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Rheological and mechanical properties of alkali-activated brick powder based pastes: effect of amount of alkali activator

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Abstract. The rheological behaviour and mechanical properties of alkali-activated materials prepared by activation of a brick powder by alkaline solution (alkali + water glass) is presented in this paper. The effect of quantity of alkaline activator on the rheological and mechanical properties of brick pastes prepared from two types of brick dust was monitored. The rheological investigation included the characterization of flow properties and viscoelastic properties of the fresh mixtures. Compression and bending strengths were determined at 7, 28, 56 and 90 days. These materials have a low yield stress and a high viscosity strongly influenced by the amount of alkali activator. The fresh brick pastes become liquid more easily with increasing amount of alkali activator. The rising amount of alkali activator leads to a decrease of the material cohesion. The results of rheological and mechanical properties investigation showed considerable differences in behaviour of particular types of brick dust.

1. Introduction

Recently, the aspiration to exploit the waste brick dust as a precursor for alkali-activated materials has been developed [1–6]. Brick dust as a waste material is available from the development of building elements for the construction of low-energy houses, or from a demolition waste. Bricks waste is usually rich of quartz and fired clays, consisting in dehydrated alumino-silicates not only in crystalline phase but also in amorphous state which is important for alkaline reaction. After an alkaline activation of aluminosilicate precursors, partially or fully amorphous inorganic polymers are formed with Si–O–Al bonds arranged through SiO₄ and AlO₄ tetrahedra into 3D-network (geopolymerization reaction). As activators, a wide range of alkaline solutions is applied: NaOH, Na₂CO₃, sodium silicate (water glass), sodium aluminate, or their potassic or lithic equivalents. The elevated temperature promotes the alkali reaction, mostly used is temperature of 60 to 90°C, although even at ambient temperature an aluminosilicate gel is formed by brick powder activation [3].

The effect of using different amounts of sodium silicate for brick powder activation has been superficially monitored [3]. Pastes obtained from brick powder activation at room temperature, cured for 7 days, exhibited bulk density of 1.5–2.0 g·cm⁻³ and water absorption of 20–27%; high content of sodium silicate solution in the mix design promoted more compact microstructures.

In recent years, an academic research on the binders has considerably extended the understanding of the geopolymerization reaction and rheological measurements greatly contributed to it. Furthermore, most of them are carried out on metakaolin, fly ashes or slags based geopolymers [7–12].

In this study, the influence of quantity of alkaline activator on the rheological and mechanical properties of brick pastes prepared from two types of brick dust (differing by content of Ca and



amorphous phase) was monitored. From the point of view of the rheology, it can assume a similar behaviour of the brick powder paste system and the metakaolin paste system. Both of them are suspensions of particles (brick powder, metakaolin) in a continuous fluid (sodium silicate solution) and their rheological behaviour can be comprehended by the physics of concentrated suspensions of rigid particles and therefore the results obtained in this study are compared with the results of similar metakaolin based geopolymers.

2. Materials

Two types of waste material from skiving of heat-insulating bricks from the HELUZ Brickworks factory, v.o.s., plant in Libochovice (L) and Hevlín (H) were the basic raw materials for alkali-activated pastes. The powder were used as-received, just the particles larger than 1 mm were removed by sieving. The chemical composition of the brick powder is shown in Table 1. The phase composition of the brick powder was studied via XRD using Rietveld analysis (Table 2) and the particle size distribution was determined using a laser diffraction analyzer (Figure 1). Fundamental physical parameters of the brick powders are summarized in Figure 1 as well.

The brick pastes were prepared by homogenization of brick powder with alkaline activator prepared from sodium silicate solution (water glass, $\text{SiO}_2/\text{Na}_2\text{O} = 1.6$, Vodní sklo, a.s., Czech Republic), sodium hydroxide p. a. (Penta Chemicals, a.s.), water and silicone defoamer (Lukosan S, Lučební závody a.s. Kolín). The $\text{SiO}_2/\text{Na}_2\text{O}$ ratio of prepared alkali activator was 1.0 and it was added in three different dosages (Table 3). For decreasing amount of sodium silicate solution a higher content of water in mixture was necessary to reach an acceptable consistency.

Fresh mixtures were cast into prismatic moulds of size $20 \times 20 \times 100$ mm. Standard conditions of sample storage were $21 \pm 2^\circ\text{C}$ and relative humidity of $50 \pm 5\%$. After 7, 28, 56 and 90 days the flexural and compressive strengths were determined.

