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MATERIAL CHARACTERISTICS OF GEOPOLYMERS BASED ON AN INDUSTRIAL WASTE PRODUCT

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ABSTRACT

Generation of industrial waste and byproducts represents a substantial challenge for the waste disposal management. Usage of such materials in building industry for development of new materials can be considered as a promising way in this field. During the last decades, an increased attention is paid to the application of waste materials for geopolymer synthesis. Moreover, the efforts aimed at alkali-activated aluminosilicates point to an overall positive environmental impact of such materials due to their lower embodied energy, as compared to the traditional binders as cement or lime. In this paper, material characteristics of geopolymers synthesized using waste brick dust as precursor and water glass-sodium hydroxide mixture as activator are studied, taking into account the influence of curing temperature and duration. The materials are cured at 21, 40, 60 and 80 °C for 6, 24, 72, and 168 hours in order to find a correlation between curing conditions and compressive and bending strength. The obtained results provide an important information for further understanding of the process of geopolymer synthesis.

Keywords: geopolymers, curing temperature, mechanical properties, waste brick dust.

INTRODUCTION

Understanding of the hydration mechanism of alkali activated aluminosilicates poses a challenging task for material engineers. Despite of decades dedicated to this topic a proper description of the formation of hydration products is still not available. Investigations in this field are driven mainly by the effort to find construction materials with multiple benefits, such as improved mechanical properties, durability, resistance to corrosion and low environmental impact (Duxson *et al.*, 2007). The ambiguity of results presented across the available literature can be assigned to the wide composition range of the prime materials (precursors) and many combinations of applied alkali activators, together with different curing conditions (Gebregziabihier *et al.*, 2016; Xu and van Deventer, 2000). On this account, efforts aimed at advanced description of factors influencing material properties are strongly needed. It was found that hydration products formed during the alkali activation are dependent on the used precursors, type and concentration of activators and the curing conditions.

A robust research focused on the investigation of the effect of used alkaline activator on the properties of geopolymers elucidated some important findings and ability of such materials to

create dense and compact structure with promising mechanical properties (Memon *et al.* 2013; Fletcher *et al.*, 2005). The excellent resistance against fire or chemical attack, low thermal contraction and conductivity are other factors which predetermine the great potential of alkali activated aluminosilicates in building industry. Notwithstanding, the current trend aimed at sustainability led to the utilization of waste materials as precursors (Naik, 2002; Karim *et al.*, 2007). Their varying chemical composition poses a substantial challenge for further research and detailed understanding of all factors with impact on material properties. Studies aimed at the explanation of the influence of molar ratios during the geopolymerization elucidated formation of different hydration products. Besides the composition of precursors and curing conditions, the finesses of the precursors is also crucial for the strength development and microstructure formation. Some authors (Chindaprasir *et al.*, 2013; Rattanasak *et al.*, 2009) generally stated that fine particles exhibited an overall better reactivity and capability to fill pores and induced formation of more dense structure with improved compressive strength.

Curing conditions were found important factors affecting the behavior of alkali-activated aluminosilicates (Yusuf *et al.*, 2014). Elevated temperatures can significantly accelerate the formation of material microstructure and their impact on final mechanical properties is also substantial. Increased mechanical parameters of formed alkali activated aluminosilicates were reported also by other authors. The explanation of this finding was proposed by Bakharev (2005), who concluded that alkali-activation reaction need to overpass a thermal activation barrier. On the other hand, some authors highlighted the importance of the duration of the curing period. They noticed that a longer thermal curing period can induce a strength decrease. A great variety of duration of curing period can be found in the literature. Some authors (de Vargas *et al.*, 2011) recommended elevated temperature curing for 5 to 12 hours, but on the other hand Ridtirud *et al.* (2011) obtained the best mechanical performance of geopolymer paste after 20 days of curing at 60 °C. Nazzari *et al.* (2011) obtained the best mechanical properties after 36 h of curing at 80 °C. Their results also pointed at the risk of mechanical properties decrease above 90 °C when high temperature could decompose the granular structure of geopolymers. The potential risk related to high temperature curing consists in dehydration of geopolymer matrix and consequent undesirable shrinkage.

