

GEOPOLYMERS BASED ON NATURAL ZEOLITE AND METAZEOLITE

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Zeolites and geopolymers have similar chemical composition, but differ in their crystallinity. In Bulgaria one of the world's largest resources of natural zeolites is located. The deposit consists of rather pure clinoptilolite (70% - 80% clinoptilolite with 10% - 15% cristobalite and some amorphous phases) and has estimated reserves in the order of 10×10^9 tons. With the aim of evaluating the suitability of this abundant raw material to serve as a precursor for geopolymeric materials, and hence applications in the building industry, an experimental study of geopolymerisation of natural zeolite was carried out.

Metazeolite was obtained by calcining the zeolite at 900 °C for one hour, at which temperature XRD analysis confirmed complete destruction of all clinoptilolite without formation of new phases, resulting in an amorphous material with a trace of residual cristobalite. Natural zeolite and calcined natural zeolite (metazeolite) were mixed with KOH, K-silicate and Na-aluminate activator solutions to produce geopolymers. In addition, also a spent Na-aluminate waste solution from the aluminium anodizing industry was used as activator. The compressive strength did increase by a factor 2 to 3 for the calcined zeolite compared to the natural zeolite. The calcined zeolite activated with K-silicate activator showed the highest compressive strength (43 MPa \pm 4 MPa), but also showed high shrinkage (7%). Shrinkage could be reduced to 2% to 3% by activation with the aluminate solutions, while compromising the compressive strength to 33 MPa – 37 MPa.

The geopolymers produced from oven-dried natural zeolite and activated with aluminate solutions showed lower shrinkage (3%) and some strength as well (10 MPa – 11 MPa). Those materials could still be of interest since they contain residual clinoptilolite and newly formed zeolite Na-P and phillipsite and therefore combine the properties of geopolymers (strength) and zeolites (ion exchange and adsorption).

The geopolymer samples were examined with X-Ray Diffraction (XRD), Fourier Transformed Infra Red (FTIR) Spectrometry and Scanning Electron Microscopy (SEM). XRD revealed that the geopolymers from the calcined zeolites remained fully amorphous when activated with KOH and K-silicate, but showed zeolite Na-P and phillipsite as newly formed crystalline phases when activated with aluminate solutions. FTIR confirmed decreasing Si/Al ratios in the geopolymers when activation was done with the aluminate solutions. SEM micrographs displayed that the samples made from the oven-dried natural zeolites had many more macro pores present compared to the geopolymers from the calcined zeolites. Probably this was due to air entrainment in the zeolites.

There was no significant difference in geopolymer product between the use of chemical grade aluminate solutions and industrial waste aluminate solutions, although using waste solutions has both economic and environmental benefits