



Manufacture of air-cooled slag-based alkali-activated cements using mechanochemical activation



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HIGHLIGHTS

- A novel mechanochemical activation procedure of air-cooled slag is presented.
- Activated air-cooled slag is successfully used to produce alkali-activated cements.
- 49.1 MPa strength was achieved with the mechanochemically treated air-cooled slag.
- Strength of mechanochemically activated air-cooled slag surpassed that of granulated one.

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ABSTRACT

This study introduces a novel activation method to manufacture alkali-activated cements (AACs) based on inactive air-cooled blast furnace slag (ACS), which is well-crystallized and hydraulically unreactive. To enhance its reactivity we developed a new mechanochemical activation procedure, analysing the effect of activation on the structure of ACS at different grinding parameters using XRD, FTIR, SEM, and PSD. The applicability of activated ACS in AACs was characterised using compressive strength at 28 days. The ACS partially amorphised by mechanochemical activation proved to be a valuable component for AACs with its maximum strength in mortar surpassing that of granulated slag.

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1. Introduction

First use of alkali activated cements (AACs) dates back to the 1940's, but great attention has been paid to them again in recent decades, mainly because of the potential use of industrial wastes [1]. A wide variety of alkali-activated cements has been developed in the last few decades. Based on the chemical composition of these binders (CaO–SiO₂–Al₂O₃ system), alkaline cements can be divided to two basic categories: high- and low-calcium cements [2]. Most commonly used starting material for high-calcium system is blast furnace slag (BFS), which can be activated using alkali hydroxides, carbonates or silicates. Alkali activation of a calcium-rich system leads to a reaction product similar to calcium silicate hydrate (C–S–H) gel (formed in hydration of ordinary Portland cement (OPC)) but with aluminium in its composition (calcium aluminium silicate hydrate (C–A–S–H) gel). The CaO/SiO₂ ratio of C–A–S–H gel is lower than that of C–S–H gel formed in Portland

cement paste [3]. AAC materials also exhibit superior properties compared to OPC, such as higher resistance to chemical attack, freezing and thawing, and high temperatures [4].

BFS is a by-product generated during the manufacture of pig iron, and comprises a CaO–MgO–Al₂O₃–SiO₂ system. Crystallinity of BFS greatly depends on the cooling method of slag melts. Well-crystallized ACS can be obtained by slow cooling of slag melts, where melilite – a solid solution of gehlenite (C₂AS) and akermanite (C₃MS₂) – is the most common mineral [5]. The cooled material is hard and dense, as well as it has little or no cementing property, that is why it is only used as coarse aggregate of asphalt pavement, railway ballast, road bases and sub-bases, and concrete [6,7]; in Hungary alone more than 1 million tons of ACS are available without large-scale utilization [8]. El-Didamony et al. [9] had only applied ACS as an inert substitute of granulated blast furnace slag (GBFS) in alkali-activated slag.

The GBFS is formed by rapid cooling of molten slag with water, which leads to the formation of disordered (vitreous) structure. These structural characteristics give GBFS moderate cementitious

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property. That is why ground GBFS (GGBFS) is used as a replacement for OPC, and as a starting material for AAC systems [4,5].

The reactivity of GBFS was improved by mechanical activation (fine grinding), which only increased the specific surface area of GBFS and did not modify its crystal structure [10,11]. Mechanical activation of fly ash and stainless steel slag was also used to enhance the effectiveness of preparation of geopolymers and hydraulic binders [12,13]. Reactivity of starting materials can significantly be improved through mechanochemical activation [14–16]. In this case, during the applied intensive dry grinding predominantly crystal structural and even physicochemical changes occur in solid materials, which are covered with the term mechanochemistry [14]. Previously, mechanochemical activation has been applied on kaolinite as a way to amorphised its crystal structure, which considerably increased its thermal and geopolymerisation reactivity [12,16,17].

To the best of our knowledge, the possibility of mechanochemical activation of ACS containing high amount of crystallized phases has not been explored yet. Present study deals with the options of mechanochemical activation, mainly for the purpose of manufacturing AAC binders using crystalline ACS. This research also investigates the changes in the ACS structure and reactivity comparing it with that of GGBFS samples. We also draw attention to the importance of “degree of amorphisation” (DOA) value and compressive strength to characterise the reactivity of various activated slag samples.

