ADHESION TEST – TESTING OF SELECTED ADHESIVES ON FIRED CLAY

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ABSTRACT. The following paper deals with the comparison of adhesion of specimens made of fired clay and selected adhesives. The adhesion was tested on specimens at the age of one, two and three days in order to determine the parameters of young masonry. Nine different adhesives (adhesives intended for masonry and adhesives for others material) were tested in total.

The above mentioned test is not standardized. The obtained results from the adhesion test determine the tensile strength between the adhesive and the fired clay and the adhesion of selected adhesive to the fired clay. The small specimens were made of two ceramic plates and an adhesive. Selected adhesives were applied to ceramic plates according to the usual standards. Eighty-one small specimens were produced in total – twenty-seven specimens for the adhesion test at each defined age of the specimens (one day, two days, three days). The produced specimens were cured in the climatic chamber with set laboratory conditions until the time of the test. The specimens were tested in the laboratory of the Heluz brick plant in Dolní Bukovsko. This paper deals with the procedure of preparation of the specimens, the course of the test and the comparison of gained results.

KEYWORDS: Fired clay, adhesion test, adhesive.

1. INTRODUCTION

The adhesion test determines the tensile strength between the adhesive and the building material (in our case fired clay) and the adhesion of selected adhesive to the building material (fired clay). Although requirement for tensile strength between a fired clay and adhesive is not defined by any regulation, the obtained results of the measurement provide information, which is important for the design of innovative construction methods. The test was performed at the age of the samples of one, two and three days. The measured values of "young" samples give an idea of the behaviour of selected adhesives available on the Czech market and indicate, which adhesives reach the required strength in a short time. A similar adhesion test (the centric adhesion test on two-unit test samples with 2C-PUR adhesive and thin-layer mortar) was performed in 2013 by the Institute of Building Materials Research (ibac) in Aachen. The performed tests were a necessary basis for the introduction of a new construction method in Germany – see [1].

Today's trend is the construction of structures in the shortest possible time, using suitable and efficient construction methods in compliance with high quality implementation, so the adhesion test is a felicitous tool for determining the required properties of tensile stressed structures.

2. MATERIALS AND SAMPLES

Eighty-one small samples were produced for the adhesion test – twenty-seven samples for adhesion test

at each defined age of the samples. The samples were made of fired clay and selected adhesive. Nine adhesives were tested in total. The selected adhesives are listed in the following table.

Sample	Description
А	HELUZ SIDI
В	HELUZ SB mortar for a thin joint
С	HELUZ TYTAN PUR foam (thin-film adhesive)
Ε	PU-contact two-component adhesive
\mathbf{F}	Den Braven – MULTI KLEBER – masonry foam
G	Den Braven – low expansion – masonry foam
Ι	Den Braven – MAMUT GLUE (High Tack)
J	Illbruck – universal chemical anchor OT120
Κ	Den Braven – wood glue – WOODFIX D3

TABLE 1. List of used adhesives [2].

The fired clay in the form of plates used for the adhesion test – size 30×30 mm and thickness 15 mm – has the same properties as HELUZ bricks. Small samples for the adhesion test were made of two plates of fired clay and selected adhesive – Table 1.

HELUZ SIDI is a silicate-dispersion masonry mortar for a thin joint, which is specified for masonry made of clay blocks HELUZ grinded. HELUZ SIDI was launched in the spring of 2020, it is a prepared mixture and is applied evenly crosswise with a structured roller [3].

HELUZ SB mortar for a thin joint is applied to the ribs of the grinded clay blocks and is applied with HELUZ applicator roller SB or by direct dipping of the bed joint in a pre-prepared mixture. The mixture is prepared from HELUZ SB and water – in a ratio of 0.45 liters of water per 1 kg HELUZ SB. HELUZ SB was applied to small samples on the whole surface of the bed joint using a spatula [4].

