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Effect of alkaline activator quantity and temperature of curing on the properties of alkali-activated brick dust

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Abstract. The properties of alkaline activated brick dust mixed with different amounts of water glass with a silicate modulus (M) of 1.0 were studied in connection with the temperature of curing. The influence of the amount of alkaline solution and the temperature of curing on the microstructure and mechanical properties of hardened materials was studied. The flexural and compressive strengths of the hardened products, as well as their microstructure, were determined. Part of the samples was stored under laboratory conditions (20 °C) for 7 and 28 days, other set of samples was stored at 60 °C for 7 and 28 days. One set of samples was treated for 7 days at 60 °C and 21 days at 20 °C. The microstructure of the hardened products was studied by means of porosimetry and scanning electron microscopy.

1. Introduction

Much research has been devoted to alkali-activated materials, which have seen a great deal of development since the 1940´s. Alkali-activated materials based on different raw materials have been developed via many works of research [1-4]. Various (especially amorphous) aluminosilicates can be used as a precursor for alkali activation. Researchers are looking for new resources from which such materials can be developed. One of the possible materials capable of alkaline activation is the waste brick dust available from the production of calibrated thermal insulating bricks. In general, waste brick dust can be used as a substitute for cement in concrete, and for the alkaline activation of aluminosilicates as well due to the pozzolanic activity it exhibits [5,6]. The results of the alkali activation of aluminosilicates are dependent on many parameters, but mainly on the relationship between the precursor and the amount of alkaline activator, the composition of both, and the temperature of curing. Robayo et al. [7] described the effect of the SiO_2/Al_2O_3 and Na_2O/SiO_2 ratio on the compressive strength of alkali-activated red clay brick waste. The addition of soluble silica to the alkaline activator had a positive effect. Da Silva et al. [8] studied the effect of the SiO_2/Al_2O_3 ratio on the early-stage reaction kinetics of the geopolymerization of metakaolin at 40 °C using alkaline solution composed from sodium silicate and sodium hydroxide. The setting time increased with the increasing SiO_2/Al_2O_3 ratio of the initial mixture. Up to a certain limit, the SiO_2/Al_2O_3 ratio was also found to be responsible for the observed high-strength gains at later stages. Their investigation demonstrates that the properties of the resulting geopolymer systems can be drastically affected by minor changes in the available Si and Al concentrations during synthesis.

Most of the authors that have produced research in this area carried out the alkali activation of brick powder at an elevated temperature. They typically cured specimens at a temperature above 40 °C for different periods of time. Tuyan et al. [9] subjected alkali-activated mixtures to six different temperatures from 50 to 100 °C for four different durations, from 1 to 7 days. A gradual increase in compressive strength was observed in the mixtures cured at a temperature of up to 70 °C for all

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periods. Reig et al. [10] investigated the properties and microstructure of alkali-activated red clay brick waste using a curing temperature of 65 °C for 7 days, along with alkaline solutions with different SiO_2/Na_2O ratios, and various water/binder ratios as well. Compressive strength increases till a SiO_2/Na_2O ratio of 1.46 is reached. Rovnaník [11] analyzed the effect of curing temperatures from 10 to 80 °C and time of curing on the compressive and flexural strengths, pore distribution and microstructure of alkali-activated metakaolin. The results have shown that the treatment of fresh mixture at elevated temperatures accelerates the development of the aforementioned strengths, but the 28-day mechanical properties are worse in comparison with results obtained at ambient temperature.
The present experimental study aims to assess the effect of the amount of alkaline solution, Si/Al, Si/Na and Al/Na ratios, and temperature of curing on mechanical properties, porosity and microstructure.

2. Materials and methodology

2.1. Characterization of raw materials

Waste powder from the manufacture of calibrated brick elements at the HELUZ Brickworks, v.o.s. factory located in Libochovice (Czech Republic) was used as a solid precursor for the purposes of this study. The chemical composition of the brick powder obtained via XRF analysis (PANalytical) is presented in table 1, while the mineralogical composition obtained via XRD analysis (SAXS - PANalytical Empyrean) is specified in table 2. The proportions of the present minerals were calculated via Rietveld analysis.

