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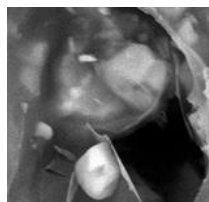
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The effect of milled basalt fibers addition on rheological and mechanical properties of alkali activated binder

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The effect of milled basalt fibers addition on rheological and mechanical properties of alkali activated binder

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Abstract. In this study, the effects of milled basalt fibers addition on rheological behaviour of alkali activated geopolymer binder were determined. This binder was synthesized by alkaline activation of calcined clay stone powder and milled blast furnace slag by potassium silicate solution. Milled basalt fibers as filler were used in range of 0-20 wt% addition to powder part of binder and the effects on rheological and mechanical properties were evaluated. The rheological properties were determined in accordance to flow properties by measurements on Ford viscosity cup and by strain controlled small amplitude oscillatory rheometry measurements of hardening process. The final properties were documented by measurements of flexural strength after 28 days and by scanning electron microscopy. The results indicate that milled basalt fibers addition has effect on flow properties of binder. The flow time increased three times and the complex viscosity is also coherently increased. The reinforcement led to two times higher values of flexural strength and the best figure of merit lied in the range of 1-5 wt% addition of milled basalt fibers.

1. Introduction

Inorganic polymer cements can be synthesized by alkali-activation of a variety of materials including thermally activated clays and coal fly ashes to produce material with mechanical and thermal properties suitable for wide range of industrial applications [1, 2]. Davidovits entitled this type of materials as “geopolymers” because of polymer like chemical structure and introduced pioneering work on alkali-activated binders based on calcined clays [3]. Geopolymers are products of chemical reaction between aluminosilicate material and liquid alkaline environment where chemical cleavage of the Si-O and Al-O bonds in parent material leads to saturation of liquid solution and subsequent polycondensation of amorphous aluminosilicate matrix [4]. In some cases slag is used as an additive for improvement of setting time and mechanical strength [5, 6].

However, by ceramic-like nature have brittle fracture character [7]. Using of fibers can change the character of fracture into quasi-brittle and prevent the crack propagation in material. Wide range of fibers is used for this purpose from organic natural and synthetic materials as PVC, PP, PVA and cellulose [7-9], inorganic carbon-based materials and mineral materials as basalt, glass, zirconia and others [10-12]. Fiber reinforcement is mainly used in form of short fibers for casting processes or as woven and non-woven composites prepared by impregnation.

This experimental research focuses on rheological and mechanical properties of geopolymeric matrix reinforced by milled basalt fibers. The binder comprises of calcined clay stone powder and milled blast furnace slag. Milled basalt fibers were applied in range of additional 0-20 wt% related to powder part and



the effect on flow properties and final mechanical strength was evaluated. Furthermore, the hardening process of individual compositions was evaluated by the small amplitude oscillatory rheometry measurements [13, 14].

2. Materials

The mixture of calcined clay stone powder and milled blast furnace slag (CCS) was used as binder. This binder was activated by potassium silicate solution with silicate module 1.61. These materials were supplied by České lupkové závody a. s, Czech Republic and their chemical composition is presented in Table 1. The basalt fibers were provided by Kamenny Vek, Russian Federation. The diameter of fibers was 14 μm and fibers were milled for 30 seconds in laboratory vibrational mill to final length. Figure 1 and Figure 2 represents morphology of binder and milled fibers respectively taken by scanning electron microscope (SEM). The overall granularity curves measured by laser light scattering are presented on insets in individual figures.

Table 1. Chemical composition of raw materials [mass %]

Composition	SiO ₂	Al ₂ O ₃	CaO	MgO	TiO ₂	Fe ₂ O ₃	K ₂ O	LoI	Others
CCS	46.37	28.72	15.46	3.40	2.01	0.93	0.56	1.92	0.63
Activator	17.60	-	-	-	-	-	17.13	65.27	-

