# THE EFFECT OF STEEL SURFACE PRETREATMENT ON THE ADHESION BOND BETWEEN FLY ASH BASED GEOPOLYMER GLUE AND STEEL SUBSTRATES

## Olena HALAS, Pavel ŠVANDA

Department of Mechanics, Materials and Machine Parts, Jan Perner Transport Faculty, University of Pardubice, e-mail: alena.halas@gmail.com

## **Abstract**

This study deals with the investigation of fly ash based geopolymer glues. The main purpose was to study the effect of steel surface pretreatment on the adhesion bond between geopolymer glue and steel substrates. The surface of metal substrates was mechanically (sand blasting) and chemically (Interlox phosphate 2325 MCIZ or UniPrep PP) pretreated. It was also prepared two types of glues with different amount of sodium hydroxide. It was conducted single lap shear strength test in order to determine adhesive bond strength between geopolymer and steel. The test results showed that type of glue didn't significantly influence on the strength, meanwhile the steel surface pretreatment had a considerable effect. The highest strength 2744KPa was reached using glue with smaller amount of sodium hydroxide and steel surface pretreatment by sand blasting and Interlox phosphate 2325 MCIZ.

## 1. Introduction

The wide study of alkali activated materials was started in the first part of the last century. A lot of experiments were made from that time in order to find new alternative materials to concrete and other silicates with good properties and lower price [1-5].

Geopolymers are a group of cement-like materials that are formed by reacting silica-rich and alumina-rich solids with a solution of alkali or alkali-salts resulting in a mixture of gels and crystalline compounds that eventually harden into a new strong compound. [6] For production of geopolymers can be used large number of primary and secondary resources. As primary resources can be used clays, volcanic deposits, as secondary – coal combustion by-products (fly ash, boiler slag, bottom ash), blast furnace slag, red mud, tungsten mine waste mud, ceramic waste materials, glass industry waste, brick scrap and many others.

Numerous investigations showed that geopolymers based on different aluminosilicates have high compressive strength and good bending tensile strength comparable with Portland cement, resistant against high temperatures, frost, and aggressive environment. These materials have also good adhesive with steel, concrete, glass. [7-13]

Adhesive properties of geopolymers haven't been studied so widely as other properties and most of existent works are mainly about metakaolin based geopolimeric glues. This article deals with a study of fly ash based geopolimeric glue. Till nowadays fly ash based glues haven't been practically studied. [10]

## 2. Experimental part

## 2.1 Materials

## 2.1.1 Geopolymer glue

Geopolymer glue was prepared using class F fly ash from Opatovice power station (Czech Republic). Its chemical composition and particle size distribution are shown in the Table 1 and Figure 1.

**Tab. 1:** Chemical composition of fly-ash, wt.%

FI	S		N	K		. N	F	Т
y-ash,	iO <sub>2</sub>	$I_2O_3$	a <sub>2</sub> O	<sub>2</sub> O	аО	gO	$e_2O_3$	iO <sub>2</sub>
0	6	3	0	2	C	1	2	0
patovice	1.4	1.1		.2	.2	.6	.6	.9

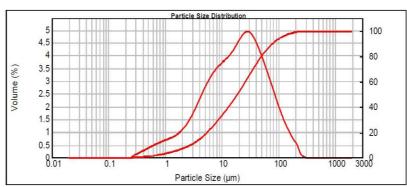


Fig. 1: Particle size distribution of fly-ash

As an alkali activator was used water glass (sodium silicate) with a silicate modulus 3.35 (content NaOH - 8%, SiO2 - 26.2%, H2O - 65.8%) and density 1340 kg/m3.

#### 2.1.2 Surface treatment

Steel substrates (carbon steel ČSN 11 373 (S235)) were previously degreased by ethanol then mechanically and chemically treated before applying the adhesive. All samples were mechanically treated by sand blasting. For chemical treatment were used two commercial products: Interlox phosphate 2325 MCIZ and UniPrep PP. INTERLOX 2325 is highly concentrated liquid phosphating compound for use by immersion phosphatizing methods. Interlox® Phosphate 2325 MCIZ produces a microcrystalline zinc-calcium phosphate coating. [14]. UniPrep PP is adhesion promoter contain no metals or acids, eliminate scale/sludge and drastically minimize waste treatment requirements [15].

The surface of first series of specimens (eight samples) was treated only by sand blasting. Further eight samples were firstly treated by sand blasting and then by Interlox phosphate. In that case the samples after sand blasting were immersed into the hot (75°C) solution (100ml of water and 4ml of Interlox® Phosphate 2325 MCIZ) for 4 minutes. Then the steel substrates were rinsed by water and dried. The surface of the third series of samples was also previously sand blasted but then immersed to the warmed (30°C) solution (100ml of water and 4ml of adhesion promoter UniPrep PP) for 1 minute. After that the steel substrates were ready for gluing.

