\sigma = -0.0006 \cdot \Delta T^{(i)} + 0.1372 \cdot \Delta W^{(i)} \exp(-0.0082 \cdot \tau^{(i)}); \quad (2)$$

$$\sigma = -0.024 \cdot \Delta T^{(i)} + 0.1151 \cdot \Delta W^{(i)} \exp(-0.00205 \cdot \tau^{(i)}) \quad (3)$$

The derived Eqs. (2) and (3) make it possible to calculate theoretically with sufficient accuracy the strength of adhesive oakwood joints bonded with non-structured and structured PVA adhesives within the temperature range of 251–306 K and humidity range of 40–100 %. Analysis of the obtained results has shown that the validity of Eqs. (2) and (3) is confirmed by a satisfactory agreement with the experimental data obtained by using long-term experimental studies (Table 1).

The changes in the strength of the adhesive oakwood joints bonded with structured and non-structured PVA adhesives during operation in the temperature range from 251 K to 306 K and humidity range from 40 % to 100 % are shown in Figs. 6 and 7, respectively. Graphs in the figures show that values of the strength calculated by using Eqs. (2) and (3) almost completely coincide with the experimental data.

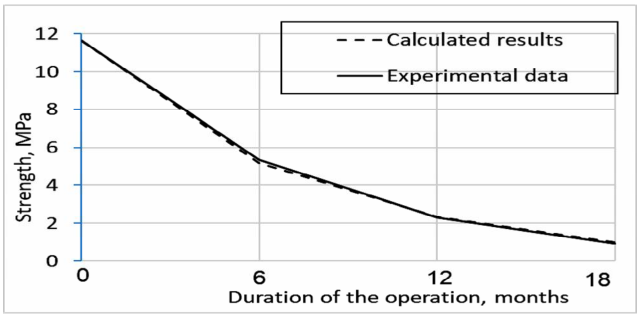


Fig. 6. Comparison of experimental and theoretically calculated (according to Eq. (2)) values of the oakwood adhesive joints strength bonded with non-structured PVA adhesive

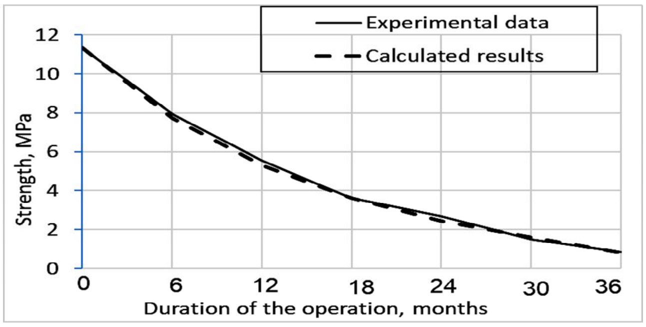


Fig. 7. Comparison of experimental and theoretically calculated (according to Eq. (3)) values of the oakwood adhesive joints strength bonded with structured PVA adhesive

The maximum relative error between the experimentally determined and theoretically calculated values of the strength has an acceptable level and does not exceed ±3.5 %. Therefore, proposed Eq. (2) and Eq. (3) allow us to calculate theoretically with high accuracy the strength of oakwood adhesive joints bonded with non-structured and structured PVA adhesives within the temperature range from 251 K to 306 K and humidity range from 40 % to 100 %.

4. Conclusions

The changes in the strength of the adhesive oakwood joints bonded with structured and non-structured PVA adhesives have been investigated by conducting long-term experiments. It was established experimentally, that the adhesive oakwood joints based on non-structured PVA adhesives with water resistance class D2 withstand the strength for 18 months with the residual strength of 0.97 MPa and the adhesive joints based on structured PVA adhesives with water resistance class D4 withstand the strength for 36 months with the residual strength of 0.85 MPa.

Based on the generalization of the experimental data and theoretical aspects regarding the strength formation aspects of the adhesive seams, Eqs. (2) and (3) have been proposed, which allow calculating theoretically (without destroying the adhesive joints) the strength of oakwood adhesive joints bonded with non-structured and structured PVA adhesives, respectively. Eqs. (2) and (3) with sufficient accuracy of ± 3.5 % reproduce experimental data within a temperature range from 251 K to 306 K and humidity range from 40 % to 100 %; therefore, they are recommended for practical use.

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ПРОГНОЗУВАННЯ МІЦНОСТІ КЛЕЙОВИХ З'ЄДНАНЬ ДЕРЕВИНИ ДУБА, З'ЄДНАНИХ ТЕРМОПЛАСТИЧНИМИ ПОЛІВІНІЛАЦЕТАТНИМИ КЛЕЯМИ

Анотація. *Серед кількох видів термопластичних клеїв, структуровані й неструктуровані полівінілацетатні (ПВА) клеї достатньо широко використовують,, зокрема для формування клейових з'єднань різних порід деревини, серед них дуба. Для забезпечення належних умов використання клейових з'єднань деревини дуба важлива наявність швидких і точних методів прогнозування їхньої міцності і довговічності. Зміни міцності клейових з'єднань деревини дуба, з'єднаних структурованими і неструктурованими ПВА клеями, вивчено за допомогою тривалих експериментальних досліджень. На основі узагальнення експериментальних даних і теоретичних прогнозів механізму утворення клейового шва запропоновано залежності, які дають змогу теоретично розрахувати міцність клейових з'єднань деревини дуба, з'єднаних неструктурованими і структурованими ПВА клеями. Запропоновані рівняння відтворюють експериментальні дані з достатньою точністю ± 3,5 % в діапазоні температур від 251 K до 306 K і вологості від 40 % до 100 %, тому рекомендовані для практичного використання.*

Ключові слова: *полівінілацетатні клеї, клейове з'єднання деревини, міцність, довговічність, клейовий шов.*