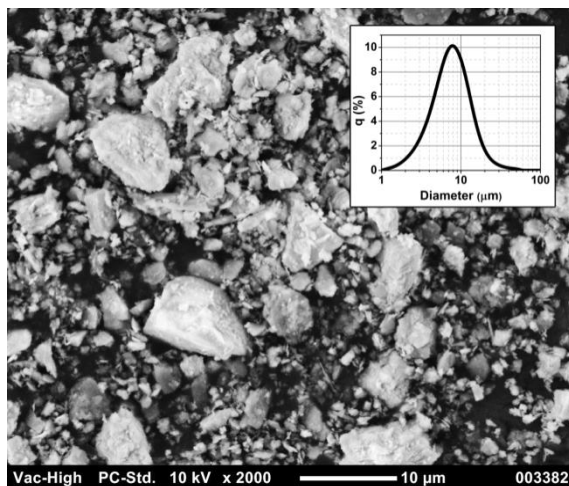


Figure 1. SEM image and distribution of binder

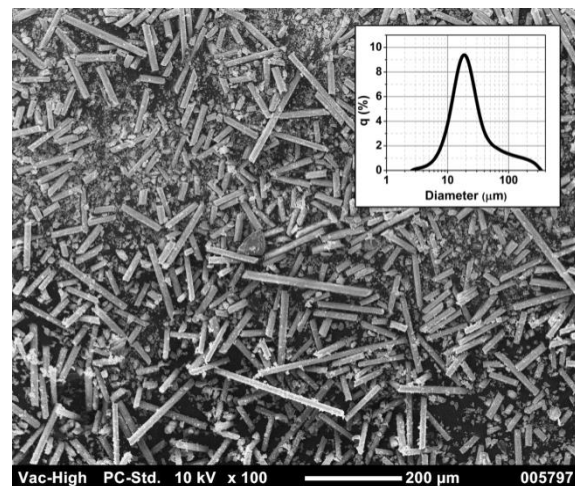


Figure 2. SEM image and distribution of milled basalt fibers

3. Procedures and results

3.1 Preparation of pastes

Binder pastes were prepared by mixing of 100 weight parts of binder powder together with 80 weight parts of activator solution. Subsequently, different amount of fibers in the range of 0 – 20 wt% was added into the paste and mixed together for 5 minutes in laboratory vacuum mixer. Pastes with different content of fibers were casted into moulds with dimensions of 120x20x20 mm. After 2 days were specimens unmounted and aged for 28 days in polypropylene bags.

3.2 Rheological measurements

In this study the complex viscosity of prepared pastes in the beginning of hardening process was determined by time-resolved oscillatory rheometric measurements with small amplitude on a strain controlled rheometer TA Instruments Ares G2 in plane-plate geometry of 40 mm in diameter with frequency of 1 rad/s and strain controlled amplitude of 0.01 %. Measurements were performed after

15 minutes from beginning of mixing. The effect of fibers addition can be clearly seen in Figure 3 where individual curves represent increase of complex viscosity in early age of pastes hardening.

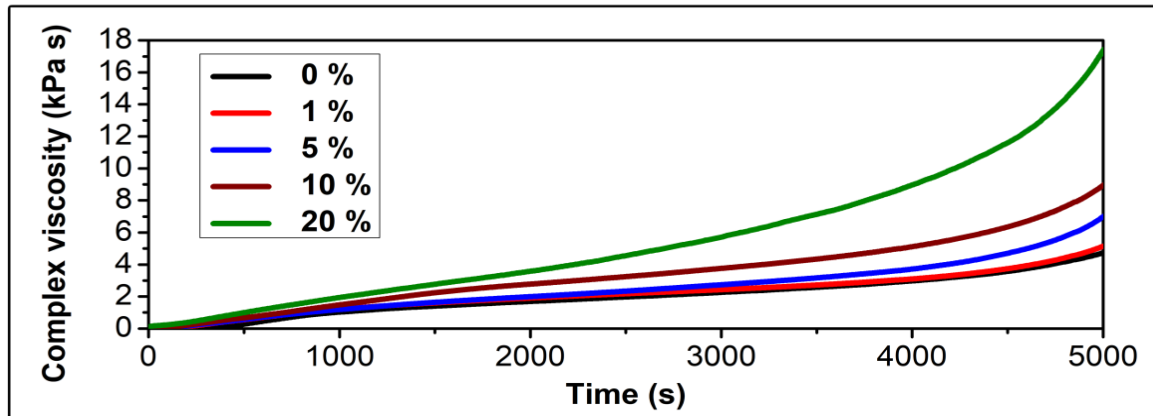


Figure 3. Complex viscosity at beginning of hardening process for individual fiber contents

As a quantification of workability the flow properties were determined on fresh pastes immediately after mixing by measurements on Ford viscosity cup with diameter of 6 mm. The results can be seen in Figure 4 described as time required to flow. It can be compared with results of complex viscosity measurements and in both cases the increasing content of fibers increases viscosity. The flow time is increased approximately three times over the investigated range. This trend is the same in measurements of complex viscosity, but this effect is not so noticeable because of different conditions of measurement when the small amplitude and frequency are applied. However, it can be concluded that even at highest investigated filling is retained good workability of paste.

