Synergistic Effects of Impregnation Agents Used in Plywood Production Relative to the Shear Strength of Bonded Joints

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The main objective of this research was to analyse the synergistic effects of impregnation agents that are most commonly used in plywood production on the strength of bonded joints. Two water-based impregnations (Teknol aqua 1410-01 and Cetol WV 885 BPD+) and one solvent-based impregnation (Gori 605) were used in this study. The tested adhesives were polyvinyl acetate (PVAC) ADHESIVE 3384 (1-component PVAC adhesive), EPI (emulsion polymer isocyanate) system 1920/1993, and polyurethane (PUR) 1968 (1-component PUR adhesive). Test specimens were made from European beech timber (*Fagus sylvatica L.*) for plywood production according to EN 205 (2017). Individual tests were conducted according to EN 204 (2001) for PVAC adhesives, and according to EN 12765 (2001) for PUR adhesives. Although the effect of impregnation on bond strength was demonstrated, it was different depending on the type of adhesive. A clear positive synergistic effect on the bond strength was shown for isocyanate-based adhesives in almost all of the tests. The synergistic effect of PVAC adhesive was very variable in each individual test and showed a negative influence of higher moisture content and dipropylene glycol content in solvent-based impregnation.

Keywords: PVAC; Isocyanate; Plywood; Impregnation agent; Adhesive; Shear strength

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INTRODUCTION

Adhesives are an integral part of the timber industry. Impregnation agents, which together with adhesives allow for better use of wood-based materials that were previously regarded as wastes, prolong its service time (Bomba *et al*. 2014). Adhesives have replaced joints where mechanical bonding measures previously had been used (Örs *et al*. 2000). However, all of these chemical compounds are inconsistent with the quality of the living environment and the terms of hygienic safety, in particular in the case of formaldehyde emissions (Böhm *et al*. 2012; Salem *et al*. 2017). Therefore, it is preferable to use adhesives that are not synthesized with formaldehyde, such as PVAC or PUR adhesives (Šmidriaková *et al.* 2015), which are popular and widely used (Král *et al.* 2015). This trend can be also seen in the plywood production industry, where the consumption of these adhesives and impregnation compounds has increased in recent years (Salem *et al*. 2013).

PVAC adhesives belong to the group of thermoplastics that was originally designed for the production of battenboard and plywood for the furniture industry (Tout *et al.* 2000; Clauß *et al.* 2011; Šedivka *et al.* 2015). PVAC adhesives, which are mostly manufactured as aqueous dispersions, have good adhesion properties (Skiest 1990). Polyurethanes are another widely used class of formaldehyde-free adhesives; they are likewise commonly used for furniture and joinery products (Desai *et al.* 2003) as well as textile, packaging, and automotive products (Vick and Okkonen 1998). Adhesives based on isocyanates are widely used for construction purposes. In particular, single-component polyurethane adhesives have become popular in recent years on the European wood adhesive market due to their ease of use. Both polyurethanes and PVAC adhesives are known for their good adhesion, flexibility, curing at low temperatures, and easy formation of hydrogen bonds to wooden surfaces (Vick and Okkonen. 1998). Joints bonded with PVAC adhesives exhibit good shear strength, conforming to the requirements for assembly bonding (Sedliačik 2005). The advantage of these adhesives is that they form a colourless bond, are easy to apply, and harden quickly without the use of heat (Frangi *et al.* 2004).

