



Hydration and properties of sodium sulfate activated slag



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ABSTRACT

Interest in alkali-activated slag as a construction material is increasing, primarily due to its environmentally friendly nature. Although strong alkaline activators, such as sodium hydroxide and sodium silicate solution, are preferred for high strength, none of them exists naturally and their manufacturing process is quite energy intensive. Whilst sodium sulfate (Na_2SO_4) can be obtained from natural resources, the early strength of Na_2SO_4 activated slag is usually low. In this paper, the effects of slag fineness and Na_2SO_4 dosage on strength, pH, hydration and microstructure were investigated and compared with those of a pure Portland cement (PC). Test results indicated that increasing the slag fineness is a more effective approach than increasing Na_2SO_4 dosage for increasing both the early and long-term strength of Na_2SO_4 activated slags. In addition, increasing the slag fineness can also increase the strength without increasing the pH of the hardened matrix, which is beneficial for immobilizing certain types of nuclear waste containing reactive metals and resins.

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1. Introduction

The construction activities in many countries around the world demand large volumes of cement production. This consumes huge amounts of energy, in particular, arising from the calcination of raw materials at around 1500 °C and the grinding of raw materials, cement clinker and gypsum [1]. The energy demand associated with Portland cement (PC) production is about 1700–1800 MJ/tonne clinker [2], which is the third largest use of energy, after that of the aluminium and steel manufacturing industries [3]. In addition, nearly 0.85 tonnes of CO_2 are released to the atmosphere per tonne of PC clinker produced. The world cement production accounts for approximately 7% of the 23,000 million tonnes of CO_2 produced annually by human activities [4].

In recent years, researchers have been trying to use different types of by-products, such as pulverized fuel ash (PFA) and ground granulated blast-furnace slag (hereafter referred to as slag), as partial replacements of PC in order to reduce the energy consumption and CO_2 emission associated with the concrete industry. However, despite this use, the vast majority of slag is still disposed of in

landfills [5,6] due to the reduction of early age compressive strength when PC is replaced with slag.

Instead of partially replacing PC with these industrial by-products, thereby indirectly activating the by-products by calcium hydroxide generated by PC hydration along with the alkalis and gypsum in PC, the by-products like PFA and slag can also be directly activated with chemical activators. Much attention has been given to alkali-activated slag (AAS) systems [6–17], including their mechanisms of hydration, hydration products, the microstructure and activators [18–24]. It has generally been agreed that the strength development of AAS depends on the type and dosage of alkali activator, fineness of the slag and the temperature of curing [25,26]. Wang et al. [21] found that the optimum amount of activator dosage, in terms of Na_2O by the mass of slag, varies from 3% to 5.5% and using waterglass with a silica modulus of 1–1.5 leads to higher mechanical strengths. Compressive strengths of 60 and 150 MPa for slag concretes activated by waterglass were achieved without heat treatment or special additives [27,28]. They also found that using powdered waterglass leads to lower strength when compared to the liquid form. Fernández-Jiménez et al. [29] also reported that the optimum concentration of the alkaline activator varies between 3% and 5% of Na_2O by slag mass and using a Na_2O amount above these limits would result in cost inefficient mixtures as well as problems with efflorescence. As a function of the strength obtained, the alkali activators used in their work maintained the following sequence: $\text{Na}_2\text{SiO}_3 \cdot n\text{H}_2\text{O} + \text{NaOH} \gg \text{Na}_2\text{CO}_3 > \text{NaOH}$. Similar findings were reported by Bakharev et al.

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[30] and Atiş et al. [31]. Krizan and Zivanovic [32] studied the early hydration of alkali-slag cements activated with waterglass at different n moduli (0.6, 0.9, 1.2 and 1.5) and sodium metasilicate ($\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$) in solution at 25 °C using isothermal conduction calorimetry. The results indicated that the cumulative heat of hydration increased with increasing modulus and dosage of waterglass. Furthermore, the compressive strength of the slag activated with waterglass of higher moduli (up to 1.5) gave higher strengths.

From the above review of the literature, it can be seen that, whilst the most commonly used activators are sodium silicate, sodium hydroxide, sodium carbonate or a mixture of sodium–potassium hydroxide (NaOH, KOH) with sodium silicate (waterglass) – potassium silicate, the mixture of sodium hydroxide with waterglass has been generally agreed to be the most effective activator and provides the best formulation for high strength and other advantageous properties. However, both the sodium hydroxide and sodium silicate do not exist naturally and they are obtained from energy intensive manufacturing processes. This is particularly the case for sodium silicate, which is produced by melting sand and sodium carbonate at 1350–1450 °C, followed by dissolving in an autoclave at 140–160 °C under suitable steam pressure [33]. As a result, even though AAS could potentially be considered as a low energy and low carbon cement system, activation with sodium silicate and sodium hydroxide may not be the best option for achieving environmentally friendly cementitious systems. As highlighted by Habert et al. [34], the objective of using alkali-activated cementitious systems is to reduce the negative effects of construction on the environment, which at present is not achieved with the use of both sodium silicate and sodium hydroxide.