3.3 Mechanical properties and microstructure

The flexural strengths of individual compositions were measured after 28 days on 120x20x20 mm samples by 3 point bending test with support span of 50 mm. Results can be seen in Figure 5 and at 20 wt% the flexural strength reaches twice as high value of unfilled binder. However, the flexural strength increasing with lower slope than time to flow through viscosity cup and thus the best figure of merit can be found in between of 1 and 5 wt% composition. The microstructure of fracture surfaces was evaluated by SEM and pure hardened paste without addition of fibers can be seen in Figure 6.

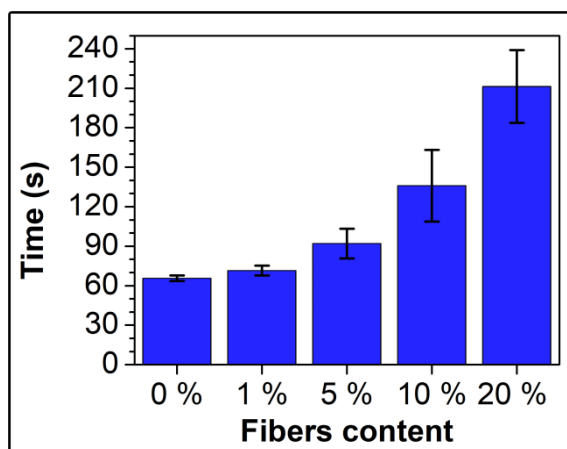


Figure 4. Time to flow through 6 mm Ford viscosity cup

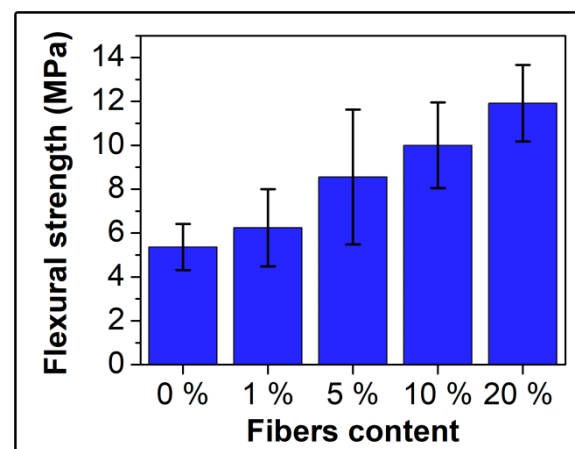


Figure 5. Flexural strength of composites after 28 days

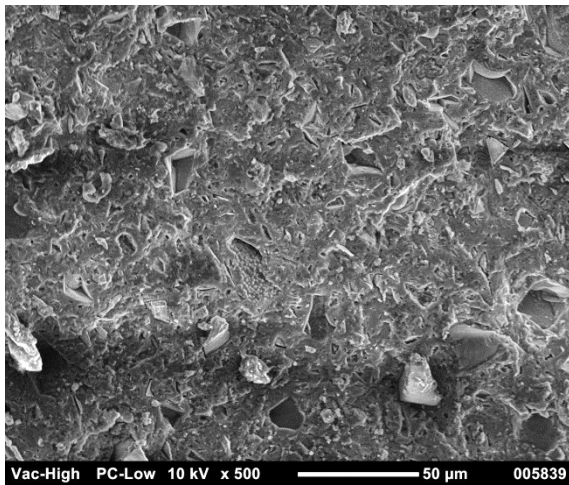


Figure 6. SEM image of hardened paste without fibers addition.



Figure 7. SEM image of hardened paste with 20 wt% of basalt fibers.

The brittle fracture surface can be seen with irregular holes after unreacted slag particles. The composition with 20 wt% of basalt fibers is presented in Figure 7 with marked uncovered fibers and imprints on fracture surface. Thus it can be concluded that reinforcing effect is predominantly produced by the pull out mechanism.

4. Conclusions

It can be summarized that with increasing content of basalt fibers in geopolymer paste are rising both the flexural strength and viscosity. At 20 wt% addition of basalt fibers rise the flexural strength 2 times and the flow time 3 times in contrast to values of unfilled paste and the complex viscosity follows the same trend. However, at highest investigated filling is still retained good workability of binder. From fracture progress can be concluded that the reinforcing effect of milled basalt fibers in binder is predominantly produced by the pull out mechanism.

Acknowledgments

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