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Effect of polyethylene glycol addition on metakaolin-based geopolymer

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Abstract

Polyethylene glycol is a non-toxic water soluble polymer often used in many industrial applications. The aim of this paper is to study the effect of polyethylene glycol with relative molecular weight ranging between 400–20000 on the properties of geopolymer mortars composed of metakaolin and sodium silicate. Polyethylene glycol was added in the amount of 0.5 to 10% by mass of metakaolin. After 28 days of curing at ambient conditions, different tests were carried out: physico-chemical (density, porosity), mechanical (flexural and compressive strength) and structural (SEM). The results showed that a maximum compressive strength of 23.9 MPa and a maximum flexural strength of 2.9 MPa were achieved by adding 10% PEG 400.

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Keywords: Metakaolin; geopolymer; polyethylene glycol; mechanical properties; microstructure

1. Introduction

Geoplymers are novel materials, which have been rapidly developed during the last decades due to ecological reasons, environmental impact and durability [1]. Geopolymer can be synthesized by mixing aluminosilicate reactive materials with CaO component and strongly alkaline solutions. In such solutions, aluminosilicate materials are rapidly dissolved to form SiO_4 and AlO_4 tetrahedral units. During the development of the reaction, mixing water is gradually split out and SiO_4 and AlO_4 tetrahedral are linked alternatively to yield three types of monolithic geopolymer products [2].

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Geopolymers have many advantages compared to OPC, such as high early strength [3], good fire and acid resistance and good durability [4–6]. Additionally, they have normally low apparent porosity, which gives them very low water permeability and thus very good resistance in freezing-thawing cycles [7].

However, disadvantages also exist. Geopolymers present a typical brittle mechanical behavior with the consequent low ductility and low fracture toughness [8]. These characteristics can limit their applications as a structural material. In order to improve these drawbacks of geopolymer materials, organic polymers are often incorporated in their structure [9].

Polyethylene glycol (PEG) is a polyether compound with many applications in industrial manufacturing. PEG is one of the most well-known water-soluble polymers, while it can also be dissolved in many organic solvents including aromatic hydrocarbons. PEG is used as a plasticizer to increase lubricity and acts as a water retention agent in ceramic mass, adhesives and binders and soldering fluxes with good spreading property [9].

In a study of Catauroa et al. [11], the influence on mechanical strength of the different percentages of PEG added to geopolymer was investigated. In absence of PEG, the tests showed overall strength regularity with aging time due to the chemical composition of sample. PEG-free samples can reach final mechanical strength faster than hybrid systems. The stretching effect of PEG, that in general provides the characteristic of elasticity to the base material together with the longer time required to reach the final structural strength, justifies the increase of flexural and compressive strengths with aging time.

Colangelo et al. [12] concluded that polymer-modified mortars have improved compressive strength in comparison to unmodified ones and polymer helps restrain micro-crack propagation. They also found out that total porosity decreases with the addition of organic polymer and this may contribute in improved durability.

Hereby the aim of this work is to explore the amount of PEG that can be added to geopolymer systems, in order to improve their properties. The rather limited literature in this field of study necessitates the expansion of knowledge on this topic.

2. Experimental

2.1. Materials used

Chemical composition of metakaolin (Mefisto K_{05} – ČLUZ a.s.) used in this study is given in Table 1. It was produced through controlled thermal processes and grain size adjustments of clay stones and floating kaolin clays. Particle sizes are d_{50} = 6.34 μm and d_{90} = 11.62 μm.

Table 1. Chemical analysis of metakaolin Mefisto K05 (*% w/w*).

SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	LOI
55.01	40.94	0.55	0.55	0.14	0.34	0.09	0.60	1.57

The metakaolin was activated with a water glass solution having a SiO_2/Na_2O ratio of 1.6. Quartz sand with a maximum grain size of 2.5 mm was used as aggregate. Different commercial types of polyethylene glycol (ROTIPURAN®, Ph.Eur), indicated as PEG later on, were used at different dosage from 0.5 to 10% by mass of metakaolin:

- PEG 400
- PEG 1000
- PEG 6000
- PEG 20000

2.2. Mortars preparation

Geopolymer mortars with and without PEG were prepared with an aggregate/binder ratio of 3/1. Each type of PEG was added at dosages of 0.5, 1, 2, 5, 7 and 10 % by mass of metakaolin. The activator and PEG were added in water and then mixed with metakaolin. Then quartz sand was added into the fresh mortar during mixing. Geopolymer mortar specimens were cast in prismatic moulds (40 × 40 × 160 mm). The specimens were left in the moulds for 2 hours, then cured in electric oven at 40 ºC for 4 hours in order to accelerate the hardening process, and finally removed from the moulds after 24 hours. After demoulding, the specimens were stored in plastic bags for 27 days at RH = 45 ± 5%.

