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10. Binary alkali-activated materials with brick powder

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Abstract: Waste brick powder represents an easily available supplementary cementitious material that can contribute to the enhanced properties of lime plasters and concretes. Furthermore, the prerequisite properties of the brick powder enable its application in alkali activated materials. In this study, brick powder was used for production of binary alkali activated binders with metakaolin, slag or fly ash. Brick powder based geopolymers were tested for their mechanical properties, porosity and microstructure; flow properties of the fresh geopolymers were evaluated by rheometric measurements. In accordance with the results, brick powder could be a suitable precursor for blended binders with metakaolin and slag. These samples exhibited good mechanical properties and microstructure characteristics. The combination with fly ash was less applicable due to a rapid setting, hardening retarder used in these binders caused significant deterioration of the mechanical properties and massive efflorescence formation.

Keywords: alkali-activated, brick powder, binder, metakaolin

10.1. Introduction

The continual growth of construction industry is closely linked with growing need of traditional Portland cement that is the second most consumed commodity in the world (Barnes 2014). Unfortunately, its production requires a high volume extraction of natural resources and energy consumption that significantly contribute to emissions of CO₂ and other harmful substances to the atmosphere (Mehta 2001). On the contrary, the technology of alkali-activated binders allows the use of a wide range of secondary or waste products (blast furnace slag, fly ash, silica fume etc.). One of the primary raw material used in geopolymers is metakaolin produced by calcination of kaolinitic clays (2–2.5 times more expensive than cement). The estimated CO₂ savings, comparing alkali-activated materials to Portland cement based on the life-cycle studies, range from 30 to 80 % (Provis 2014) and together with reduced storing capacities needed for unutilized industrial by-products are promising values for

the future. The difference strongly depends on the market conditions and availability of source materials in particular region (Van Deventer, 2010).

Alkali-activated materials result from the reactions of raw materials with a high content of amorphous aluminosilicate phase and the alkaline activator solution. Alkaline activator, in most cases alkaline hydroxide and/or alkaline silicate, ensures the appropriate conditions for the raw materials dissolution and formation of new phases. The first theoretical background of alkaline activation was developed by Glukhovskiy in 1959 (Glukhovskiy 1959). Afterward, in 1981, prof. Davidovits introduced the term “geopolymer” after studies concerning the alkaline activation of a burnt mixture of kaolinite, limestone, and dolomite resulting in a material with regular polymeric three-dimensional structure (Davidovits 1981). Besides the better environmental performance, engineering properties of alkali-activated binders are comparable or superior to Portland cement in terms of strength, chemical and thermal resistance (Duxson 2007).

Ceramic waste represents a huge amount of industrial waste materials produced. Mostly, it comes from the processing of construction and demolition waste, such material is frequently contaminated by other constituents, e.g. concrete, masonry mortar, etc. Thus, it is primarily used as an inexpensive recycled aggregate for road construction and terrain works. The other source of material, more suitable for use in binder technology, is represented by fine ground brick powder that is recently widely produced during the high-precision plane grinding process of clay blocks (Keppert 2014). Use of waste brick powder does not involve additional costs and environmental impact. In spite of present effort to maximize re-utilization of ceramic waste, the major part is still directed to disposal in landfills (Pachego-Torgal 2010).

Since the ancient times, the finely ground brick powder is used as a pozzolanic admixture to lime plasters to enhance their mechanical performance, heat insulation properties and durability (Baronio 1996). In a similar way, it is nowadays applied in concrete technology. Various studies confirmed higher strength and durability of concrete with brick powder that exhibits lower chloride permeability, reduced autogenous shrinkage and volume changes caused by alkali-silica reaction (Turanli 2003). Use of recycled ceramic aggregate in structural concrete is rather limited due to its higher water absorption and therefore reduced freeze-thaw resistance.

