



Effect of thermal treatment on reactivity and mechanical properties of alkali activated shale–slag binder



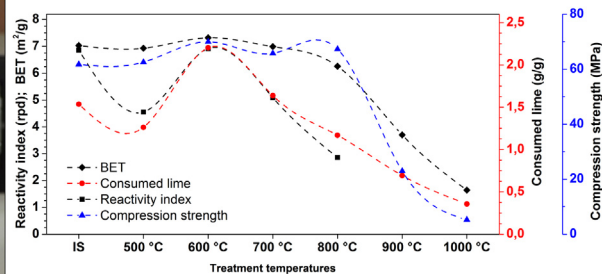
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HIGHLIGHTS

- Investigation of structural changes of thermally treated shale–slag powder in temperature range of 500–1000 °C.
- Application of small amplitude dynamic rheometry for geopolymerization investigations.
- Powder mixture treated at 600 °C for 4 h exhibits highest reactivity.
- The geopolymer binder reached ultimate compression strength of 80.52 Mpa.

GRAPHICAL ABSTRACT



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ABSTRACT

This paper is aimed at investigating the properties of calcined shale–slag as an alternative alkali-activated constructional binder hardened by geopolymerization reaction with sodium silicate solution. Focus is put on possibilities of thermal treatment in temperature range of 500–1000 °C and investigation of structural changes. These investigations were done by X-ray diffraction (XRD), scanning electron microscopy (SEM), infrared spectroscopy (FTIR), measurement of specific surface area, pozzolanic activity and mechanical strength of prepared geopolymer mixtures. Reaction rates were measured by small amplitude dynamic rheometry as a relatively new method for investigation of geopolymerization process. The best compression strength of 82.52 MPa and highest reactivity, according to the pozzolanic activity and dynamic rheometry, was achieved by thermal treatment at 600 °C for duration of 4 h.

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1. Introduction

Inorganic polymer binders can be synthesized by alkaline activation of a variety of materials including thermally activated clays, coal fly ash and blast furnace slag to produce a solid material with mechanical and thermal properties potentially suitable for wide range of industrial applications [1–3], such as binders for building

applications [4–7], fire resisting parts [8–10], soil and earths stabilizers [11–13], foams [14,15] and many more. Alkali-activated pozzolanic materials were discovered by Purdon and Glukhovskiy in the 1940s and systematically studied up to the 1960s [16–18]. Their scientific work was devoted to the synthesis of alkali-activated materials, usually with high-calcium metallurgical slags (non-Portland cement). The first fundamental principles of production of hydraulic binders by alkaline activation were set by them. Glukhovskiy called these aluminosilicates “soil cements” (in Russian “gruntosilikaty”). The research in this field has developed

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in conjunction with types of utilized raw materials, methods of synthesis and also terminology. Davidovits termed this type of materials as “geopolymers” and introduced pioneering alkali-activated calcium-free systems based on calcined clays [19–21]. The alkaline activation of an aluminosilicate material can be described as blend of a highly concentrated alkaline solution and a reactive solid silicates and aluminates [22,23]. From a general point of view aluminosilicate binders are classified as geopolymers activated by alkali hydroxide solutions (NaOH [24,25], KOH [26,27], CsOH [28]), carbonate and sulfate solutions [29] or alkali silicate solutions [2,3]. Sodium silicate solutions are widely used as geopolymer constituent controlling the disaggregation processes of the solid phase via combination of partial dissolution of solid particles and cleavage of the Me–O, Si–O–Si, Al–O–Al, Al–O–Si bonds [30]. The dissolution rate depends on the pH, chemical composition, concentration, structural state, and distribution of silicate species in the system [31]. The following gel precipitation and solid phase formation is driven by polycondensation mechanism [32,33]. The process of structural changes leading to the formation of amorphous aluminosilicate matrix depends on the starting material, the type of reactive silicate solution and hardening conditions. Not only calcined kaolinite, but many aluminosilicates (may containing alkaline earth oxides as CaO) can be hardened with this mechanism. Examples can be shales and other clay minerals [34–37], fly ashes [38], slags and their mixtures [16,39–44].

In this study, the effect of thermal treatment of a pre-calcined shale dust mixed with calcium-rich slag has been investigated in relation to reactivity, structural changes, mechanical properties and rheology. Changes in chemical reactivity and crystal structure due to thermal treatment in temperature range 500–1000 °C are presented. Results are supported by X-ray diffraction (XRD), scanning electron microscopy (SEM) and infrared spectroscopy (FTIR) regarding the morphology changes, and with Chapelle test, specific surface area and dynamic rheometric analysis concerning the reactivity. Finally, mechanical properties of hardened specimens are investigated for comprehensive overview.

