

## Strength Increase Pattern in Joints Bonded with PVAc Adhesives

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This research project deals with PVAc adhesives, their properties, and conditions of use. The primary objective of this study is to verify the dependence of the strength increase in a bonded joint on the curing time. The bonding process used the following PVAc adhesives: Kleiberit 303, Propellerleim 3W, 4B Plus, and Provotil, conforming to resistance classes D3 and D4 pursuant to ČSN EN 204. The test objects and processing of results were in accordance with ČSN EN 205. The readings were converted into charts demonstrating the strength increase in the bonded joints. The strength required in these adhesives by the standard is 10 MPa, and it is prescribed that the test should be performed over the 7 days following the assembly bonding (ČSN EN 204). The tested adhesives achieved this value in a time period of 75 min to 165 min, depending on the adhesive type. It also follows from the measurement that the results differ depending on the sort of adhesive. Comparison of the results with those of other authors (Košíček 1974; Eisner 1966; Eisner *et al.* 1983; Sedliačik 2005) indicates that the strength increase pattern is not changed with product development and that the differences are due to adhesive modifications by the manufacturers.

*Keywords:* Adhesive; Strength increase; PVAc; Dispersion; Time

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### INTRODUCTION

Adhesives are an integral component of industrial production, and the wood processing industry is no exception. They are constantly undergoing improvement, and consumption of different adhesive types has been growing significantly.

Today, there is also an emphasis on the non-toxicity of adhesives, which has led to efforts to replace formaldehyde-based industrial adhesives with adhesives of different compositions, such as PVAc dispersions (Tout 2000; Kim and Kim 2005). Synthetic PVAc-based adhesives have been developed for industrial production, and their range of application has been growing over the last two decades, mainly due to their ever increasing application in the paper, textile, and wood processing industries (Verma and Bisarya 1986; Örs *et al.* 2004). Their chief advantage is their short curing time at temperatures starting at approximately 10 °C, their resistance to both organic and inorganic influences, their simple application, and the fact that no harmful substances (such as formaldehyde) are released (Kaboarani *et al.* 2012; Qiao and Eastal 2001; Sedliačik 2005; Eisner and Berger 1958). They are applicable both in do-it-yourself work at home and industrial batch production (Örs *et al.* 1999). PVAc adhesives, sometimes referred to as “white glue,” are made by polymerisation of the vinyl acetate monomer and then stabilised with other polymers into a copolymer (Kim and Kim 2006). The

properties of different types of PVAc adhesives are modified by the addition of various kinds of plasticisers (*e.g.*, dibutyl phthalate or tricresyl phosphate) (Habenicht 2006), filling agents (various types of nanoparticles and nanofibres) (Prolongo *et al.* 2010; Zhou *et al.* 2007), and hardening agents (*e.g.*, Al<sup>3+</sup> and Cr<sup>3+</sup> salts) (Sedliačik 2005) to improve their properties with respect to their application purpose. The adhesive curing time and time for further processing depends on the types and quantities of plasticisers and hardening agents added (Ochigbo *et al.* 2009). The curing time is one of the key parameters in the industrial application of PVAc adhesives (Eisner *et al.* 1983), and there is a requirement to reduce this time to a minimum (Brown 1999). The chief reason for this is a requirement to achieve hardness and dimensional stability of the bonded joint for further processing of the bonded components (handling, milling, grinding, cementing, painting, cutting, *etc.*) as soon as possible (Atar *et al.* 2009; Keskin *et al.* 2009). At present, manufacturers indicate the minimum pressing time, but they typically do not specify the time for further processing. Košíček (1974) studied the dependence of the strength increase in a bonded joint on time (Fig. 1); Eisner *et al.* (1966; 1983) and Sedliačik (2005) adopted his findings. No studies have been conducted more recently. Moredo and Sakuno (1991) dealt with strength measurements of three types of adhesives on apitong (*Dipterocarpus* spp.) in certain periods of time, though in this case they observed the effect of resin content in the wood on the strength of bonded joint. However the composition and production process of PVAc adhesives has been developed since that time, so their properties may differ. The hardening indicator is important in terms of planning the efficiency of continuous production, prevention of potential downtime and manufacturing defects, and minimisation of risk related to occupational safety (Uysal 2005). Working bonded components too soon after bonding may lead to the destruction of the bonded joints; conversely, too long of a curing time may result in manufacturing process delays, which are not desirable from an economic viewpoint (Adamopoulos *et al.* 2012).