Although the ceramic matrix dominantly consists of the crystalline phase (quartz, cristobalite) and the reactive vitreous content is generally lower, studies focused on the alkaline activation of red ceramic waste already confirmed its reactivity in the geopolymerization process (Robayo 2016). The lower rate of reactions could be increased by a higher specific surface of the material

(additional grinding), tailored alkaline activator or thermal treatment during setting but this is not necessary. When alkali silicate is used for the geopolymerization, mechanical properties show significant improvement (Sun 2013). The application of brick powder in blended alkali-activated materials was predominantly studied in binders with slag; slag–brick powder system allows alkaline activation of brick powder with insufficient reactivity for one-component geopolymers (Rakhimova 2015).

10.2. Materials and methods

The alkali-activated materials were composed as a mixture of brick powder with a maximum grain size of 1 mm and one other basic aluminosilicate precursor (metakaolin, ground granulated blast furnace slag or coal fly ash) in different mass ratio. Brick powder content was 0, 25, 50, 75 and 100%. The chemical composition of the aluminosilicate precursors is introduced in Table 10.1. A commercial sodium silicate solution with $\text{SiO}_2/\text{Na}_2\text{O} = 1.6$ was used as an activator and lower silicate moduli ($M_S 1.3$, $M_S 1.0$) were attained by mixing the water glass with pure solid NaOH (98%). The alkali-activated mortars were prepared using quartz sand with a maximum grain size of 2.5 mm as aggregate.

Table 10.1. Chemical composition of aluminosilicate precursors

Material	Brick powder	Metakaolin	Slag	Fly ash
SiO ₂ (%)	55.39	55.01	36.72	49.82
Al ₂ O ₃ (%)	17.07	40.94	8.54	24.67
Fe ₂ O ₃ (%)	4.49	0.55	0.10	7.50
CaO (%)	13.15	0.55	39.80	3.91
MgO (%)	2.37	0.14	9.93	2.68
S _{total} (%)	1.96	0.34	1.01	0.91
Na ₂ O (%)	0.69	0.09	-	0.70
K ₂ O (%)	2.79	0.06	-	2.78
MnO (%)	0.06	-	0.24	-
P ₂ O ₅ (%)	0.24	-	0.03	-
TiO ₂ (%)	1.30	-	0.33	-

The raw mix composition presented in Table 10.2 was designed according to the preliminary study when the water content (Fig. 10.1) varied in order to reach the same workability of mortars (EN 1015-3 1999). Fresh brick powder-fly ash blended binders exhibited extremely short setting time, so it was necessary to use a retarding agent (potassium citrate monohydrate) to keep the prolonged workability. This agent was used taking into account its possible adverse effect

on the mechanical properties with workability extended for more than 30 minutes, sufficient for mixing and moulding. The initial setting time of binders with metakaolin or slag was more than 60 minutes.

Table 10.2. Mix composition of alkali-activated mortars

Mixture	Aluminosilicate precursor (g)				Sodium silicate (g)	Sand (g)	K-citrate (g)
	BP	MK	S	FA			
MK 0	700	0	-	-	630 (Ms1.6, Ms1.3)	2100	-
MK 25	525	175					
MK 50	350	350					
MK 75	175	525					
MK 100	0	700					
S 0	700	-	0	-	280 (Ms1.6, Ms1.3)	2100	-
S 25	525		175				
S 50	350		350				
S 75	175		525				
S 100	0		700				
FA 0	700	-	-	0	490 (Ms1.0)	2100	122.5
FA 25	525			175			
FA 50	350			350			
FA 75	175			525			
FA 100	0			700			

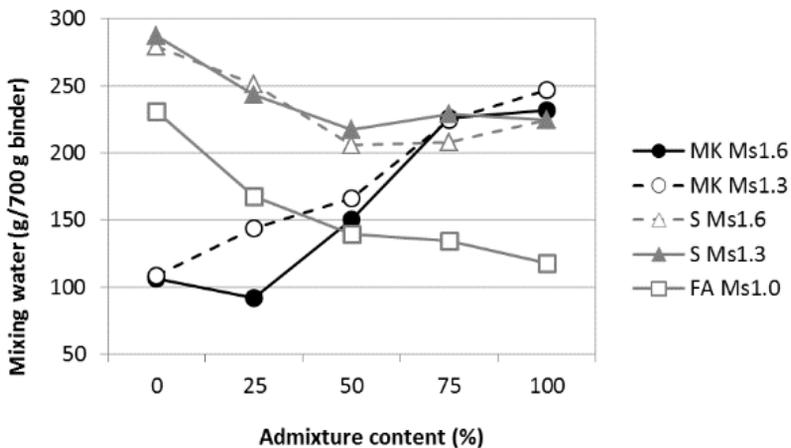


Fig. 10.1. Additional mixing water in alkali-activated mortars with various content of metakaolin, slag or fly ash