2. Materials and methods

2.1. Materials

In this study, a commercially available powder mixture of shale and slag supplied under trademark Baucis L160 from Ceske luppove zavody a.s., Nove Straseci, Czech republic (CLUZ a.s.) was examined. The shale dust is pre-calcined to metakaolin state and then mixed with fine amorphous slag. The mixing proportions are 60/40 weight percents. The chemical composition was determined by XRF technique

and is listed in Table 1. The mineralogical composition was determined by XRD analysis and the results indicate a dominant presence of natural quartz and anatase as a crystalline part of calcined shale and akermanite with merwinite as a crystalline part of slag. Also mullite and grossite are present in trace amounts. According to Rietveld refinement technique on X-ray diffraction data (not presented here), the main part of this material is in amorphous state.

For alkaline activation a commercial aqueous solution of sodium silicate with silicate module 1.71 and content of water soluble solids 32.28% was used. As a filler was used calcined chamotte with trademark Chamotte NP special with a granulometry range of 0–0.5 mm. These materials are also supplied by CLUZ a.s. [45].

2.2. Characterization methods

The elemental analysis of the initial material was carried out by XRF using a Bruker AXS S4 Explorer spectrometer calibrated for aluminosilicate materials. The XRD analysis was conducted on a Pananalytical X'Pert Pro diffractometer (operating with Cu-K α radiation). The chemical reactivity was examined by the standard Chapelle test of pozzolanic activity and the active surface was measured by the BET technique using an SEA inverse gas chromatograph from Surface Measurement Systems Ltd. The setting time and reaction kinetics were determined by time-resolved dynamic rheometric measurements with small amplitude on an Ares G2 rheometer from TA Instruments in plane–plane geometry. SEM imaging was performed with a FEI Quanta200 instrument. The mechanical strength was measured by a Zwick Roell Z330E testing machine with a rate of 0.25 mm/min. FTIR analysis was conducted on a Thermo Scientific Nicolet iS5 FT-IR spectrometer in ATR mode with ZnSe crystal and the spectra were corrected on single-bounce ATR environment.

2.3. Thermal treatment

The experiment was carried out as follows. For preparation of reference samples, Baucis L160 as raw material was used after a mild drying at 125 °C to constant weight. Samples of treated material were prepared from the dried raw material by thermal treatment in standard electric muffle furnace with a heating ramp rate 3 °C/min and a soak time 240 min at selected temperature. Immediately after that period, the samples were taken out of the furnace and stored in a desiccator. Fig. 1 shows photographs of the powder samples in initial state and after thermal treatment in the range 500–1000 °C.

3. Measurements and results

In this section, individual measurements with parameters and experimental setups are presented. The results are presented individually in short and will be discussed together in detail in the discussion section for a better overview.

3.1. Dynamic rheology

The rheometry measurements in small amplitude dynamic mode were performed according to the references [46,47]. The procedure is as follows.

Raw or thermally treated binder in accurate amount of 50 g was poured into 40 g of activator and the mixture was then mixed in

Table 1
Chemical composition of raw material Baucis L160 [mass %].

SiO ₂	Al ₂ O ₃	CaO	MgO	TiO ₂	Fe ₂ O ₃	K ₂ O	Na ₂ O	Others	LOI*	Total
46.54	27.08	17.07	3.52	2.04	0.86	0.55	0.08	0.35	1.91	100

* Loss on ignition at 1000 °C.

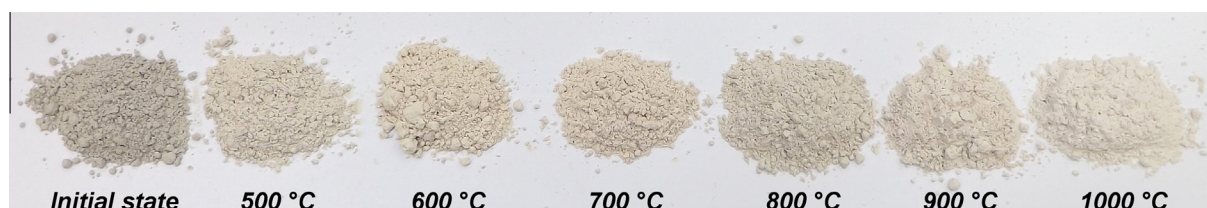


Fig. 1. Powder samples of raw and thermally treated material.

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