The hardening of PVAc dispersions involves no chemical reaction or structural change. It is only a physical process in which the wood absorbs water from the PVAc adhesive, producing a continuous film on its surface. If a hardening agent is added to the adhesive, the adhesive mixture hardens partly physically and partly chemically (Liptáková and Sedliačik 1989).

A process known as coalescence occurs when the dispersion dries. The dispersion loses water and the polymer particles gradually come closer together until the substantial capillary forces compress them with such force that they pervade each other (bond). This produces a continuous film of polymer that is insoluble in water (Kim *et al.* 2007). Most dispersion films are colourless once they dry. Coalescence only occurs if the drying takes place at temperatures over a certain limit, known as the “minimum film-forming temperature.” At lower temperatures, the particles will not merge into a continuous film and the dry film layer remains chalk-white and crumbly (Eisner *et al.* 1983; Liptáková and Sedliačik 1989). Conversely, the mechanical strength of PVAc adhesives decreases with increasing temperature of drying; the adhesive loses its strength completely when dried at a temperature above 70 °C (Uysal 2005).

Dispersion adhesives are characterised by a relatively rapid strength increase in the bonded joint even at normal temperatures. Typically, they can achieve more than 50% of their final strength within the first hour (Eisner *et al.* 1983). This paper describes the exact process. The aim of this study is to determine the values of strength of bonded joint with use of PVAc adhesives within certain time periods, and further to compare PVAc

adhesives from various manufacturers and find out differences among them. Another objective is to compare results with other authors.