Table 1. Chemical composition of brick powder.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	TiO_2	SO_3	Sum
Mass [%]	54.5	16.8	5.9	13.5	2.6	2.5	0.9	0.2	0.7	1.7	99.3

Table 2. Mineralogical composition of brick powder.

Mineral	Formula	[%]
Anorthite	$CaAl_2Si_2O_8$	13.4
Gehlenite	$Ca_2Al[AlSiO_7]$	2.6
Hematite	Fe_2O_3	2.7
Orthoclase	$KAlSi_3O_8$	8.9
Quartz	SiO_2	29.4
Illite 2M1	$K_{1.67}(AI_{3.29}Fe_{0.38}Mg0_{0.32})[(Si_{6.68}Al_{1.32})O_{20}(O)_{4}]$	19.7
Calcite	$CaCO_3$	1.5
Amorphous phase	-	21.8

The pozzolanic activity of the brick powder was determined using the modified Chapelle test. The result representing the amount of calcium hydroxide consumed in the reaction per 24 hours is 328 mg $Ca(OH)_2$ per 1 gram of brick powder. The particle size distribution of the brick powder with grains less than 1 mm in diameter is d_{10}= 1.753 µm, d_{50}=11.052 µm, and d_{90}=52.167 µm.

2.2. Composition of the tested mixtures

An alkaline solution (M_s=1.0) was prepared by mixing 70 g of sodium water glass (26.43% SiO_2, 16.61% Na_2O, and 56.96% H_2O) (Vodní sklo, a. s., Czech Republic), 9.5 g of NaOH, and 50 g of water. The mixture composition is presented in table 3. The Si/Al, Si/Na, and Al/Na ratios in the mixture are presented in table 4. While the Si/Al ratio slightly increases as the amount of alkaline solution grows, the Si/Na and Al/Na ratios markedly decrease in this case.

Table 3. Composition of mixtures.

Mixture	Brick powder [g]	Alkaline solution M_s =1.0 [g]	Water [g]
FL-151	200	30	87
FL-152	200	50	67
FL-153	200	70	47
FL-154	200	90	27

Table 4. Ratios of Si/Al, Si/Na, and Al/Na in the mixtures.

	FL-151	FL-152	FL-153	FL-154
Si/Al	2.86	2.93	3.01	3.08
Si/Na	9.43	6.56	5.09	4.19
Al/Na	3.29	2.24	1.69	1.36

3. Experimental procedure

The samples were treated as follows: two sets of samples were stored at the temperature 20 °C for 7 and 28 days, 2 sets of samples were stored at the temperature 60 °C for 7 and 28 days and one set of samples was treated for 7 days at 60 °C and 21 days at 20 °C.

The flexural and compressive strengths of 20×20×100 mm test specimens were determined using a hydraulic press (Tonindustrie, Germany). The porosity of the alkali-activated brick powder was assessed using a PoreSizer 9310 mercury porosimeter (Micromeritics). Cumulative pore volume was determined for samples cured at a temperature of 60 °C. Detailed microstructure images were taken via a MIRA3 XMU environmental scanning electron microscope (Tescan).

4. Results and discussion

4.1. Mechanical properties

The bulk densities grow from sample 151 to 154, and increase with the temperature and time of curing of individual samples. The bulk densities of the samples cured at 20 °C were determined to be between 1648 and 1825 kg·m^{-3}, while the heated samples had bulk densities from 1184 to 1613 kg.m^{-3}.

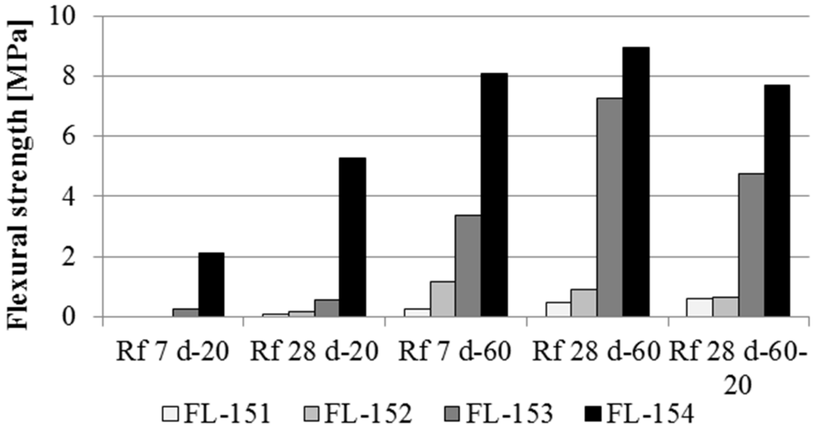


Figure 1. Flexural strengths.

