



# Available online at www.sciencedirect.com

# **ScienceDirect**

Procedia Engineering

Procedia Engineering 151 (2016) 108 - 113

www.elsevier.com/locate/procedia

International Conference on Ecology and new Building materials and products, ICEBMP 2016

# Blended alkali-activated fly ash / brick powder materials

Pavel Rovnaník\*, Bohuslav Řezník, Pavla Rovnaníková

Brno University of Technology, Faculty of Civil Engineering, Veveři 331/95, Brno 602 00, Czech Republic

### Abstract

Aluminosilicate materials can be transformed into very compact binding material by the process called alkaline activation. The paper presents results of alkaline activation of fly ash and waste fine-grained brick body. Prepared geopolymers were tested for the compressive and flexural strengths, bulk density, and microstructure was examined by means of SEM. The results of investigated parameters showed the differences in relation to the ratio of fly ash to brick binder.

© 2016 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

Peer-review under responsibility of the organizing committee of ICEBMP 2016

Keywords: Brick powder; fly ash; alkaline activation; compressive strength; flexural strength; microstructure

# 1. Introduction

Alkaline activation of aluminosilicate materials is known from the thirties of the last century. Alkali-activated materials (geopolymers) are good alternative to Portland and blended cements. Geopolymers are formed by the chemical reaction between aluminosilicate materials and alkali-silicate solution. The result is partially or fully amorphous inorganic polymers with Si–O–Al bonds arranged through SiO<sub>4</sub> and AlO<sub>4</sub> tetrahedra into three dimensional structure. The great attention has been paid especially to the alkaline activation of metakaolin, clays, fly ashes, slags. Nowadays, there have been published several papers that studied the possibility of using fine grain ceramics for alkaline activation. Reig et al. [1,2] studied effect of SiO<sub>2</sub>/Na<sub>2</sub>O ratio on a short-time compressive strengths and microstructure of mortars prepared from brick powder, alkali-activator of silicate modulus from 0.0 to 2.0, and sand. Mortars were stored at 65 °C and relative humidity 90–95% for 3 and 7 days. Although thermogravimetric analysis identified initial zeolitic structure, this trend disappeared with increasing concentration

<sup>\*</sup> Corresponding author. Tel.: +420-541-147-632; fax: +420-541-147-667. E-mail address: rovnanik.p@fce.vutbr.cz

of alkaline solution. The best results showed mortar with SiO<sub>2</sub>/Na<sub>2</sub>O ratio 1.60. Mortar reached compressive strength of 30 MPa after 7 days. The ceramic material derived from municipal waste collection as a potential alkali-activated material was studied by Sun Zengqing et al. [3]. The ceramics was crushed and pulverized; the mean particle size was 30 μm. The ceramic dust was activated either by sodium silicate solution of SiO<sub>2</sub>/Na<sub>2</sub>O molar ratio 3.2 or sodium silicate solution with various concentration of sodium (potassium) hydroxide. Samples were cured at 65 °C for 28 days. The geopolymer of optimum mix design gave the highest compressive strength of more than 70 MPa. Higher strengths were acquired after 2 hours of calcination at 1000 °C.

Komnitsas et al. [4] applied the demolition waste ceramic materials for the preparation of geopolymers. Waste material was collected, cleaned, dried, and pulverized to reach  $d_{50} = 35 \mu m$ . Sodium silicate and sodium hydroxide solutions of different concentration were used as activating agents. The samples were cured at temperatures 60, 80, and 100 °C for 7 days. Brick powder was successfully geopolymerized reaching a compressive strength up to 50 MPa, and after heating to high temperature (800 °C) compressive strengths moderately decreased.

Rakhimova and Rakhimov [5] used blended binder prepared from granulated blast furnace slag (GBFS) and brick waste powder (BP) at different ratio by alkali-activation using mixed solution of sodium silicate, sodium hydroxide and sodium carbonate. The compressive strength of mixture with GBFS/BP ratio 60/40 was higher than for one component binders. The compressive strength reached 120 MPa when GBFS and BP were milled together.

Nowadays, the heat-insulating bricks have to be fairly plain; hence, they are skived. The waste brick powder arises during this process; it is partially re-used as opening material in the brick production but great amounts are disposed. This material has very fine grains with no inappropriate substances; therefore, further modification is not needed. With respect to lower pozzolanic activity of brick powder, mixtures prepared from fly ash and brick powder were studied. Characterization of the composition, results of compressive and flexural strengths and microstructure reached at an ambient temperature are described in this paper. The study is in agreement with trends in concrete technology where blended binders are preferred.