The mortars were cast into prismatic moulds (40 × 40 × 160 mm) and covered with a plastic sealant to avoid moisture loss. After 24 h in the ambient conditions, the specimens were heated at 40 °C for 4 h. After demoulding, the hardened specimens were stored in the laboratory conditions (22 ± 2 °C, φ = 45 ± 5 %) till the age of 7 or 28 days, respectively.

The mechanical properties (compressive and flexural strength) were tested after 7 and 28 days of curing. The microstructure and porosity of the geopolymers (7 days) were evaluated using Micromeritics Poresizer 9310 and scanning electron microscope (SEM) Tescan MIRA3 XMU.

Alkali-activated pastes for rheology testing were prepared with brick powder of the maximum grain size of 0.125 mm and the water-to-binder ratio remained constant for each binary system to evaluate the influence of both solid precursors and water glass (dosage and viscosity) on the rheological properties of the paste. Mix composition is shown in Table 10.3. The fresh pastes were tested using Discovery HR-1 (TA instruments) hybrid rheometer and TRIOS 4.0.2 software was used for a data evaluation. The measurements were performed in a Peltier Concentric Cylinder system with a DIN rotor at 25 °C. The standard gap for DIN cylinder system (5.917 mm) was adopted. The paste was introduced into the rheometer at the end of the mixing cycle and the measurement followed the procedure listed in Table 10.4. Parameters (yield stress, viscosity, rate index, reliability) were estimated from the decreasing shear branch of the flow curves using Herschel-Bulkley model:

$$\tau = \tau_0 + K\dot{\gamma}^n \quad (10.1)$$

where τ is shear stress, τ_0 is yield stress, K is coefficient of consistency, $\dot{\gamma}$ is shear rate and n is rate index. Investigations were performed at a constant temperature of 25 °C.

Table 10.3. Mix composition of alkali-activated pastes for rheology measurements

Mixture	Aluminosilicate precursor (g)				Sodium silicate (g)	K-citrate (g)	Water (ml)
	BP	MK	S	FA			
MK 0	50	0	-	-	42	-	21
MK 25	37.5	12.5					
MK 50	25	25					
MK 75	12.5	37.5					
MK 100	0	50					
S 0	50	-	0	-	20	-	15
S 25	37.5		12.5				
S 50	25		25				
S 75	12.5		37.5				
S 100	0		50				
FA 0	50	-	-	0	35	8.75	14
FA 25	37.5			12.5			
FA 50	25			25			
FA 75	12.5			37.5			
FA 100	0			50			

Table 10.4. Procedure of the rheological measurements

Step	Duration	Description
Sample conditioning	60 s	Temperature equilibration (25 °C)
	60 s	Pre-shear, shear rate 100 s ⁻¹
	60 s	Equilibration
Flow Sweep	600 s	Linear sweep, shear rate 0,1–150 s ⁻¹

10.3. Results and discussion

10.3.1. Brick powder–metakaolin geopolymer

Mechanical properties of brick powder–metakaolin geopolymers are presented in Fig. 10.2. Generally, both compressive and flexural strength of the pure brick powder geopolymer (MK 0) were quite low. The strengths of the mixture with silicate modulus of 1.3 were slightly higher to a higher alkaline content, and thus better ability to dissolve aluminosilicate network. The flexural strength of these mixtures decreased with time which can be attributed to the microcrack formation caused by drying shrinkage. A partial replacement of brick powder with metakaolin significantly improved the mechanical performance. Blended binder made from brick powder and metakaolin also exhibited a rather intensive synergistic effect in strength. The best mechanical properties were achieved with metakaolin content of 50% (M_S1.6) or 75% respectively (M_S1.3).