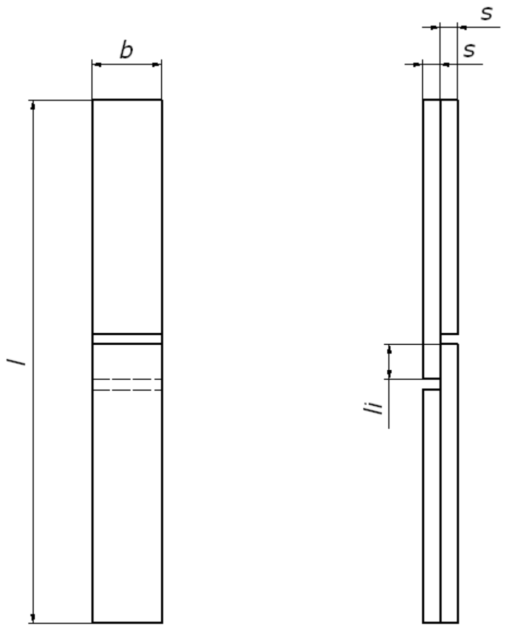
Wood is a natural organic material that can be degraded by biological agents (*i.e.* bacteria, fungi, insects) and by physical effects such as fire. Wood use is therefore limited by its sensitivity to such factors, which can damage or completely destroy its structure and worsen its properties (Reinprecht 2010). For this reason, wood materials should be coated with a protective agent suited for wood material and its use (Örs *et al*. 2004; Uysal 2006; Keskin *et al.* 2009). When wood materials are impregnated, their lifespan in outdoor applications may be seven to eight times greater than that of non-impregnated wood materials (Özçifçi and Okçu 2008), and impregnation agents therefore also play important role in wood technology and are very often used in combination with PVAC and PUR adhesives in formaldehyde-free plywood production (Böhm *et al*. 2012). A very popular adhesive system is the combination of both PVAC and polyurethane adhesives – an emulsion polymer isocyanate system that takes advantages of both of the mentioned adhesives (Šedivka *et al.* 2015). The most common agents used for impregnation of veneers for plywood production are those based on a derivate of 1,2,4-triazole such as propiconazole or tebuconazole, based on carbamates, for instance 3-iodo-2-propynyl-butylcarbamate or based on thiazole or benzothiazole derivates such as 1,2-benzothiazole-3(2H)-one (Reinprecht 2012). It can be expected that water based agents will affect the shear strength of glue bonds. It is also likely that some of the compounds contained in agents can initiate a reaction with adhesives resulting in either a positive or negative impact on shear strength. These phenomena are able to cause serious problems in the manufacturing processes, leading to lower quality of final wood products. The main objective of this work was to detect, measure, and analyse the effect of the previously mentioned impregnating agents on the shear strength of bonded joints.

EXPERIMENTAL

Materials

The measurements and preparation of test specimens were performed according to standards EN 205 (2017), EN 204 (2001), and EN 12765 (2001) (Fig. 1).

The test specimens for experimental measurements were made according to the procedure defined in standard EN 205 (2017). A total of 14 test specimens were produced for each measured group. The specimens were made from a strong un-steamed and conditioned board made from beech (*Fagus sylvatica* L.) with straight fibres, a nominal density of 700 kg/m^3 ± 50 kg/m^3, and a moisture content of 12% ± 1%. The angle between the annual rings and the surface of the panel varied between 30° and 90°. For test specimens that were bonded using PVAC adhesives, three types of tests were performed according to EN 204 (2001) for the D4 durability class. Three types of tests were performed for specimens that were bonded using the PUR adhesive, and they are specified in EN 12765 (2001) for the C4 durability class.



l= 150 mm ± 5 mm
li= 10 mm ± 0.2 mm
b= 20 mm ± 0.2 mm
s= 5.0 mm ± 0.1 mm

Fig. 1. Test specimens according to EN 205 (2017)

The test specimens were bonded using PVAC and EPI adhesives in the D4 durability class, and one PUR adhesive in the C4 durability class. The PVAC adhesive was PVAC 3384, which was a single-component polyvinyl acetate adhesive with properties that corresponded to EN 204-D4 (2001).

Table 1. Properties of Tested Adhesives (in accordance with datasheets)

Adhesive	PVAC ADHESIVE 3384	EPI system 1920/1993	PUR 1968
Adhesive durability class	D4	D4	C4
Application Quantity [g/m^2] (Adhesive applied to both adherents)	200	200	200
Viscosity at 20 °C [mPas]	3000 to 9000	4000 to 9000	2000 to 4000
Min. Processing Temperature [°C]	+ 10	+ 5	+5
Open Time [min]	8	15	25
Wood Moisture when gluing [%]	10 - 14	10 - 14	10 - 14
Pressure [MPa]	0.8	0.8	0.8
Density [kg/m^3]	1100	*	1160
Pressing time at 20 °C joint bonding [min]	40	40	40

Note: * Characteristic not stated by the supplier

The EPI adhesive that was used was EPI 1920/1993, which was a two-component polyvinyl acetate adhesive with addition of isocyanate and its properties corresponded to EN 204-D4 (2001). The third used adhesive was PUR 1968, which was a single-component polyurethane with properties that corresponded to EN 12765-C4 (2001). The specimens were impregnated with three impregnating agents. Two of the impregnating agents were water-based Teknol aqua 1410-01 (Finproduct, Prague, Czech Republic) Cetol WV 885 BPD+ (AkzoNobel, Brno, Czech Republic), and one impregnating agent was solvent-based Gori 605-00 (Finproduct, Prague, Czech Republic). The parameters of the adhesives and impregnations are specified in Tables 1 and 2.

