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FURAN RESINS AS POTENTIAL STRUCTURAL ADHESIVES FOR WOOD

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Abstract:

The adhesives compositions for wood gluing have to complied special requirements to be called structural adhesives. Three adhesive compositions based on polycondensation, namely an urea-formaldehyde adhesive, a mixed furan resin and a mixture of these in equal proportions, were tested in terms of their compliance to the requirements imposed for structural adhesives by SR EN 301:2006 and their classification as type I or type II based on their expected performance in service in different specified environmental conditions. Two tests were employed for this purpose: determination of the influence of acid attack on the wood fibres on the transverse tensile strength and the determination of the effects of wood shrinkage on gluing strength.

The results shown that: the adhesive composition based on urea formaldehyde resin, included in this research only as a reference, does not comply to the requirements for structural adhesives, the mixed furan resin complies entirely to the requirements for structural adhesives of type I. The adhesive composition based on mixture equal proportion of these resin demonstrated a significantly improved performance compared to the reference urea- formaldehyde adhesive, but could not reach the requirements for the structural adhesives. However, it shown a much better resistance to climatic cyclic aging compared to the UR reference, which clearly demonstrates the positive effect of the mixed furan resin.

Key words: *wood gluing; structural adhesive classification; furan resin.*

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INTRODUCTION:

A more extensive utilisation of wood in construction is a worldwide sustainable approach, reflected also in Romania. For instance the market value of the wooden materials used in constructions in 2012 was about 3.65 billion lei (~830000 EURO), according to the Association of manufacturers of construction materials from Romania (http://www.agendaconstructiilor.ro).

Building with wood brings some well acknowledged advantages, such as: sustainability, good strength to weight ratio, earthquake resistance, thermal and acoustic insulation, low price, durability and long-term savings, alongside the versatility of the modern wooden materials for constructions adaptable to any architectural style, from traditional to contemporary (*Isopescu 2011).*

a b c

Fig.1

Some examples of wood utilisation in modern constructions (**http://en.wikipedia.org*** ***).***

"Structural Wood Panels (glulam, plywood, osb boards etc.), have a major interest in different applications for a wooden structure, Fig. 1, from them we can mention: wooden beams, roof cover, floors and walls, tensioned panels etc., obtained from glued products."

The use of adhesives in construction dates from 1906, when a German carpenter named Otto Hetzer produced the first glued wooden structures for this purpose, later standardized as *glulam*, considered at the origin of glued laminated timber (Patent 197773/ 1906, Lăzărescu 2004, Büren 2005).

The glued wooden products for constructions require special adhesives forming high strength and durable bonds, so that to ensure an appropriate behaviour of the reconstituted material as a whole for the entire lifetime of the wooden structures (Charles 1999).

These adhesives are called *structural adhesives* and they must comply with the requirements stated by SR EN 301, 302. According to these norms, structural adhesives are:

▪ Adhesives of *type I* - adhesives used in conditions which involve prolonged exposure to high temperatures (higher than 50ºC), or adhesives used at temperatures below 50ºC in applications totally exposed to the action of the natural climatic factors, equivalent to a controlled climate with a relative air humidity of 85% at the temperature of 20ºC.

▪ Adhesives of type II - adhesives used indoors, in ventilated and heated constructions, or in outdoor conditions, protected from or exposed only for short periods of time to the direct action of the climatic factors, conditions equivalent to a controlled climate with a relative humidity less than 85% at 20ºC, conditions leading to a moisture content in solid wood of approximately 20% and a little lower in the wood-based panels.

A certain adhesive is certified as structural of type I or II following 4 specific tests (shear strength by longitudinal traction, resistance to delamination, determination of the influence of acid attack on the wood fibres to transversal traction strength and determination of shrinkage of wood on shear strength) carried out on glued sample artificially aged according to specified procedures.

The most widely employed structural adhesives are the polycondensation adhesives of phenol-formaldehyde, resorcin-formaldehyde and phenol-resorcin-formaldehyde type (Mara 2012, Frangi 2004)

However, research (Lavisci 2001, Andreica *et al.* 2007, Frihart 2011) and even specific standards (EN 14080: 2006: *Timber structures - Glued laminated timber - Requirements*) suggest the use of some other types, more expensive, adhesives, such as epoxy and polyurethane adhesives for structural applications.