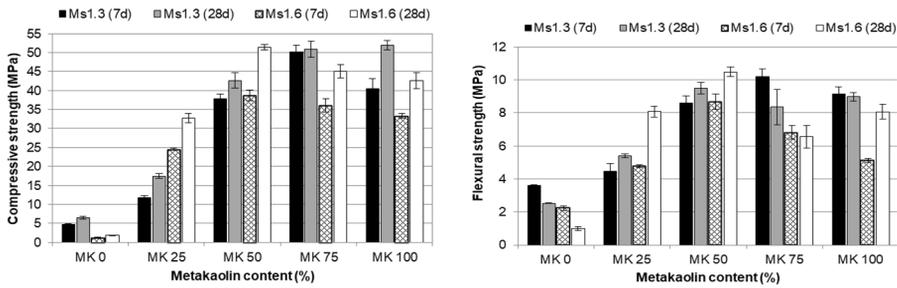


Fig. 10.2. Compressive (left) and flexural (right) strength of brick powder–metakaolin geopolymers at the age of 7 and 28 days

Mechanical properties are in good accordance with a pore size distribution of the samples (Fig. 10.3). When the metakaolin content rose above 50%, the binder was characterized by an increased number of gel pores and reduced volume of macropores. Although the total porosity of these samples was slightly higher, it influenced negatively neither compressive nor flexural strength. The porosity of binders with 75% of metakaolin was lower compared to the pure metakaolin geopolymer (in case of both types of water glass), which means that the brick powder in low dosage modifies pore structure and involves in strength enhancement of the material. Water in geopolymers with low Ca content has only a rheological purpose and it is not involved in geopolymerization process. Higher water demand in metakaolin binders was associated with its high specific surface but did not affect mechanical performance.

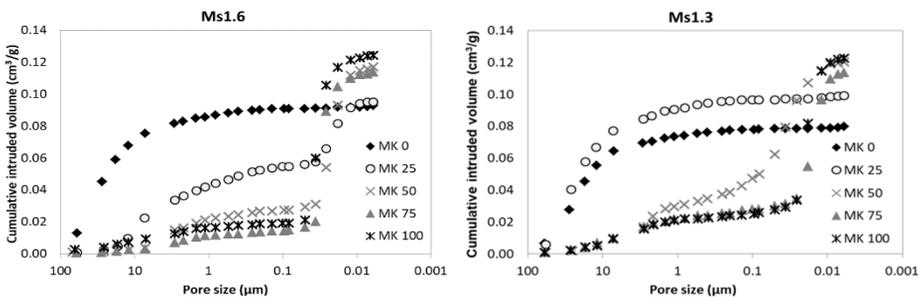


Fig. 10.3. Cumulative intruded volume of early brick powder–metakaolin geopolymers

Matrix homogeneity highly depended on the ratio of input materials as can be seen from the first two images of the same magnification (MK 25 and MK 75) in Fig 10.4. The structure of the binder consisted of intact brick particles and residual layered metakaolin particles bound by an aluminosilicate gel (Fig. 10.4, right).

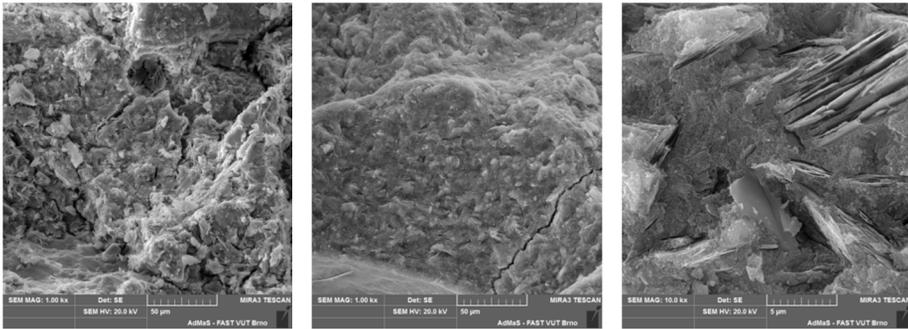


Fig. 10.4. SEM images of brick powder–metakaolin geopolymers (scale: MK 25: 50 μm , MK 75: 50 μm , MK 75: 5 μm)