Table 2. Properties of Impregnation (in accordance to datasheets)

Impregnation	Teknol aqua 1410-01	Cetol WV 885 BPD+	Gori 605-00
Active Substances Concentration	3-iodo-2propynyl-butylcarbamate (0.3-0.8%) Propiconazole (0.3-1%) 1,2-Benzothiazole-3(2H)-one (<0.05%)	3-iodo-2propynyl-butylcarbamate (0.6%) Propiconazole (0.9%) Tebuconazole (0.3%)	3-iodo-2propynyl-butylcarbamate (0.8%) Propiconazole (0.3-1%) 1,2-Benzothiazole-3(2H)-one (<0.05%)
Main Solvent	Water	Water	Naphtha (Heavy), Tall oil, Dipropyleneglycol methylether
Temperature during the use [°C]	20 ±1	20 ±1	20 ±1
Wood Moisture [%]	12 ±1	12 ±1	12 ±1
Drying Time [h]	2.5 h	4.5 h	3
Consumption [ml/m^2]	120	120	120

Methods

The measurement consisted of the fact that when using all impregnations, three types of tests were performed for each adhesive according to the relevant standards. A total of 14 test specimens were created for each conditioning sequence.

Conditioning sequences 1, 3, and 5 were performed for the PVAC adhesives according to EN 204 (2001) for durability class D4. In conditioning sequence 1, after 7 days of conditioning under normal conditions (temperature 20 °C ± 2 °C and humidity of 65% ± 5%), the specimens were tested on test machine UTS Testsysteme 50 (Denkendorf, Germany), and here the strength had to be ≥ 10 MPa. In conditioning sequence 3, after 7 days of conditioning under normal conditions, the specimens were placed into water at a temperature of 20 °C ± 5 °C for 4 days, and here the strength of the bond had to be ≥ 4 MPa. In conditioning sequence 5, after 7 days of conditioning under normal conditions, the specimens were placed into boiling water for 6 h, and then into water at a temperature of 20 °C ± 5 °C for an additional 2 h. For this test, the strength must be ≥ 4 MPa.

Tests with conditioning sequence types 1, 2, and 4 were performed for PUR adhesives according to EN 12765 (2001) for durability class C4. Type 1 proceeds in the same manner as for PVAC adhesives. In the type 2, after 7 days of conditioning the specimens were placed into water for 1 day at a temperature of 20 °C ± 5 °C, and for this test the strength had to be ≥ 7 MPa. In type 4, after 7 days of conditioning the specimens were placed into boiling water for 3 h, and then into water at a temperature of 20 °C ± 5 °C, and for this test the strength had to be ≥ 4 MPa.

The reference test specimens were first created for each adhesive type, *i.e.* specimens where impregnation was not used. For samples to be impregnated, were those first dipped into the impregnation and then bonded with adhesive to simulate use in real production.

A shear tensile test was conducted according to EN 205 (2003), wherein the specimen was encumbered until it was breached, and the highest exerted force was recorded in Newtons.

Test equipment

The test equipment used was a testing machine (UTS Testsysteme, Denkendorf, Germany) with a constant feed rate as described in ISO 5893 (2012).

Transverse tensile test according to EN 205

Test specimens were torn apart in a test machine (UTS Testsysteme, Denkendorf, Germany), where the specimens were affixed in clamps on both ends at a length of 40 mm to 50 mm. The specimens were encumbered using tensile force until they were breached, and after breaching, the highest F_{max} force in Newtons (N) was recorded. The feed speed of the test machine for PVAC adhesives was 50 mm/min, and for PUR the strength of the feed was determined between 6 mm/min to 12 mm/min, in this case the selected speed was 10 mm/min.