#### 2. Materials and methods

The fly ash (FA), waste material from Power plant Chvaletice (CZ), and brick powder (BP), waste material from skiving of heat-insulating bricks (Heluz, Dolní Bukovsko, CZ), were the basic raw materials for alkali-activated binder. No modification process of fly ash and brick powder were required. Chemical composition is presented in Table 1.

Table 1. Chemical composition of fly ash and brick powder.

Material	SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	S tot.	LOI (1100 °C)
FA	52.21	29.59	8.44	1.82	1.16	1.66	0.3	0.86	2.12
BP	57.67	14.91	5.02	9.81	3.74	3.20	1.45	1.86	0.00

Table 2. Mineralogical composition of brick powder.

Component	Content (%)
Quartz	47.32
Microcline	15.65
Muscovite	9.80
Albite	11.30
Orthoclase	4.83
Hematite	3.26
Rutile	2.88
Anatase	2.75
Sanidine	2.21
Amorphous phase	18.49

Quantitative XRD analysis was carried out for brick powder, mineralogical composition of fly ash was assessed only qualitatively. Sample of fly ash contained quartz, anatase, mullite, hematite, magnetite, anorthite, albite and larnite. A significant diffuse peak in the XRD pattern gives evidence of amorphous phase. Mineralogical composition of both materials is different; while the fly ash contains mullite and larnite, brick powder has a significant amount of feldspars. Results of quantitative XRD analysis of brick powder are presented in Table 2.

Fig. 1 shows typical round grains of fly ash, whereas the brick powder consisted of sharp-edged grains. With agreement to grain size and the specific surface the pozzolanic activity of fly ash determined by the modified Chapelle test is higher than pozzolanic activity of brick powder (Table 3).

	FA	BP
$d_{10}(\mu m)$	5.1	5.0
$d_{50}(\mu m)$	20.1	25.6
$d_{90}(\mu m)$	114.9	130.3
Specific surface $(m^2/kg)$	483	462
Pozzolanic activity	535	345
(mg Ca(OH)2/1 g of pozzolan)		

Table 3. Particle size, specific surface and pozzolanic activity of raw materials.

The testing specimens were prepared by mixing of fly ash and brick powder, and further homogenized with alkaline activator prepared from sodium silicate solution (water glass,  $SiO_2/Na_2O = 1.6$ ), sodium hydroxide and water (Table 4). The  $SiO_2/Na_2O$  ratio of prepared alkaline activator was 1.0 and it was added in dosage of 25 wt. % with respect to a dry mixture. Behavior of fresh mixtures changed during process of mixing; for increasing amount of brick powder a higher content of water in mixture was necessary to reach an acceptable consistency. Reference samples contained only FA or BP, respectively.

Table 4. Mix design for fly ash	and brick powder.
---------------------------------	-------------------

-		•			
Mixture	FA (g)	BP (g)	Sodium silicate solution (g)	NaOH (g)	Water (g)
FA	700	0	156	19	78
FABP1	525	175			96
FABP2	350	350			111
BP	0	700			110

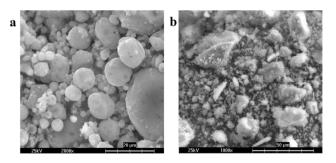


Fig. 1. Micrographs of raw materials: (a) fly ash; (b) brick powder.

Fresh mixtures were cast into prismatic moulds of size  $20 \times 20 \times 100$  mm. Standard conditions of sample storage were  $21 \pm 2$  °C and relative humidity of  $50 \pm 5\%$ . After 7, 28 and 90 days the compressive and flexural strengths were determined.

The microstructure of solid materials was investigated by means of scanning electron microscope Jeol JSM-840. The micrographs were taken in SE mode from dry samples that were sputtered with carbon. Accelerating voltage was set to 25.0 kV.

#### 3. Results and discussion

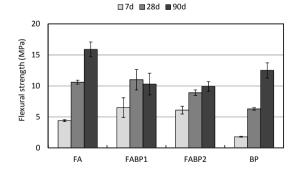
Bulk density and shrinkage of the geopolymer pastes at the age of 28 days are present in Table 5. Bulk density increased with a higher amount of brick powder. The reason of increasing bulk density is probably a different specific gravity of fly ash (2036 kg/m³) and brick powder (2680 kg/m³). An increased shrinkage of the mixtures with brick powder can be related to the amount of mixing water; brick powder consumed by 40 % more water compared to fly ash.

Table 5. Bulk delisity and similkage.					
Mixture	Bulk density	Shrinkage			
	28 d	28 d			
	$(kg/m^3)$	(%)			
FA	1657	1.9			
FABP1	1665	2.7			
FABP2	1673	3.7			
BP	1756	5.7			

Table 5. Bulk density and shrinkage.

