Andrija Bogner, Boris Ljuljka, Ivica Grbac

Methods for Testing the Resistance of Wood Hotmelt Adhesives to Temperature Changes and Weathering

Metode ispitivanja otpornosti taljivih ljepila za drvo na temperaturne i klimatske promjene

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SUMMARY • *The paper presents a research on various types of hotmelt adhesives with regard to their temperature resistance and durability. The tested adhesives were based on ethylene/vinyl acetate (EVA); polyamide (PA); polyurethane (PU); and polyolephine (PO), while the adherents were solid beech, 5 mm thick (T-shaped samples); beech veneer, 0.6 mm; PVC strip, 3 mm; paper strip, 0.6 mm; HPL laminate strip, 0.9 mm.*
The adhesives were comparatively tested with various testing methods for their resistance to weather and temperature change. All adhesives exhibited different characteristics under temperature changes; prolonged higher temperature; and cyclic climatic changes.
The aim of the research was to study the bond strength of various edging materials glued with four different hotmelts, with joints exposed to different climatic conditions, and to establish the most suitable methods for the bond strength assessment.
Key words: *hotmelts, edge-bonding strength, testing methods*

SAŽETAK • *U radu su prikazane različite metode ispitivanja otpornosti i trajnosti taljivih ljepila, kao i rezultati dobijeni tim metodama. Taljiva ljepila najčešće se koriste za lijepljenje*

The authors are a senior lecturer, a professor and an associate professor respectively, at the Faculty of the Zagreb University.
Autori su redom docent, redovni profesor i izvanredni profesor na Šumarskom fakultetu Sveučilišta u Zagrebu.
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različitih rubnih materijala na rubove ploča ili za lijepljenje aplikacija na površinu ploča. Cilj rada bio je pronalaženje najpogodnije metode pa je korištena standardna metoda, ali su razvijene i vlastite nove metode, koje su po mišljenju autora bliže realnim spojevima i uvjetima u upotrebi gotovih proizvoda finalne drvne industrije. Od standardnih metoda korišćena je metoda s uzorcima "T" oblika prema WPS 68, a novorazvijene metode se baziraju na mjerenju čvrstoće na smik odnosno čvrstoće na savijanje na uzorcima izrađenim od različitih materijala i podvrgnutim različitim temperaturnim i klimatskim promjenama. Od adheziva korišćena su ljepila na bazi etilenvinilacetata (EVA), poliamida (PA), poliuretana (PU), i poliolefina (PO). Od adherenada korišćeni su masivna bukovina, iverica, bukov furnir, PVC folija, papir HPL (High Pressure Laminate) laminat.

Ključne riječi: *taljiva ljepila, čvrstoća rubnog spoja, metode ispitivanja*

1. INTRODUCTION
1. Uvod

Since they were first introduced in the fifties, hotmelt adhesives have been increasingly used in wood processing, which may be connected with the first application of chipboards the production of furniture, where a suitable method of edge treatment and application of edge-straps was required. There are several advantages of these adhesives:

- they are simple to use and enable fast processing,
- they produce technologically firm and durable joints in just a few seconds after the application of the adhesive and edge material;
- they are environmentally friendly, and do not lose substance in the process of hardening;
- they exhibit high adhesion to wood and synthetic materials;
- there are great possibilities in chemical formulation of the adhesive in order to meet specific requirements;
- acceptable price.

With melting and hardening through cooling as the main parameters of the procedure, most adhesives are suitable thermoplasts whose melting and hardening points meet the technological requirements of the product use, and the melting temperature does not decompose them. The suitable materials for production of hotmelts are (Ljuljka, Šonje, 1990; Minford, 1991): copolymers ethylen / vinylacetate EVA, copolymers ethylen / ethylacetate EEA, polyacryls, polyamides PA, polyolephyne PO, polyurethane PU, polyesther PE.

The basic adhesive usually contains resins which improve the cohesion, adhesion, and elasticity, and reduce the price. Waxes improve water-resistance and adhesion, and reduce the price. Filling materials reduce shrinkage, increase bond strength and reduce the price of the glue. Adhesives also contain additional plastification and stabilization materials.

