

SEM/EDS characterization of fly ash based geopolymers

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Fly ash (FA) is the major combustion residue produced by burning of pulverized coal in thermal power plants and collected by electrostatic precipitators. FA is dustlike heterogeneous material represented by particles generally below 100 μ m in size. The basic oxides in chemical composition of FA samples are SiO₂, Al₂O₃, CaO and Fe₂O₃. FA contain significant amount of amorphous matter and some crystalline phases, such as quartz, feldspar, mullite, hematite and anhydrite. Currently, only a part of this waste is utilized, mostly by cement and concrete industry, the remainder being directly placed in dumps. There is a growing urge for investigation of the new possibilities for FA utilization. In recent years, great scientific attention is focused on a new class of high performance materials for construction and waste immobilization, called geopolymers [1].

Geopolymers are a class of aluminosilicate materials generally synthesized at ambient or slightly elevated temperatures by reaction of a solid aluminosilicate powder with a concentrated alkali metal silicate or hydroxide solution [1,2]. Geopolymer technology is an attractive way of producing high performance binder from waste materials such as FA. Few conceptual models of geopolymerisation reaction are suggested [1,2]. Dissolution of the solid aluminosilicate material by alkaline solution produces (mostly) monomeric silicate and aluminate species. Dissolution of amorphous aluminosilicates is rapid at high pH, and this process is followed by oligomerisation and polymerisation, creating aluminosilicate polymer. Final product of the geopolymerisation reaction is X-ray amorphous aluminosilicate gel [3,4]. Depending on the starting aluminosilicate material, alkali activator and reaction condition, some zeolite structures as reaction products can also be formed.

In this study, FA sample from Serbian power plant was treated with sodium silicate solution. Two different solutions were used, where SiO₂/Na₂O ratio in the solution were 0.5 and 1.5. Calculated quantities of FA and sodium silicate solution were mixed so the concentration of Na₂O in pastes was 10% (mass). Pastes were packed in plastic bags and cured for 24h at room temperature and 6 days at elevated temperature (55°C). Another series of the pastes was prepared with sodium silicate solution, SiO₂/Na₂O=1.5, and cured at room temperature for 1, 28 and 180 days. Fracture surface of the pastes was studied by SEM (VEGA TS 5130 MM, Tescan) and EDS (INCAPentaFET-x3, OXFORD Instruments). Samples were Au/Pd coated.

Figures 1a and 1b show the microstructure of the alkali activated FA treated with solutions with different content of silicate. Pastes consist of the nonreacted and partially reacted FA particles embedded in gel formed as a result of geopolymerisation reaction. When the activating solution contains high concentration of silicate, dissolved aluminate and silicate species from FA react with already present silicate in solution forming oligomers. The reaction is rapid and formed polymer will not only condense on the surface on the FA particles, but will migrate into the bulk solution (Figure 1b). On the other hand, if the activating solution contain lower content of silicate, it is more likely that the small dissolved

aluminosilicate species will precipitate on the surface of FA particle where they may either redissolve or become fixed to the surface as new units condense over them. The presence of small aluminate species can enhance zeolite formation and crystal growth (Figure 1a).

Figure 1c shows the results of the EDS analysis of the pastes prepared with sodium silicate solution containing high concentration of silicate and cured for 1, 28 and 180 days. Si/Al atomic ratio in the products of the geopolymerisation reaction in these samples decreases with time. This is probably due to the high content of silica present in activating solution. Dissolved aluminate and silicate species from FA react rapidly with silica already present in the solution and create polymer. Pastes are setting and hardening quickly. Once a gel is formed, dissolution process and movement of the species in the paste is significantly obstructed. The reaction products formed at the beginning of the reaction contain high level of Si and high Si/Al atomic ratio due to the silicate present in activating solution. With time, Si/Al ratio decreases.

There are many aspects of geopolymer technology that remain incompletely understood. The key tool that has the potential to provide a detailed understanding of geopolymer microstructure and its evolution with time is electron microscopy.

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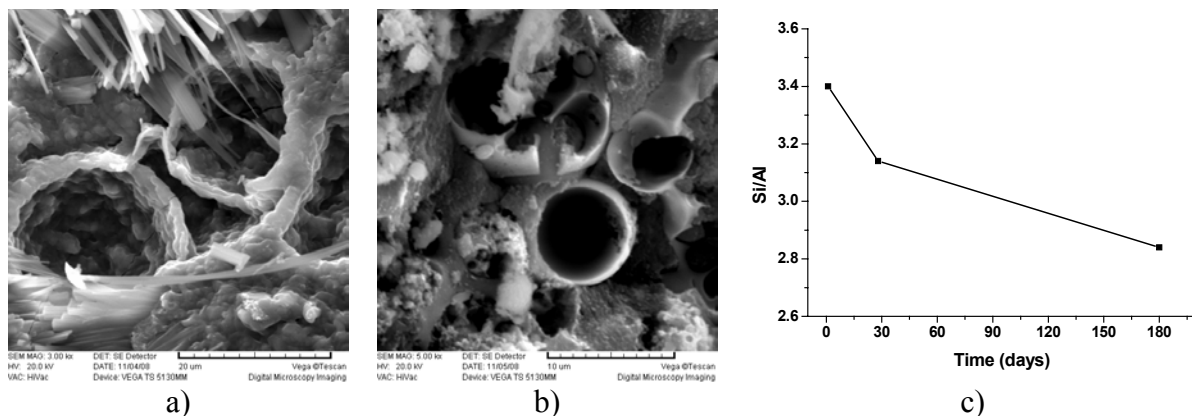


Figure 1. a) SEM image of the FA paste activated with sodium silicate solution, $\text{SiO}_2/\text{Na}_2\text{O}=0.5$. b) SEM image of the FA paste activated with sodium silicate solution, $\text{SiO}_2/\text{Na}_2\text{O}=1.5$. c) EDS results: Si/Al atomic ratio in FA pastes cured for 1, 28 and 180 days