Flexural and compressive strengths of geopolymers prepared with variable fly ash and brick powder ratio are presented in Fig. 2. Strengths of all samples were rising over time until the age of 90 days. Alkali-activated fly ash showed the highest long-term strengths, although the 7-days strengths were lower than for the blended mixtures. Brick powder geopolymer exhibited the lowest early strengths but after longer period the values reached those observed for blends. These results are slightly in contradiction with results presented by Rakhimova [5] that blended mixtures exhibit better mechanical properties than one-component materials.

The microstructure of alkali-activated materials was studied using scanning electron microscope. Microstructure of alkali-activated fly ash is presented in Fig. 3. Spherical grains of fly ash grow into alkali-activated matrix and are coated with newly formed binder. However, even after 90 days the original fly ash spheres are recognizable. While the fly ash particles are spherical, the brick powder contains sharp-edged grains (Fig. 4). Microstructure of alkali-activated brick powder is less compact and more porous. The sharp-edged grains are coated with alkali-activated matrix but the degree of reaction seems to be much lower.



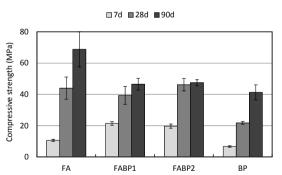


Fig. 2. Flexural and compressive strengths of prepared geopolymer pastes at the age of 7, 28 and 90 days (standard deviation presented as error bars).

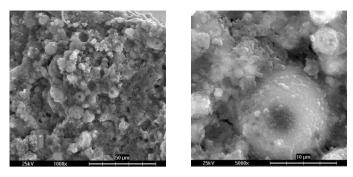


Fig. 3. Micrographs of activated fly ash paste at different magnifications.

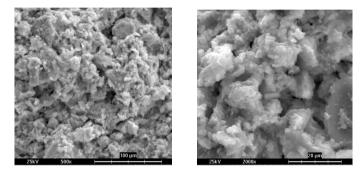


Fig. 4. Micrographs of activated brick powder paste at different magnifications.

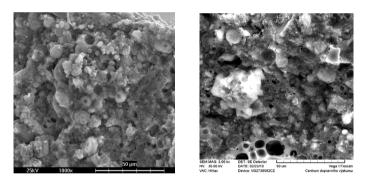


Fig. 5. Micrographs of blended geopolymer pastes: (left) FA: BP = 75: 25; (right) FA: BP = 50: 50.

The microstructure of blended pastes is very similar to the one with fly ash alone (Fig. 5). Although a number of unreacted fly ash particle can be still observed the matrix looks rather dense and compact. It can be concluded that microstructural observations are in a good accordance with the mechanical properties.

#### 4. Conclusion

This study has presented blended alkali-activated materials prepared from fly ash and brick powder. From the experimental results and the discussion above, the following conclusions are made:

 Alkali-activated fly ash reached higher flexural and compressive strengths as a result of larger specific surface, higher pozzolanic activity and more compact microstructure.

- Lower flexural and compressive strengths of alkali-activated brick powder material resulted from much lower pozzolanic activity and non-compact microstructure.
- Contrary to blended alkali-activated materials prepared from blast furnace slag and brick powder, materials based
  on the fly ash and brick powder showed lower flexural and compressive strengths than materials based on fly ash
  alone.
- The microstructure of alkali-activated fly ash and blended materials is very compact while alkali-activated brick powder exhibited less compact structure with more pores located between sharp-edged grains.

# Acknowledgements

This research has been supported by the Czech Science Foundation, under project No 16-02862S.

# References

- L. Reig, L. Soriano, M.V. Borrachero, J. Monzó, J Payá, Properties and microstructure of alkali-activated red clay brick waste, Constr. Build. Mater. 43 (2013) 98–106.
- [2] L. Reig, M. M. Tashima, L. Soriano et al., Alkaline activation of ceramic waste materials. Waste Biomass Valor. 4 (2013) 729-736.
- [3] Z. Sun, H. Cui, H. An, D. Tao, Y. Xu, J. Zhai, Q. Li, Synthesis and thermal behaviour of geopolymer-type material from waste ceramic, Constr. Build. Mater. 49 (2013) 281–287.
- [4] K. Komnitsas, D. Zaharaki, A. Vlachou, G. Bartzas, M. Galetakis, Effect of synthesis parameters on the quality of construction and demolition wastes (CDW) geopolymers, Adv. Powder Technol. 26 (2015) 368–376.
- [5] N.R. Rakhimova, R.Z. Rakhimov, Alkali-activated cements and mortars based on blast furnace slag and red clay brick waste, Mater. Design 85 (2015) 324–331.