Besides wood industry (furniture and building), hotmelts are used in leather and shoe manufacture, textile and car industry, packaging and book-binding. Wood industry initially mostly used EVA glues, later introduced the PA and PU, and only recently the POs.

Accepting many differences among them, it is important to know the characteristic of each particular adhesive type according to its supposed use.

Chemistry, general properties and the use of the adhesives are described in the references (Ljuljka, Šonje, 1990; Minford, 1991). The general properties of the EVAs and PAs in final wood processing were analyzed by Ljuljka, Šonje, 1978; Ljuljka, 1978. The properties of the reactive PU hotmelt combined with different materials as the final product joint models were described by Pizzi 1989 and Zeppenfeld 1991. Comparative research on bond strength and joint durability with different types of glue that are used today was carried out by Šonje and Ljuljka in 1994. The aim of the research was to find the most suitable method for testing the glues. The method should be simple, capable of simulating the conditions of the product's use, and it should give reliable results that would be the base for making the choice of the proper adhesive.

2. RESEARCH METHODS AND SAMPLES
2. Uzorci i metode ispitivanja

There are diffrent methods for testing the resistance of hotmelt adhesives to the conditions that exist in the use of the finished furniture products whose various parts had been glued with the hotmelts. E.G., - we sawed a sample out of a board upon which the edge material had been glued. We put the sample into an oven, and increased the temperature by 10°C every hour. The temperature at which the edge material becomes detached is the maximum temperature for the combination board/adhesive/edge material. The method is simple, and the testing conditions similar to those in practical use. However, the evaluation of the results is somewhat subjective, because the changes are those of quality rather than quantity.

- Another method is the one named after Hessen and in more details described in the references (Ljuljka, 1978). It consists of ungluing the material and winding it on a spool, at which the strength needed for the ungluing process is measured.

- A modified Martes method acording to Ljuljka (1978) requires solid wood samples exposed to bending stress with fork shape samples, the stress ranging from 1.0 to 5.0 N/mm^2, 50°C. Ungluing begins either with a fracture, or when the fork end is lowered by 10 mm.

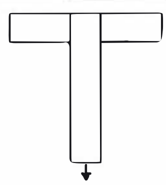
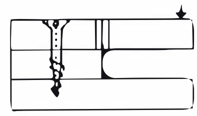
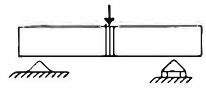
As the described methods are either complex, or they test models that present actual conditions in the plant, the samples in this research were prepared otherwise.

Table 1 is a clear presentation of testing done on some samples of adhesives and edge materials of a particular form and pattern. In Test 1, Table 1, the T-shaped samples were made of beech, 5 mm thick and a moisture contnet 10±2%. On a 400 mm^2 gluing surface the shear strength was measured on a testing machine after a particular sample treatment. The gluing process was done in accordance with the manufacturer's instructions.

The stability of the adhesives was tested for temperature change and weathering. The former was tested so that a group of 8 samples was warmed or cooled for one hour at various temperatures: -20, -10, 0, 10, 20, 30, 40, 50, 60, 70, and 80°C. Shear strength was measured on the tensile testing machine, so that no more than 5 seconds passed between the moment of removing the sample from the climatization device and the process of testing.

Weathering stability was tested so that the samples werre exposed to room temperature (20°C) and the relative air humidity R.H. of 65%, for seven days.

Table 1
Methods for hotmelt testing • Metode ispitivanja taljivih ljepila

TEST NO. *POKUS BR.*	ADHESIVES *LJEPILO*	GLUED MATERIALS *LIJEPLJENI MATERIJAL*	SAMPLE FORM *OBLIK UZORKA*	TEST TYPE *VRSTA ISPITIVANJA*
1	EVA PA PO PU	beech 5 mm *bukovina 5 mm*		-stability to temperature change -stability to weathering *-postojanost na temperaturne promjene* *-postojanost na klimatske utjecaje*
2	EVA PA PO PU	PVC 3 mm paper 0.6 mm laminate HPL 0.9 mm on particleboard edge *PVC 3 mm* *papir 0.6 mm* *laminat HPL 0.9 mm* *na rub iverice*		-stability to temperature change *-postojanost na temperaturne promjene*
3	EVA	beech veneer upon particleboard edge *bukov furnir na rub iverice*		-stability to increased temperatures -stability to weathering and increased temperatures *-postojanost na povišene temperature* *-postojanost na klimatske utjecaje i povišene temperature*
4	EVA	beech veneer upon particleboard edge *bukov furnir na rub iverice*		-stability to lower temperatures -stability to weathering and lower temperatures *-postojanost na niske temperature* *-postojanost na klimatske utjecaje i niske temperature*