Expression of results according to EN 204

The shear strength of a bonded joint (τ) was expressed in MPa and calculated according to Eq. 1,

$$\tau = \frac{F_{\max}}{l_2 \times b} \tag{1}$$

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

The evaluation of the results was conducted at a significance level of $\alpha = 95\%$ using analysis of variance ANOVA (Statistica 12, Tulsa, USA). A total of 14 test specimens were tested for each combination and tested for extreme values using the Dixon test. For test specimens where an error was detected (*e.g.*, a small split groove), the measured values were immediately discarded; however, there was a minimum of 10 valid samples in every case.

RESULTS AND DISCUSSION

The results for the individual adhesives and impregnation are specified in Tables 3 through 5, which show the values of the basic statistical indicators for each combination of adhesive and impregnation agent.

Table 3. Overview of Measurement Readings for PVAC Adhesive 3384

PVAC 3384						
Type of Impregnation	Reference			Teknol aqua 1410-01		
Conditioning sequence	1	3	5	1	3	5
X [MPa]	15.44	5.19	3.36	10.68	2.56	DL
Max. [MPa]	17.13	6.36	4.28	12.87	4.03	DL
Min. [MPa]	13.88	4.2	3.15	9.19	0.87	DL
SD	1.24	0.59	1.17	1.24	0.95	DL
CV [%]	8	11.36	34.79	11.61	37.00	DL
Type of Impregnation	Cetol WV 885 BPD+			GORI 605		
Conditioning sequence	1	3	5	1	3	5
X [MPa]	10.55	DL	DL	11.89	4.56	DL
Max. [MPa]	11.65	DL	DL	13.48	6.82	DL
Min. [MPa]	8.63	DL	DL	10.20	3.02	DL
SD	0.87	DL	DL	1.21	1.33	DL
CV [%]	8.21	DL	DL	10.16	29.25	DL

Note: X- mean value; Max.- maximum measured value; Min.- minimum measured value; SD- standard deviation; CV - coefficient of variation; DL – delaminated or non-measurable value

It is obvious from Table 3 that the PVAC adhesive is not designed to stand direct water contact in conditioning sequence 3 (Bomba *et al.* 2014), nor its combination with high temperature in conditioning sequence 5 (Šedivka *et al.* 2015). The test with conditioning sequence 5 resulted in delamination for most of the specimens, and the rest of them, which remained integrated, were too disturbed to be able to measure any value.

Table 4. Overview of Measurement Readings for EPI System 1920/1993

EPI system 1920/1993						
Type of Impregnation	Reference			Teknol aqua 1410-01		
Conditioning sequence	1	3	5	1	3	5
X [MPa]	12.88	3.34	DL	11.72	5.21	DL
Max. [MPa]	14.16	5.75	DL	13.52	6.27	DL
Min. [MPa]	11.64	1.67	DL	9.41	4.12	DL
SD	0.91	1.18	DL	1.51	0.93	DL
CV [%]	7.09	35.31	DL	12.89	17.77	DL
Type of Impregnation	Cetol WV 885 BPD+			GORI 605		
Conditioning sequence	1	3	5	1	3	5
X [MPa]	15.39	4.27	DL	12.01	5.12	DL
Max. [MPa]	17.74	5.75	DL	13.66	6.67	DL
Min. [MPa]	10.91	2.67	DL	9.00	3.15	DL
SD	2.09	1.00	DL	1.66	1.19	DL
CV [%]	13.59	23.31	DL	13.81	23.27	DL

Note: X- mean value; Max.- maximum measured value; Min.- minimum measured value; SD- standard deviation; CV - coefficient of variation; DL – delaminated or non-measurable value

The results of the EPI adhesive are summarized in Table 4, from which is clear that the isocyanates contained in the adhesive provided better moisture resistance than in the case of PVAC. Although as expected, since the isocyanate contend was still low, the dispersion part of the adhesive could not handle the immersion of the samples in water during conditioning sequence 3, and certainly not high temperatures as in the case of PVAC. What is most interesting is the fact that one can observe a slight positive synergy effect of impregnating agents when comparing the results to those of the reference.