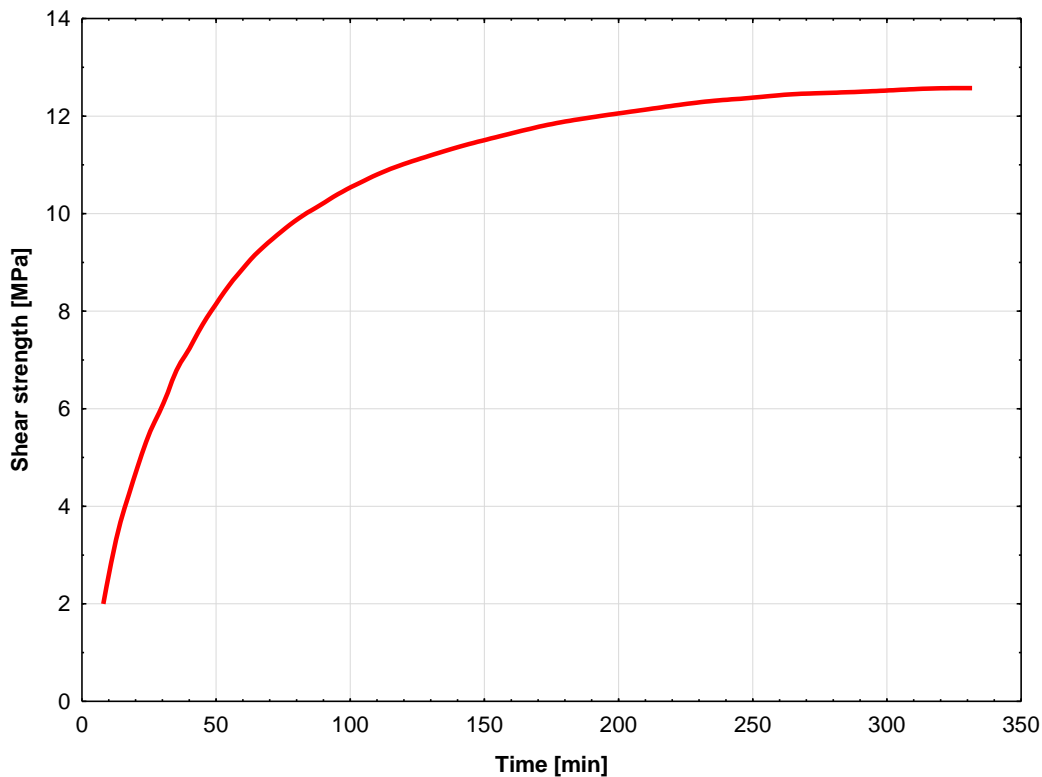


Fig. 1. Increasing strength of a joint bonded with Duvilax LS 50 over time (Košíček 1974)

## EXPERIMENTAL

### Measurement Methodology

The measurement and the preparation of tested objects were in compliance with ČSN EN 205 “Test methods for wood adhesives for non-structural applications – Determination of tensile shear strength of lap joints”.

ČSN EN 205 describes two procedures for manufacturing test objects of beech (*Fagus sylvatica*) wood with a straight direction of fibres with a nominal density of  $700 \pm 50 \text{ kg/m}^3$  and moisture content of  $12 \pm 1\%$ . The angle between annual rings and glued surface must be in range of  $30^\circ$  to  $90^\circ$  (radial or semi-radial surface). The surfaces intended for bonding must be worked by planar milling or grinding no earlier than 24 h before the bonding. Dust must be removed carefully. The surfaces prepared in this way must not be touched or dirtied.

The test objects were made by simple lapping method (Fig. 2). It is recommended that the bonded parts of these test objects are not attached to one another with the same direction of annual rings. When clamping the objects in the test machine, the tensile force must act centrally and at the plane of the adhesive layer. These conditions are achieved by using spacer inserts or through an appropriate arrangement of the clamping jigs.

This method was chosen because it enables testing of bonded samples even after a short time, unlike the first method. This fact was critical for the test in question.

The conditions of bonding (application quantity, open time, pressure) were held in accordance with values given by manufacturers of the adhesives. The glueing was performed at the temperature of  $18 \pm 2$  °C and relative humidity of 65%. For the first 90 min, the pressing times were identical with the time periods of measurements. Then the pressing time ranged from the minimal value given by manufacturer to approximately 2 h. After this time the pressing was finished and the bonded specimens were held in the above-mentioned conditions until the examination was done.

The strength of the bonded joints was measured after 5, 7, 10, 15, 20, 25, 30, 40, 50, 60, and 90 min, after 2, 3, 4, 6, 8, 12, 24, and 48 h, and after 7 days. The times shown are times after the start of pressing following the start of the test in the tensile testing machine.

The strength increase in the bonded joint was measured for three commonly available polyvinyl acetate dispersion adhesives, specifically, Kleiberit 303, made by Kleiberit; Propellerleim 3W, 4B, made by Rhenocoll; and Provotil, made by Collanti Concorde.

**Table 1.** Properties of Tested Adhesives

Adhesive	Kleiberit 303	Rhenocoll 3W, 4B	Protovil D4
Bond quality	D3	D3	D4
Application quantity [g/m <sup>2</sup> ]	100 - 200	120 - 150	180 - 250
Viscosity at 20 °C according to RVT Brookfield	13.000 ± 2.000	Not given	6.000 ± 2.000
Chalk point [°C]	+ 5	3 - 5	+ 4
Open time [min]	6 - 10	5 - 8	10
Wood moisture [%]	10 - 14	10 - 12	8 - 12
Pressure [MPa]	0.7 - 1	0.2 – 0.5	0.3 – 0.5
Pressing time at 20 °C joint bonding [min]	min. 15	6 - 8	15 - 30

#### *Test principle*

A symmetrically bonded, simply lapped joint of two symmetrical wooden adherents was conditioned under the prescribed conditions and strained with a tensile force acting parallel to the adherent fibre structure until failure (ČSN EN 205).

