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RHEOLOGY PROPERTIES OF METAKAOLIN ACTIVATED BY LITHIUM WATER GLASS

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Abstract. Alkali-activated materials play an important role in the current industry, because they offer alternative way towards low carbon technologies. However, their crucial utilization still lays in the field of composites resistant to high temperatures, especially in the heavy machinery and chemical industry. The current paper documents rheological properties of set of pastes on the basis of Czech metakaolin as precursor, which was activated by lithium water glass exhibiting various silicate modulus. The rheological properties were studied in terms of standard flow test and optimized composition exhibiting similar workability were subsequently investigated by using rotary rheometer. The rheology was monitored in time to document performed changes during the geopolymerization. The attained flow curves were fitted by Herschel-Bulkley model and basic rheological characteristics of optimized pastes were derived. The research confirmed prolonged workability of the studied paste activated by lithium water glass and better. The pastes activated by lithium water glass of lower silicate modulus exhibited lower values of yield stress.

Keywords: Lithium water glass, metakaolin, rheology, flow curves.

1. Introduction

Last decades the scientific activity is focused on the development and improvement of alternative binding systems exhibiting lower carbon dioxide emissions in comparison with traditional systems suchlime and Portland cement (PC). The activation of aluminosilicates is a promising way offer, so called geopolymers [1]. These materials are well known for more then a century. The first systematic research was published by Kuhl in 1930, however the patent by the similar author was filled at 1908. These initial works were focused on the utilization of blast furnace slag as a precursor and sodium hydroxide as an activator [2]. With increasing interest in alkali-activated materials (AAM) in last decades were intensively studied the other possibilities of these binders with the aim to replace PC in a production of buildings and structures. AAM could be formulated on the basis of different precursors [3, 4]. Promising results were attained with blast furnace slag, however in relation to gradual reduction of heavy metallurgy in regions such a West and Middle Europe. Hence, the attention is currently focused on calcined clays, which seem to perspective materials for such an application, because of their wide availability [5, 6]. Besides number of successful utilizations of AAM in building structures, the main importance lies in the field of high temperature application, because of superior resistance of such formulated composites [7–9].

The final properties are logically influenced by the mineral composition of used precursor. Thus, the properties of hardened composite could be highly different. In addition, the microstructure and entire performance of AAM based composite is also determined by the used activator. Aluminosilicate could be successfully activated by the water glass, usually potassium or sodium), or by sodium hydroxide or a combination of both [10]. The relatively fast process of polymerization leads to the gradual loss of the workability, what limits standard industrial application of AMM. In technical praxis is preferable applied potassium-based activator, due to prolonged workability time [11]. Incorporation of lithium ions is relatively rare, because of higher price of such materials, however intensive research was performed in the field of utilization of lithium slag, however the lower reactivity was monitored [12]. The present paper is focused on the experimental study dealing with monitoring of rheology properties of pastes on the basis of metakaolin activated by the lithium water glass, which was selected due to its better performance in terms workability of AAMs.

2. Experimental program

The experimental program was focused on the systematic description of rheology properties of the set of alkali-activated pastes. Czech metakaolin was used as a precursor and lithium water glass of different modulus served as an activator. The used precursor is metakaolin Mefisto K05 (ČLUZ a.s., Nové Strašecí, Czech Republic) originating by calcination of kaolinitic clay at approximately 750 °C. Its chemical composition is introduced in Table 1. The previous

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	SO_3
49.1	47.3	0.9	0.2	0.1	0.5	0.0	1.6	0.1

Table 1. Chemical composition of used metakaolin.

Water glass	Li 2.7–5.0	Li 3.0–3.5	Li 3.7–4.2	Li 3.7–5.0
Middle value	3.85	3.25	3.95	4.35

Table 2. Used lithium water glass.

research confirmed, that amorphous content is just about 74.5 % [13] and poccolanic activity in terms of Chapelle test 1 967 mg/$Ca(OH)_2$ per a gram of pozzolan [14]. The used lithium water glass, produced by Vodní sklo, Ltd., exhibited different silicate modulus, which are introduced in Table 2.

The pastes of AAM were prepared by mixing of metakaolin and the activator by 1:1 for 2 minutes in a laboratory mixer. Subsequently was determined spreading in accordance with ČSN EN 1015-3 using traditional Hägermann flow table, Figure 1a. The resulting spreading represents diameter of the pastes after vibration, Figure 1b. The consistency was monitored after mixing, 15, 60 and 150 minutes to assess the stability in time. The second phase of the laboratory campaign was focused on the optimization of the dosage of an activator to achieve similar consistency. The targeted spreading was approximately 310 mm.