2.3. Tests

The effect of type of PEG on compressive and flexural strength was evaluated at first. Flexural and compressive strengths of the specimens were measured at the age of 28 days. Flexural strengths were determined by using a standard three-point-bending test, while compressive strengths were measured on both residual pieces obtained from the flexural strength test.

Total porosity and pore size distribution were determined in metakaolin based mortars by means of mercury intrusion porosimetry using Micromeritics Poresizer 9300 porosimeter. The microstructure of geopolymer mortars was investigated with scanning electron microscope (SEM) (Tescan MIRA3 XMU), applying acceleration voltage of 20 kV.

3. Results and discussion

3.1. Mechanical properties

Development of mechanical strength of the reference metakaolin-based geopolymer mortar (REF) and of the polymer-modified geopolymer mortars are given in Fig. 1 (compressive strength) and Fig. 2 (flexural strength). The maximum compressive strength was observed for the composition with 10% of PEG 400, being by 57% higher than that of REF. The composition containing 1% of PEG 1000 showed the minimum compressive strength value, which was by 45% lower than that of REF. Also one of the lowest values was recorded for the specimen with 7% of PEG 20000. In general, the addition of PEG 20000 resulted in lower compressive strength values compared to REF composition. On the contrary, PEG 400 added at the amount of 5, 7 and 10% contributed to compressive strength increase. Flexural strengths showed the same trend as was observed for compressive strength. The best results were achieved with addition of 5, 7 and 10% PEG 400, whereas the lowest strength showed geopolymer with PEG 20000.

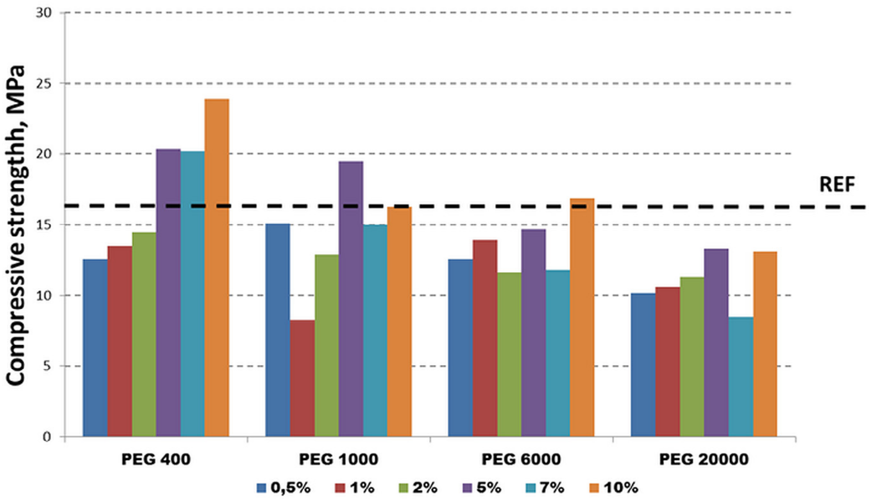

Fig. 1. Compressive strength of geopolymer mortars with different contents of different types of PEG.