Flow sweep curves recorded for the pastes with different metakaolin/brick powder ratio are shown in Fig. 10.5. With growing metakaolin content, pastes exhibited higher stress, yield stress and consistency coefficient. Pastes MK 0 and MK 25 (with both types of activating solution) were characterized by almost zero yield stress and rate index approaching 1 (Table 10.5) which refers to Newtonian fluids. In this case, the consistency coefficient can be considered a plastic viscosity. Increasing distance between two branches of flow curves for the pastes with increasing metakaolin content is caused by the thixotropic behaviour. Besides the metakaolin/brick powder ratio, all parameters of the fresh pastes with constant water content are also influenced by the silicate modulus (therefore viscosity) of alkaline activator solution. The consistency coefficient of mixtures with water glass of lower silicate modulus ($M_s1.3$) is almost doubled with respect to the mixtures with other type of water glass ($M_s1.6$). The rate index increased with higher metakaolin content indicating a slightly shear-thickening behaviour of metakaolin.

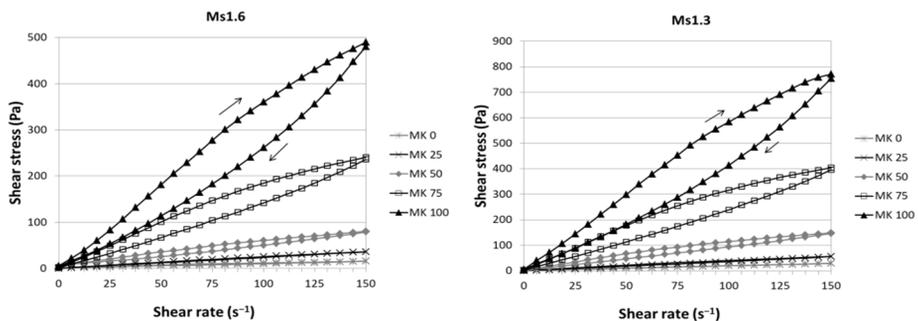


Fig. 10.5. Flow sweep curves of brick powder–metakaolin pastes

Table 10.5. Yield stress and viscosity of brick powder–metakaolin pastes

Mixture	Yield stress (Pa)		Consistency coefficient (Pa·s)		Rate index (-)	
	M _S 1.6	M _S 1.3	M _S 1.6	M _S 1.3	M _S 1.6	M _S 1.3
MK 0	0.40	0.06	0.10	0.20	0.99	0.99
MK 25	0.24	0.54	0.22	0.29	1.02	1.04
MK 50	5.55	2.13	0.21	0.66	1.16	1.07
MK 75	5.03	9.38	0.54	0.93	1.20	1.20
MK 100	15.55	26.93	0.32	0.53	1.44	1.44

10.3.2. Brick powder–slag binder

Alkali-activated brick powder binders with slag exhibited an apparent trend in mechanical strength evolution with a decreasing brick powder/slag ratio depicted in Fig. 10.6. Slag was responsible for high compressive strength (around 80 MPa) but binder with 50% of brick powder also reached a satisfactory value of around 50 MPa. Considerable influence of silicate modulus of the alkaline activator was not observed; the only difference consisted in slower compressive strength gains. Mechanical properties after 28 days of curing were comparable while 7 days strength was slightly higher for M_S1.3 mixture. The flexural strengths followed a similar trend and were quite comparable with brick powder–metakaolin blends. Porosity changed in accordance with the results mentioned. Total pore volume of brick powder geopolymer was twice as high as the one of alkali-activated slag and the maximum of volume is associated with large capillary pores between 0.5–20 μm (Fig. 10.7). In case of slag-rich binders, the water demand is lower but it is partly consumed during the hydration of Ca-rich slag to form C-A-S-H binding products.

