



# Influence of limestone content, fineness, and composition on the properties and microstructure of alkali-activated slag cement



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## ABSTRACT

The influence of the fineness, concentration, and chemico-mineralogical composition of limestone on the workability, reaction kinetics, compressive strength, microstructure, and binder gel characteristics of sodium carbonate-based waste-activated waste slag cement pastes was investigated in this work. Alkali-activated slag cements incorporated with limestone, containing 33–100% of calcite, at a content of up to 60% with a 28-day compressive strength of 26.2–48.8 MPa were proposed. The main reaction products of hardened alkali-activated cement pastes and those incorporated with limestone are C–S–H, CaCO<sub>3</sub>, Na<sub>2</sub>Ca(CO<sub>3</sub>)<sub>2</sub>·5H<sub>2</sub>O, and Na<sub>2</sub>CaSiO<sub>4</sub>. “Physically active” limestone does not chemically react with the binder gel but it can improve the physical structure. The higher packing density of mixed cement, without an increase in the water demand, the satisfactory binding strength of limestone with the binder gel lead to the improvement in the physical structure and compressive strength of alkali-activated slag paste.

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## 1. Introduction

Alternative or non-traditional cements form a large group of binders, differing significantly from Portland cement and from each other by the composition and type of raw materials, nature of hardening products, mechanism of hardened cement paste formation, and the research experience, adoption, and application of each type of cement [1]. Some types of alternative binders also termed new cements for the 21st century, new cementitious matrixes, combining higher, in comparison with PC, ‘ecological compatibility’ and some technical characteristics are proving to be increasingly promising in terms of partial replacement of ordinary Portland cement (OPC) in the present climate of ‘sustainable development’ [1,2]. The non-clinker non-fired alkali-activated cements (AACs) belong to this category of cement [3–5]. Such materials usually consist of two components: an aluminosilicate precursor and an alkaline activator, with a variety of industrial by-products and aluminosilicate solids having been used as raw

materials. At the present stage of development of AACs, ground granulated blast furnace slag (GGBFS) and fly ash are accepted as attractive precursors for large-scale industrial production of AACs [6]. However, fly ash and metallurgical slag are not available everywhere nor has the supply chain for their distribution been established in local markets; this is because the supply chain has an interdependence on the existence of a ready market [4]. According to past studies, a wide range of natural materials and by-products is suitable for the replacement of GGBFS, which also has valuable technical advantages and economic benefits [7–10]. The cost and environment footprint of the alkali activator also holds importance in the further development of alkali-activated slag cement (AASC), which is why the application of widely accessible mineral admixtures that can effectively substitute for GGBFS in AASC activated by appropriate alkali components, including alkali-containing wastes, is of scientific and practical interest.

One of the most popular mineral admixtures for OPC is limestone (LS) owing to its abundance and chemical-mineralogical composition [11,12]. Portland–LS cements are the most widely used cements in Europe. Two classes exist in EN 197-1 designated as CEM II/A-L and CEM II/B-L, in which the maximum contents of limestone are 20 and 35%, respectively. Several studies in the past

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few years have stated the attractiveness of LS for blended non-clinker cements [13,14], including AACs [15–23]. Developed by Sakulich et al. [16], alkali-activated slag/limestone fine aggregate concrete had a compressive and tensile strength of up to 45 and 4 MPa at 28-day age, respectively. According to Bilek [17], the addition of up to 33% ground LS filler, with a  $S_{sp}$  of 360 m<sup>2</sup>/kg, to GGBFS activated by a blend of sodium water glass and potassium hydroxide improved its workability, decreased early strength, and almost did not affect the 28-day strength when compared to a reference sample. Gao et al. [19], investigating the properties of alkali-activated slag–fly ash blends with LS addition, stated that the LS powder mainly shows physical modifications with slight but non-ignorable chemical modifications of alkali-activated slag–fly ash blends. The additional nucleation sites provided by LS powder slightly accelerated the induction and acceleration/deceleration stage of the reaction; it also increased the total heat evolution within the tested 72 h and improved the strength of the blended alkali-activated binder system. The gel-characteristic analyses show that the reaction products of alkali activated slag–fly ash–limestone blends are governed by the chain structured C–(A)–S–H type gels. The presence of LS powder did not lead to the formation of new phases, but slightly increased both the physically and chemically bound water content. Yip [20] confirmed the dissolution of Ca<sup>2+</sup> from calcium carbonates in alkali activated systems. It should also be noted that satisfactory surface binding between the LS powder and alkali-activated materials was suggested by Cwirzen [21]. Thus, according to the above-mentioned studies, LS is a promising mineral supplementary material, which has a positive effect on the properties of AAC mainly due to the “physical filler effect”. At the same time, the properties of AACs incorporated with supplementary materials depend on a number of influencing factors, including important factors of supplementary materials such as its chemical-mineralogical composition and fineness [8].