#### *Test device*

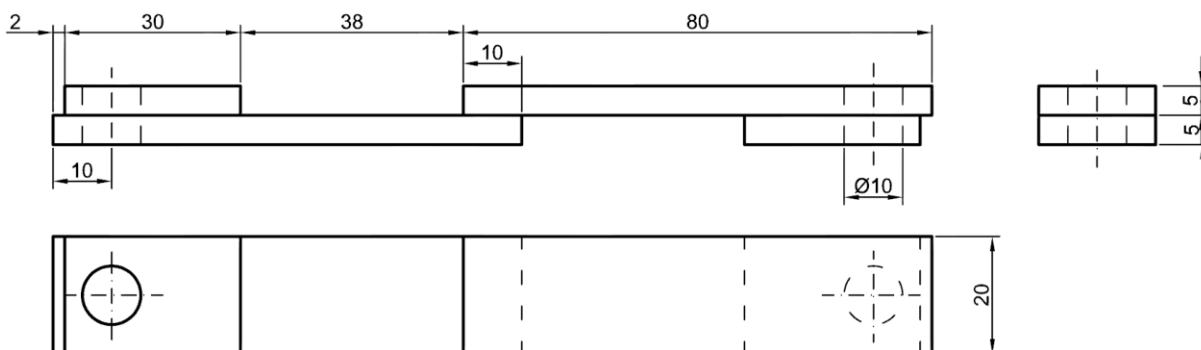
The test device was a testing machine with a constant speed of thrust as described in ISO 5893.

#### *Number of test objects*

The test was performed with enough test objects so that each test would produce at least 10 valid results. Invalid results are those where the failure occurs in the wood rather than in the adhesive layer, or where it is visually evident that the adhesive was not applied properly.

### Tensile shear test

The test objects were evaluated in a device such that both ends of the object were clamped in the machine jaws along a length of 40 to 50 mm. Arrangements were made so that the force would act centrally and along the bonding plane. The test object was strained with a tensile strength until failure, and the greatest exerted force,  $F_{\max}$ , was recorded in newtons (N). The thrust speed of the tensile testing machine was constant at 50 mm/min (ČSN EN 205).



**Fig. 2.** Diagram of the lapped test sample

### Expression of results

The strength of the bonded joint ( $\tau$ ) is expressed in newtons per square millimetre ( $\text{N}/\text{mm}^2$ ) and calculated using the following formula,

$$\tau = \frac{F_{\max}}{l_2 \times b} \quad (1)$$

where  $F_{\max}$  is the greatest exerted force in newtons (N),  $l_2$  is the length of the bonded test surface in millimetres (mm), and  $b$  is the width of the bonded test surface in millimetres (mm).