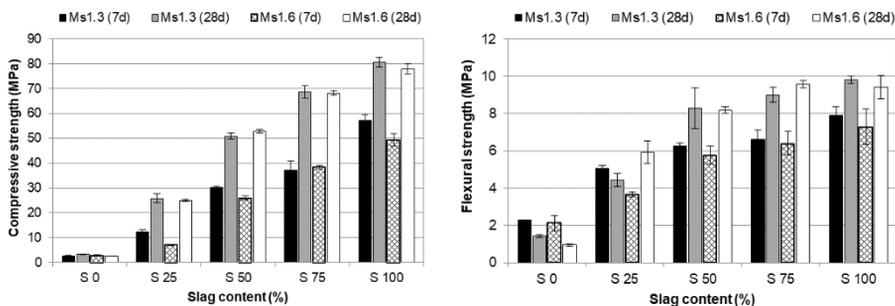


Fig. 10.6. Compressive (left) and flexural (right) strength of brick powder–slag geopolymers at the age of 7 and 28 days

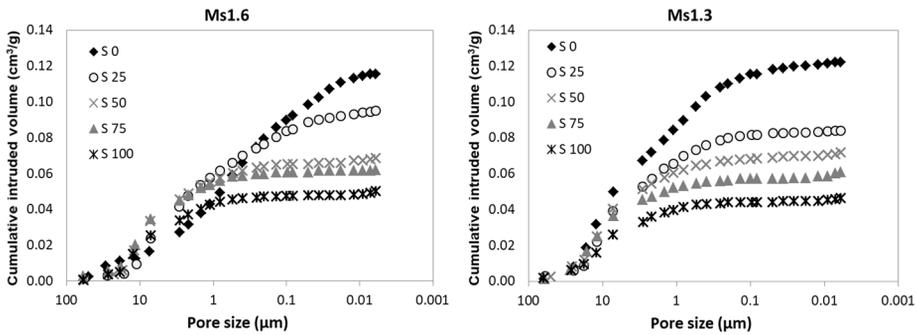


Fig. 10.7. Cumulative intruded volume of early brick powder–slag geopolymers

The SEM images in Fig. 10.8 show less homogenous structure with microcracks due to drying during the sample preparation procedure and needle-like crystals indicating the initial carbonation process. Carbonation of some metakaolin-based samples was also observed.

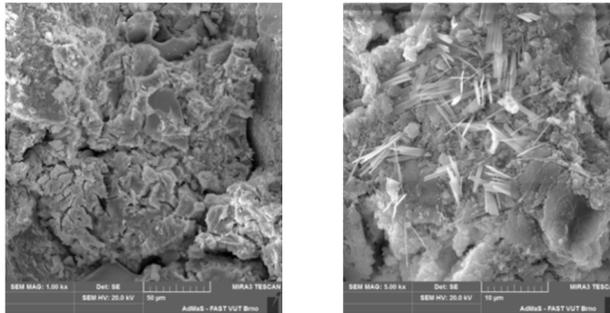


Fig. 10.8. SEM images of brick powder–slag geopolymers (scale: S 25: 50 μm , S 25: 10 μm)

Slag addition showed an adverse effect on the rheology of geopolymers (compared to metakaolin) and reduced the shear stress of the geopolymer pastes (Fig. 10.9). All pastes in the $M_{S1.3}$ series containing slag showed similar yield stress and slightly decreasing consistency coefficient with a higher slag content (Table 10.6). However, mixture S 0 showed the worst workability because its coefficient of consistency is more than five times higher. Since rate index of all mixtures is very close to 1.0, the consistency index can be considered a plastic viscosity of geopolymer pastes. Pastes with activator $M_{S1.6}$ showed similar trend with even lower viscosity of mixtures having more than 25% of slag. The exception was a pure brick powder mixture (S 0) for which the measurement was not completed due to the accelerated setting time. The maximal stress ascended to more than 1250 Pa and did not decrease during measurement of

decreasing branch of the flow curve. Therefore, it was not possible to assess the rheological parameters.

