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Mechanical properties of non-woven glass fiber geopolymer composites

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Abstract. This experimental research focuses on mechanical properties of non-woven glass fabric composites bound by geopolymeric matrix. This study investigates the effect of different matrix composition and amount of granular filler on the mechanical properties of final composites. Matrix was selected as a metakaolin based geopolymer hardened by different amount of potassium silicate activator. The ceramic granular filler was added into the matrix for investigation of its impact on mechanical properties and workability. Prepared pastes were incorporated into the non-woven fabrics by hand roller and final composites were stacked layer by layer to final thickness. The early age hardening of prepared pastes were monitored by small amplitude dynamic rheology approach and after 28 days of hardening the mechanical properties were examined. The electron microscopy was used for detail description of microstructural properties. The imaging methods revealed good wettability of glass fibers by geopolymeric matrix and results of mechanical properties indicate usability of these materials for constructional applications.

Introduction 1.

Alkali-activated aluminosilicate cements were discovered by Purdon and Glukhovsky already in 1940s [1] and are developed up to day. 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 [2]. 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 [3]. It is known that geopolymers can have high mechanical strength [4] and are inflammablebut by ceramic-like nature have brittle fracture character [5]. Using of fibers can change it into ductile one 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 [5-7], inorganic carbon-based materials and mineral materials as basalt, glass, zirconia and others [8-10]. Fiber reinforcement is mainly used in form of short fibers for casting processes or as woven and non-woven composites prepared by impregnation.

This study focuses on mechanical properties of composites prepared by infiltration of geopolymeric binder into the non-woven glass fabric. The effect of different matrix composition and amount of granular filler on the mechanical properties of final composites are evaluated and documented by scanning electron microscopy. Furthermore, the hardening process of individual binder compositions was evaluated by the small amplitude oscillatory rheometry measurements [11, 12].

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2. Materials

In this study, a calcined shale (CS) as a metakaolin source and calcined chamotte with granularity 0-0.1 mmas ceramic filler were used. These materials were supplied by Ceske lupkove zavody a.s, Nove Straseci, Czech Republic and by Kotouc Stramberk a. s. in the case of milled blast furnace slag. The chemical compositions were determined by the XRF technique and are listed in Table 1.

 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
CS	53.02	39.9	0.11	0.12	2.33	1.22	0.58	2.05	0.67
Activator	17.60	-	-	-	-	-	17.13	65.27	-

Granularity CS and filler were determined by static light scattering technique and results are graphically presented in figure 1 b). For alkaline activation an aqueous solution of potassium silicate with silicate module 1.61 and content of water soluble solids 34.73 % was used.



Figure 1. SEM images of calcined shale (a), particle size distribution (b) – black (CS) and red (filler) and SEM image of filler (c)

3. Procedures and results

3.1. Preparation of composites

In this study, the experimental approach was as follows. The non-woven glass fabric how it can be seen in figure 2 a) was incorporated by geopolymeric matrix with different composition in the range 100:70 - 100:100 wt.% of CS to activator solution respectively. Moreover, matrix 100:100 was filled by ceramic filler in range 20 - 100 wt.% in relation to CS for investigation of its impact on mechanical properties. Matrices were prepared by mixing of constituents in vacuum mixer for 5 minutes and incorporated into non-woven sheets with thickness of 4 mm by hand roller as can be seen in figure 2 a). The microstructure of non-woven fabrics is presented in figure 2 b). Five sheets were stacked layer by layer on each other for final specimen thickness and aged for 28 days in polypropylene bags.

3.2. Rheological measurements

Changes in complex viscosity were measured by small amplitude oscillatory rheometry. The measurements on unfilled pastes were performed in plain-plate geometry on an Ares G2 rheometer from TA Instruments with strain controlled to 0.01 %. Individual pastes were prepared by vacuum mixing and measured after 15 minutes at 30 °C isotherm.



Figure 2. Image of hand roller and non-woven fabric (a), SEM of non-woven glass fabric (b), tested specimens (c)

The changes of complex viscosity presented in figure 3 describe hardening rates of individual matrices and it can be deduced that increasing amount of activator leads to reduction of hardening rate by increasing liquid to solid ratio and delaying of saturationand polycondensation, but also to decreasing of matrix viscosity. The composition 100:100 was selected for filling experiment, based on obtained data where the initial viscosity reduction of two orders of magnitude was achieved in comparison with matrix composition 100:70.



Figure 3. Complex viscosity of individual matrices

3.3. Measurements of mechanical properties

For flexural strength investigation, prepared non-woven sheets were divided by diamond saw to strips with dimensions of 100 x 10 mm which can be seen in figure 2 c) and were subsequently tested. Figure 4 summarizes flexural strengths of individual compositions measured after 28 days. How it can be seen the composition of pure matrix influences final strength and the best performance was achieved with composition 100:90:0. It can be attributed to sufficient hardening reaction of matrix and lower water content. In the case of matrices 100:100 increasing amount of filler improves the mechanical strength. However, the high viscosity of filled matrix at composition 100:100:100 prevents sufficient infiltration of matrix into the fabrics structure.

This effect is documented by scanning electron microscopy. In figure 5 a) the matrix 100:90 is fully infiltrated into the fabrics structure but in the case of matrices 100:100:80 and 100:100:100 the material stay also in between of individual sheets as can be seen in figure 5 b) and c) respectively. In last case this effect is highly significant and leads to decrease of overall flexural strength.







Figure 5. SEM micrographs of composites 100:90 (a), 100:100:80 (b) and 100:100:100 (c)

4. Conclusions

It can be concluded that in this study the best composition of matrix is 100:90 wt.% of calcined shale to activator solution. In the case of 100:100 matrixes the increasing amount of filler retains mechanical strength up to 80wt% of filler. Further increasing of filler amount leads to a considerable decrease in flexural strength. This effect is caused by remaining matrix in between of individual fabrics which is caused by increase of matrix viscosity. It can be described as main limitation of used impregnation process when high viscosity matrix cannot be completely expelled from stacked composite interspaces.

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