EVALUATION OF STRUCTURAL WOOD GLUED JOINT QUALITY, DEPENDING ON SELECTED **TYPE OF AHDESIVE**

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Abstract: Structural wood glued joints are used to bond components of lamellar or agglomerated load-bearing members. Factor that influences the quality of the joint is the sensitivity of wood mass to ambient humidity changes. Cyclic wetting and drying of the timber leads to its volume changes and causes shear stress in the area of the bond line. Therefore, selected types of adhesives were tested according to a standard EN 302-1. In addition to shear strength assessment, a wood failure of specimens was observed. To get comparative samples for further durability evaluation, the infrared absorption spectroscopy analysis was performed. Further research will continue with examination of the joint quality of specimens in long-term external exposition and macromolecular changes in adhesive structure.

Keywords: Wood glued joint, shear strength testing, infrared absorption spectroscopy.

1 Introduction

The quality evaluation of wood glued joints adjusts standard ČSN EN 302-1 that prescribes design, parameters and preparation of the specimen and type of wood that should be used as supporting substrate. After curing, the specimen are exposed to accelerated aging test and their shear strength is evaluated in five types of exposition causing stress in the bondline area due to increasing moisture degree in combination with drying.

The expositions should simulate adverse conditions, which affect the usage of glued structural member in exterior. According to achieved shear strength value in every expositions, the adhesive is assigned to specific class of utilization.

In this case, the specimens were only under influence of exposition A1 (specimen air-conditioned at the temperature of 20 °C and humidity of 65 % for 7 days), which provides favourable conditions. As the accelerated test does not cause macromolecular structure changes, this type of exposition will be suitable as referential for the long-term testing of durability. Specimens designed to long-term testing will be tested afterwards, after two years of exposition to natural external weather conditions.

The macromolecular structure changes are consequence of crosslinking damage caused by environment work, especially the UV radiation. Such changes can be revealed using infrared absorption spectroscopy. First, there have to be determined the possibilities of the method - the sampling, the measurement technique and its accuracy.

2 Materials and methods

2.1 Specimens according to ČSN EN 302-1

The specimens were prepared by joining two plates made from beech wood (Fagus sylvatica) using three types of adhesives. All three adhesives are distributed by Akzo Nobel. Two of them the one with melamine-urea-formaldehyde (MUF) and the one with emulsion-polymer-isocyanate (EPI) basis - are twocomponent adhesives, the other with polyurethane basis is a onecomponent adhesive.

Besides the properties of adhesives, another important factor that influences the quality of forming bond line, is the density of supporting wood mass. If the surface is more porous, the adhesive will be able to penetrate deeper to the structure and make stronger bond. Therefore, the density of wood plates was measured before gluing. Bonding parameters are shown in Table

Table 1 Bonding parameters of all tree tested adhesives

Adhesive	Mixing ratio	Hardener	Pressure [MPa]	Pressing time [hours]	Glue spread [g/m ²]	Wood density [kg.m ^{.3}]
PUR	-	-	0.8	1	200	690.9
EPI	100:15	1993	0.8	0.5	180	715.3
MUF	100:100	7557	0.8	3.5	400	700.2

After curing, the specimens were cut to required dimensions with prunings at the edges of tested area and air-conditioned in A1 exposition according to standard ČSN EN 302-1, i.e. 7 days at the temperature of 20°C and the humidity of 65%. All specimen were tested on the Testometric M350-20CT machine.

2.2 Samples for infrared absorption spectroscopy

Infrared absorption spectroscopy is an analytical method that is based on the fact that the interaction of the mass and infrared radiation causes the dipole moment changes of molecules of the mass. This changes induce vibrations of the molecules that absorb part of radiation energy, which will make a curve peak on the spectrogram. The wavenumber, on which the peak (spectral band) arises, is dependent on the length of chemical bond between the atoms in the molecule, so every chemical substance has its typical curve shape. If the chemically pure substance is analyzed, the bands can be exactly identified. Bands on the spectrograms of mixture can overlap. This fact greatly hinders their identification. That is the reason, why the infrared absorption spectroscopy is commonly provided as a comparative analytical method. [1]

There are several techniques of sample measurement, but most widely used are ATR (attenuated total reflectance) and transmission. The ATR technique does not require involved sample preparation, solid (altogether or grounded to the powder) or liquid materials can be analyzed. The ATR spectrograms are less accurate than the spectrograms obtained by transmission technique. Samples analyzed by transmission are only solid. If there is a possibility of making a thin film (polymer samples), the samples are measured that way, if not, samples have to be grounded to a fine powder and after mixing with the KBr compress to a tablet.