Table 1. Chemical composition of brick powders examined by XRF spectroscopy (mass %).

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	TiO_2	SO_3	sum
L	54.2	17.2	4.4	14.1	1.7	2.8	0.3	0.2	1.0	1.1	97.0
H	55.5	17.0	5.8	10.5	2.4	2.8	0.7	0.2	0.8	1.9	97.6

Table 2. Phase composition of brick powders (mass %).

Mineral	Formula	L	H
Quartz	SiO_2	20.0	26.2
Hematite	Fe_2O_3	2.0	2.3
Albite	$\text{NaAlSi}_3\text{O}_8$	3.5	13.0
Microcline	KAlSi_3O_8	4.2	3.6
Orthoclase	KAlSi_3O_8	6.5	3.5
Muscovite	$\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$	4.4	12.5
Illite	$\text{K}_{0.65}\text{Al}_2(\text{Al}_{0.65}\text{Si}_{3.35}\text{O}_{10})(\text{OH})_2$	3.1	3.8
Diopside	$\text{CaMgSi}_2\text{O}_6$	1.6	4.4
Akermanite	$\text{Ca}_2\text{MgSi}_2\text{O}_7$	2.9	2.8
Mullite	$\text{Al}_6\text{Si}_2\text{O}_{13}$	2.9	–
Anhydrite	CaSO_4	2.0	–
Amorphous	–	47.0	27.8

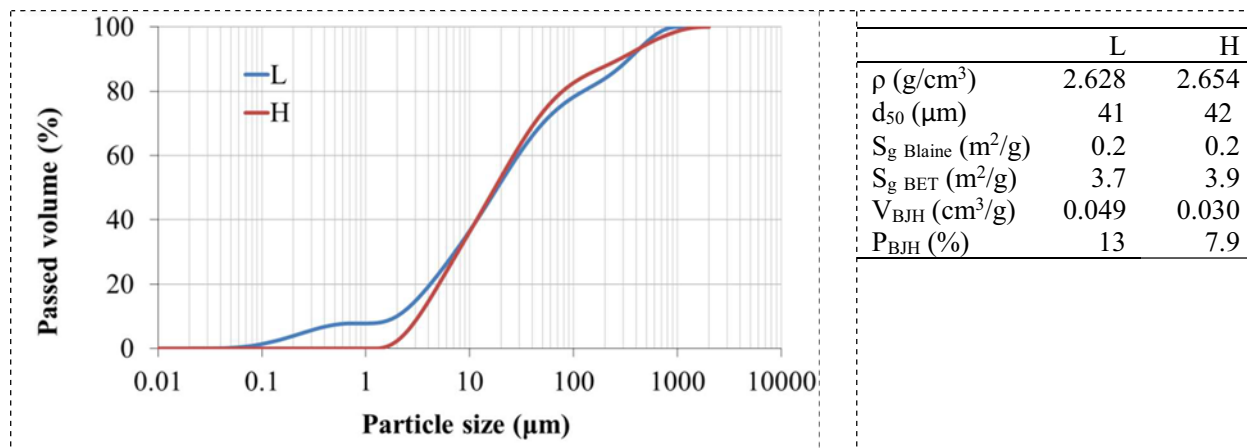


Figure 1. Particle size distribution and fundamental physical parameters of brick powders.

3. Experimental procedure

3.1. Rheological properties

Rheological tests were carried out on the hybrid rheometer Discovery HR-1 (TA Instruments) in the Peltier Concentric Cylinder system with a DIN rotor and TRIOS 4.0.2.30774 software was used for a data evaluation. All the measurements have been done at 25°C.

3.1.1. Flow properties. The brick suspension was introduced into the measurement system at the end of the mixing cycle. The rheological measurements were started after a period of rest of 60 s. The testing routine comprised a shear rate increase (from 0.1 to 100 s⁻¹) applied through 30 steps with 15 s of measuring time at each shear rate followed by a decrease of shear rate on the same conditions. The results were expressed as shear rate vs. shear stress (flow curves) and the Herschel-Bulkley model (1) was applied to descending branches of flow curves to fit the experimental data and used to describe the pastes rheological behaviour [13]:

$$\tau = \tau_0 + k\dot{\gamma}^n \quad (1)$$

where τ is the shear stress, $\dot{\gamma}$ is the shear rate, τ_0 corresponds to the yield stress, k is the consistency coefficient and n is the fluidity index which characterizes shear-thinning ($n < 1$) or shear-thickening ($n > 1$) behaviour of a material.