Table 5. Overview of Measurement Readings for PUR 1968

PUR 1968						
Type of Impregnation	Reference			Teknol aqua 1410-01		
Conditioning sequence	1	2	4	1	2	4
X [MPa]	11.36	2.50	1.60	12.92	5.94	6.29
Max. [MPa]	12.15	3.81	2.40	14.17	7.82	7.66
Min. [MPa]	10.32	1.24	0.53	11.20	4.14	5.30
SD	0.58	0.87	0.66	0.83	1.20	0.79
CV [%]	5.12	34.68	41.45	6.44	20.22	12.58
Type of Impregnation	Cetol WV 885 BPD+			GORI 605		
Conditioning sequence	1	2	4	1	2	4
X [MPa]	12.41	5.12	4.25	12.72	2.44	2.28
Max. [MPa]	14.83	5.84	6.40	13.98	3.23	3.93
Min. [MPa]	10.70	4.00	2.46	11.28	1.31	0.58
SD	1.14	0.67	1.24	0.97	0.66	1.12
CV [%]	9.22	13.17	29.16	7.59	27.25	49.03

Note: X- mean value; Max.- maximum measured value; Min.- minimum measured value; SD- standard deviation; CV - coefficient of variation

In Fig. 2, the bond strength values for all adhesives for conditioning sequence 1 test are specified; here, the testing procedure for PVAC and PUR was the same. Other types of tests differ for the PVAC and PUR adhesives.

According to EN 204 (2001), the bond strength value should have been ≥ 10 MPa, and therefore, for conditioning sequence 1, all of the adhesives met the standards when all of the impregnations were used. The red line on Figs. 2 and 3 shows the value below which strength must not fall.

It is clear from Fig. 2 that the PVAC 3384 adhesives without impregnation exhibited the highest strength. Adversely, the PVAC 3384 adhesives exhibited the lowest strength in cases where water-based agents Teknol aqua 1410-01 and Cetol WV 885 BPD+ were used. This is most likely caused by the fact that despite drying process according to the technical data sheets, there could still be higher moisture content in the wood samples during gluing process (Bomba *et al.* 2014). However, one can observe a reasonable drop in shear strength in the combination of PVAC adhesive and solvent-based agent where there is no moisture increase expected. This can be a result of using the solvent dipropylene glycol methyl ether, whose part dipropylene glycol is often used as plasticizer in PVAC adhesives (Skeist 1990). Dipropylene glycol is also known for hydrogen bond breaking (Cho *et al.* 2009).