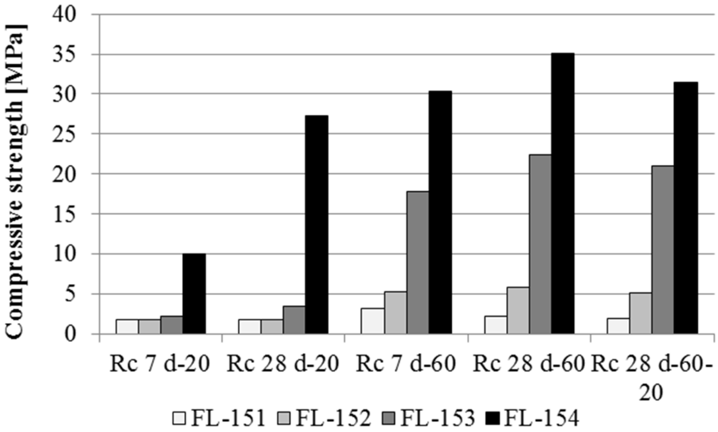


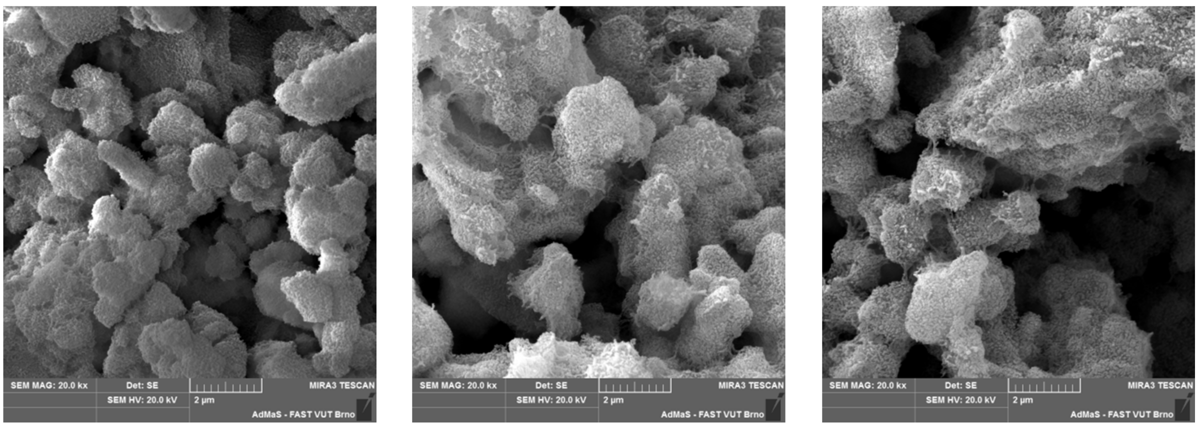
Figure 2. Compressive strengths.

The flexural strengths (Figure 1) increased with curing time at the same temperature, and with higher curing temperature as well. Significantly higher strengths were reached with higher proportions of alkaline solution (samples 153 and 154). Compressive strengths recorded the same development (Figure 2). Higher amounts of alkaline solution are related to the diminishing of Si/Na and Al/Na ratios, i.e. to the enhancement of sodium ion content, and the corresponding OH^- ion content.

4.2. Morphology of microstructure and pore structure

The microstructure of the samples differs depending on the composition of the fresh mixture. A higher amount of alkaline activator results in denser microstructure. Sample FL-151 has the least dense microstructure and the pores which are present are of a significant size for all curing temperature modes (Figure 3), and similar images were obtained for sample FL-152 (Figure 4). The microstructure of sample FL-153 (Figure 5) looks like that of the two previously named samples but the pore size is considerably smaller and little formations are visible on the surface. Sample FL-154 (Figure 6) features dense microstructure without pores, and only some cracks are visible; the images are quite different.