This research aims to study the influence of LS content, fineness, and composition on the properties and microstructure of sodium carbonate–based waste-activated slag cement.

## 2. Experimental details

GGBFS was obtained from the Chelyabinsky factory. Three types of local LS differing in chemical and mineralogical composition were used as supplementary material. The chemical composition of GGBFS and LS is shown in Table 1. The main difference in the mineralogical composition of LSs is in the calcite content, which is 90%, 33%, and 100% in LS1, LS2, and LS3, respectively. LS1 also contains 9% of quartz and 1% of albite; LS2 includes 1% of quartz and 66% of dolomite.

The GBFS was ground in a laboratory planetary mill to a  $S_{sp}$  of 300 m<sup>2</sup>/kg, and the LSs were ground to  $S_{sp}$  ranging from 200 to 600 m<sup>2</sup>/kg (Blaine). The particle size distribution and the  $S_{sp}$  of the GGBFS and LS powders were tested using a Fritsch Particle Sizer ANALYSETTE 22 laser particle size analyser and a Blaine air-permeability apparatus.

**Table 1**  
Chemical composition of starting materials.

Starting material	Component (mass % as oxide)												
	SiO <sub>2</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	MgO	MnO	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	CO <sub>2</sub>	LOI
GGBFS	37.49	36.22	11.58	8.61	0.50	0.16	1.80	0.64	0.95	0.01	2.00	–	–
LS1	14.26	43.31	2.44	0.84	–	1.11	0.12	0.38	0.51	0.08	–	35.62	36.28
LS2	1.26	39.79	0.20	12.94	–	0.08	0.01	0.08	0.07	<0.01	–	44.33	45.50
LS3	0.47	55.06	0.07	0.49	–	0.05	0.01	0.03	0.01	0.02	–	43.66	43.83

The alkaline activation of the AACs was carried out using alkaline solid waste, forming because of incineration of sewage of petrochemical company “Nizhnekamskneftekhim” (Russian Federation) which produces synthetic rubbers and plastics (Table 2). It consists mainly of sodium carbonate (91.4%). The alkaline solid waste content was 10 g per 100 g of GGBFS or GGBFS + LS.

Vicat tests were carried out according to EN 196–3 to establish the effect of LS on the standard consistency of the fresh AASC pastes.

All test mixtures were prepared by using an electrically driven mechanical mixer. Initially, GGBFS and LS were mixed in a dry state for a minute and then the activator solution was gradually added while mixing continued for about 3 min. Fresh pastes were cast into steel cubic moulds (2 × 2 × 2 cm). The compressive strengths of the cubes were tested after a 28-day storage under normal conditions (room temperature, humidity 95–100%). Average compressive strengths of six cubes were obtained from each mixture at each testing age.

Isothermal calorimetry experiments were carried out using the metering equipment “Thermochron”. The pastes were mixed externally, loaded into the sealed glass ampoule, and loaded into the calorimeter. The time elapsed between the instant the activating solution was added to the powder and the paste being loaded into the calorimeter was around 2–3 min. The tests were run for 200 h.

The mineralogical composition of the LS and hardened AASC pastes was determined using a D8 ADVANCE X-ray diffractometer (XRD) with CuK $\alpha$  radiation operating at 40 kV and 30 mA. Fragments of selected 28-day cured samples were sprayed with an Au/Pd alloy for observation under a scanning electron microscope (SEM, Merlin of CARL ZEISS).

For qualitative analysis, a set of etalons established in the program Aztec has been used (reference standards for X-RAY micro-analysis “Registered Standard No. 8842”).

## 3. Results and discussion

### 3.1. The influence of LS on the properties of fresh and hardened AASC pastes

For study of the influence of LSs on the properties of fresh and hardened AASC pastes, the content of the LS was varied from 0 to 70%. Normal consistency of fresh AASC and alkali-activated slag–limestone cements (AASLSC) did not significantly increase with the content and  $S_{sp}$  of LS, and was about 25–26%. These results are in agreement with the results stated by Tsvivilis et al. [24] about lowering the water demand of PC paste incorporated with LS powder and with those stated by Gao et al. [19] concerning the positive effect of increasing LS powder content on the flowability of alkali-activated slag–fly ash–limestone blends. This is explained by the microvoids of the paste being filled by fine LS particles, which leads to better particle packing and release of water lubricating the particles.

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