3.1.2. Viscoelastic properties. Time evolution of the viscoelastic properties of pastes were evaluated by small amplitude (0.01%) oscillation tests measuring dynamic moduli, G' and G'' , represent the elastic and viscous behaviour of a material. If the elastic behaviour dominates the viscous one ($G' > G''$), the sample exhibits a certain rigidity. Conversely, if $G'' > G'$, the sample shows the character of a liquid without a consistent chemical or physical network-of-forces [13]. Since one of the purposes of the this study is to determine the variation over time of the viscoelastic parameters according to the geopolymer composition, it is important to determine the linear viscoelasticity region (LVR) in order to prevent structural decomposition of the geopolymerized network. The LVR is the region in which measurements are nondestructive to the microstructure of a material and it ends at a critical strain γ_c . The strain of 0.01% used in the oscillation tests is lower than the critical strain for all brick pastes studied. The rheological characterization was completed by frequency sweep tests with increasing the frequency from 0.1 Hz to 10 Hz. Experimental data were reported in terms of complex modulus, G^* , and loss tangent, $\tan(\delta)$, defined as follows (2–3):

$$G^* = \sqrt{(G')^2 + (G'')^2} \quad (2)$$

$$\tan(\delta) = G''/G' \quad (3)$$

Table 3. Composition of mixtures.

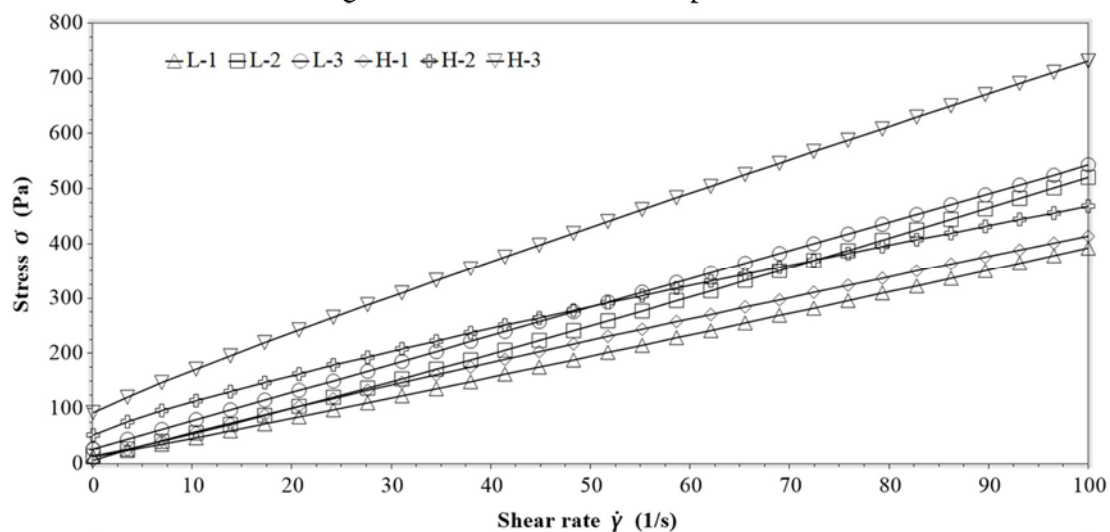
Mixture	Brick powder (g)	Sodium silicate, $M_s = 1.0$ (g)	H ₂ O (ml)	Lukosan S 1% (ml)
L-1	100	65.5	–	2
L-2	100	57.3	5	2
L-3	100	49.1	10	2
H-1	100	65.5	–	2
H-2	100	57.3	5	2
H-3	100	49.1	10	2

4. Results and discussion

4.1. Rheological properties

4.1.1. Flow properties. Figure 2 illustrates the descending branches of flow curves of the fresh brick powder suspensions. The shape of curves indicates nearly Newtonian behaviour of brick pastes as confirmed by the flow index values, n , varying around 1 (Figure 3). It is apparent that the yield stress of pastes increase with decreasing dosage of sodium silicate solution (Figure 3). This is expected because the lower silicate solution content in paste limits the solubility of the precursor particles and the paste is less fluid. Likewise, low yield stresses has been observed at metakaolin based geopolymers [10, 11, 14]. Lower value of the yield stress for sample H-1 (versus L-1) may be caused by lower content of calcium in this brick powder. The presence of divalent cations can result in attractive ion correlation forces (as in C-S-H particles). Brick powder H responded to the decreasing amount of activator by a significant increase in the yield stress.