## RESULTS AND DISCUSSION

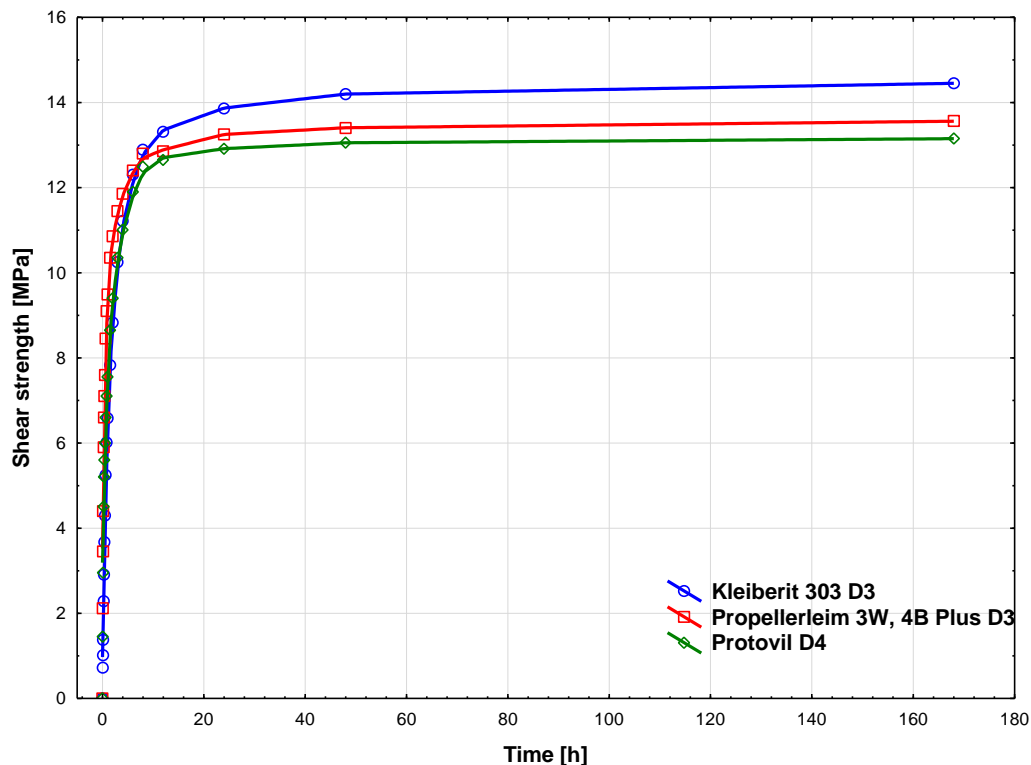
Dispersion adhesives are characterised by a relatively fast strength increase in the bonded joint, even at normal room temperatures (20 °C) and a relative humidity of around 65%. Typically, they can achieve more than 50% of their final strength within the first hour (Eisner *et al.* 1983). This statement is confirmed by the results of our measurements summarized in Tables 2, 3, and 4.

For Kleiberit 303 and Provotil, the average strengths of the bonded joints after 1 h of curing were 6.58 MPa and 7.53 MPa, respectively. Propellerleim 3W, 4B shows a remarkably fast strength increase in the bonded joint, achieving 9.49 MPa after 1 h of curing; this is near the standard minimum strength of 10 MPa, which is usually measured after 7 days of curing.

Figure 3 shows the exact process of strength increase in the bonded joint between 5 min and 7 days of curing.

**Table 2.** Overview of Measurement Readings for Kleiberit 303

Time	5 min	7 min	10 min	15 min	20 min	25 min	30 min	40 min	50 min	60 min
$\chi$ [MPa]	0.72	1.01	1.37	2.28	2.91	3.67	4.29	5.25	6.01	6.58
Max. [MPa]	0.94	1.39	1.89	2.90	3.63	4.76	5.19	6.22	7.38	8.68
Min. [MPa]	0.51	0.62	0.97	1.87	2.14	2.66	3.34	3.66	4.26	5.21
SD	0.14	0.25	0.34	0.38	0.53	0.63	0.69	0.96	1.08	1.11
$v$ [%]	19.80	24.64	25.04	16.58	18.18	17.24	16.13	18.21	18.04	16.92
Time	90 min	2 h	3 h	4 h	6 h	8 h	12 h	24 h	2 days	7 days
$\chi$ [MPa]	7.83	8.83	10.24	11.11	12.30	12.89	13.31	13.86	14.19	14.45
Max. [MPa]	9.42	10.91	12.08	13.16	15.33	15.29	15.49	16.41	15.80	16.16
Min. [MPa]	5.80	6.91	7.61	8.99	9.65	10.53	11.56	12.19	12.19	13.02
SD	1.09	1.43	1.75	1.18	1.57	1.37	1.25	1.26	1.09	1.01
$v$ [%]	13.98	16.18	17.14	10.61	12.76	10.65	9.41	9.08	7.71	6.97

**Fig. 3.** Strength increase in bonded joints over time, 5 min to 7 days

It is evident from Fig. 3 that the strength increase process was almost the same for the three adhesives tested for the period from 5 min to 8 h, and that the difference was only perceptible between 8 h and 7 days of curing. Maximum strength after 7 days reached 13.15 to 14.45 MPa. The most important practical fact is the exact pattern from the start of the curing to the point where 10 MPa is reached. Figure 4 shows this pattern.

