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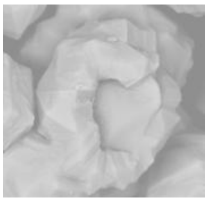

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The effect of short carbon fibers on rheological behaviour and mechanical properties of metakaolin-slag geopolymer binder

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Abstract. This study investigates the effects of short carbon fibers addition on rheological behaviour of geopolymer binder and final mechanical strength of prepared composite. The pure geopolymer binder and variously carbon fiber reinforced composites were tested. The pure geopolymer binder was synthesized by alkalination of calcined claystone powder and milled blast furnace slag by potassium silicate solution. The binders reinforced by carbon fibers were prepared under the same conditions. Mixtures were accordingly reinforced by 20 wt% of short and milled fibers, respectively. The effect of fibers length (3 mm and ≤ 0.5 mm) was studied with regard to rheological behaviour of prepared mixtures and mechanical properties of final composites. The rheological properties were determined in accordance to flow properties by rotational rheometry. The final properties were determined by measurements of flexural strength after 3, 28 and 280 days and by means of optical and scanning electron microscopy. The results indicate that milled carbon fibers had a slight effect on flow properties compared to pure binder. In contrast, 3 mm long carbon fibers significantly increased viscosity and noticeably decreased workability by interlocking mechanism of individual fibers. Moreover, it was proved that addition of milled fibers had a low reinforcement effect on composite samples and led only to slight improvement of stress-strain behaviour. On the contrary, the reinforcement by 3 mm carbon fibers improved the stress-strain properties and significantly increase ultimate flexural strength (approximately 5 times) compared to pure geopolymer binder in all studied time intervals.

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] and produce sufficient mechanical properties [4]. 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 [5-7]. In some cases slag is used as an additive for improvement of setting time and mechanical strength [8, 9].

However, by ceramic-like nature have geopolymeric binders brittle character of fracture [10]. 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 [10-12], mineral materials as basalt, glass, zirconia and others to high performance carbon-based materials [13-16]. Fiber reinforcement of inorganic binders 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 carbon fibers of different length. The binder comprises of calcined clay stone powder and milled blast furnace slag. Carbon fibers in length 3 mm and ≤ 0.5 mm were applied in amount of 20 wt% related to powder part and the effect on flow properties and final mechanical strength was evaluated.

2. Materials

The mixture of calcined claystone powder and milled blast furnace slag (CCS) was used as a binder and the morphology can be seen in Figure 1. The slag part of binder is represented mainly by bigger particles, consisting of amorphous glassy material with minor accessory minerals merwinite and akermenite. Calcined claystone part consists almost completely of reactive metakaolin with trace amount of accessory minerals quartz and mullite. The granularity, with characteristic volumetric values d10, d50 and d90 of 0.37, 5.03 and 11.37 μm respectively, was determined by laser light scattering technique. For alkaline activation of geopolymerization reaction an aqueous solution of potassium silicate with silicate module 1.61 was used. These materials were supplied by České lupkové závody a. s, Czech Republic and their chemical composition is presented in Table 1. The reactivity of this binder was studied in details in references [16, 17].

The short carbon fibers with initial length of 3 mm and diameter of 14 μm, provided by GRM Systems s.r.o., Czech Republic, were used and marked as "sCF". The milling process of fibers was conducted in laboratory vibrational mill for 30 seconds and resulted fibers with final length below 0.5 mm were marked as "mCF". Images of used carbon fibers, before and after milling process, can be seen in Figure 2 and 3 respectively.

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

Composition	**SiO_2**	**Al_2O_3**	**CaO**	**MgO**	**TiO_2**	**Fe_2O_3**	**K_2O**	**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	-

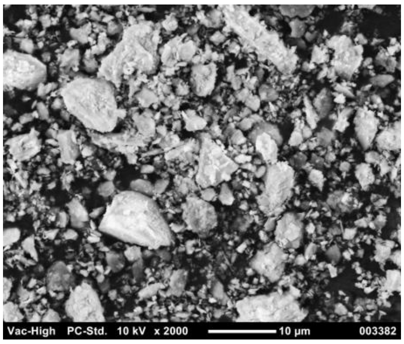

Figure 1. SEM image of CCS binder.

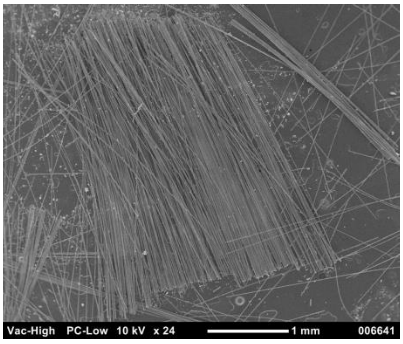

Figure 2. SEM image of short carbon fibers.