HELUZ TYTAN PUR foam was applied with the application gun on the whole surface of the bed joint [5]. PU-contact two-component adhesive is composed of two components – adhesive and accelerator – and it was applied on the whole surface of the bed joint of both parts of the sample. The adhesive was applied by a pneumatic spray gun. The application of PU-contact two-component adhesive has to be fast due to the presence of the accelerator. The increase of adhesion and application of the adhesive are very fast.

Den Braven masonry foams were selected to compare the results with HELUZ TYTAN PUR foam and were also applied by application gun [6, 7]. All samples with masonry foam were not loaded fast enough – the foam has swelled up, and the thickness of bed joint was greater than stated in the technical datasheets of adhesives. Swelling of the foam negatively affects its declared values, the probability of failure at the site of adhesive increases and therefore the values obtained from the adhesion test are on the side of safety.

MAMUT GLUE is a one-component adhesive MS polymer-based. This adhesive has a declared high initial adhesion (up to 500 kg/m²) and has been applied by application gun [8].

Illbruck – universal chemical anchor OT120 is a two-component resin without styrene. This chemical anchor is intended for anchoring rods, bolts and bushings to concrete and common building materials. This chemical anchor is also suitable for anchoring in hollow masonry. The chemical anchor is applied by an application gun for silicone sealants [9].

The wood glue – WOODFIX D3 – is an aqueous dispersion glue and is intended for gluing wood [10].



FIGURE 1. The fired clay in the form of plates and the produced samples.

The produced samples $(9 \times 3 \times 3)$ for the adhesion test were cured in the climatic chamber until the time of the test. The climatic chamber was set to



FIGURE 2. The selected adhesives: B – HELUZ SB mortar for a thin joint, A – HELUZ SIDI, C – HELUZ TYTAN PUR foam (thin-film adhesive), E – PU-contact two-component adhesive.



FIGURE 3. The selected adhesives: F – MULTI KLE-BER – masonry foam, G – Low expansion – masonry foam, I – MAMUT GLUE (High Tack), J – Illbruck – universal chemical anchor OT120, K – Wood glue – WOODFIX D3.

laboratory conditions – constant temperature 23 °C, constant relative humidity 50 %. The test samples were loaded by steel plate (m = 280 g) in the chamber. The steel plate is a component for the adhesion test. Each sample from a series of test samples was equipped with steel plates with hitches for attachment to the test apparatus before the adhesion test. Steel plates measuring 50×50 mm were glued by epoxy glue (Sikadur®-31 CF Rapid) to the lower and upper surface of the test sample.

A small number of samples with the selected adhesive at each defined age (three test samples) were selected due to the total number of test samples (eightyone test samples). The adhesion test was performed mainly for the selection of unsuitable adhesives. Adhesives with satisfactory results from this test will be subjected to further testing in the future.



FIGURE 4. Application of epoxy to samples for fixing steel plates.

3. Adhesion measurement

The adhesion test was performed at the age of the samples one day, two and three days – each series of 27 samples at a defined age. The series of samples at a defined age was transferred from the climatic chamber to the testing laboratory in Dolní Bukovsko on the day of the test. At first the cross-sectional area A_i of each test sample was measured using caliper. Then the samples were placed in a test apparatus and after that loaded with a tensile force. The used test apparatus was apparatus named TIRAtest 2803, which can exert a force of up to 3 kN. this force is sufficient for the purpose of the adhesion test.



FIGURE 5. The sample preparation in a test apparatus.

At first the sample in a test apparatus was fixed manually. After that the sample was loaded with tensile force – the value of increase of tensile force was set to 10 N/s – until the sample failed. The measured tensile force $F_{ti,\max}$ and the type of failure of the test sample were recorded in the prepared measurement table.

The possible types of failure of the sample are failure in the adhesive, in the fired clay, in a combination of fired clay and adhesive or in the epoxy between the fired clay and the steel plate. The named failure types and scheme of adhesion test are depicted in the following figures.

From the tensile force $F_{ti,\max}$ and from the crosssectional area A_i the tensile stress σ_{ti} , the average stress σ_{tm} and finally the characteristic stress σ_{tk} was were calculated. The characteristic stress σ_{tk} was



FIGURE 6. The scheme of adhesion test.