Two types of samples were prepared for the infrared absorption analysis:

- Referential drops of mixed adhesives cured on polyethylene sheet with the diameter of about 1 cm.
- Tested samples taken from the shear strength specimen fractions.

The preparation of referential sample was different for each used adhesive. As these samples should show the accuracy of tested sample analysis by comparison of the spectrograms, they had to be analyzed using the transmission technique. PUR adhesive made a thin film during curing, which was cut from the drop. MUF adhesive is brittle enough to be grounded to a powder. EPI adhesive is very plastic and hardens slowly, so it had to be deep frozen and then grounded to a powder. MUF and EPI were analyzed in a tablet.

The spectrograms of tested samples were obtained by ATR technique. The fractions of the shear strength specimen had to be soaked in water for one day and then mechanically disconnected in the bond line area. The sample was put onto a measuring crystal with sufficiently smooth and flat cleaned surface of the adhesive layer.

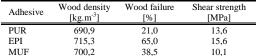
Besides these two types, a spectrogram of pure wood was taken, so its bands could be excluded from the analysis.

3 Results and discusion

3.1 The shear strength testing

The shear strength of the joints was evaluated as the highest achieved force acting on tested area. Besides, the wood failure upon this area had to be considered, because, if its value was higher than 0%, it could be assumed that full or part loads was carried by the supporting wood mass. Also, the wood density had to be assessed as a factor influencing the increase of shear strength of the joint. Achieved results are shown in Table 2 and following graphs.

Table 2 Results of the shear strength testing



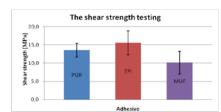


Figure 1: Graph of the shear strength testing evaluation

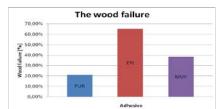


Figure 2: Graph of wood failure evaluation

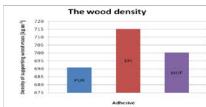


Figure 3: Graph of wood density evaluation

The highest shear strength was achieved by the specimens glued with EPI adhesive. These specimens were also mostly violated outside the bond line. Thus, it can be assumed that the real shear strength of the joint is even higher.

The specimens glued with PUR adhesive were spread on the most porous support, so the quality of the joint should be best according to the thickest layer. Most of these specimens violated in the bond line. Therefore, it can be considered that PUR joints has lower quality than the ones glued with EPI adhesive, because measured values are approximately corresponding to the real shear strength of the bond.

The lowest value of shear strength achieved the specimens bonded with MUF adhesive. The wood failure of these specimens and density of supporting wood mass are suggesting that these values of shear strength are corresponding to real strength of the bond too.

3.2 The infrared absorption spectroscopy analysis

The description of infrared spectra obtained from prepared samples was the first step of durability evaluation. In following period, the specimens exposed to the long-term aging test will be analyzed and their spectrograms will be compared to these ones. The appearance or disappearance of several bands will indicate structural changes in chemical bonds of the polymers.

Spectrogram of tested sample of each adhesive was compared to its referential sample spectrogram to make sure that all important bands can be measured using ATR technique, and to the pure wood sample to exclude bands of wood mass.

Important bands that will be monitored by structure changes evaluation are shown in Figure 4 (PUR), Figure 5 (EPI) and Figure 6 (MUF).

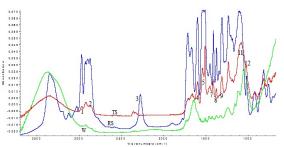


Figure 4 Spectrograms of PUR adhesive (TS – tested sample, RS – referential sample, W – wood curve)

Spectral bands of PUR adhesive that will not be overlapped by spectrum of wood support and should be checked on the spectrogram of long-term tested samples are in Table 3.