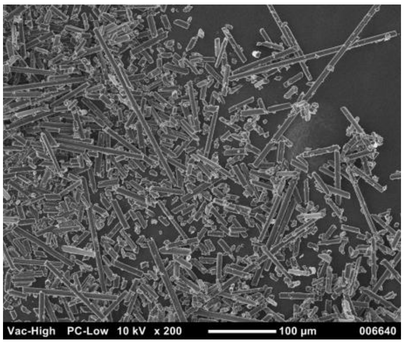

Figure 3. SEM image of carbon fibers after milling process.

3. Procedures and results

3.1. Preparation of binder and composites

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

3.2. Flow properties

The rheological properties were determined in accordance to flow properties by rotational rheometry on the rheometer TA Instruments Ares G2 in plane-plate geometry of 40 mm in diameter. Measurements were performed after 15 minutes from beginning of mixing. The difference in stress behaviour according to shear rate can be clearly seen in Figure 4 and the sCF highly increases the viscosity and reduces workability.

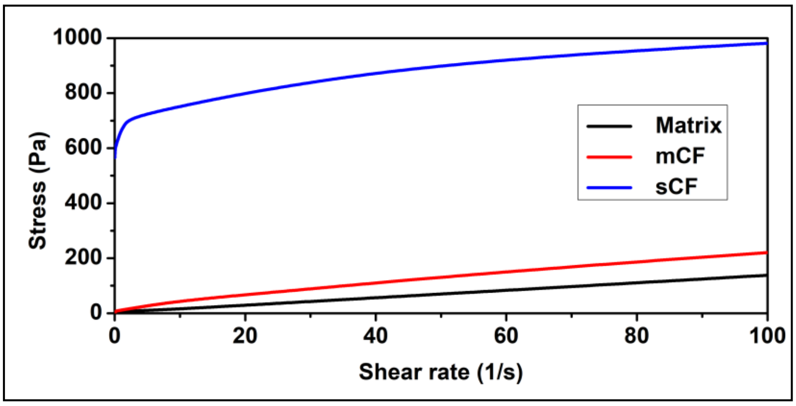

Figure 4. Stress-shear rate behavior of pure matrix and matrix with short and milled carbon fibers.

3.3. Flexural properties and microstructure

The flexural strengths of individual compositions were measured after 3, 28 and 280 days respectively on 20x20x120 mm samples. How it can be seen in Figure 5 that the effect of carbon fibers is remarkable in both cases and the flexural strength is affected by the length of fibers.

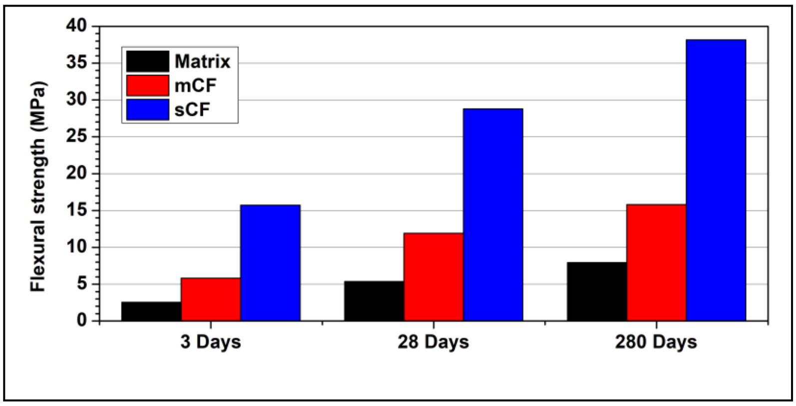

Figure 5. Flexural strength of individual compositions measured in time intervals of 3, 28 and 280 days.

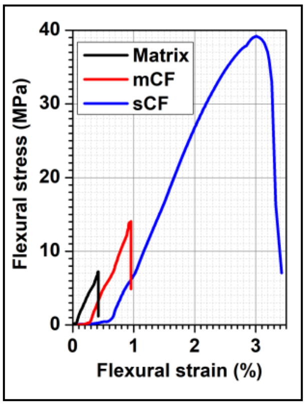

Figure 6. Stress-strain behaviour of individual compositions after 280 days.