**Table 3.** Overview of Measurement Readings for Propellerleim 3W, 4B

Time	5 min	7 min	10 min	15 min	20 min	25 min	30 min	40 min	50 min	60 min
X [MPa]	2.12	3.45	4.40	5.90	6.60	7.10	7.60	8.45	9.10	9.49
Max. [MPa]	2.43	3.75	4.89	6.59	7.30	8.26	8.91	9.42	10.44	11.19
Min. [MPa]	1.81	2.92	3.82	4.91	5.64	6.16	6.44	6.86	7.36	7.21
SD	0.18	0.31	0.37	0.46	0.60	0.67	0.81	0.96	1.05	1.30
v [%]	8.74	8.86	8.43	7.84	9.05	9.40	10.69	11.31	11.53	13.67
Time	90 min	2 h	3 h	4 h	6 h	8 h	12 h	24 h	2 days	7 days
X [MPa]	10.35	10.85	11.45	11.85	12.40	12.80	12.85	13.25	13.40	13.56
Max. [MPa]	11.92	12.91	13.06	13.86	15.43	14.76	15.00	15.36	14.80	16.32
Min. [MPa]	8.30	9.16	9.56	9.69	10.20	11.45	11.16	11.39	11.19	10.79
SD	1.07	1.40	1.28	1.20	1.38	0.88	1.15	1.22	1.09	1.55
v [%]	10.34	12.92	11.15	10.14	11.14	6.86	8.92	9.32	8.29	11.66

**Table 4.** Overview of Measurement Readings for Provotil

Time	5 min	7 min	10 min	15 min	20 min	25 min	30 min	40 min	50 min	60 min
X [MPa]	1.45	1.90	2.95	4.50	5.21	5.62	6.02	6.59	7.13	7.53
Max. [MPa]	1.69	2.19	3.39	5.10	5.90	6.76	7.31	7.51	8.44	9.71
Min. [MPa]	1.19	1.42	2.56	3.77	4.34	4.75	4.84	5.06	5.36	6.18
SD	0.15	0.27	0.32	0.46	0.52	0.65	0.79	0.91	1.03	1.01
v [%]	10.39	14.41	10.73	10.13	9.88	11.59	13.19	13.79	14.44	13.47
Time	90 min	2 h	3 h	4 h	6 h	8 h	12 h	24 h	2 days	7 days
X [MPa]	8.65	9.39	10.35	11.01	11.90	12.49	12.65	12.91	13.06	13.15
Max. [MPa]	10.22	11.22	11.96	13.01	14.93	14.68	14.80	15.21	14.65	14.98
Min. [MPa]	6.60	7.76	8.46	8.84	9.70	11.75	10.96	11.24	11.04	11.66
SD	1.07	1.32	1.28	1.19	1.38	0.81	1.20	1.28	1.18	0.94
v [%]	12.38	14.06	12.33	10.85	11.61	6.51	9.49	9.92	9.07	7.17

X – average value

Max. – maximum measured value

Min. – minimum measured value

SD – standard deviation

v – coefficient of variation

The fastest strength increase was seen in Propellerleim 3W, 4B, which achieved 10 MPa after 75 min of curing. Kleiberit 303 showed the slowest strength increase, achieving the standard-defined 10 MPa in 165 min. The growth curve for Provotil was in between the aforementioned adhesives, achieving the standard-defined value in 150 min. The situation changed after some time; Kleiberit 303, while showing the lowest increase over short times, equaled the strength of Provotil at 200 min and showed a greater strength increase after that. At 405 min, its strength equaled that of Propellerleim 3W, 4B, and it subsequently maintained the greatest strength until 7 days after bonding.