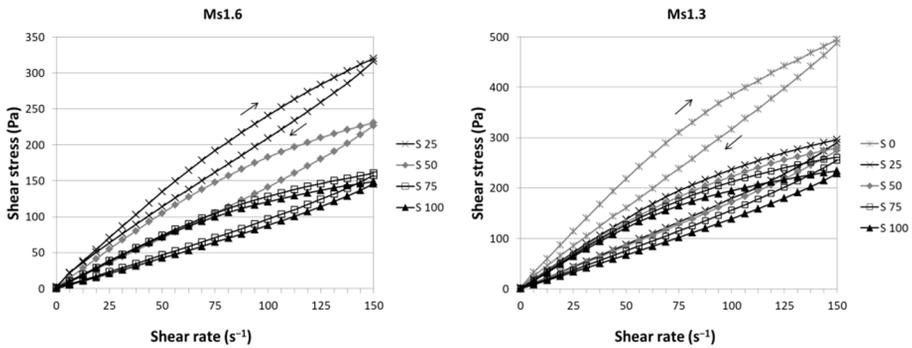


Fig. 10.9. Flow curves of brick powder–slag pastes

Table 10.6. Yield stress and viscosity of brick powder–slag pastes

Mixture	Yield stress (Pa)		Consistency coefficient (Pa·s)		Rate index (-)	
	Ms1.6	Ms1.3	Ms1.6	Ms1.3	Ms1.6	Ms1.3
S 0	-	0.59	-	5.62	-	0.89
S 25	7.43	4.86	2.46	1.02	0.96	1.12
S 50	4.07	s86	0.87	1.10	1.10	1.09
S 75	2.87	4.43	0.48	0.78	1.15	1.15
S 100	2.82	4.11	0.39	0.66	1.17	1.16

10.3.3. Brick powder–fly ash binder

While the brick powder–fly ash pastes tested in the preliminary study achieved excellent mechanical properties (more than 60 MPa in compression and 13 MPa in flexure after 7 days), addition of retarding agent to blended mortars caused an extreme drop in both compressive and flexural strength which varied within 7–13 MPa and 3.7–4.7 MPa, respectively (Fig. 10.10). Nevertheless, the combination of precursors did show a considerable synergistic effect. Samples with 50 and 75% of fly ash reached comparable properties but increased brick powder content resulted in lower 7 days strength. Fly ash-based binders usually exhibit good workability and required the lowest water content despite the higher viscosity of water glass with low silicate modulus (Ms1.0). Poor performance of brick powder–fly ash mortars was associated with a higher content of macropores (Fig. 10.11) and lower binding ability of constituent parts

probably affected by the retarding agent. This fact was also evidenced by SEM images in Fig. 10.12 illustrating low degree of particles dissolution and matrix

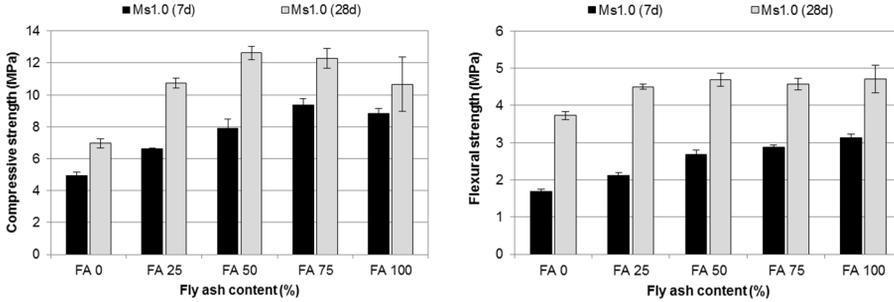


Fig. 10.10. Mechanical properties of brick powder–fly ash geopolymers (FA 1.0) after 7 and 28 days of curing

inhomogeneity. Moreover, the excess of alkalis that were not consumed during the geopolymerization process was reflected in a massive efflorescence formation (Fig. 10.12, right).

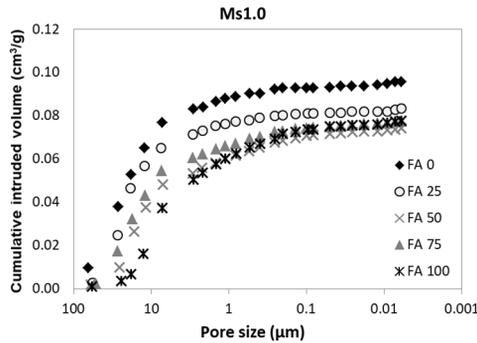


Fig. 10.11. Cumulative intruded volume of early brick powder–fly ash geopolymers

Addition of fly ash to the brick powder paste also decreased the stress but the differences were very small (Fig. 10.13). The pastes also showed less thixotropic behaviour compared to mixtures with slag and metakaolin. Yield stress of pure brick powder paste was slightly higher (1.32 Pa) than the one obtained for the pastes containing fly ash (0.66–0.76 Pa). In general, all the values could be considered as zero yield stress typical for Newtonian fluids (Table 10.7). Newtonian behaviour is also supported by the values of rate index which are almost equal to 1.00. Viscosity almost linearly decreased with the