The second group of samples was exposed to cyclic lab climatic conditions as follows:

- Climate 1 (20°C/65%) for one hour;
- Climate 2 (70°C/95%) for five hours;
- Climate 3 (-10°C) for five hours;
- Climate 4 (40°C/30%) for four hours.

Since we needed adequate intervals to achieve the mentioned climatic conditions, one cycle lasted 24 hours. All samples from this group were treated in seven cycles.

The third group of samples, was continuously exposed to climate of 50°C/30% R.H., lasting 21 days.

After treating the samples in the mentioned way, shear strength was measured on the testing machine and the percentage of wood failure on the fracture surface estimated for each adhesive layer and contact surface.

In Test 2 Table 1, the research was done on an 18 mm chipboard, with the same edge materials and adhesives. After testing several different sample forms, the best was found to be the one shown in Table 1. After sample climatization in room conditions, groups of 8 were exposed to varying temperatures lasting one hour each: 0, 10, 20, 30, 40, 50, 60, 70, and 80°C. The samples were inserted into the testing machine quickly, to avoid any significant change of temperature, and then tested in compression to induce shear stress.

In Test 3, the samples were made from veneer boards and bare chipboards whose edges were machine-coated with beech veneer. After applying veneer onto the edges, 74mm x 30mm x 18 mm sample halves were cut out. By gluing two halves together over the edge veneer, we made samples. The gluing was done with the PVAC adhesive. The samples were mounted to supports whose ends were loaded by the same force that caused a stress of 1 MP in the joint. Such samples were heated up to 50°C during 1.5 hours, upon which the temperature was raised by 10°C every hour in two sessions. The ungluing was monitored with regard to its time and temperature. The fracture surface was analyzed according to the percentage of the crack in the chipboard, adhesive layer and the surface between the adhesive and edge material. The second sample group in Test 3 was exposed to cyclic set of laboratory climatic conditions by the following scheme:

- Climate 1 (75°C/90%) for four hours;
- Climate 2 (-15°C) for 2.5 hours;
- Climate 3 (70°C/30%) for one hour;
- Climate 4 (20°C/65%) for one hour.

After being exposed to climatic changes, the samples were fixed on supports, loaded and heated to the temperatures of 40, 50 and 60°C for one hour. The ungluing was monitored as to its time and temperature, and the fracture surface was analyzed. In Test 4, the samples were made in the same way as in Test 3. The bending strength of samples that had been left for 8 hours at -24°C was measured in room conditions, and their fracture surface analyzed.

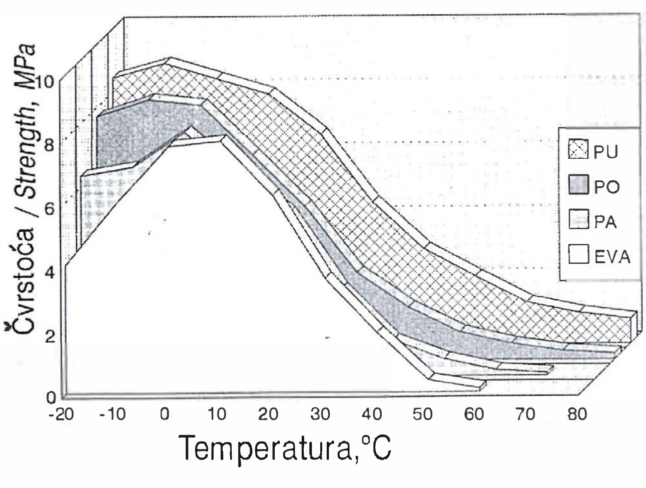
3. RESULTS AND DISCUSSION
3. Rezultati istraživanja i diskusija

3.1. Test 1
3.1. Pokus 1

The results are shown in Figure 1, showing the strength of the tested adhesives depending on temperature.