The consistency coefficient (which can be compared to a plastic viscosity of the material) increased with decreasing dose of alkali activator (Figure 3) which was caused by decrescent amount of mixing solution. H powder exhibited a higher consistency coefficient. This is due to the different content of the amorphous phase in the precursors, where mixtures with higher crystalline content are more reluctant to flow. The consistency coefficients of the pastes were higher than of cement pastes or metakaolin-based geopolymer pastes despite of bigger particle sizes of brick powders than of common cement or metakaolin [14, 15]. This is due to the viscosity of sodium silicate solution which is 10 to 100 times higher than the viscosity of water. As a consequence the viscosity of these fresh pastes is expected to be 10 to 100 times higher than of standard cement pastes.

**Figure 2.** Descending branches of flow curves of brick powder suspensions.

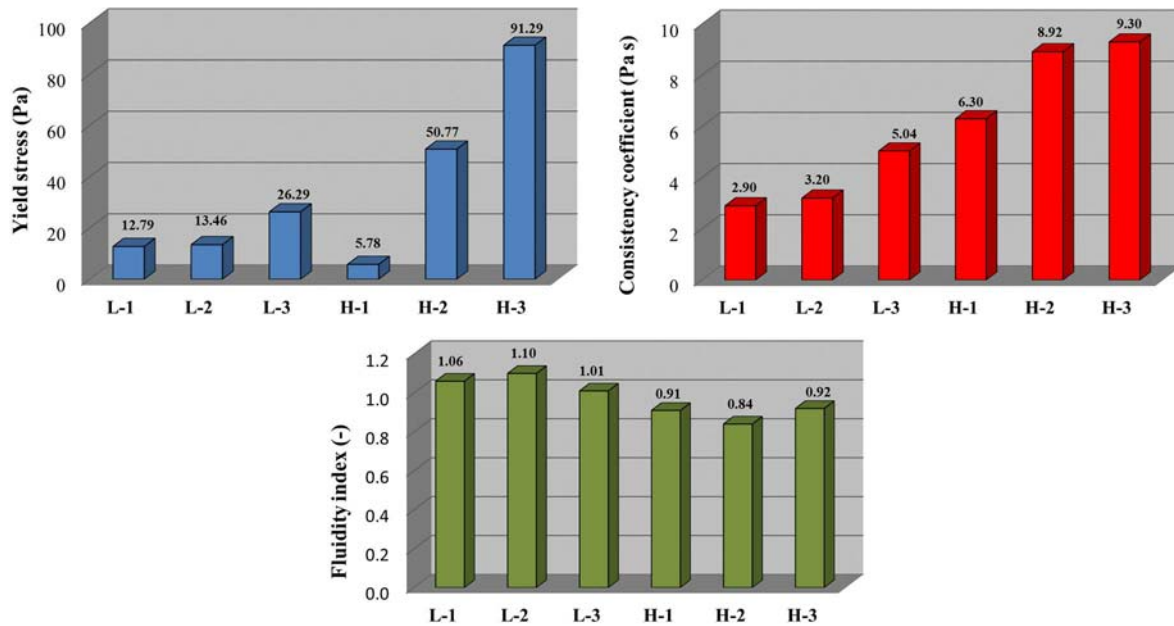


Figure 3. Comparison of yield stresses (τ_0), consistency coefficients (k) and fluidity indexes (n) of brick powder suspensions.

4.1.2. Viscoelastic properties. Figure 4 shows the time evolution of the viscoelastic parameters (G' and G'') during a small amplitude (0.01%) oscillation test. Geopolymer gel formation was monitored by a crossover point $G' = G''$ (gel point). Before the gel point, samples show the behaviour of a liquid with $G'' > G'$, after the gel point, the samples show gel-like or solid behaviour with the existence of interactions between constituents [13]. The results showed that the geopolymer gel formation is faster in the brick powder paste with higher content of calcium and amorphous phase (L). It confirmed the enhanced reactivity of amorphous precursors for alkali activated materials. Decreasing amount of alkali activator in the mixtures led to an acceleration of the gel formation probably due to stronger interactions between particles in more concentrated aluminosilicate solution. L-3 mixture exhibited the most rigid structure.