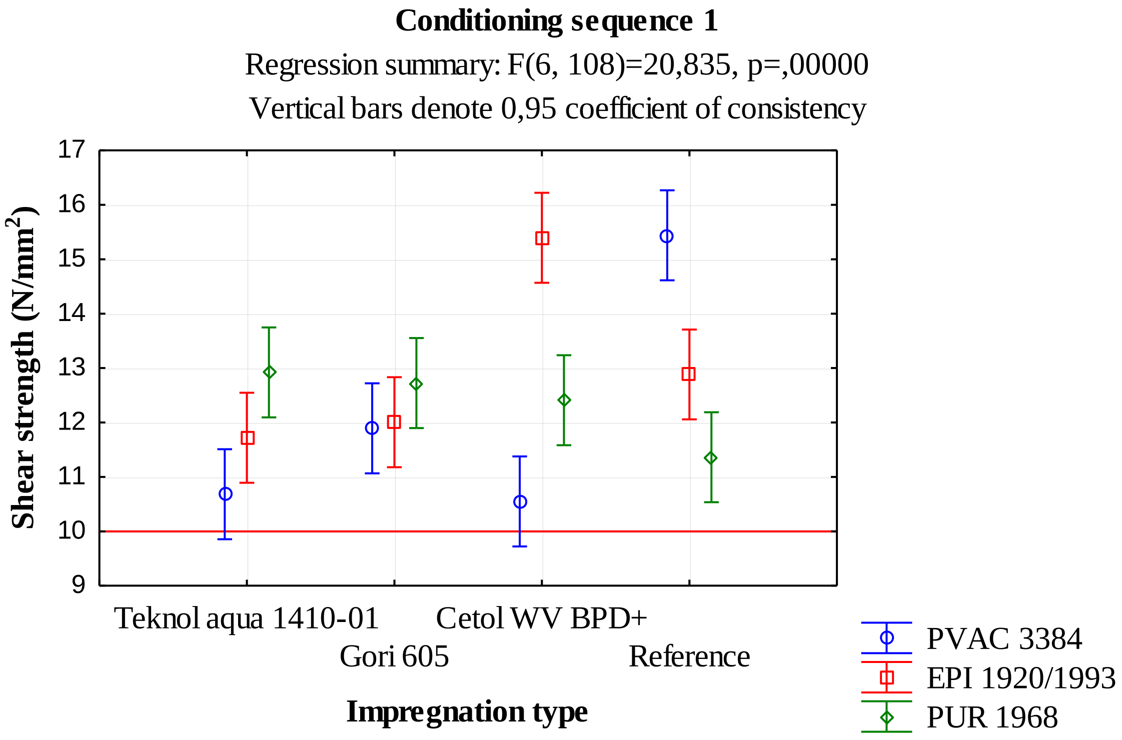


Fig. 2. Bond shear strength for conditioning sequence 1

For EPI 1920/1993 with the use of Cetol WV 885 BPD+, an extreme increase in shear strength was observed. As expected, the reactions with other impregnation agents were slightly better than the behaviour of PVAC due to isocyanates used as a hardener in EPI 1920/1993. Nevertheless, the high increase in strength in combination with Cetol WV BPD+ was surprising. One possible explanation involves a chemical reaction between the Tebuconazole substance and isocyanates, which is the only different substance compared to the other water-based agent. However, further testing is needed to investigate this phenomenon.

The shear strength values for PUR 1968, in cases where impregnations were used, were higher than for the reference test specimens, which is likely caused by the same phenomenon that decreased the strength of PVAC, as polyurethanes harden when receiving moisture (Bomba *et al.* 2014). The highest strength value was found during the use of Teknol aqua 1410-01, wherein strength increased by 13.7%. However, what is interesting is that the strength was also increased for the solvent-based agent. A possible source of this increase is again the dipropylene glycol contained in the GORI 605 solvent, which is known as a chain extender and crosslinker of polyurethanes (Martin and Murphy 2000). In practice, this creates polymer enforcement, as isocyanates and extenders form a stiff, unmoving polymer component called the called hard segment (Blackwell *et al.* 1982). These segments, which are oriented perpendicularly to the soft segment, have a covalent bond to the soft segment of polyurethanes, which are formed mostly from polyols (Desai *et al.* 2004). When loaded, the soft segment is stressed, and the hard segment is aligned in the stress direction (Blackwell and Gardner 1979). This phenomenon leads to an increase in strength and tear resistance (Musselman *et al.* 1998; Lin *et al.* 2017). This is also partially caused by 3-Iodo-2-propynylbutylcarbamate (IPBC) contained in all of the tested impregnation agents. IPBC has very good bonding ability to both wood and isocyanates, as IPBC is a carbamate group, in other words, the group of urethanes. Such compounds are known for their significant mutual conversion ability, and they therefore are often used as additional fungicides in adhesives (Mazela *and* Perdoch 2012).