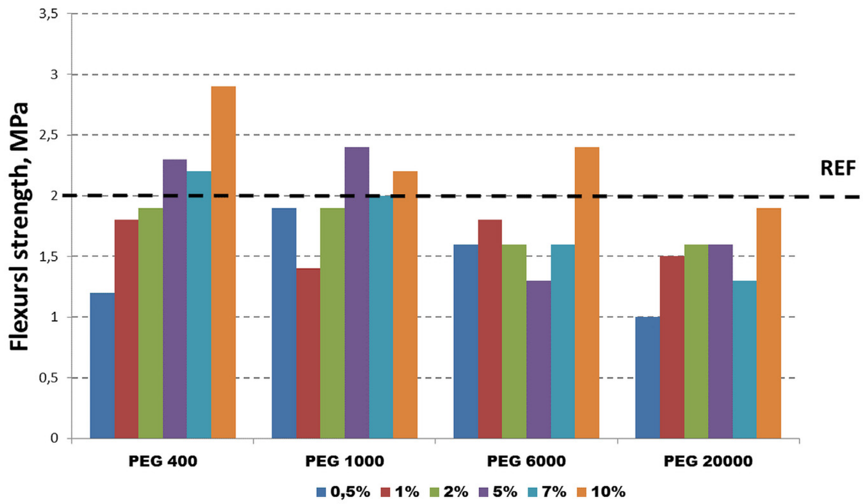

Fig. 2. Flexural strength of geopolymer mortars with different contents of different types of PEG.

3.2. Porosity

Fig. 3 illustrates the pore size distribution of geopolymer mortars. Porosity measurements were performed only on reference specimen and specimens with the highest and lowest compressive strength. According to Fig. 3, the least porous material is the REF specimen. The largest pore volume is associated with pores between 0.l–1 µm in diameter. For PEG 1000 the curve is shifted to bigger pores, while in case of PEG 400 smaller porous are predominant. Since porosity strongly influences the mechanical properties of the material, these data correspond to the mechanical characteristics of the tested geopolymers.

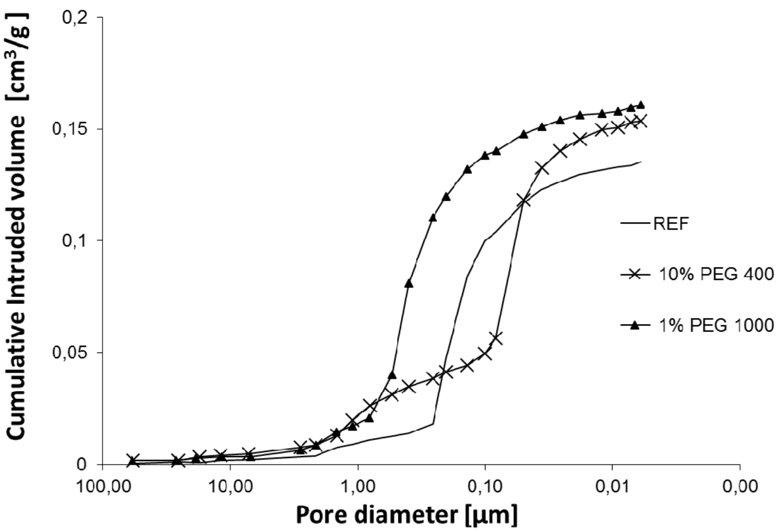

Fig. 3. Porosity of geopolymer mortars with different content of PEG.

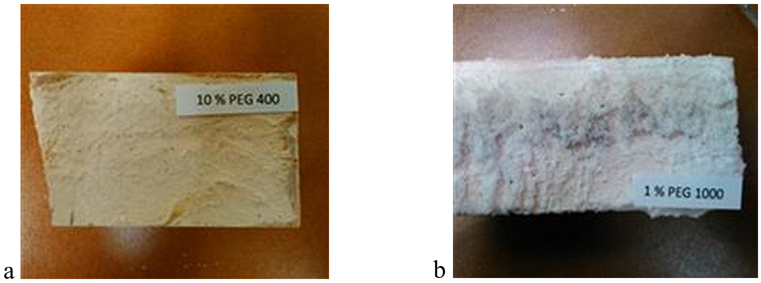

Fig. 4. Geopolymer mortar specimens containing (a) 10% PEG 400 and (b) 1% PEG 1000.