FIGURE 7. The possible types of failure of the sample in the adhesion test.

calculated using the standard deviation s, the coefficient of variation V and the uncertainty coefficient k_n (2.01 (n=2), resp. 1.89 (n=3)). The individual parameters were calculated according to the following formulas.

$$A_i = A \cdot B \ [\text{mm}^2] \tag{1}$$

$$\sigma_{ti} = \frac{F_{ti,\max}}{A} \quad [MPa] \tag{2}$$

$$\sigma_{tm} = \frac{1}{n} \cdot \sum \sigma_{ti} \, \left[\text{MPa} \right] \tag{3}$$

$$\sigma_{tk} = \sigma_{tm} \cdot (1 - k_n \cdot V) \tag{4}$$

$$s = \sqrt{\frac{1}{n-1} \cdot \sum (\sigma_{ti} - \sigma_{tm})^2} \tag{5}$$

$$V = \frac{s}{\sigma_{tm}} \tag{6}$$

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4. Results and discussion

The values and the information obtained from the adhesion test in the following tables state which adhesives performed the best in the adhesion test. The assessment of the adhesion test depends mainly on the measured values of the tensile force at the failure of the sample, but the variance of the results and the character of the failure of the sample are also important. The type of sample failure gives us an idea of what limits the measured value of tensile stress and whether the used adhesive can resist greater tensile stress then was measured. The failure of the sample in the fired clay says that the used adhesive is likely to resist greater tensile stress than the fired clay used to production of the samples; however, this type of failure did not prevail in any set of samples.

The samples that demonstrated satisfactory results in the adhesion test are the samples made of adhesives Den Braven – MAMUT GLUE (High Tack), HELUZ SIDI, PU-contact two-component adhesive and Den Braven - wood glue - WOODFIX D3. MAMUT GLUE (High Tack) performed the best in the adhesion test and the samples with this adhesive failed in the adhesive or in a combination of fired clay and the adhesive - it can be assumed that the adhesive is limited by tensile stress values close to the values measured by us. The negative factors of MAMUT GLUE (High Tack) are the unpredictability of sample failure and its price. A typical failure of PU-contact two-component adhesive is failure in the adhesive at different deformation. This adhesive is very malleable, with reduced adhesion when applied unprofessionally and is applied in a thin layer.

Very surprising results were measured on samples with applied wood glue – WOODFIX D3. The measured values of tensille stress were higher for wood glue than for some samples with aplicated adhesives intended for masonry.

The low expansion foam (Den Braven) was the worst of the tested masonry foams. The worst of all the samples were those with the adhesive Illbruck – universal chemical anchor OT120 and HELUZ SB mortar for a thin joint. The principle of the chemical anchor consists in its expansion into the borehole; this was not possible in the case of application to the contact area of small samples. The samples with the chemical anchor were broken in the adhesive in all cases, which did not adhere sufficiently and reached low resistance to tensile stress.

The performed adhesion test is not completely valid, because the number of test samples for series at a defined age was small (three samples for every adhesive at a defined age). Relatively high standard deviations in individual series of measurements (small number of test samples) caused a significant decrease of the characteristic stress of some samples aged two days and three days, respectively. The large variance of the measured values is also the reason for the negative value of the characteristic stress of samples with adhesive named HELUZ SB mortar for a thin joint at the age of one day. It would be necessary to produce and test more small samples to obtain valid values of the characteristic stress.

5. CONCLUSION

This article introduces the results of the adhesion test performed on the small samples, which were made of fired clay in the form of plates and selected adhesive. The obtained results are not completely valid (a small number of test samples), but the results give an idea of the tensile properties of selected adhesives applied to the fired clay. Therefore they are useful for the selection of adhesives for a future, more detailed study.

The adhesion test was performed at an early age of the samples to determine the adhesion and tensile strength between the fired clay and the selected adhesive. These ascertained properties are important for the progress of innovative construction methods – not only for the construction of brick buildings.

Surprising results were measured on samples with applied adhesives that are not intended for masonry. We must be careful about these results because we do not know the development of their properties in contact with masonry over a longer period of time. The research will continue by deeper examination of several selected adhesives.