Frangi *et al*. (2004) shows that the advantages of using polyurethanes adhesives result from the fact that they are efficient, easy to apply, colourless, and possibly more resistant, assumption based only of shear strength evaluation.

In this context, the study of the possibilities of using some mixed furan resins, obtained by condensation from furfuryl alcohol, urea and formaldehyde, as structural adhesives for wood gluing is an opportunity, considering their competitive price, the possibility of cross-linking at environmental temperature and the good resistance to water and temperature, specific generally to the furan resins (Hoydonckx 2009).

Literature data concerning the use of different types of furan resins as wood adhesives are limited. Schneider (2002) used a simple furan resin based on furfuryl alcohol (obtained by its self-condensation) and patented an adhesive mixture for plywood. Mixed furan resins based on furfuryl alcohol condensed with other reagents were studied with good results as wood adhesives for plywood (Schultz 1990) and other wood-based products such as particleboard, fibreboards, OSB (Goodman 1998, Patent 1983, Moon 2001).

In the present research a mixed furan resin, cross-linkable at room temperature in acidic medium, namely Urelit FC2 produced by S.C. Viromet S.A, was tested as a potential structural wood adhesive according to the specific norms. The tests referred to in this paper are the influence of the acid attack on the wood fibres on the transverse tensile strength and the determination of the effects of wood shrinkage on gluing strength, the other two specific tests being the subject of another publication (Zeleniuc and Varodi 2013).

MATERIALS AND METHODS

Two types of resins, Urelit FC2 and Urelit R), produced in Romania by Viromet SA, were used to prepare 3 adhesive compositions tested in the present research, as follows:

- UR/FC2/NH_4Cl-20 – based on urea-formaldehyde resin and the mixed furan resin Urelit FC2 type in equal weight proportion;
- FC2/NH_4Cl-20 - based only on mixed furan resin type Urelit FC2.
- UR/IRs- based on cold-setting urea-formaldehyde resin Urelit R type, as a reference product.

The use of Urelit R as a reference gluing product was motivated by the fact that it is the only cold-setting polycondensation synthetic resin for solid wood gluing produced and currently used in Romania. This is prepared for utilisation by adding an acidic catalyst (coded IR).

The composition and the characteristics of the adhesives tested are presented in Table 1. Rye flour was added as extender in a constant proportion of 12.33% reported to the liquid resin in all these compositions.

Table 1

Physical-chemical characteristics and adhesive composition used in experimental tests

Adhesive specifications	Unit	Quantity of adhesive/hardener		
		UR/IRs	FC_2/ NH_4Cl-20	UR/FC_2 NH_4Cl-20
Adhesive composition:				
- urea-formaldehyde resin Urelit R (UR),	g	100	-	50
- mixed furan resin Urelit FC2 (FC2),	g	-	100	50
- ammonium chloride, 20% solution (NH_4Cl – 20),	ml	-	25	25
- solid hardener IR type,	g	1.5	-	-
- rye flour (F),	g	12.33	12.33	12.33
- water	ml	1	-	-
Physical and chemical characteristics:				
- solid content	%	73.03	61.32	62.00
- initial pH	-	4	4	4
- viscosity –flow time, FORD cup, Φ 8 mm at 20ºC	s	95	31	11
- jellification time at 20ºC, t_{gel}	min	27	67	37
- stickiness disappearance time at 20ºC, t_{dl}	min	-	10	6
- solidification time at 20 ºC (viability), t_s.	min	86	143	90

Determination of the effect of acid damage to wood fibres by temperature and humidity cycling on the transverse tensile strength

The sample for this test consists of a glued joint between 2 pieces of spruce wood (*Picea abies*) using a thick layer (0.5mm) of adhesive. The test samples resulted by mechanical processing of glued assemblies of two wooden boards, the gluing parameters being: temperature 20°C, pressing time 4 hours, specific pressure 0.6±0.1MPa. The glued pieces were conditioned for 7 days at 20±2°C and 65±5% relative humidity in a FEUTRON climatic chamber before further mechanical processing to obtain the actual final test samples (Fig. 2).