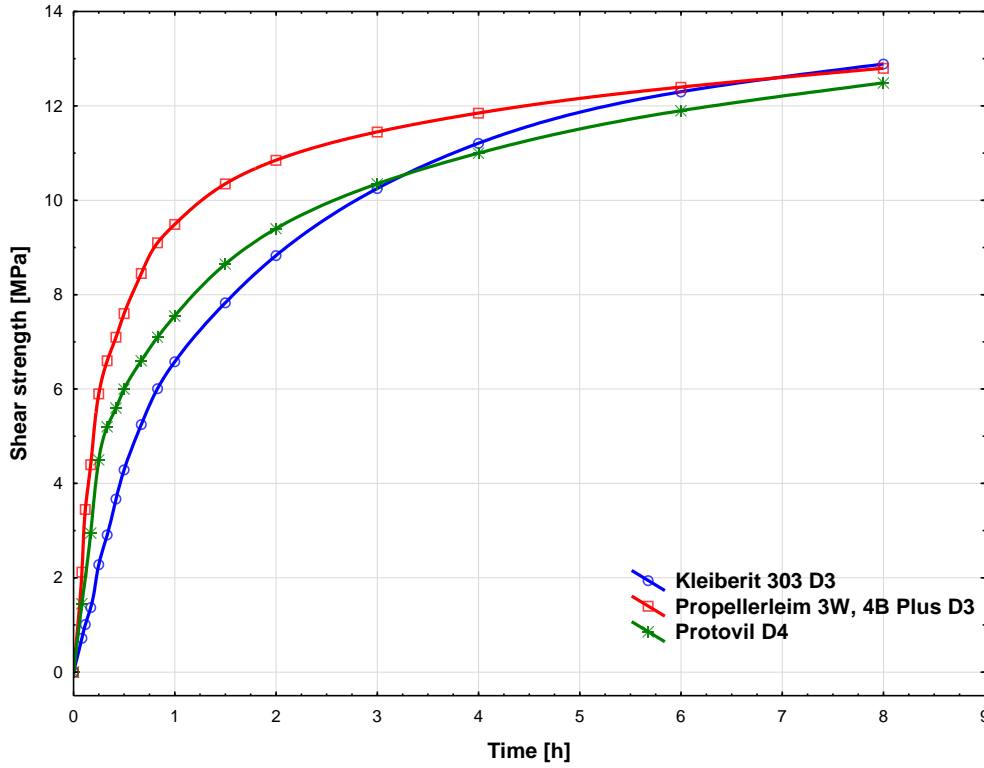


Fig. 4. Strength increase in bonded joints over time, 5 min to 8 h

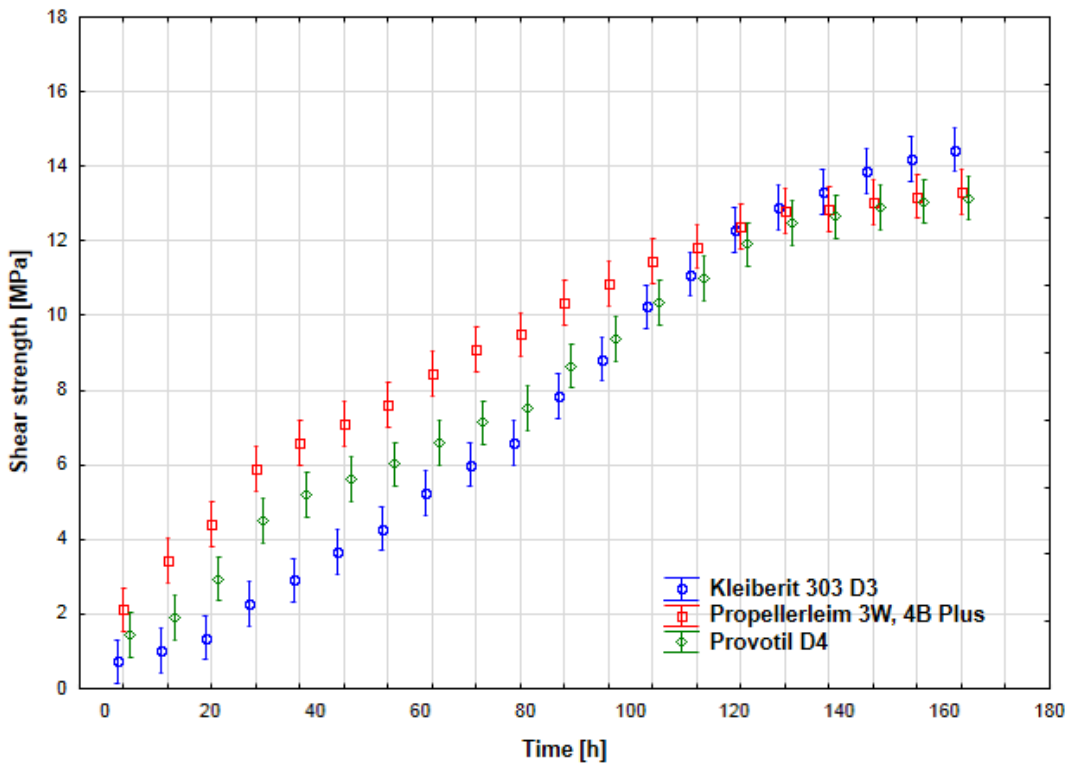


Fig. 5. ANOVA – Vertical columns stand for 0.95 reliability intervals



Comparing the results with those reported by Košíček (1974; Fig. 1), it is evident that there are types of PVAc adhesives with a strength increase greater than the Duvilax LS 50 adhesive tested in that study. Although Duvilax LS 50 achieved the strength of 10 MPa after 90 min, Propellerleim 3W, 4B reached that value in 75 min. The other two adhesives (Kleiberit 303 and Provotil) achieved the value later (150 and 165 min, respectively). This means that PVAc adhesives have evolved since 1974, but the evolution has not affected the speed of the strength increase in the bonded joint.

The differences between the adhesives were tested using a multi-factor ANOVA (Fig. 5). Propellerleim 3W, 4B Plus showed a statistically significant difference between 7 and 180 min of pressing time, over which range it showed the highest strengths. After 180 min of pressing time, its strength decreased and fell below the values of Kleiberit 303. The strength of this adhesive demonstrably differed from that of Propellerleim 3W, 4B Plus from the very start of measurement (5 min) until 180 min: it showed the lowest strength values. From 2 to 7 days of curing, its strength demonstrably increased compared to the other adhesives tested. Provotil demonstrably differed from Propellerleim 3W, 4B Plus between 7 min and 180 min of curing; it differed from Kleiberit 303 between 7 min and 50 min of curing and then from 2 to 7 days of curing.

## CONCLUSIONS

1. PVAc adhesives are characterized by a very fast strength increase, which begins in the initial curing period. The standard strength for these adhesives is defined as 10 MPa, and ČSN EN 204 requires that the test be performed 7 days after bonding the assembly. These adhesives achieve the value in a time period from 75 min to 165 min, depending on the adhesive type.
2. The results indicate that the ultimate strength does indeed only stabilize after 7 days of curing; nevertheless, the standard-defined value can be achieved in a time 134 times shorter with one selected adhesive (Propellerleim 3W, 4B). The ultimate strength of the adhesives tested stabilised at values from 13.15 MPa (Provotil) to 14.45 MPa (Kleiberit). It was determined that the adhesive with demonstrably the highest strength increase in the bonded joint may not necessarily show the highest final strength (Propellerleim 3W, 4B), conversely, the adhesive with the slowest strength increase may well achieve the highest final value (Kleiberit 303).
3. Furthermore, it follows from the measurements that the results differ from manufacturer to manufacturer. Comparison of the results with those of other authors (Košíček 1974) indicates that the strength increase pattern has not changed with technical advancement and that the differences are due to the chemical composition set by the manufacturer.

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