## 2.2 Samples preparation

There were prepared two types of geopolymer adhesives with different amount of sodium hydroxide (Tab.2).

Tab. 2:	Composition	of the samples
---------	-------------	----------------

Component Sample's name	FI y ash (wt.%)	Wate r glass (wt.%)	So dium hydroxide (wt.%)	s i/Al	N a/Si	N a/Al
G5	6	30.0	1.5	2	0	0
	8.5				.16	.31
G8	6	29.2	2.3	1	0	0
	8.5			.995	.18	.36

Sodium hydroxide was firstly dissolved in water glass. Then the alkali solution was added to fly ash. Geopolymer mixture was mixed 15 minutes and then adhesive was applied to steel substrates (Fig.2).

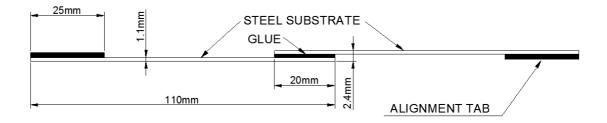


Fig. 2: Samples geometry

The glues were curing at room temperature and constant load 0.1 MPa. Single lap shear strength was measured on universal testing machine ZD 10/90 with data collection on PC. The test was conducted after 21 days of curing.

#### 2.3 Results and discussion

Table 3 and figure 3 show the single lap shear test results. It can be observed that there wasn't practically difference between glues G5 and G8, the shear strength between adhesive G5 and steel was a little bit higher than between adhesive G8 and steel. Meanwhile chemical pretreatment of steel substrates surface improved the adhesion between steel and geopolymer adhesive. The highest impact had the pretreatment by INTERLOX Phosphate 2325. Maximum reached single lap shear strength was 2744 KPa. In case of adhesive promoter UniPrep PP application the maximum strength was 2460 KPa. The lowest shear strength was reached for samples with sand blasting pretreatment of the steel surface only. The strength was nearly the same for both geopolymer glues – 1823KPa in case of G5 glue application and 1794KPa in case of G8 glue application.

**Tab. 3:** Single lap shear test results

glue/steel surface pretreatment	T <sub>sls</sub> [kPa]	Standard deviation σ [KPa]	Failure type
G5/sand blasting	1823	299	adhesive
G5/INTERLO X 2325	2744	195	adhesive
G5/UniPrep PP	2460	283	adhesive
G8/sand blasting	1794	334	adhesive
G8/INTERLO X 2325	2433	723	adhesive
G8/UniPrep PP	2279	228	adhesive

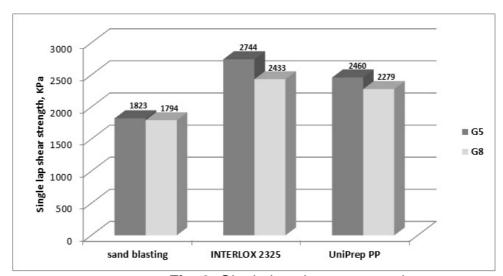


Fig. 3: Single lap shear test results

Earlier geopolymer glues based on fly ash weren't practically studied. Temuujin et al. studied glues based on fly ash. It was evaluated two parameters which could influence on the adhesion between geopolymer glue and steel substrates: molar Si:Al ratio and water content. The authors came to the conclusion that adhesive strength of the coating to steel strongly depended on the chemical composition of the coating. The water content influenced on the ease with which geopolymer can be applied on metal substrate. And the maximum bond strength was reached between high silica containing composition and mild steel (more than 3.5MPa). In tote the adhesive strength between geopolymer glue and mild steel varied from 0.25 MPa (Si:Al ratio 1) to more than 3.5MPa (Si:Al ratio 3.5) and between stainless steel and geopolymer adhesive – from 0.4MPa (Si:Al ratio 1) to 1.4 (Si:Al ratio 3.5). [10]

Glues based on metakaolin were investigated more widely. S. De Barros et al. studied adhesive properties of geopolymers based on metakaolin to steel and aluminum substrates previously mechanically (grit-blasting or sand blasting) and chemically (nitro-phosphoric acid or silanization) pretreated. The test results showed that there was better adhesion between geopolymer glue and steel substrate than between geopolymer glue and aluminum substrate. In both cases the mechanical pretreatment of the substrate surfaces was more efficient than chemical. The highest

strength had the steel joints with grit blasting pretreatment of the surface – around 5 MPa and with sand blasting pretreatment – 4 MPa. The chemical pretreatment of the steel substrate didn't give positive results. The strength of the samples with chemical pretreatment was significantly lower than strength of the samples with mechanical pretreatment only. The strength of aluminum joints was more than two times lower than strength of steel joints – around 2 MPa. The chemical pretreatment didn't affect positively too, though there wasn't so significant difference in strength as in steel joints. [11] Besides geopolymer glues based on fly ash Temuujin et al. also studied glues based on metakaolin. They investigated the bond strength between geopolymer and steel (mild and stainless). They observed that after curing adhesion of glues with Si:Al ratio 1 and 2 to steel was very week. Meanwhile, samples with Al:Si ratio 2.5 showed strong adhesion behaviour with values recorded as >3.5 MPa for both stainless and mild steel substrates.[12,13]