The pastes of optimized composition were investigated using rotary rheometer to attain basic characteristic describing properties of this type of fluid. The instrument produced by Anton Paar with ball apparatus was used for this propose, Figure 2. The flow characteristics were measured using dynamic procedure 5 and 60 minutes after mixing. The acquired flow curves were then plotted by Herschel-Bulkley model (1),

$$\tau = \tau_0 + K\gamma^n, \quad (1)$$

where τ is a shear stress (Pa), τ_0 is an yield stress (Pa), K is a consistency index (-), γ is shear rate (s^{-1}) and n is the flow index (-). This methodology was successfully applied in previous research [15].

3. Results and discussion

The performed experimental program was focused on the detailed description of rheology properties of AAM on the basis of metakaolin activated by lithium water glass. The importance of rheology properties has relation to technical praxis, where the sudden loss of workability limit further utilization of AMM in the industry. The results of the first phase of the project are shown in Figure 3. It is evident, that changing silicate modulus of used lithium water glass influenced the final flow of studied pastes. Specific feature of fresh AAM is sudden loss of workability, when sodium or potassium activator is applied [10, 11]. However, thanks to used lithium water glass slight gradual improvement of the workability was monitored within 150 minutes.

(a).

(b).

Figure 1. (A) Flow table (B) final spreading.

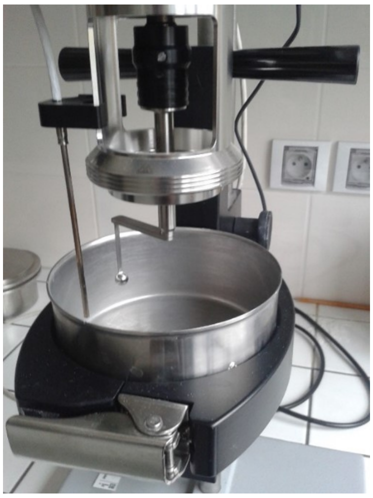


Figure 2. Used configuration of used rotary rheometer.

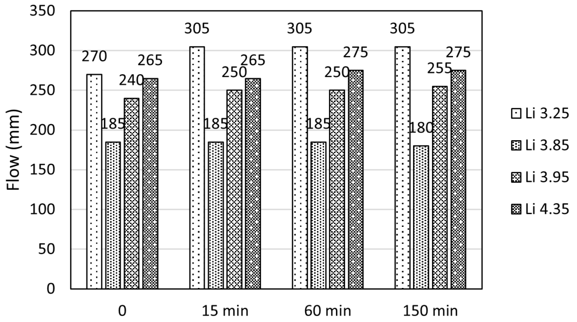
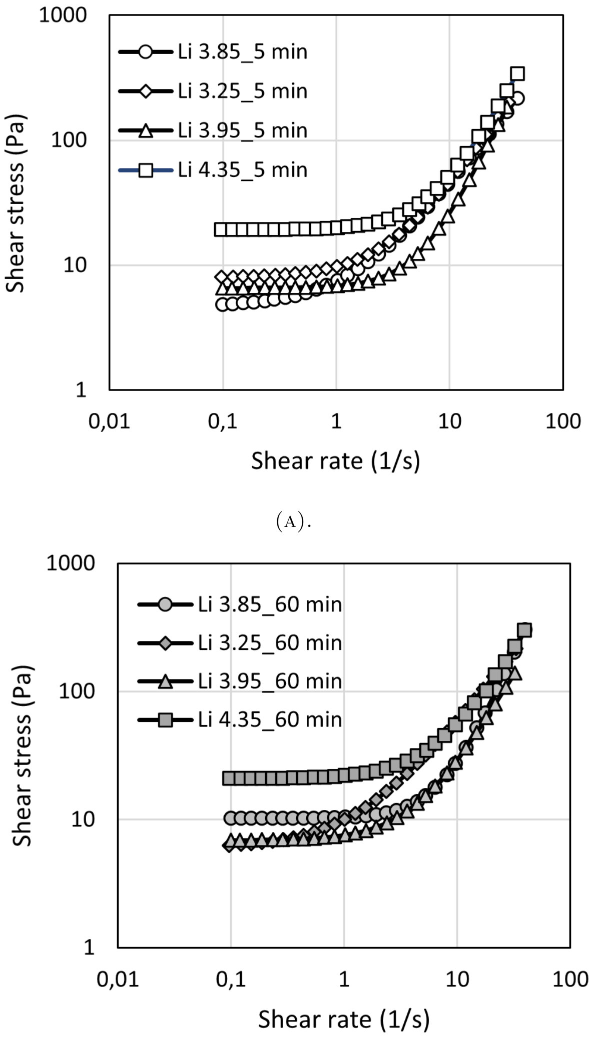


Figure 3. Flow of AAM pastes in time.