This study is aimed at the investigation of geopolymers based on waste brick dust which are alkali activated by the mixture of sodium silicate and sodium hydroxide. The experimental work is focused on the effect of different curing conditions on the properties of geopolymers.

MATERIALS AND METHODS

Manufacturing and grinding of thermal insulation bricks produces a substantial amount of waste brick powder with presumably favorable chemical composition and ready-to-use properties. The material obtained from Heluz, Czech Republic was only gathered and dried at 105 °C for 48 h to remove redundant moisture and sieved.

An Analysette 22 Micro Tec plus (Fritsch) device was used for determining the brick powder particle distribution. The measurement is based on the laser diffraction principle and can be used for the particle range from 0.01 µm to 2 mm; red laser is employed for coarse particles, while green laser beam for fine particles. Figure 1 shows that the used brick powder had a suitable finesses, with the maximum located close 55 µm.

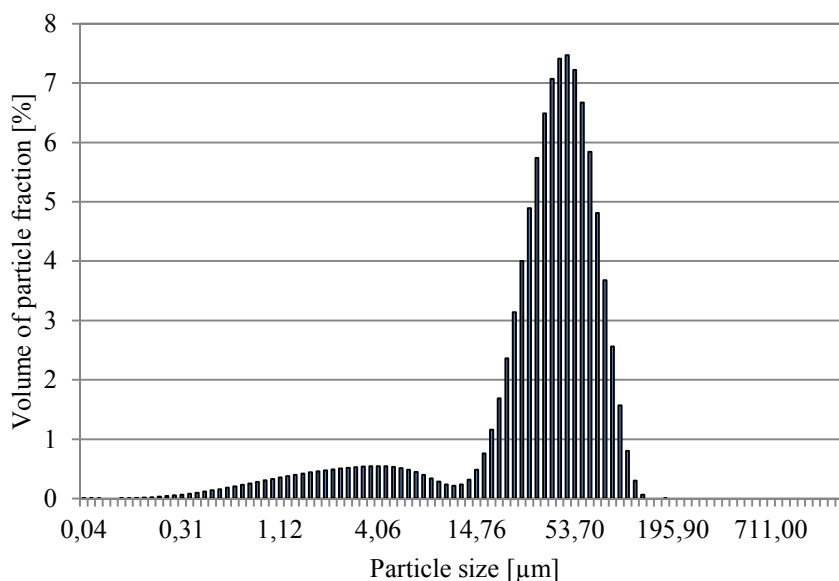


Fig. 1 - Particle size distribution of brick powder

The chemical composition of the studied waste brick powder was determined by X-ray fluorescence (XRF) analysis. The results presented in Figure 2 showed that the necessary oxides were present in substantial amounts.