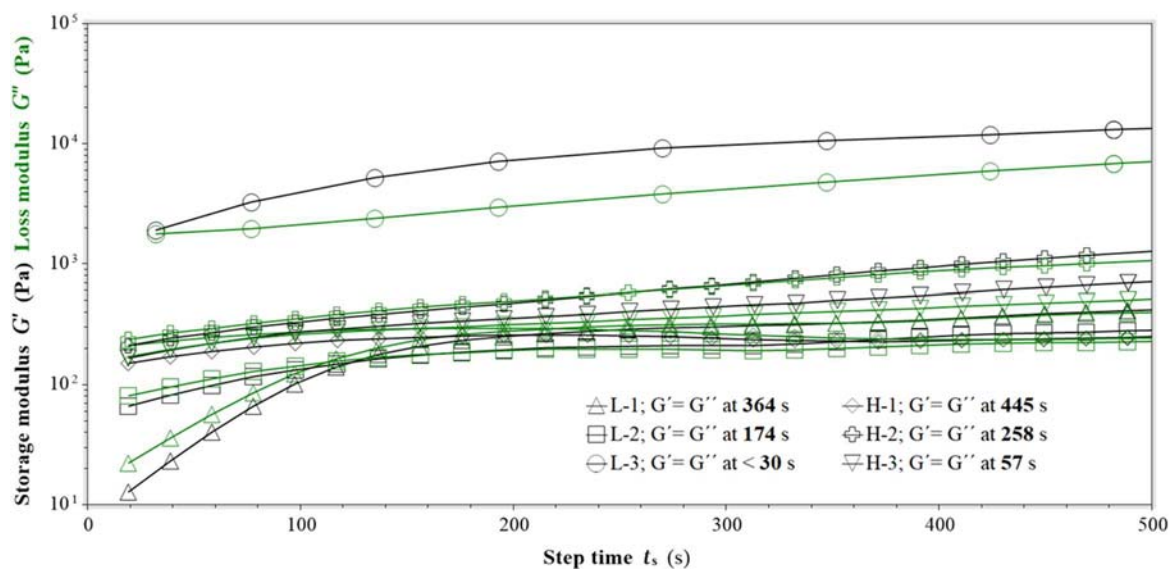


Figure 4. Time evolution of dynamic moduli for the brick powder based pastes.

The results of frequency sweep tests proved the connection between the amount of alkali activator and the stiffness of brick pastes; the ratio between dissipation and elasticity increased (growing G^*) with decreasing amount of alkali activator and the pastes became more rigid and more viscous (Figure 5). Since the $\tan(\delta)$ of the pastes vary around 1, the brick paste showed the viscoelastic behaviour with almost the same ratio of the viscous and elastic portions. This does not apply to mixtures L-3 and H-3 which exhibited lower $\tan(\delta)$ values of about 0.5, indicating a higher proportion of the elastic component (G'), suggesting that these pastes have a stronger structure more resistant to external interference.