LIST OF SYMBOLS

- A, B Horizontal dimension of the sample
- A_i Cross-sectional area
- $F_{ti,\max}$ Tensile force
- *n* Number of measurements
- σ_{ti} Tensile stress
- σ_{tm} Average stress
- σ_{tk} Characteristic stress
- s Standard deviation
- V Coefficient of variation
- k_n Uncertainty coefficient

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A –	HELU	Z SIDI							
Day	Name	Cross- sectional area	Tensile force	Tensile stress	Type of failure	Nr. of measure- ments	Avg. stress	Std. deviation	Char. stress
		$\frac{A_i}{[\mathrm{mm}^2]}$	$\begin{array}{c} F_{ti,\max} \\ [\mathrm{N}] \end{array}$	$\frac{\sigma_{ti}}{[\text{MPa}]}$		\overline{n}	σ_{tm} [MPa]	$s \; [MPa]$	σ_{tk} [MPa]
	A1	961.61	543.22	0.565	adhesive				
1	A2	959.31	575.86	0.600	adhesive	2	0.583	0.025	0.532
	A3	951.04	-	-	adhesive (when clamping)				
2	A1	956.97	958.41	1.002	$^{3/4}$ adhesive + $^{1/4}$ fired clay	3	0.954 0.171	0.171	0.630
	A2	945.87	1036.55	1.002	fired clay				
	A3	944.93	721.25	0.763	fired clay				
	A1	945.55	779.50	0.824	fired clay				
3	A2	965.87	773.15	0.801	fired clay	3	0.687	0.219	0.273
	A3	960.64	417.10	0.434	$^{4/5}$ adhesive + $^{1/5}$ fired clay				

B – HELUZ SB mortar for a thin joint

Day	Name	Cross- sectional area	Tensile force	Tensile stress	Type of failure	Nr. of measure- ments	Avg. stress	Std. deviation	Char. stress
		$\begin{array}{c} A_i \\ [\mathrm{mm}^2] \end{array}$	$F_{ti,\max}$ [N]	σ_{ti} [MPa]		n	σ_{tm} [MPa]	s [MPa]	σ_{tk} [MPa]
1	B1 B2 B3	953.53 952.96 945.78	$270.47 \\ 144.19 \\ 555.59$	$0.284 \\ 0.151 \\ 0.587$	adhesive adhesive adhesive	2	0.341	0.224	-0.082
2	B1 B2 B3	951.72 942.94 946.13	$219.49 \\ 302.15 \\ 462.38$	$\begin{array}{c} 0.231 \\ 0.320 \\ 0.489 \end{array}$	adhesive adhesive adhesive	3	0.347	0.131	0.099
3	B1 B2 B3	946.79 966.27 956.36	300.20 229.42	0.311 0.240	when clamping adhesive adhesive	2	0.275	0.050	0.175

C – HELUZ TYTAN PUR foam (thin-film adhesive)

Day	Name	Cross- sectional area	Tensile force	Tensile stress	Type of failure	Nr. of measure- ments	Avg. stress	Std. deviation	Char. stress
		$\begin{array}{c} A_i \\ [\mathrm{mm}^2] \end{array}$	$\begin{array}{c} F_{ti,\max} \\ [\mathrm{N}] \end{array}$	σ_{ti} [MPa]		n	σ_{tm} [MPa]	s [MPa]	σ_{tk} [MPa]
1	C1 C2 C3	$956.04 \\ 956.31 \\ 935.68$	$325.55 \\ 477.45 \\ 162.82$	$0.341 \\ 0.499 \\ 0.174$	adhesive adhesive adhesive	3	0.338	0.163	0.031
2	C1 C2 C3	958.21 954.40 952.93	$177.41 \\183.67 \\188.39$	$\begin{array}{c} 0.185 \\ 0.192 \\ 0.198 \end{array}$	adhesive adhesive adhesive	3	0.192	0.006	0.180
3	$\begin{array}{c} C1\\ C2\\ C3 \end{array}$	955.73 956.85 942.39	308.77 296.97 243.62	$\begin{array}{c} 0.323 \\ 0.310 \\ 0.259 \end{array}$	adhesive adhesive adhesive	3	0.297	0.034	0.233