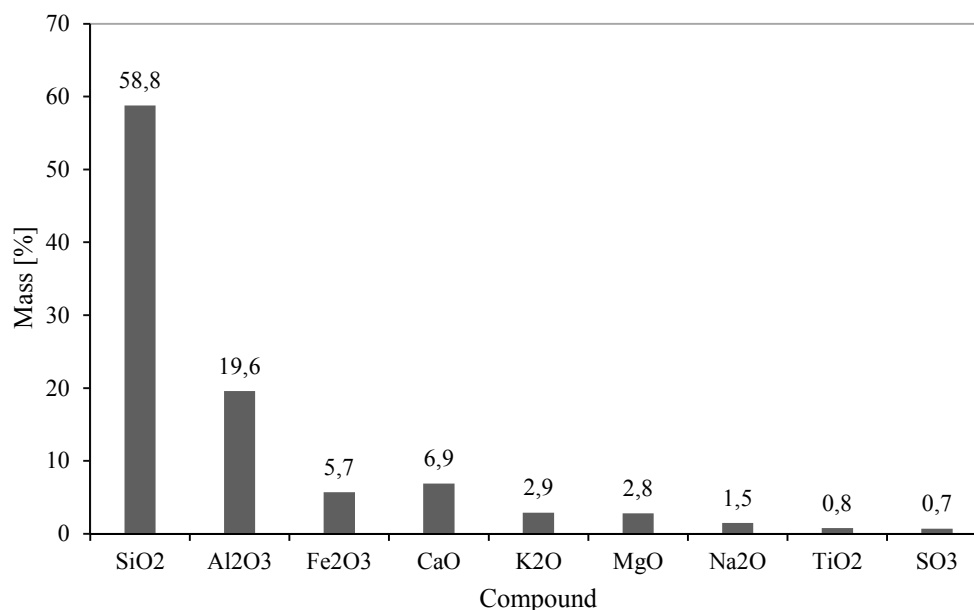


Fig. 2 - Chemical composition of waste brick powder

X-Ray diffractograms of the applied brick powder were obtained using a Panalytical XPert Pro diffractometer. The content of amorphous phase was determined by help of an added internal standard (20 % of ZnO). The obtained results are showed in Table 1. The amorphous part of waste brick powder constituted about 28 mass% of the sample. Among the most distinct crystalline compounds belonged quartz (26.2 mass%), albite (13.0 mass%), and muscovite (12.5 mass%)

Table 1 - Mineralogical composition of waste brick powder (XRD)

Substance	Chemical composition	(% by mass)
Amorphous part	-	27.8
Quartz	SiO ₂	26.2
Hematite	Fe ₂ O ₃	2.3
Albite	NaAlSi ₃ O ₈	13.0
Microcline	KAlSi ₃ O ₈	3.6
Orthoclase	KAlSi ₃ O ₈	3.5
Muscovite	KAl ₂ (AlSi ₃ O ₁₀)(OH) ₂	12.5
Illite	K _{0.65} Al ₂ (Al _{0.65} Si _{3.35} O ₁₀)(OH) ₂	3.8
Diopside	CaMgSi ₂ O ₆	4.4
Akermanite	Ca ₂ MgSi ₂ O ₇	2.8

The alkali activation was done by a mixture of sodium silicate and sodium hydroxide with the silicate modulus of 1.0, according to the recommendation of Law *et al.* (2014). During the geopolymer preparation, sodium hydroxide pellets (Fichema a.s.) were firstly dissolved in water and consequently mixed with sodium silicate (Vodní Sklo, a.s.). The brick powder was added afterwards and mixed in. The composition of prepared mixtures and description of curing conditions is summarized in Table 2.

Table 2 - Composition and curing conditions of geopolymer mixtures

Material	Brick powder (g)	Water glass (g)	NaOH (g)	Water (ml)	Silicate modulus (-)	Temperature of curing	Duration of curing
AA-21-6	200	70	9.5	50	1	21 °C	6 h
AA-21-24	200	70	9.5	50	1		24 h
AA-21-72	200	70	9.5	50	1		72 h
AA-21-168	200	70	9.5	50	1		168 h
AA-40-6	200	70	9.5	50	1	40 °C	6 h
AA-40-24	200	70	9.5	50	1		24 h
AA-40-72	200	70	9.5	50	1		72 h
AA-40-168	200	70	9.5	50	1		168 h
AA-60-6	200	70	9.5	50	1	60 °C	6 h
AA-60-24	200	70	9.5	50	1		24 h
AA-60-72	200	70	9.5	50	1		72 h
AA-60-168	200	70	9.5	50	1		168 h
AA-80-6	200	70	9.5	50	1	80 °C	6 h
AA-80-24	200	70	9.5	50	1		24 h
AA-80-72	200	70	9.5	50	1		72 h
AA-80-168	200	70	9.5	50	1		168 h

Cast samples were further divided into several categories and cured in different conditions. Four temperatures were chosen, namely 21, 40, 60 and 80 °C and the curing took 6, 24, 72 and 168 hours.

Among the basic physical properties of studied geopolymers, the bulk density, matrix density and open porosity were measured. The bulk density was determined by weighing the samples with known volume which was obtained using a digital caliper. The matrix density was measured by a Pycnomatic ATC (Thermo Scientific) helium pycnometer. The total open porosity was calculated on the basis of the knowledge of bulk- and matrix density.