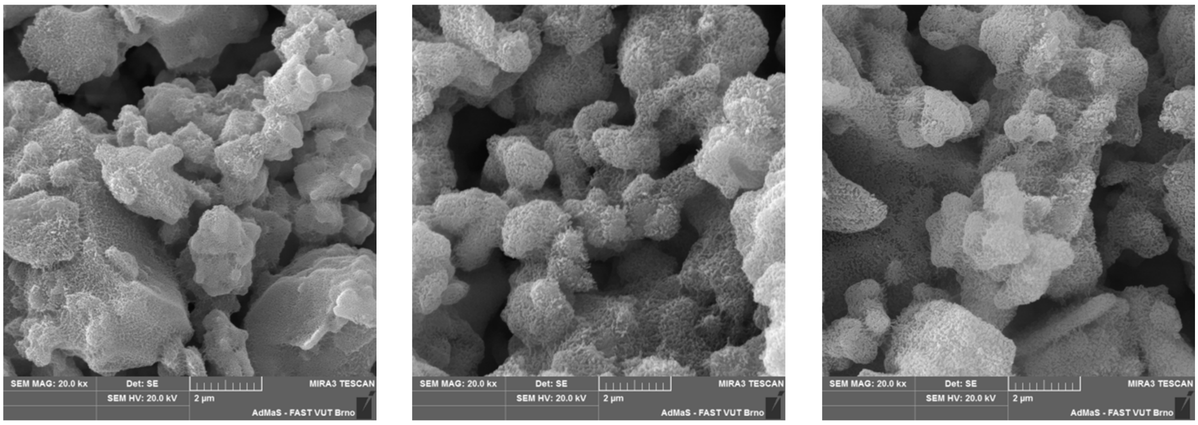
Pore structure was measured for all samples cured at a temperature of 60 °C for 28 days. The results for cumulative pore volume are presented in Figure 7. Pore volume decreases as the amount of alkaline solution in the mixture rises.



FL-151 – 28 d – 20 °C FL-151 – 28 d – 60 °C FL-151 – 28 d – 60 and 20 °C

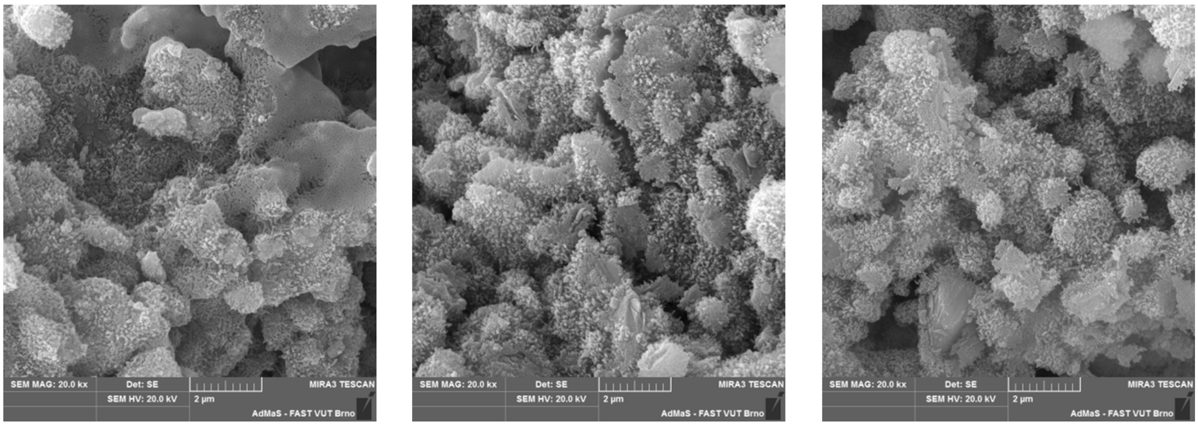
Figure 3. Micrographs of FL-151 samples cured at different temperatures.

The sample with the lowest concentration of alkaline solution (FL-151) has the largest pores and greatest cumulative volume of them all. The sizes of the pores and their cumulative volume fall as the concentration of alkaline solution in the mixture increases.