The test samples were subjected to cyclic variations of temperature and humidity (4 cycles of 3 stages A, B, C presented in Table 2) and then reconditioned at 20±2°C and 65±5% relative humidity until constant weight, before testing to tensile perpendicular to the glue line until failure. During the transverse tensile test the samples were freely sitting in a special testing device (Fig. 2). The test was carried out employing a universal testing machine type VEB THŰRINGER INDUSTRIEWERK RAUENSTEIN by the application of a

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progressively increasing tensile force at a speed of (10±1) kN/min until the breaking of the sample, when F_{max} was determined.

For each adhesive composition 5 replicate samples were tested in tensile after cyclic variations of temperature and humidity and another 5 test pieces originating from neighbouring positions in the initial glued assembly were kept in the conditioning chamber as controls until testing.

Table 2

Climatic conditions of cyclic exposure of samples for the determination of acid damage to wood fibres on the transverses tensile strength

Step	Duration, h	Temperature, °C	Relative humidity, %
A	24	50±2	87.5 ±2.5
B	8	10±2	87.5 ±2.5
C	16	50±2	≤ 20

The transverse tensile strength f_1, was calculated in accordance to equation (1):

$$f_1 = \frac{F_{max}}{A}\left[N/mm^2\right] \quad (1)$$

where:
f_l - transverse tensile, in N/mm^2;
F_{max} - maximum breaking force, in N;
A - surface of the glued area, in mm^2.

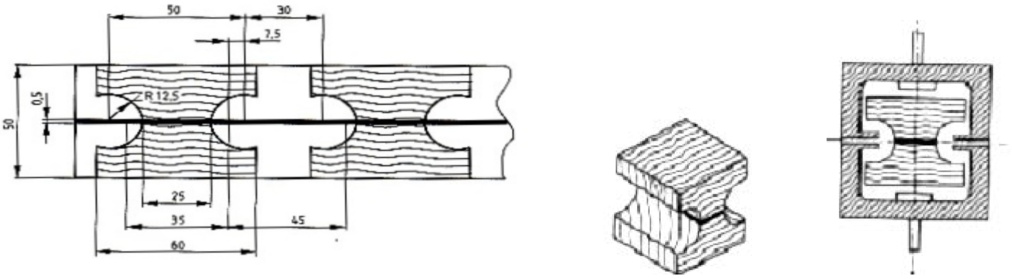
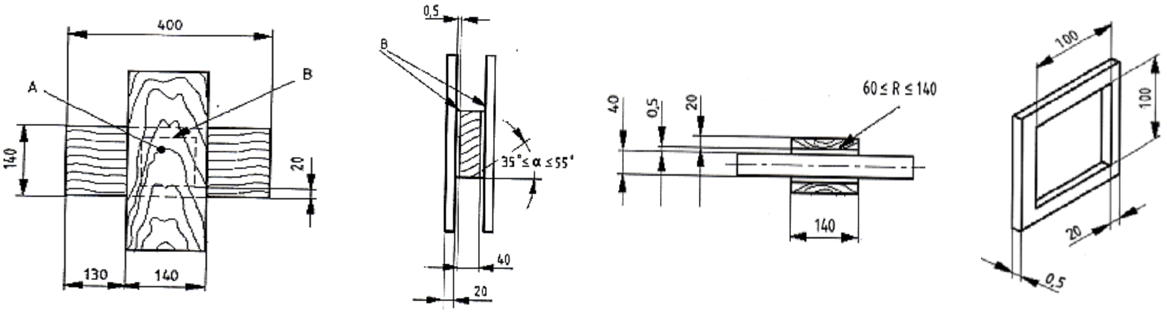


Fig. 2

The final shape and dimensions of the test sample for determining the effect of acid damage to wood fibres by temperature at humidity cycling on the transverse tensile strength and its positioning in the testing device.