A hydraulic testing device VEB WPM Leipzig having a stiff loading frame with the capacity of 3000 kN was employed for the measurement of compressive and bending strength.

RESULTS AND DISCUSSION

The basic physical properties are given in Table 3. Two basic trends were observed in the changes of open porosity; it decreased with increasing curing temperature and increasing curing duration. The bulk density showed the opposite trends, the matrix density was found basically unaffected by curing conditions.

Table 3 - Basic physical properties of studied materials

Mixture	Bulk density [kg/m ³]	Matrix density [kg/m ³]	Total open porosity [%]
AA-21-6	1760	2672	34.70
AA-21-24	1786	2664	32.96
AA-21-72	1812	2658	31.83
AA-21-168	1874	2649	29.26
AA-40-6	1801	2666	32.45
AA-40-24	1836	2657	30.90
AA-40-72	1865	2658	29.83
AA-40-168	1881	2651	29.05
AA-60-6	1812	2648	31.57
AA-60-24	1842	2645	30.36
AA-60-72	1870	2658	29.65
AA-60-168	1899	2641	28.10
AA-80-6	1820	2647	31.24
AA-80-24	1850	2642	29.98
AA-80-72	1885	2648	28.81
AA-80-168	1895	2650	28.49

As it was reported by Altan and Erdogan (2012), the increased temperature curing can induce the formation of a more dense structure compared to the mixtures cured at ambient conditions. Hydration at the elevated temperature can, according to the research performed by Nazzari *et al.* (2011), affect the condensation and re-solidification periods, which plays an important role for the development of microstructure and mechanical properties. The basic physical properties obtained in this paper were thus in a good qualitative agreement with these findings.

The lowest values of compressive strength were obtained for mixtures cured at ambient conditions (Figure 3). The increase in curing temperature led to a significant improvement of compressive strength. The curing at 40 and 60 °C brought a continuous increase in compressive strength with curing time up to 26.75 MPa and 57.31 MPa, respectively. For curing at 80 °C, the compressive strength in the initial time periods was significantly higher than for other curing temperatures but the increase was stopped after 72 h at 47.94 MPa. According to the findings of Topark-Ngarm *et al.* (2014) the observed improvements can be assigned to the higher degree of geopolymerization.