## **Conclusions**

It hasn't been paid much attention on the investigation of adhesive properties of fly ash based geopolymers yet. The main purpose of this article was to investigate the influence of steel surface pretreatment on the adhesive bond between steel and fly ash based geopolymer glue. The single lap shear test showed that the most positive effect had chemical pretreatment of the steel surface, especially pretreatment by Interlox phosphate 2325 MCIZ. Maximum reached shear strength was 2744 KPa. The type of the glue G5 or G8 didn't have significant impact on the strength. Tests results described in this article are comparable with results described earlier [10].

#### **REFERENCES**

- [1] Kuhl. Zement-Chemie. Thechnik: band III ved verlag . 1958.
- [2] Purdon, A.O. The action of alkalis on blastfurnace slag. Journal of the Society of Chemical Industry. September 1940, Volume 50.
- [3] Глухоский, В. Грунтосиликаты. Киев : Государственное издательство литературы по строительству и архитектуре УССР, 1959. 125 с.
- [4] Глуховский, В.; Пахомов, В. Шлакощелочный цементы и бетоны. Киев : Будівельник, 1978. 184 с.
- [5] Beneš, L., Halas, O., Minař, L. Alkali-activated materials: history review, fly ash as raw material for alkali activated materials. In BORKOWSKI, S., SHEVTSOVA, O. (ed.). Process innovation. 1st ed. Dnipropetrovsk, 2010, p. 10–19. ISBN 978-966-1507-33-2.
- [6] Van JAARSVELD, J.G.S., J.S.J. Van Deventer and L. Lorenzen. Factors affecting the immobilisation of metals in geopolymerised fly ash. Research Report, Department of Chemical Engineering, University of Stellenbosch, South Africa, 1995.
- [7] ŠKVÁRA, F. Alkali activated material geopolymer. In 2007 International Conference Alkali Activated Materials Research, Production and Utilization. Prague: Česká rozvojová agentura, 2007. p. 661 676. ISBN 978-80-86742-18-2.
- [8] LATELLA, B.A., PERERA, D.S., ESCOTT, T.R., CASSIDY, D.J. Adhesion of glass to steel using geopolymer. J MATER SCI. 2006, No. 41, p. 1261 1264. DOI: 10.1007/s10853-005-4234-3.

- [9] ZHANG, Zuhua, Xiao YAO and Huajun ZHU. Potential application of geopolymers as protection coatings for marine concrete: I. Basic properties. Applied clay science. 2010, No. 49, p. 1-6. DOI: 10.1016/j.clay.2010.01.014.
- [10] TEMUUJIN, Jadambaa, Amgalan MINJIGMAA, William RICKARD, Melissa LEE, lestyn WILLIAMS and Arie van RIESSEN. Fly ash based geopolymer thin coatings on metal substrates and its thermal evaluation. Journal of Hazardous Materials. 2010, No. 180, p. 748 752. DOI: 10.1016/j.jhazmat.2010.04.121.
- [11] DE BARROS, S., J.R. DE SOUZA, K.C. GOMES, E.M. SAMPAIO, N.P. BARBOSA and S.M. TORRES. Adhesion of geopolymer bonded joints considering surface treatments. The journal of adhesion. 2012, p. 364-375. ISSN 0021-8464. DOI: 10.1080/00218464.2012.660075.
- [12] TEMUUJIN, Jadambaa, Amgalan MINJIGMAA, William RICKARD, Melissa LEE, Iestyn WILLIAMS and Arie van RIESSEN. Preparation of metakaolin based geopolymer coatings on metal substrates as thermal barriers. Applied Clay Science. 2009, No. 46, p. 265-270. DOI: 10.1016/j.clay.2009.08.015.
- [13] TEMUUJIN, Jadambaa, William RICKARD, Melissa LEE and Arie VAN RIESSEN. Preparation and thermal properties of fire resistant metakaolin-based geopolymer-type coatings. Journal of Non-Crystalline Solids. 2011, vol. 357, no. 5, p. 1399-1404. ISSN 00223093. DOI: 10.1016/j.jnoncrysol.2010.09.063.
- [14] Atotech CZ, a.s., Jablonec nad Nisou. INTERLOX® PHOSPHATE 2325 MCIZ (Data Sheet). 2007. 4 p.
- [15] Atotech CZ, a.s., Jablonec nad Nisou. UniPrep PP (Data Sheet). 2007. 3 p.