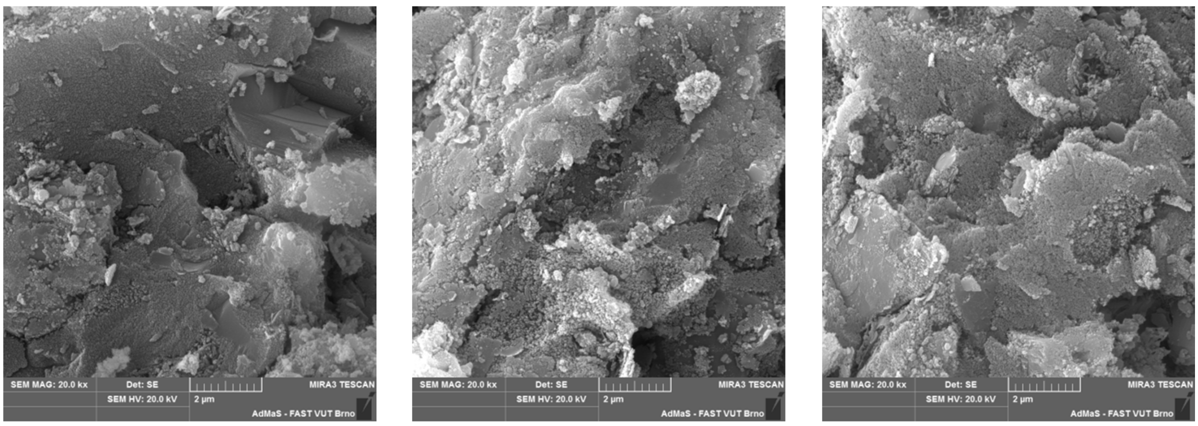
FL-152 – 28 d – 20 °C FL-152 – 28 d – 60 °C FL-152 – 28 d – 60 and 20 °C

Figure 4. Micrographs of FL-152 samples cured at different temperatures.



FL-153 – 28 d – 20 °C FL-153 – 28 d – 60 °C FL-153 – 28 d – 60 and 20 °C

Figure 5. Micrographs of FL-153 samples cured at different temperatures.



FL-154 – 28 d – 20 °C FL-154 – 28 d – 60 °C FL-154 – 28 d – 60 and 20 °C

Figure 6. Micrographs of FL-154 samples cured at different temperatures.

The sample with the highest concentration of alkaline solution (FL-154) contains the finest pores; their cumulative volume is the smallest of all the assessed samples. The results of porosimetric measurements, i.e. of pore size and cumulative pore volume, are in good agreement with the images of the samples in the micrographs above.

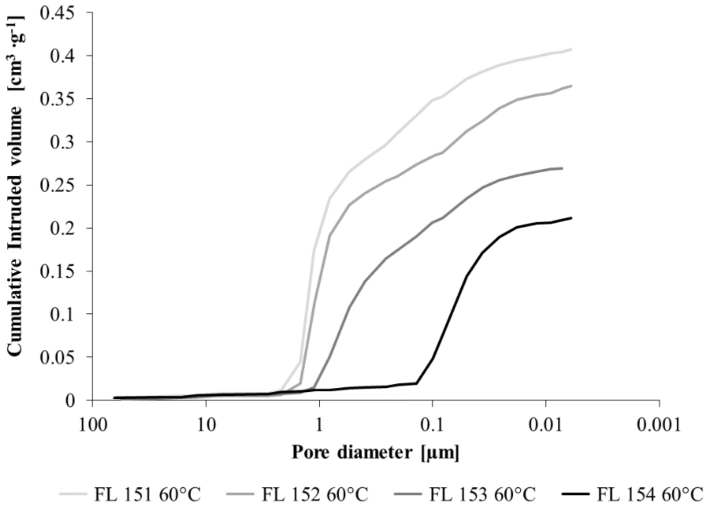


Figure 7. Cumulative intruded volume of pores.

5. Conclusion

This study has demonstrated the effect of the amount of alkaline solution and temperature on the microstructure and mechanical properties of alkali-activated brick powder. The following conclusions may be drawn from this study:

- Flexural and compressive strengths increase and microstructure is more comprehensive with growing Si/Al ratio.
- Decreasing Si/Na and Al/Na ratios mean a greater content of alkaline solution, resulting in a rise in the strengths and compactness of matter.
- The temperature of curing increases the strength of the hardened product. The most significant increase in strength was found in samples with the highest content of alkaline solution.
- Cumulative pore volume and pore diameter decrease with an increasing concentration of alkaline solution in the mixture.

Acknowledgments

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