E –	PU-co	ntact two	o-compo	nent adh	esive				
Day	Name	Cross- sectional area	Tensile force	Tensile stress	Type of failure	Nr. of measure- ments	Avg. stress	Std. deviation	Char. stress
		$\begin{array}{c} A_i \\ [\mathrm{mm}^2] \end{array}$	$F_{ti,\max}$ [N]	σ_{ti} [MPa]		n	σ_{tm} [MPa]	s [MPa]	σ_{tk} [MPa]
1	E1 E2	944.30 964 10	-	-	adhesive (when clamping) adhesive	2	0.724	0.078	0.568
	E3	964.41	750.96	0.009 0.779	adhesive				
2	E1	939.39	526.22	0.560	adhesive				
	E2	947.68	382.27	0.403	$^{3}/_{4}$ adhesive + $^{1}/_{4}$ fired clay	3	0.443	0.103	0.248
	E3	926.90	338.60	0.365	adhesive				
3	E1	980.25	27.68	0.028	adhesive (did not adhere)	9	0 534	0.029	0.457
5	E2	946.78	530.87	0.561	adhesive	2	0.004	0.038	0.457
	E3	946.88	479.58	0.506	adnesive				
F –	Den B	raven – I	MULTIF	LEBER	– mansory foam				
Day	Name	Cross- sectional area	Tensile force	Tensile stress	Type of failure	Nr. of measure- ments	Avg. stress	Std. deviation	Char. stress

Day	Name	area	force	stress	Type of failure	ments	501055	deviation	501655
		$\frac{A_i}{[\mathrm{mm}^2]}$	$\begin{array}{c} F_{ti,\max} \\ [\mathrm{N}] \end{array}$	σ_{ti} [MPa]		n	σ_{tm} [MPa]	s [MPa]	σ_{tk} [MPa]
1	F1 F2 F3	991.60 968.45 962.23	$211.40 \\ 157.38 \\ 248.28$	$0.213 \\ 0.163 \\ 0.258$	adhesive adhesive adhesive	3	0.211	0.048	0.121
2	F1 F2 F3	723.23 730.33 948.33	$368.26 \\ 176.21 \\ 406.53$	$0.509 \\ 0.241 \\ 0.429$	adhesive adhesive adhesive	3	0.393	0.137	0.133
3	F1 F2 F3	$859.60 \\ 744.07 \\ 948.64$	$253.21 \\ 457.25 \\ 350.27$	$\begin{array}{c} 0.295 \\ 0.615 \\ 0.369 \end{array}$	adhesive adhesive adhesive	3	0.426	0.167	0.110

G – Den Braven – low expansion – masonry foam

Day	Name	Cross- sectional area	Tensile force	Tensile stress	Type of failure	Nr. of measure- ments	Avg. stress	Std. deviation	Char. stress
		$\begin{array}{c} A_i \\ [\mathrm{mm}^2] \end{array}$		$\frac{\sigma_{ti}}{[\text{MPa}]}$		\overline{n}	σ_{tm} [MPa]	$s \; [MPa]$	σ_{tk} [MPa]
	G1	969.70	182.85	0.189	adhesive				
1	G2	956.29	109.63	0.115	adhesive	3	0.165	0.044	0.082
	G3	855.50	164.46	0.192	adhesive				
	G1	885.25	94.46	0.107	adhesive				
2	G2	989.78	267.68	0.270	adhesive	3	0.198	0.084	0.040
	G3	755.55	164.08	0.217	adhesive				
	G1	969.96	328.94	0.339	adhesive				
3	G2	915.55	255.04	0.279	$^{5}/_{6}$ adhesive + $^{1}/_{6}$ fired clay	3	0.417	0.190	0.058
	G3	822.25	521.58	0.634	$^{5/6}$ adhesive + $^{1/6}$ fired clay				