decrease of brick powder/fly ash ratio. This is can be explained by mostly spherical fly ash particles which increase the fluidity of the system.

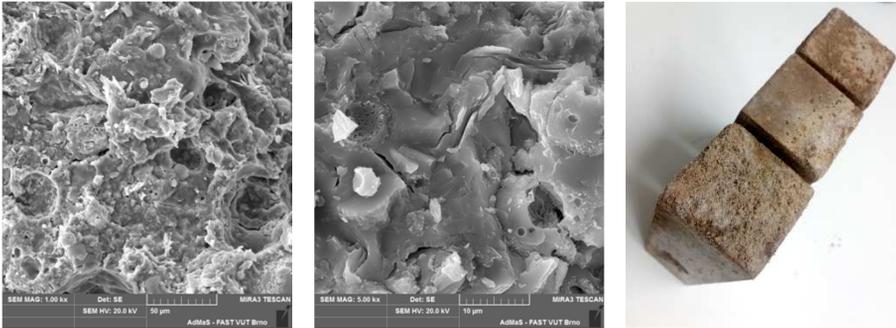


Fig. 10.12. SEM images of brick powder–fly ash matrix (scale: FA 50: 50 μm , FA 50: 10 μm) and photograph of geopolymers sample with efflorescence.

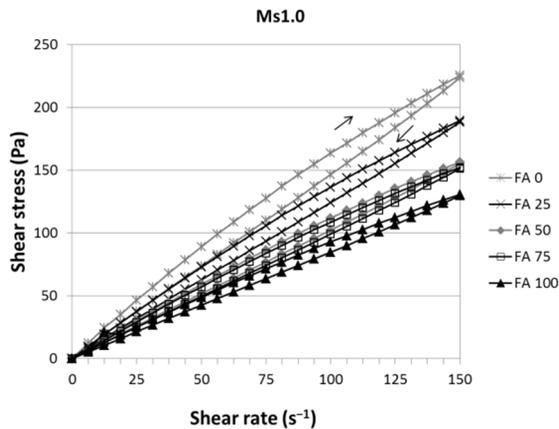


Fig. 10.13. Flow curves of brick powder–fly ash pastes

Table 10.7. Yield stress and viscosity of brick powder–fly ash pastes

Mixture	Yield stress (Pa)	Consistency index (Pa·s)	Rate index (-)
FA 0	1.32	1.35	1.02
FA 25	0.76	1.21	1.01
FA 50	0.66	0.98	1.01
FA 75	0.75	0.89	1.02
FA 100	0.70	0.75	1.03

10.4. Conclusions

This study aimed to analyse the possibility of use of red clay brick powder in a binary alkali-activated binder with metakaolin, slag and fly ash. The best performance in terms of mechanical properties was observed in case of brick powder–metakaolin and brick powder–slag matrix, both of these reached the compressive strength of more than 40 MPa with 50% brick powder content. Brick powder geopolymers blended with slag exhibited lower porosity, and therefore, enhanced mechanical properties. Although the total pore volume of samples with higher metakaolin content was higher, it did not weaken the binder structure because of their small diameter (gel pores). Brick powder–fly ash geopolymers required highly concentrated alkaline activator solution which caused significant shortening in the setting time. The application of potassium citrate as retarding agent resulted in a considerable drop of strength accompanied by the formation of strong efflorescence. Metakaolin was the only precursor that increased yield stress and viscosity of fresh brick powder geopolymer pastes during rheological measurement, fly ash and slag caused their decrease. While the pastes with slag and a high percentage of metakaolin were thixotropic, parameters of all other mixture proved Newtonian fluid-like behaviour.

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