2. Materials and methods

2.1. Materials

Two different batches of GGBFS (I. and II.) and ACS were obtained from DUNAFERR Ltd., Hungary. Their chemical and phase compositions are presented in weight percentage (wt%) in Tables 1 and 2. The phase composition of original GGBFS and ACS samples was identified by X-ray diffraction (XRD) analysis, and the quantitative analysis was carried out using the Rietveld method. The XRD analyses indicated (Fig. 1) that the ACS was completely crystalline, and its major constituent was the solid solution of akermanite and gehlenite (77 wt% – hereafter referred to as akermanite). In the case of GGBFS samples the amount of vitreous phase was different for the two batches (70 wt% and 90 wt% for GGBFS I. and II., respectively), and the main crystalline phase was merwinite, while the minor constituents were akermanite and quartz.

Sodium silicate (28.6 wt% SiO₂, 6.8 wt% Na₂O, and 64.5 wt% H₂O) and laboratory-grade sodium hydroxide pellets were applied as alkaline activators. The AAC mortars were prepared using stan-

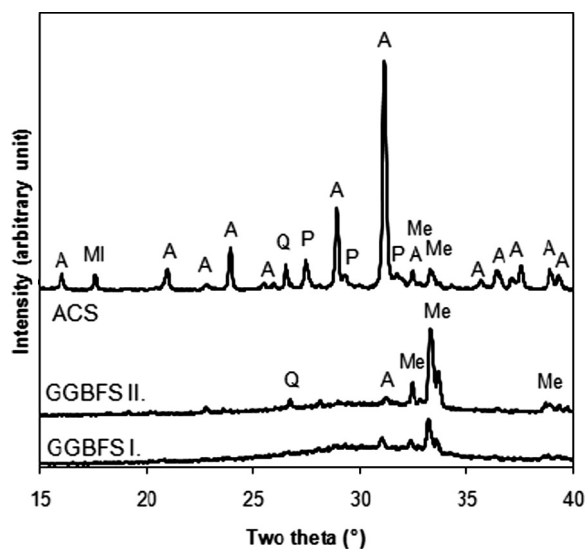


Fig. 1. XRD patterns of original slag samples. (A: akermanite PDF 1-88-778, Me: merwinite PDF 1-74-382, Q: quartz PDF 1-75-6052, MI: melilite (Fe-doped) PDF 1-75-7913, P: pseudowollastonite PDF 1-74-0874).

ard quartz sand (Lasselsberger-Knauf Ltd.) (with particle size up to 2 mm) as aggregate.

2.2. Sample preparation

ACS samples were ground by Fritsch Pulverisette 6 type planetary ball mill for the purpose of mechanochemical activation, but prior to this activation, the samples had been crushed by a jaw crusher in order to obtain an average particle size of 300 μm (medium value of the PSD (D50)). Mechanochemical activation was carried out using 37 g samples, 500 cm³ zirconia grinding bowl, and zirconia balls (10 mm diameter). The used grinding parameters are listed in Table 3.

GGBFS samples were also ground in the planetary ball mill to achieve an average particle size similar to that of ACS samples. Grinding parameters were the same for both GGBFS samples: 120 min grinding time, 400 rpm rotation speed, and 1:1 ms:mgv value. The obtained D50 values of GBFS I. and II. samples were 10.9 μm and 5.3 μm, respectively.

Preparation of AAC mortar (with sand) was based on the European standard EN 196-1. The composition of AAC binder was chosen according to a parallel research and was the same for every slag sample. The composition of the activator solution was

Table 1
Chemical composition of slag samples.

	Major compounds (wt%)						Minor compounds (wt%)
	CaO	SiO ₂	Al ₂ O ₃	MgO	Fe ₂ O ₃	SO ₃	MnO ₂ , TiO ₂ , Na ₂ O, K ₂ O
GGBFS I.	40.0	40.4	7.4	7.2	–	2.3	2.7
GGBFS II.	39.6	34.6	11.0	9.0	–	3.4	2.4
ACS	34.2	42.7	8.6	8.1	1.2	3.1	3.4

Table 2
Phase composition of slag samples.

	Constituents (wt%)					
	Akermanite	Merwinite	Quartz	Pseudo-wollastonite	Melilite (Fe-doped)	Vitreous phase
GGBFS I.	1	8	–	–	–	91
GGBFS II.	1	28	–	–	–	71
ACS	77	10	2	10	1	–

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