Table 3	Important	bands	that	should	be	monitored	on	long-term	tested
samples	of PUR ad	hesive							

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No.	Region	Chemical bond	Type of vibration
1	2940 cm ⁻¹	СН	asymmetrical stretching
2	2860 cm ⁻¹	CH	symmetrical stretching
3	2400 - 2200 (2280) cm ⁻¹	N=C=O	isocyanate – hardener
4	1600 cm ⁻¹	C=C	aromatic - stretching
5	1540 cm ⁻¹	NH	amine - bending
6	1500 cm ⁻¹	C=C	aromatic - stretching
7	1470 cm ⁻¹	CH_2	bending, scissor
8	1360 cm ⁻¹	C-N	amine - stretching
9	1350 cm ⁻¹	CH_2	bending, wagging
10	1280 cm ⁻¹	OCONH	urethane - stretching
11	1140 cm ⁻¹	-O-C-	urethane - stretching
12	1000 cm ⁻¹	OCONH	urethane - stretching

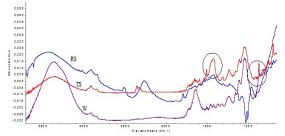


Figure 5 Spectrogram of EPI adhesive (RS – referential sample, TS – tested sample, W – wood curve)

The only two bands that could be checked on the long-term tested specimen are marked in Figure 5 (1430cm⁻¹ and 880cm⁻¹). Both bands belong to an inorganic filler, which covers greater part of the adhesive mass.

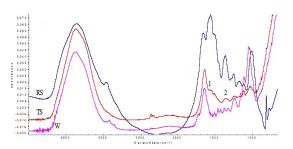


Figure 6 Spectrograms of MUF adhesive (TS – tested sample, RS – referential sample, W – wood curve)

Bands of MUF adhesive that will not be overlapped by spectrum of wood support and should be checked on the spectrogram of long-term tested samples are in Table 4.

Table 4 Important bands that should be monitored on long-term tested samples of MUF adhesive

No.	Region	Chemical bond	Type of vibration
1	1540 cm ⁻¹	NH	amine - bending
2	1340 cm ⁻¹	CH	bending, wagging

For the macromolecular structure changes evaluation, used method of sample preparation for infrared absorption spectroscopy analysis is suitable only for the PUR adhesive according to the accuracy. In its case, the long-term tested samples can be prepared the same way as the tested samples, as the amount of identified bands was big enough.

Spectrograms of EPI based adhesive show only the bands of its inorganic filler. To get the spectrum of pure polymer base of the adhesive, another treatment of the samples is needed. Further research will continue with preparation of referential sample by removing of the inorganic part and providing a transmission analysis of deep frozen and grounded adhesive base. Afterwards, the methodology of tested and long-term tested sampling will have to changed. The fraction of specimens will be mechanically disconnected in the bond line area. A piece of the specimen containing the adhesive will be deep frozen, then grounded to a fine powder and then analyzed using transmission technique too. This spectrogram will be compared to a spectrogram of wood mass removed from the same specimen to exclude the bands that belong to the wood support.

Because of the low amount of identified bands in the spectrogram of MUF tested sample, the sampling and sample preparation methodology will have to be change too. The tested and the long-term tested samples will have to be prepared as the ones glued with EPI based adhesive.

4 Coclusion

Three types of adhesives were evaluated according to standard ČSN EN 302-1 A1 exposition – PUR, MUF and EPI based adhesive. From these three types, best result of the shear strength testing achieved the specimens glued with the EPI based adhesive (average value of 15.6 MPa), the worst results were ejected by the MUF adhesive. Besides the shear strength testing, two more factors were monitored – the density of the supporting wood mass (as the porosity of the surface helps to create stronger bond) and the wood failure in the shearing area. The major part of EPI based adhesive glued specimen failed in the wood mass (65%), so it can be assumed that the shear strength of the joint is even higher than the determined. The values achieved by other two types of adhesives responded approximately to the real shear strength of the joints, as the wood failure of the specimens was low.

Further research will continue with the long-term aging test. A1 exposition tested specimens will be used as referential to the long-term aged ones to get a comparison of the strength and macromolecular structure changes. Therefore, an infrared absorption spectroscopy analysis of the specimens was performed. Sufficient results were given only by the samples prepared from the specimens glued with PUR adhesive. The analysis of the two other adhesives will have to be repeated on the samples obtained by changed methodology of preparation.

Literature:

1. Svehla, G.: *Comprehensive analytical chemistry VI.* 1. issue. Amsterdam: Elsevier scientific publishing company, 1976. 555 p. ISBN 0-444-41165-8

Primary Paper Section: J

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