The highest strenght in a wide temperature range has been found with the PU adhesive, then the PO, PA, and EVA. At 10-40°C, the EVA has higher strength than the PA and PO, but it loses this strength more rapidly outside this temperature range. The highest strength at high temperatures was exhibited by the PU, immadiately followed by the PO adhesive.

Besides the bond strength, fracture form is another significant feature. For each sample the type and fracture proportion was



Slika 1. *Utjecaj temperature na čvrstoću spoja • Influence of temperature on bond strength*

determined. Three fracture types were established: adhesion fracture (the one on the edge of contact surface and adhesive); cohesion fracture (fracture spreads within the adhesive); wood fracture (fracture line progresses through wood). The highest proportion of fracture through wood with all adhesives was found within the temperature range of -20 to +20°C. However, the highest percentage of wood failure was seen on the PU-glued samples. This adhesive proved to have equal adhesion and cohesion connections at all temperatures. The PO adhesives had a high percentage of interface fractures up to 60°C. Beyond this, at higher temperatures, the glue fracture proportion increased. The EVA-glued samples had lower fracture proportion over contact surface at low and high temperatures. The great variablity of results with PA adhesives made any reliable conclusion almost impossible.

Bond strength with samples exposed to climatic changes is shown in Figure 2.

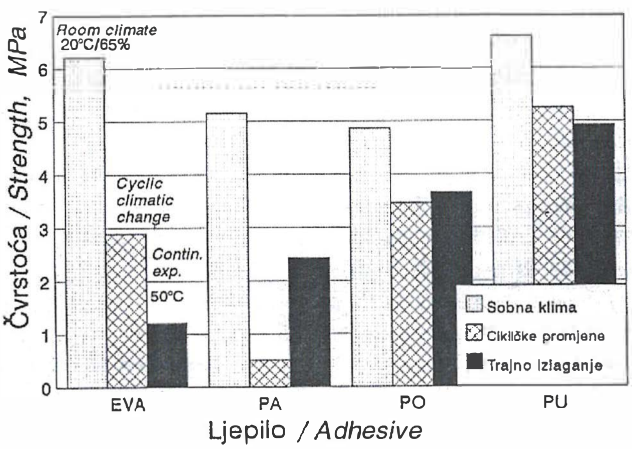
Figure 2 shows that the EVA and PA adhesives are more susceptible to long-term higher temperatures and cyclic climatic changes, where the EVA reacts to such permanent exposure with considerably lower bond strength. In contrast, the PA's reactions to cyclic changes are stronger. Both treatments of PO and PU do not cause bigger changes (25 to 30% mostly), and the bond retains a satisfactory strength. Wood fracture (the strongest link) was recorded only with PU adhesives, which is another proof of their quality. A permanent impact of temperature and cyclic changes causes decreased adhesion capabilities ofthe adhesives in the bond, particularly of the EVAs, then POs and PAs.

3.2. Test 2.
3.2. Pokus 2

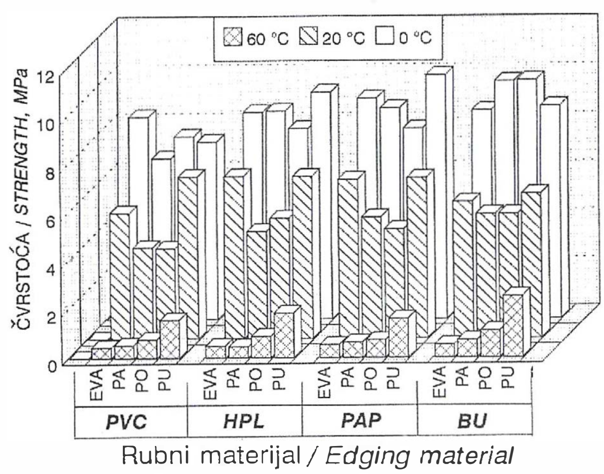
Figure 3 shows that the PU adhesives makes the strongest joints with all glued materials. When analyzing the joints with different edge materials, we found that the weakest were those with PVC. Medium strength was found in the joints with HPL, the strongest with paper and beechwood. At medium and high temperatures, adhesives were more important for the joint strength than the edge material. When analyzing the types and proportions of fracture, we found that the bonds with the EVA and PU adhesives falied within wood.