Determination of influence of shrinkage of wood on shear strength

The final test samples for this test (Fig. 4) result from cross type double overlap joints (Fig. 3) made of 3 pieces of spruce wood (*Picea abies*) glued together at 20°C, specific pressure 0.6±0.1MPa, pressing time 4 hours. Application of the adhesive was made using a special aluminium frame (Fig. 3d) to ensure a constant thickness of 0.5mm and a glued area of 100mmx100mm.



a) **b)** **c)** **d)**

Fig. 3

Intermediate phases in the preparation of the test samples employed for determination of the influence on wood shrinkage on the shear strength- a) front view; b) lateral view; c) top view; d) aluminum frame.

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The intermediate glued pieces were conditioned for 7 days at 20±2°C and 65±5% relative humidity in a FEUTRON climatic chamber. After conditioning the test samples were subjected at a drying stage in the same climatic chamber at a temperature of (40±2)°C and relative humidity of the air (30±2%) until their content of moisture determined by weighing was (8 ±1)%.

In a further stage, these intermediate joints were processed by cutting 2 parts of the overlap and gluing 4 additional parts (dimensions of 220x130x30mm) made of softwood on the centrepiece at a distance of 3 mm from the overlapped vertical pieces, as shown in Fig. 4. This is necessary to ensure a balanced load and to allow a free movement of the wooden pieces detached during compression testing.

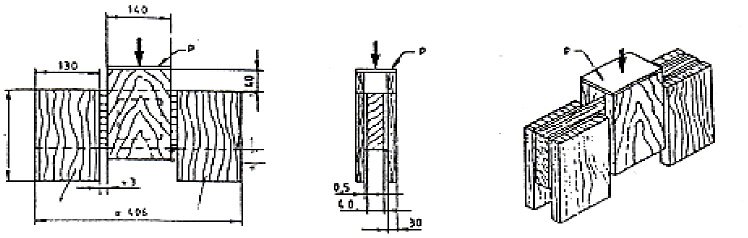

Fig. 4
The shape and the dimensions of the final test samples used for determining the influence of wood shrinkage on shear strength by compression.

The test final samples were reconditioned in standard atmosphere (temperature 20±2°C and 65±5% relative humidity) for 14 days prior testing. A universal testing machine LLOYD LS 100 PLUS (Fig. 5), equipped with a special device for applying progressively a compression load (20±5kN/min) until failure (Fmax), was employed for testing. Breaking was usually achieved in about 70 seconds.

a ***b***

Fig. 5
The universal testing machine type LLOYD LS 100 PLUS employed for determining the influence on wood shrinkage on shear strength by compression:
a – test sample placed on the stand; b - sample under the test.

The shear strength τ_c, was calculated in accordance to equation (2):

$$\tau_c = \frac{F_{max}}{A}\left[N/mm^2\right] \qquad (2)$$

where:
τ_c - shear strength, in N/mm^2;
F_{max} - maximum breaking force, in N;
A - surface of the glued area, in mm^2.

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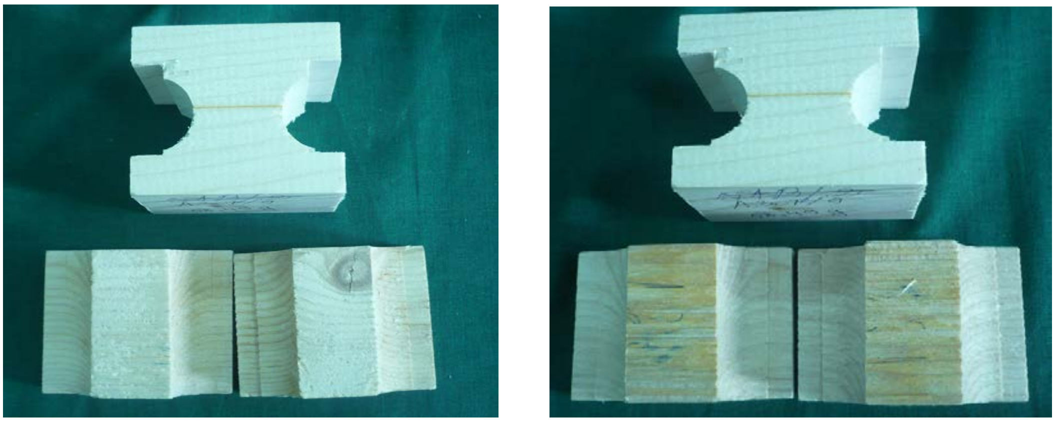
RESULTS AND DISCUSSION