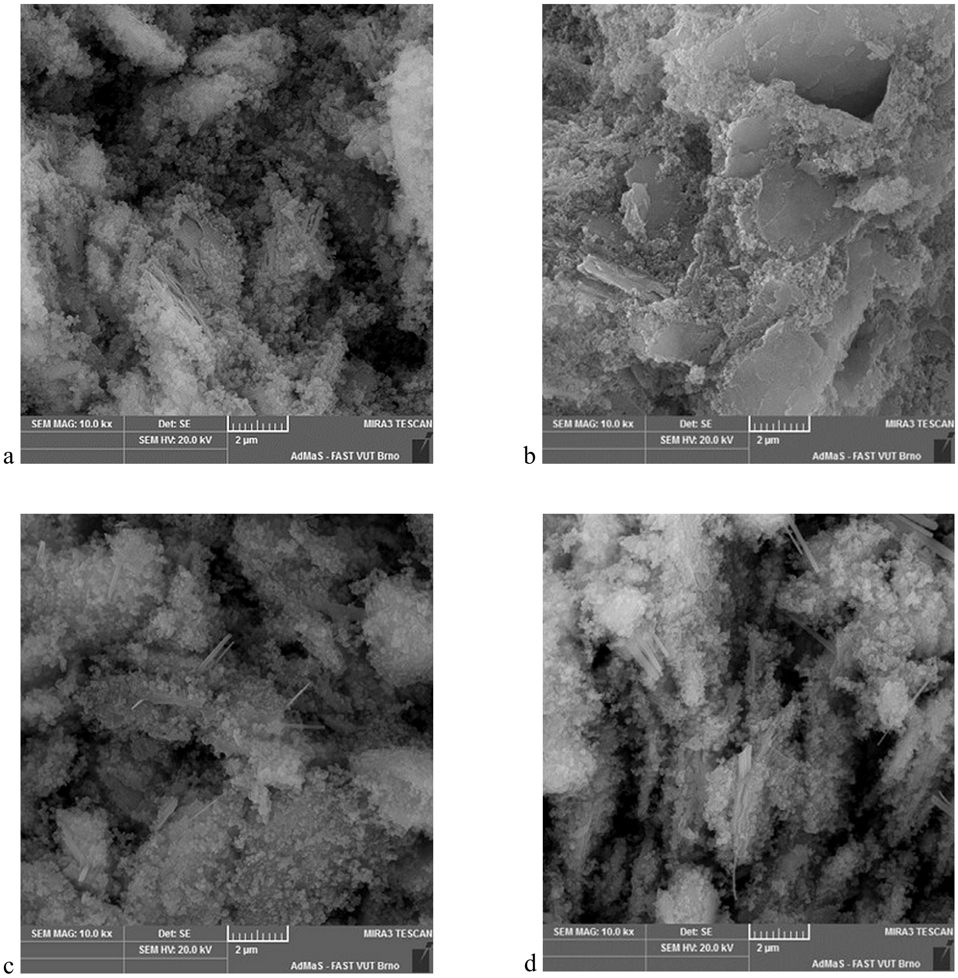

Fig. 5. SEM photos (10000× magnification) of (a) REF geopolymer mortar, (b) 10% PEG 400, (c) 0.5% PEG 20000, (d) 1% PEG 1000.

3.3. Microstructure

Fig. 4 shows that no efflorescence was observed on the surfaces of the samples with 10% of PEG 400. Nevertheless, intensive efflorescence was formed on the surfaces of the samples with 1% of PEG 1000.

Micrographs of samples obtained from the mortars with 10% PEG 400 showed that the structure is solid and compact (Fig. 5b), whereas the structure of REF is more porous and open-grain (Fig. 5a). The structure of the specimens with 0.5% PEG 20000 and 1% PEG 1000 (Fig. 5c, d) is the most porous, therefore shows the lowest compressive strength. It is also apparent that the specimen with 1% of PEG 1000 contains needle-shaped crystals of alkali carbonates. This corresponds to the efflorescence observed in Fig. 4b. However, the structure of geopolymer mortar with 10% PEG 400 (which had the maximum compressive strength) did not show any efflorescence on its surface (Fig. 4a), which may explain its high strength. SEM analysis proved that the PEGs affect the structure of metakaolin based geopolymers.

4. Conclusions

The present work focused on the effect of the addition of different types and amounts of PEG in metakaolin geopolymer mortars. Several tests were carried out: physical (pore size distribution), mechanical (flexural and compressive strength), and microstructural (SEM).

The maximum compressive and flexural strengths were achieved by adding 10% of PEG 400. The minimum value of compressive and flexural strength was obtained for the specimens containing PEG 1000 at dosage 1% and PEG 20000 at dosage 0.5%, respectively. These results were confirmed by SEM observations and porosity measurements. The specimen (10% PEG 400) with the highest strength had dense structure and was less porous. This was a preliminary study and further work and more detailed studies should be done to explain the mechanisms, what role PEG plays in geopolymerization reaction.

Acknowledgements

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