3.3. Test 3
3.3. Pokus 3

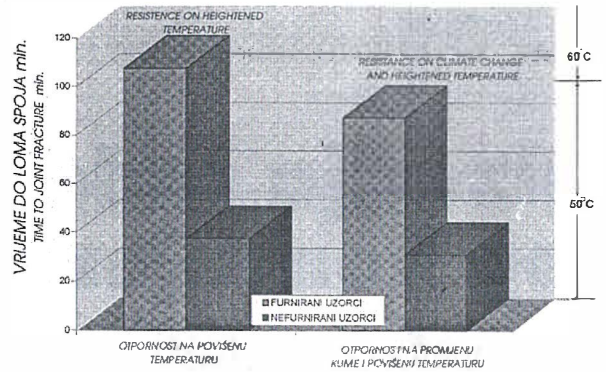
Figure 4 shows the results of testing the resistance of the EVA glue to increased



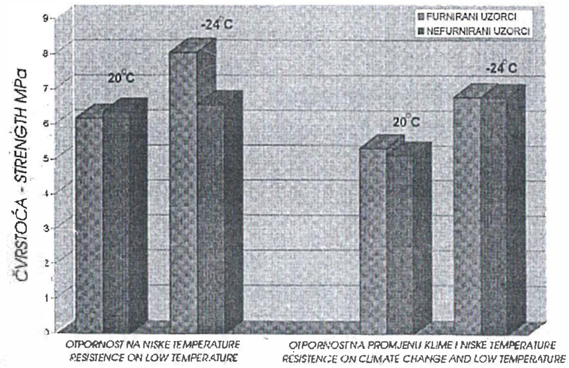
Slika 2. *Otpornost taljivih ljepila na povišenu temperaturu i cikličke klimatske promjene* • *Resistance of hot melt adhesives to elevated temperature and cyclic climatic change*



Slika 3. *Čvrstoća spojeva pri 0°, 20°, i 60°C* • *Strength of joint at 0°, 20° and 60°C*



Slika 4. Otpornost taljivog ljepila (EVA) na povišenu temperaturu i klimatske promjene • Resistance of hotmelt adhesive EVA to elevated temperature and climatic changes



Slika 5. Otpornost taljivog ljepila (EVA) na niske temperature i klimatske promjene • Resistance of hotmelt adhesive (EVA) to low temperature and climatic changes

temperature and to the change of climate with increased temperature in combination.

3.4. Test 4
3.4. Pokus 4.

Figure 5 shows the results of testing the resistance of the EVA glue to low temperatures and to the change of climate at low temperature.

Figure 5 shows that veneer samples have better resistance. An interesting fact is that the samples showed higher resistance after having been exposed to low temperature compared to the results of room temperature testing. When testing the resistance to climatic change and low temperature, the decrease in joint strength proved to be somewhat greater than with samples tested only for low temperature. At room temperature, fracture occurred mostly along the border-line between the glue and chipboard, while at lower temperature it spread along the chipboard.

The general conclusion is that in all tests permanent exposure to high temperatures or cyclic climatic changes caused a decrease in joint strength, which eventually led to joint destruction. The resistance of hotmelt adhesives in varying conditions of use is not the same. Accordingly, the most suitable adhesive may be chosen. The advantage of most tests is that they imitated real conditions of finished product in use: permanently increased temperature in the cupboards near the cooker, or in the furniture pieces transported in hot countries, or the cyclic changes on kitchen tables and cupboards, between the cooker and sink, etc. The methods used in tests 1, 2, and 4 gave quantitative indicators, while the one used in Test 3 contained qualitative indicators of the adhesive resistance to temperature and climatic changes.

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