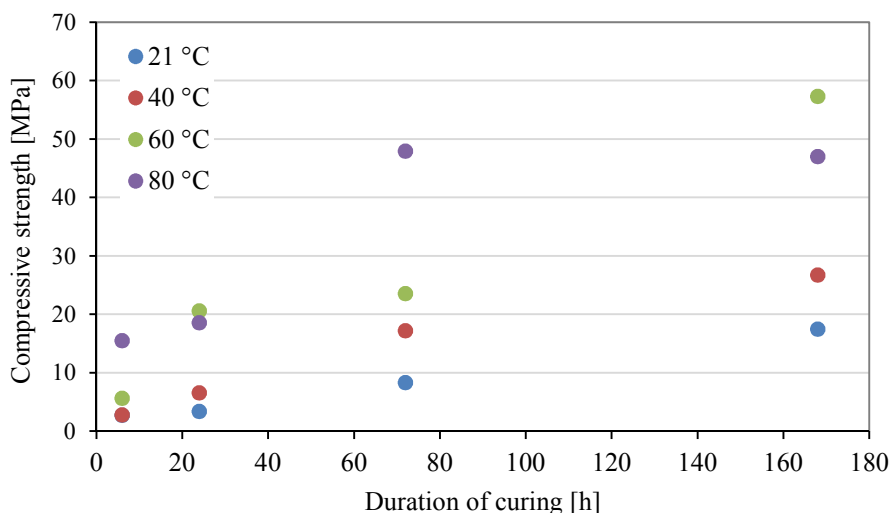


Fig. 3 - Compressive strength in dependence on curing temperature and duration

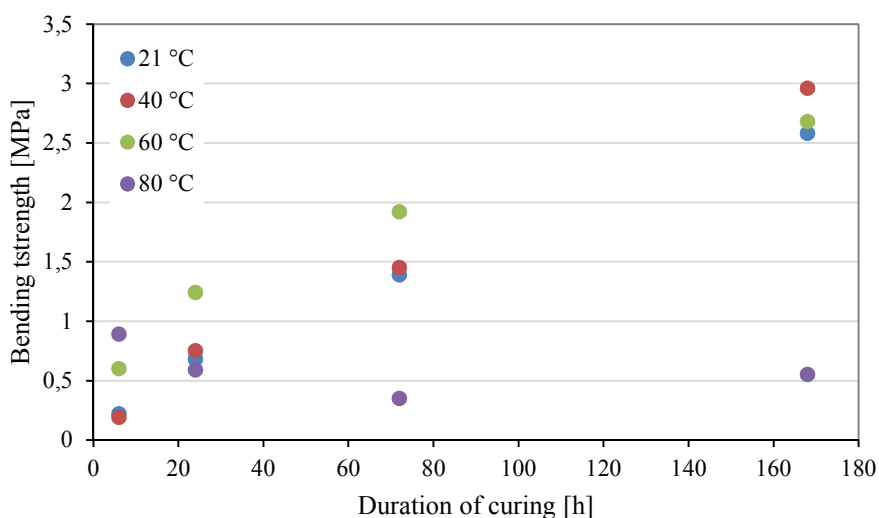


Fig. 4 - Bending strength in dependence on curing temperature and duration

The bending strength increased with the increasing curing temperature and curing time only up to 60 °C and 72 h (Figure 4). The highest bending strength after 168 h showed the geopolymer cured at 40 °C. Curing at 80 °C was effective only up to 6 h; for longer curing times the bending strength was even lower than for geopolymers cured at ambient conditions. The obtained results were in accordance with the findings of Nazzari *et al.*, (2011), who described a decrease of mechanical parameters as a result of prolonged curing period due to

excessive shrinkage caused by the dehydration of geopolymer matrix. Apparently, for the geopolymers analyzed in this paper, the decrease of bending strength was a consequence of the crack appearance caused by shrinkage which was more pronounced than for the compressive strength.

Summarizing the results obtained for the compressive and bending strengths, the optimal combination of curing conditions for the studied geopolymers was the curing temperature of 60 °C and curing duration of 168 h when the strength values of 57.31 MPa and 2.68 MPa, respectively, were achieved. In a comparison with the common curing at ambient temperature, which after 168 h resulted in the compressive strength of 17.45 MPa and bending strength of 2.58 MPa, it was for the compressive strength a remarkable improvement but for the bending strength only a negligible increase.

CONCLUSIONS

The effect of various curing regimes on the properties of geopolymers based on the utilization of waste brick powder as precursor was investigated in this study. The chemical composition and the particle size distribution of the raw material pointed at its suitability for alkaline activation. The experimental assessment of material properties of analyzed geopolymers showed that curing temperature had a major influence on the compressive strength which after curing at 60 °C increased up to three times, as compared with curing at ambient conditions. Apparently, this increase was related to the compaction of microstructure of the geopolymer caused by the higher degree of geopolymerization, which was manifested by the decrease of open porosity. On the other hand, the improvements in bending strength due to the increase of curing temperature up to 60 °C were only marginal, which could be attributed to the negative effect of enhanced shrinkage accompanied by crack appearance competing with the microstructure compaction. Curing duration up to 168 h had a positive effect on both compressive strength and bending strength for the curing temperatures up to 60 °C. Curing at 80 °C was effective only for the compressive strength in shorter time periods; the bending strength was affected negatively already after 24 hours of curing time.

Summarizing the results obtained in this paper, one can conclude that the increase of curing temperature up to 60 °C can lead to a substantial increase of the mechanical performance of geopolymers based on waste brick powder, which can also increase their application potential in the building industry. However, the obtained contradictory results for compressive and bending strength indicate a need for further, more detailed investigations.

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