(a).

(b).

Figure 4. (A) Flow curve of pastes after 5 minutes (B) Flow curve of pastes after 60 minutes.

The second phase of the experimental program was dealt with the optimization of the dosage of the activator. With respect to the results of initial testing, different amount of activator is necessary to achieve given value of flow. Hence, the further set of paste was prepared in order to reach flow approximately 310 mm. The detailed results in Table 3 document reduction of the activator in such designed experiment. The derived flow characteristics from Equation 1 on the basis of rheology measurement are introduced in Table 4.

The derived values of yield stress (Pa) well document differences between single studied pastes and used activators. The value of this property describes the minimal stress needed to flow. From the plotted curves illustrated in Figure 4a and Figure 4b is evident, that the paste with the highest applied silicate modulus exhibited the highest value of the yield stress. That was monitored for both time intervals. It can be assumed, that remaining mixtures exhibited negligible differences. From this point of view lower silicate modulus seems to more suitable for application requiring better workability of the mixture.

Similar findings were reported by Vance et al. [16], who described the mechanism of improved workability in case of AAM pastes with low values of silicate modulus. It was concluded, that changing charge on the surface of used precursor and ionic species in the activator. Similar mechanism was reported by Rovnaník et al. [17], who studied rheology properties of AAM pastes on the basis of metakaolin activated by sodium water glass. They mentioned higher reactivity of metakaolin due to its mainly amorphous character, thus immediate gelation accompanied by the increase of yield stress in time. However, the increase of yield stress in this research program studying effect lithium water glass has not been monitored. Liang et al. [18] studied combination of metakaolin and lithium slag as precursors activated by sodium water glass and sodium hydroxide. The yield stress and workability ware increasing with increased incorporation of lithium slag, however the setting time was decreased. This behavior is caused by the increase of Si/Al ratio of used precursor, which proportionally accelerate setting of the paste. The initial better workability in mixtures incorporating lithium slag was influenced by its lower water adsorption.

Designation	**Used activator**	**Metakaolin [g]**	**Water glass [g]**	**Flow [mm]**	**Dosage change [%]**
Li 3.25	Li 3.0–3.5	500	500	330	0
Li 3.85	Li 2.7–5.0	500	615	305	23
Li 3.95	Li 3.7–4.2	500	600	310	20
Li 4.35	Li 4.5–5.0	500	500	300	0

TABLE 3. Flow of AAM pastes with optimized dosage of the activator.

	5 minutes				**60 minutes**			
	K [-]	n [-]	τ_0 [Pa]	R^2	K [-]	n [-]	τ_0 [Pa]	R^2
Li 3.25	1.74	1.34	7.98	0.98	3.98	1.13	6.01	0.97
Li 3.85	2.78	1.18	4.69	0.98	0.19	2.00	10.25	0.97
Li 3.95	0.26	1.88	6.59	0.97	0.68	1.52	6.90	0.97
Li 4.35	0.74	1.65	19.23	0.98	3.39	1.09	15.40	0.98

TABLE 4. Flow characteristics of studied pastes.

4. CONCLUSIONS

The performed experimental program was focused on the rheology properties of AAM paste activated by lithium water glass, which introduce interesting possibility for enhancement of suitable technological properties. The study was focused on the determination of basic rheological properties such as flow and yield stress, of which values were plotted using Herschel-Bulkley model. The attained results declared increased workability of studied pastes in time, thus the sudden loss of workability has not been reported within 150 minutes. This feature differs from other research works using traditional ways of metakaolin activation like sodium/potassium water glass. From the rheology characteristics point of view, the lower silicate modulus led to the more promising values of yield stress, however the differences between single mixtures were negligible. The lower silicate modulus seems to more suitable also due to expected higher mechanical properties of hardened composites.

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