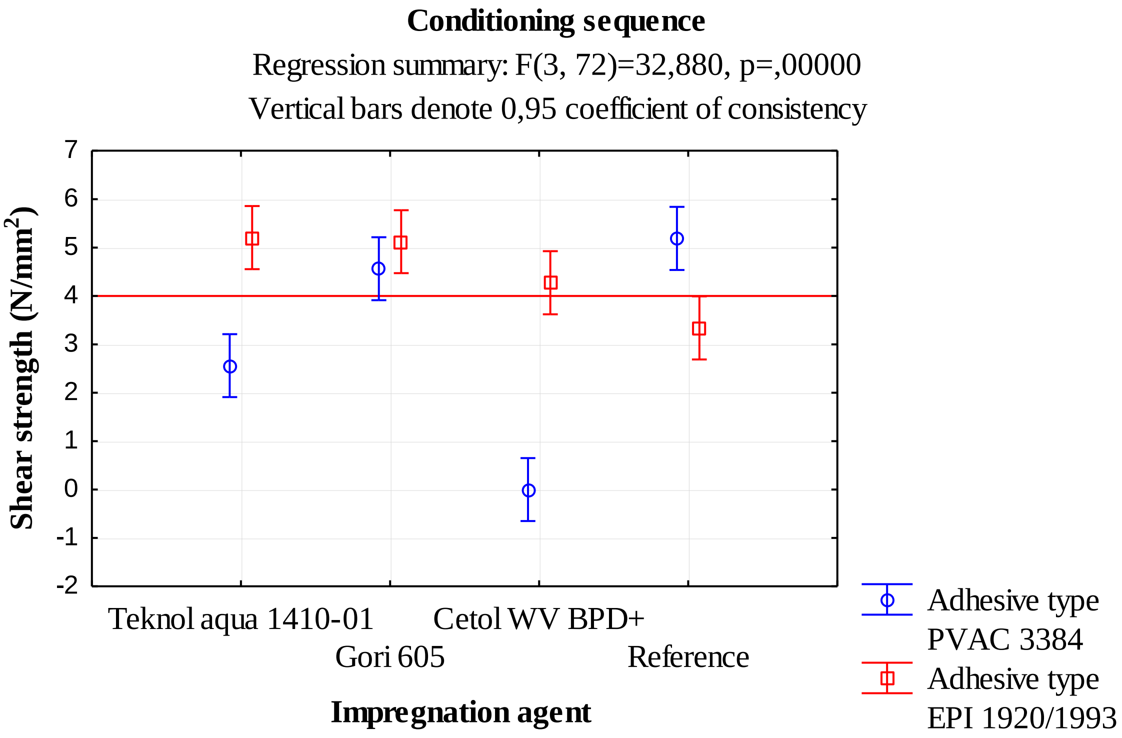


Fig. 3. Bond strength for conditioning sequence 3

Figure 3 shows a comparison of the strengths of PVAC 3384 and EPI 1920/1993 for conditioning sequence 3, where the samples were in cold water for 4 days. According to the standard, the strength value should have been ≥ 4 N/mm^2, but this value was not achieved for all of the used impregnations.

As expected, the PVAC adhesive only met the EN 204 (2001) standard in case of the reference test specimens and when solvent-based impregnation GORI 605 was used. This is again caused by the absence of slightly higher moisture during the gluing of samples as in conditioning sequence 1 (Bomba *et al.* 2014). For Cetol WV 885 BPD+, during the test, most of the specimens delaminated, or nothing was measured on the test machine. When the Teknol aqua 1410-01 impregnation was used, the strength value was under 4 N/mm^2. When compared to conditioning sequence 1, the results fit very closely and also corresponds to the results published in the work of Bomba *et al.* 2014.

EPI 1920/1993 met the standard for all of the used impregnations, but it did not meet it for reference test specimens for which the strength value was under the standard limit of 4 N/mm^2. The highest strength was found for Gori 605 and Teknol aqua 1410-01, and when these two impregnations were used, the strength value was approximately the same. This test confirms the theory that an increase in strength is caused by the 3-iodo-2-propynylbutylcarbamate (IPBC) contained in all of the tested impregnation agents.

There is no graph shown for conditioning sequence 5, as a value was only measured for PVAC adhesive 3384, where impregnation was not used.

CONCLUSIONS

1. The highest values of glue bond shear strength of the PVAC 3384 adhesives were measured in case of the reference test specimens for all types of conditioning sequences. Adversely, the worst result was achieved when using the Cetol WV 885 BPD+ impregnation, where values were only measured for type 1.
2. For EPI system 1920/1993 adhesives, the highest values were measured for Cetol WV 885 BPD+ for conditioning sequence 1, and for Teknol aqua 1410-01 for conditioning sequence 3. A strength value was not measured for any test specimens for conditioning sequence 5. The reason for the large differences when Cetol WV 885 BPD+ was used need to be further investigated.
3. With the exception of one case, where a decrease in strength was not significant, all of the impregnation agents in all of the tests of PUR 1968 increased shear strength. The standard value of shear strength was meet for conditioning sequence 1 in all cases and, for conditioning sequence 4, the strength values met the standard when Cetol WV 885 BPD+ and Teknol aqua 1410-01 were used.
4. It was evident from the results that all of the impregnations used in this work had a negative impact on the strength of a bonded joint when the adhesive PVAC 3384 was used. Gori 605-00 and Teknol aqua 1410-01 did not have a significantly negative impact on EPI system 1920/1993 for conditioning sequence 1. For conditioning sequence 3, all of the used impregnations had a positive effect.

5. For all cases there was a clear synergistic effect of IPBC on strength when adhesives containing isocyanates are used.

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