The determination of the effect of acid damage to wood fibres by temperature and humidity cycling on the transverse tensile strength

The result of this test was expressed as the average transverse tensile strength of 8 replicate samples, excluding the cases where the break occurred in wood to values below a minimum admissible for the respective wood species (as required by SR EN 301:2006) or for which visual examination showed that the adhesive was not correctly applied (Table 3). Moreover, the appearance of the breaking area was a visually examined to determine the percent areas corresponding to the three types of failures defined by SR EN 302-3/2005: **A** - solid wood failure; **B** – failure along the glue line, with a fine cover of fibres visible in the failure zone; **C** – failure in or along the glue line without a fine cover of fibres visible in the failure zone. For each sample the A, B, C areas were estimated and rounded off to the nearest 10%, summing up to a total of 100%.

The pictures in Fig. 6 are examples for a failure of **type C** (for test pieces glued with the reference adhesive composition) and a combined failure of **type A** and **B** (for test pieces glued with the adhesive composition based on furan resin type URELIT FC2).



a *b*

Fig.6
The aspect of breaking area for two test-pieces after determining the effect of acid attack on the fibres on the transverse tensile strength:
a – failure of type C (100%) for reference adhesive composition UR/IRS, b- combined failure A and B (50% =50%) for adhesive composition based on mixed furan resin FC2/NH4Cl-20.

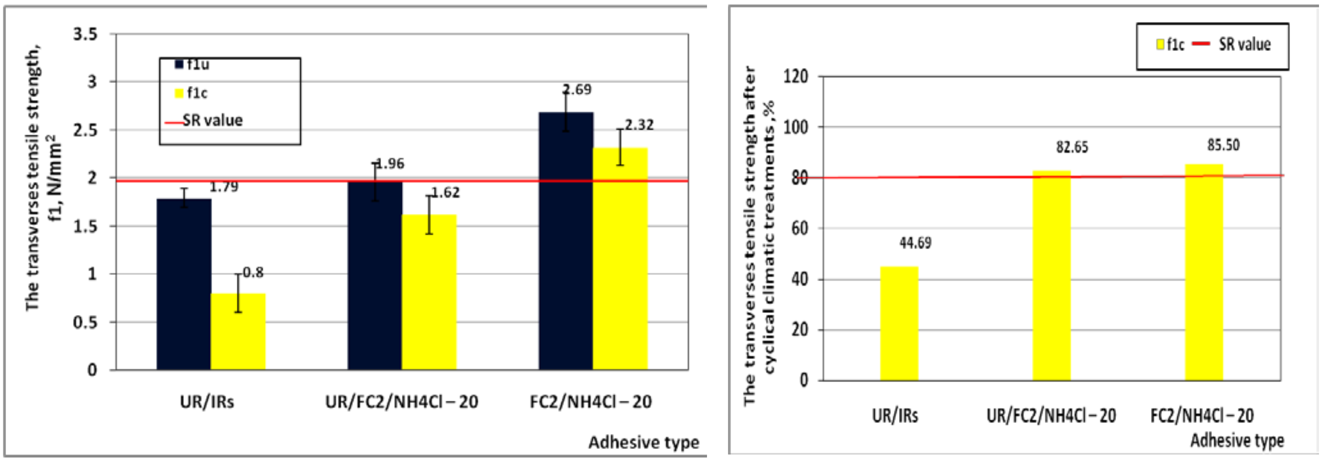
Table 3

The transverse tensile strength for the studied adhesive compositions as a result of acid attack on the wood fibres for test-pieces in dry state and after ageing under cyclic standardised conditions