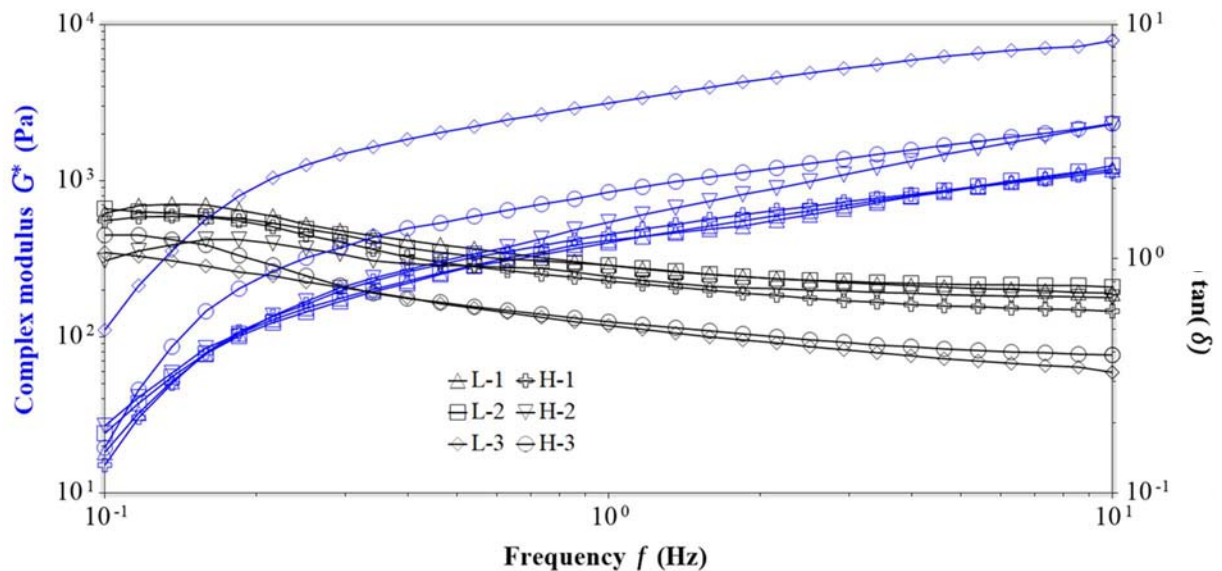


Figure 5. Evolution of G^* and $\tan(\delta)$ as a function of frequency for the brick pastes.

4.2. Mechanical properties

Obviously, because of binder hardening, the mechanical strengths of pastes increased with curing time (Figure 6). It can be stated that the pastes made of the brick powder with higher content of calcium and amorphous phase (L) have better strength properties than the other pastes. This again confirms the advantage of using a precursor with high content of amorphous phase for the production of alkali-activated materials. H mixtures showed a gradual increase in strengths with decreasing amount of alkali activator. A different trend can be observed for L mixtures where the best strength results were obtained for L-2 sample. When comparing the strength characteristics of the pastes in a fresh and hardened state, it can be concluded that already in the fresh state, the L brick powder has better properties than the H brick powder. However, the sample with the strongest structure in the fresh state, did not achieve the highest strength after hardening.

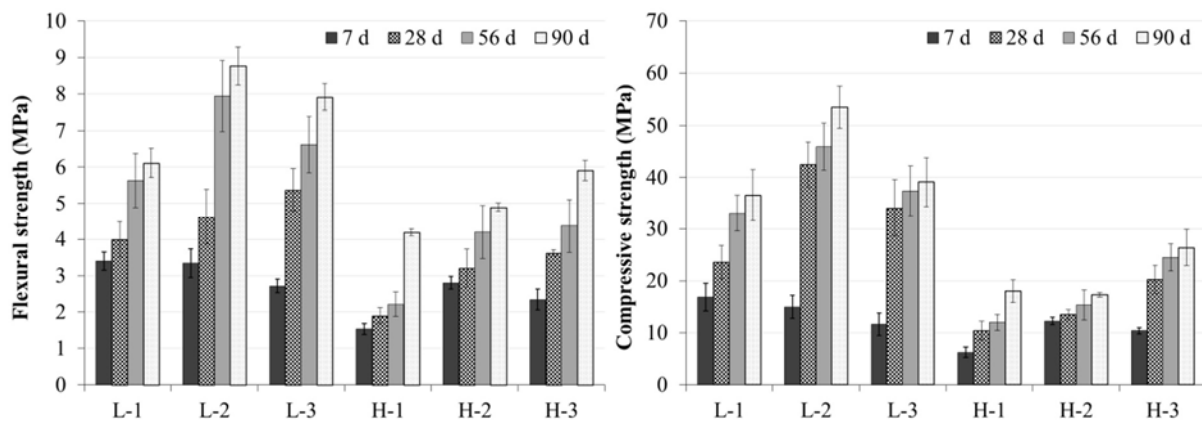


Figure 6. Flexural and compressive strengths of brick powder based pastes.

5. Conclusions

This paper has focused on the effect of alkaline activator quantity on the rheological and mechanical properties of brick pastes prepared from two types of brick powder differing by content of Ca and amorphous phase. The main conclusions are follow:

- Brick powder based pastes prepared in this study are nearly Newtonian fluids with relatively low yield stresses and high viscosities that increase with decreasing dosage of sodium silicate solution.
- Increased values of yield stress and consistency coefficient of H brick powder are caused by higher content of crystalline phase in this precursor, since mixtures with higher crystalline content are more reluctant to flow.
- The geopolymer gel formation in brick paste is faster if higher content of calcium and amorphous phase is present in a brick powder. Decreasing amount of alkali activator in the mixtures leads to an acceleration of the gel formation.
- The pastes made of the brick powder with higher content of calcium and amorphous phase (L) have better strength properties than the H pastes. This confirms the advantage of using brick powder precursors with high content of calcium and amorphous phase for the production of alkali-activated materials.

Acknowledgments

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