Addition of mCF remarkably increases flexural strength in contrast to pure Matrix. From stress-strain behaviour can be concluded that in the case of mCF this effect is mainly by bridging of initial short cracks as it can be seen on stress-strain curve in Figure 6. When the ultimate flexural stress is reached the stress sharply decreases without bridging effect and in this case the mCF behaves more like the particle than fiber filler. The effect of sCF is different in stress-strain behaviour and further increase of ultimate stress can be seen. Moreover, effect of longer fibers and the reinforcement by the pull-out mechanism can be assumed from residual stress. This assumption is further supported by analysis of microstructure.

The microstructure of fracture surfaces was evaluated by scanning electron microscopy and by optical microscopy in bright field (BF). The SEM images are presented in Figures 7-9. The Figure 7 represents the structure of pure geopolymer binder. In Figure 8 the presence of carbon fibers with length of tens of micrometers can be clearly seen with residual imprints in geopolymer matrix. The observed length corresponds to stress-strain behaviour and explains the negligible bridging effect. In the case of sCF presented in Figure 9 the short carbon fibers can be seen going across the field of view without breakage what confirms the pull-out mechanism of reinforcement. From the microstructure of imprints in mCF and sCF can be concluded that the wetting of carbon fibers by this geopolymer binder is sufficient. This situation is further documented in Figures 10-12 taken by optical microscopy in BF where the contrast between light geopolymer matrix and dark carbon fibers is better and more intensive.

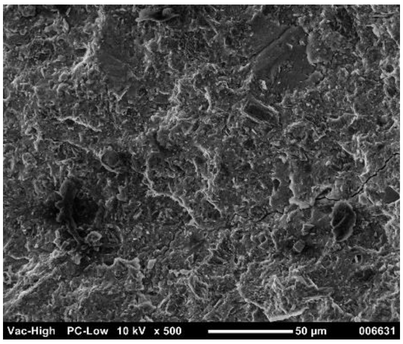

Figure 7. SEM image of pure Matrix.

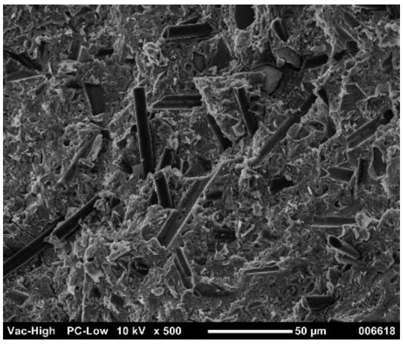

Figure 8. SEM image of matrix with mCF.

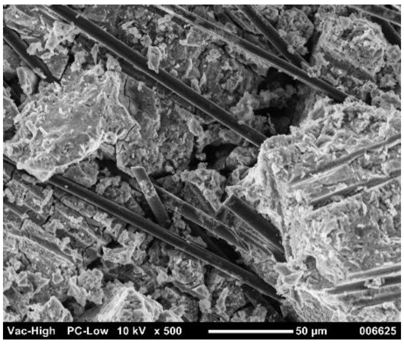

Figure 9. SEM image of matrix with sCF.

Figure 10. Image of Matrix in BF.

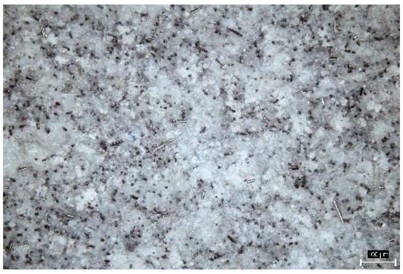

Figure 11. Image of matrix with mCF in BF.

Figure 12. Image of matrix with sCF in BF.

4. Conclusions

In this study the effect of carbon fiber addition on flow properties of prepared suspensions and stress-strain behavior was investigated. It can be concluded that the length of carbon fibers play important role in flexural strength, flow properties and workability. The addition of carbon fibers raises both the ultimate flexural strength and viscosity. Both these effects are more significant with increasing of fibers length. Addition of sCF produces good composite material with residual flexural strength by bridging effect of fibers and with very good flexural strength of 15.7, 28.8 and 38.1 MPa after 3, 28 and 280 day respectively. It is in contrast to values of 5.8, 11.9 and 15.8 MPa for mCF composite and 2.6, 5.4 and 7.2 MPa in the case of pure Matrix. On the other hand, these remarkable properties are on the cost of decreased fluidity and workability. From this study can be moreover concluded that the wetting of carbon fibers by studied geopolymer binder is good and sufficient and this effect can be assigned to surface properties of used potassium silicate solution.

Acknowledgments

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