No.	Adhesive compositions (Resin Type/type of catalyst)	The transverses tensile strength, f1, N/mm2				
		Required values SR EN 301:2006		Experimentally obtained Values		
		in dry state	After cyclic climatic treatments	in dry state	After cyclic climatic treatments	
					N/mm^2	% From dry state
1	UR/IRS	Min. 2	Min. 80% of the mean value obtained from the blank (dry conditions)	1.79 (0.1)	0.80 (0.2)	44.69
3	UR/FC2/NH4Cl-20			1.96 (0.2)	1.62 (0.2)	82,65
4	FC2/NH4Cl-20			2.69 (0.2)	2.32 (0.19)	85.50
Note: Standard deviations in brackets						

Numerical data show that in the case of the reference adhesive composition, **UR/IRS,** the transverse tensile strength in the dry state (without cycling) was 1.79MPa, a value slightly lower (with about 10%) than the minimum value of 2N/mm^2 imposed by SR EN 301:2006. After cyclic climatic treatments this value was reduced to approximately 44% from the initial value. The failure of the test samples was predominantly of **type C (100%) or combined of type C and B (70% / 30 %),** in both dry state and after climatic treatments (Fig. 7a).

Accordingly, it can be stated that the urea-formaldehyde adhesive Urelit R does not comply with the requirements for structural adhesives. This is not a total surprise, nor a problem, as this type of adhesive is employed for different utilisations, others than the structural ones in constructions. However, these results are taken as reference to compare the expectable performance of the mixed furan resin studied within this research.



a b

Fig. 7

The transverse tensile strength for the studied adhesive compositions as a result of acid attack on the wood fibres for test-pieces: a- values in dry state (f1u) and after cyclic ageing (f1c): b - values after cycling ageing as percentage from the initial values in dry state.

The adhesive composition based on equal weight proportion of the mixed furan resin type URELIT FC2 and urea resin URELIT R type, **UR/FC2/NH4Cl-20**, presented a higher value of transverse tensile strength in dry state (1.96MPa), compared to UR, but still slightly lower (by approximately 2%) than the minimum imposed value of 2MPa. The influence of climatic cycling was obviously less drastic, so that the value after cycling was 1.62MPa, which is more than 80% from the initial value in dry state and actually is just about the corresponding value for 80% from the minimum 2.0MPa. The test samples presented failure of **type B and C (70% / 30%)** with approximately, 70% of the surface covered with wood fibres.

In the same context, the adhesive composition based only on the mixed furan resin (URELIT FC2), coded **FC2/NH4Cl-20,** presented the highest transverse tensile strength value, respectively 2.69MPa, exceeding with approximately 34% the minimum limit imposed by SR EN 301:2006 in the dry state. After cyclic climate treatments this value was reduced to a lower extent than admissible to 85.5% from the original value. The samples failure type was also of **type B and C (80% / 20%)**, but at least 80% of the surface of the breaking area was covered with wood fibres.

Determination of influence of shrinkage of wood on shear strength.

a b

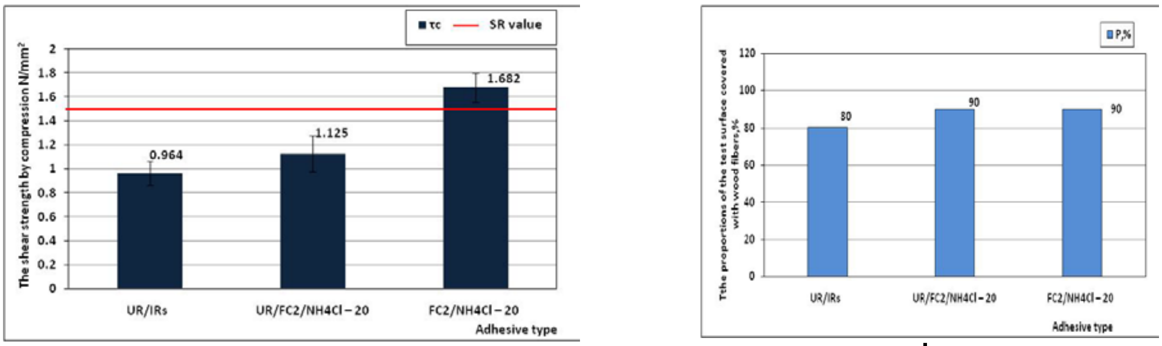
Fig. 8

Samples used for determination the influence of shrinkage of wood on shear strength by compression after testing: a – general aspect; b – front view showing the breaking area with wood failure.