I - I	I – Den Braven – MAMUT GLUE (Hight Tack)											
Day	Name	Cross- sectional area	Tensile force	Tensile stress	Type of failure	Nr. of measure- ments	Avg. stress	Std. deviation	Char. stress			
		$\begin{array}{c} A_i \\ [\mathrm{mm}^2] \end{array}$	$\begin{array}{c} F_{ti,\max} \\ [\mathrm{N}] \end{array}$	σ_{ti} [MPa]		n	σ_{tm} [MPa]	s [MPa]	σ_{tk} [MPa]			
1	I1	937.27	1098.71	1.172	adhesive	0	1.049	0 119	0.910			
1	I2	979.38	922.21	0.942	adhesive	2	1.042	0.116	0.819			
	I3	980.92	992.69	1.012	adhesive							
	I1	938.44	1076.41	1.147	$^{3/4}$ adhesive + $^{1/4}$ fired clay	3	1 004	0.176	0.672			
-	I2	980.00	791.50	0.808	$^{2/3}$ adhesive + $^{1/3}$ fired clay	0	1.001	0.110	0.012			
	I3	965.33	1022.11	1.059	adhesive							
	I1	957.52	560.67	0.586	fired clay							
3	I2	974.93	841.90	0.864	adhesive	3	0.769	0.159	0.468			
	I3	958.48	823.55	0.859	$^{1/3}$ adhesive + $^{2/3}$ fired clay							

J –	J – Illbruck – universal chemical anchor OT120												
Day	Name	Cross- sectional area	Tensile force	Tensile stress	Type of failure	Nr. of measure- ments	Avg. stress	Std. deviation	Char. stress				
		$\begin{array}{c} A_i \\ [\mathrm{mm}^2] \end{array}$	$\begin{array}{c} F_{ti,\max} \\ [\mathrm{N}] \end{array}$	σ_{ti} [MPa]		n	σ_{tm} [MPa]	s [MPa]	σ_{tk} [MPa]				
1	J1	959.45	158.83	0.166	adhesive								
	J2	974.06	-	-	adhesive (when clamping)	2	0.244	0.110	0.022				
	J3	1005.73	323.34	0.321	adhesive								
2	J1 J2 J3	$1003.29 \\ 667.99 \\ 970.94$	$214.63 \\ 176.98 \\ 256.95$	$0.214 \\ 0.265 \\ 0.265$	adhesive adhesive adhesive	3	0.248	0.029	0.192				
3	J1 J2	942.78 962.20	$265.29 \\ 338.38$	$0.281 \\ 0.352$	adhesive adhesive	2	0.317	0.050	0.217				
	J3	962.54	-	-	adhesive (when clamping)								

K –	K – Den Braven – wood glue – WOODFIX D3											
Day	Name	Cross- sectional area	Tensile force	Tensile stress	Type of failure	Nr. of measure- ments	Avg. stress	Std. deviation	Char. stress			
		$\begin{array}{c} A_i \\ [\mathrm{mm}^2] \end{array}$	$\begin{array}{c} F_{ti,\max} \\ [\mathrm{N}] \end{array}$	σ_{ti} [MPa]		n	σ_{tm} [MPa]	s [MPa]	σ_{tk} [MPa]			
	K1	947.99	344.52	0.363	adhesive							
1	K2	955.68	2.84	0.003	fired clay (when clamping)	2	0.449	0.121	0.206			
	K3	981.25	524.34	0.534	adhesive							
2	K1 K2 K3	974.64 961.84 956.02	980.99 647.83 912.48	$1.007 \\ 0.674 \\ 0.954$	adhesive fired clay adhesive	3	0.878	0.179	0.540			
3	K1 K2 K3	981.08 992.86 946.76	440.06 656.44 822.06	$0.449 \\ 0.661 \\ 0.868$	adhesive fired clay ³ /4 adhesive + ¹ /4 fired clay	3	0.659	0.210	0.263			

TABLE 2. The values and the information obtained from the adhesion test [1].



FIGURE 8. Summary of results from the adhesion test at the age of the samples one, two and three days [1].

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