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Influence of heat treatment on the pore structure of some clays - precursors for geopolymer synthesis

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Abstract

The aim of the presented study was the heat treatment response of the pore structure of the aluminosilicate materials (bentonite, kaolin and zeolite) and the resulted characteristics of the metaproducts, presented as the geopolymer raw materials. The treatment of the studied clays at 650 °C during 4 hours caused a high pore structure response represented by the occurrence of the creation of a new pore area on the pore size distribution histograms beginning over 2000 nm pore radius. Its size was strongly dependent on the clay species. The found relation between compressive strength of the related geopolymers and the histograms area size the assumption entitles to valuate that response of the pore structure of the clays to the heat treatment as a factor significantly conditioning the effectivity of the alkali activation and the process of the geopolymer synthesis.

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1. Introduction

The inorganic binders based on the alkali activation of reactive aluminosilicates as geopolymer cements are known. As geopolymer cements raw materials, the solid aluminosilicate industrial wastes and thermally treated clays are used [1,2]. The thermal treatment of clays results in the dehydration and dehydroxilation in the temperature range between 500–800 °C, depending on the type of the clay mineral [3]. The reactivity of the raw material is based

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on the content of the metastable, nearly amorphous aluminosilicate phases able to dissolute in alkaline solutions [4–6].

A further significant factor conditioning the reactivity of the amorphous aluminosilicate phase, among others, is its pore structure. Its significance is based on the fact that it determines the possibility of the transport and the contact conditions of the alkali solution with the activated material. Therefore, the pore structure represents a factor directly conditioning the intensity of the alkali activation process and the development of the engineering properties of the resulted geopolymer [7,8]. However, there are only few published works concerning the effects of thermal treatment on the pore structure [9]. This fact was the motivation for the presented study.

2. Experimental

2.1. Test specimen's preparation and their testing

The objects of the study were bentonite, kaolin and zeolite and their product of the heating at 650 °C during 4 hours. The subjected clays were naturally mined and dry grinded to the maximal particle size 90 mμ. The grinding produces important changes in powdered material and influences its behavior in further processing steps [10]. The metaproducts obtained for the pressure compacted and reference test specimens 20 mm-edge cubes were used. Their preparation conditions were as it follows:

- Pressure compacted paste – alkali activator solution/metaproduct ratio (l/s) 0.08, pressure compaction at the application of the uniaxial pressure of 300 MPa with the endurance of 1 min at the fresh mixture, directly after its preparation.
- Reference paste - alkali activator solution/metaproduct ratio (l/s) 0.7, the current hand compaction, as an alkali activator the sodium hydroxide was used; its portion corresponded to 7 wt. % of the metaproduct weight.

The preparation of the pastes represented an intensive mixing of the mixture for 2 min using electrical mixer. The test specimens hardened 24 hours in the forms at the temperature 20 °C and 95% relative humidity. After the demolding their testing followed.

2.2. Tested properties and test methods used

The test specimens used for the testing were dried at 105 °C. The following properties were tested and these test methods were used:

Compressive strength - destructive test under the use of a loading press.

Chemical composition – current chemical analytic procedures were used.

Phase composition:

- Thermal analysis - Thermal analysis METTLER TOLEDO, TGA/DSC 1, STAREe System was used. The sample mass 45 mg, heating rate 10 °C/min, ramping from ambient temperature to 1000 °C in an air atmosphere were the conditions of the test.
- X-ray analysis - STOE Powder Diffraction System (STADI P) using Co Kα radiation, operating at 40 kV and 30 mA was used. Data were collected over 2θ between 5 and 40. Assignment of lines was made by comparison with JCPDS (Joint Committee on Powder Diffraction Standards) files.
- Pore structure – used equipment was Pore Master 60 (Quantachrome, UK) automated mercury porosimeter under the use of the wetting contact angle 141.3° and mercury surface tension 0.48 $N \cdot m^{-1}$. The porosimeter generates pressure to 414 MPa enabling the pore size analysis from 3.6 nm to 950 000 nm. Particles morphology – scanning electron microscope JEOL 6400 type enabling 30 Angstroms resolution was used.