The results of this test is expressed by the average shear strength of the 4 replicate samples alongside the percentage of the breaking area representing wood failure, assessed by visual examination with an accuracy of 10% (Fig. 8). These results are presented in Fig. 9 as comparative graphs for the three adhesive compositions employed in this research.

The results of this test is expressed by the average shear strength of the 4 replicate samples alongside the percentage of the breaking area representing wood failure, assessed by visual examination with an accuracy of 10% (Fig. 8). These results are presented in Fig. 9 as comparative graphs for the three adhesive compositions employed in this research.

These values show that for the reference adhesive composition UR/IRS, the shear strength by compression was 0.96MPa, a value smaller (with about 35%) than the minimum value of 1.50N/mm^2 imposed by SR EN 301:2006 (Fig. 9a). Approximately 80% of the surface in the breaking area represented wood failure (Fig. 9b).



a b

Fig. 9
The shear strength by compression (a) and the proportions of the test surface covered with wood fibres for all adhesive compositions studied after tests (b).

The adhesive composition based on equal weight proportion of the mixed furan resin type URELIT FC2 and urea resin URELIT R, **UR/FC2/NH4Cl-20**, presented a higher value of shear strength (1.12MPa), compared to UR, but still lower (by approximately 19%) than the minimum imposed value of 1.50MPa (Fig. 9a). Approximately 90% of the surface in the breaking area represented wood failure (Fig. 9b).

In the same context, the adhesive composition based only on mixed furan resin type (URELIT FC2), **FC2/NH4Cl-20,** presented the highest shear strength of 1.68MPa, exceeding with approximately 12% the minimum limit imposed by SR EN 301:2006 (Fig. 9a). Approximately 90% of the surface in the breaking area represented wood failure (fig 9b). Compared with the reference samples based on urea resin **UR/IRS,** the **FC2/NH4Cl-20** composition had higher shear strength, with approximately 70%.

CONCLUSIONS

The experimental results allowed some conclusions referring to the performance of the tested adhesives and the potential use of mixed furan resins in the formulation of adhesive compositions for structural applications.

The adhesive composition based on urea formaldehyde resin (UR/IRS), included in this research only as a reference, does not comply to the requirements for structural adhesives; both the actual values in dry state of the transverse tensile strength and shear strength and their decrease after climatic cyclic aging were bellow the imposed limits. This is in an expectable result.

The adhesive composition based on the mixed furan resin (FC2/NH4Cl-20) complies to the requirements for structural adhesives and can be considered, according to these tests, as a structural adhesive of type I**.**

The mixture composition based on urea-formaldehyde resin and the mixed furan resin **(**UR/FC2/NH4Cl-20) demonstrated a significantly improved performance compared to the reference urea-formaldehyde adhesive, but could not reach the requirements for the structural adhesives. The transverse tensile strength in dry state was only slightly lower (about 2%), while the shear strength by compression was less than minimum allowable limit with approximately 19%. However, it was obvious a much better resistance to climatic cyclic aging compared to the UR reference, which clearly demonstrates the positive effect of the mixed furan resin.

It has to be mentioned that the adhesive composition based on the mixed furan resin employed in this research complies entirely to the requirements for structural adhesives of type I, as demonstrated by the results of the other two tests imposed by the EN 301/2006: the resistance to delamination and the shear strength by tension, presented in other publications of the authors. Concluding, it can be said that the mixed furan resins have a potential as structural adhesives for wood. However, more research is needed to improve their performance by the recipe formulation to can actually compete with other types of structural adhesives for wood with better performance.

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