Unlike sodium silicate and sodium hydroxide, sodium sulfate (Na_2SO_4) can be obtained either from natural occurring sodium-sulfate-bearing brines, crystalline evaporate deposits or as a by-product during the manufacture of various products such as viscose rayon, hydrochloric acid and silica pigments. Thus, by using sodium sulfate as an activator, a more environmentally friendly cementitious system could be formulated. Furthermore, compared to other alkaline activators, sodium sulfate is usually less expensive and less harmful. However, surprisingly, compared to other activators, there have been very limited studies addressing the activation of slag with sodium sulfate. One possible reason for its low popularity could be due to its low early strength. As reported by Wang et al. [21] the early compressive strength of AAS mortars activated with sodium sulfate is lower than other activators such as Na_2CO_3 , NaOH and waterglass. The recorded compressive strengths of the mortars activated with 2 M Na_2SO_4 were 1.2, 5.1, 10.2 and 20 MPa at the ages of 1, 3, 7 and 28 days, respectively. These results of compressive strength were lower than those obtained from other activators, in particular the early age strength. However, recent work carried out by Rashad et al. [35] indicated that Na_2SO_4 activated slag has a better resistance than PC systems to the degradation caused by exposure to elevated temperature up to 600 °C [35]. Sodium sulfate activated slag has also been considered as a potential binder for dealing with some nuclear wastes containing reactive metals due to its reduced pH, reduced heat generated from hydration compared to PC-based blends and less free water content due to the formation of ettringite [35,36]. Nonetheless, to satisfy the requirements of the construction industry, its low early strength must be addressed. Although, by adding alkaline additives, such as lime and PC clinker, the early strength of Na_2SO_4 activated slag could be improved [21], it may again increase the embodied energy consumption and CO_2 emission. On the other hand, the addition of alkaline additives would increase the pH of the activated slag. Although the increased pH would be preferred for construction industry due to the enhanced stability of the reinforcing steel, it may cause concerns in some nuclear waste immobilization applications, as the increased pH could lead to the

reactions between activated slag with certain metallic wastes, such as aluminium, causing expansive corrosion of metals and excessive generation of hydrogen, along with attacks on zeolites and glass which may exist in certain waste forms [37].

It is well-established that by increasing the fineness of the materials, the reactivity can be enhanced due to the increased surface area and surface energy. Therefore, the primary objective of this study was to investigate the effect of the fineness of slag and the dosage of sodium sulfate on the hydration, pH and compressive strength of sodium sulfate activated slag in an attempt to increase its early-age strength without increasing the pH. In addition, it is expected that an improved understanding on the hydration mechanism of sodium sulfate activated slag system could be achieved.

2. Experimental

2.1. Materials

Industrial grade of Na_2SO_4 was used as an activator. The slag supplied by UK Hanson Cement was used as the raw material to produce the activated slag paste. Its chemical composition was characterised by X-ray fluorescence (XRF) spectrometry. Two degrees of slag fineness with specific surface areas of, 2500 and 5000 cm^2/g were used. Portland cement, CEMI Class 42.5R, with a Blaine surface area of 2700 cm^2/g (complies with BS EN 197-1:2001), was used as a reference. The corresponding oxide contents of the slag and PC, calculated from elemental compositions determined by XRF, are shown in Table 1.

2.2. Mixture proportions

Four activated slag pastes and a control PC paste were prepared. The activated slag pastes were formulated with two different dosages of sodium sulfate (1% and 3% Na_2O equivalent of Na_2SO_4 by the mass of slag) and the slag with two different finenesses. All mixing proportions are detailed in Table 2. A water binder ratio (w/b) of 0.3 was fixed for all mixtures.

2.3. Method

The slag was dried for 6 h in a special dryer at 40 ± 1 °C before mixing. The sodium sulfate solution was prepared and used at 40 ± 1 °C. The slag was added to the sodium sulfate solution that has been previously placed in the bowl of a Hobart planetary mixer, over a 5 min period after which time the mixer was stopped and any unmixed powders was scraped from the sides and the paddle into the mixing bowl. Mixing was then continued for 5 min before casting into $25 \times 25 \times 25$ mm moulds and vibrating for 1 min to remove air bubbles. The same mixing regime was used for the PC

Table 1
Oxide composition of PC and slag (% by mass) after calculation from XRF results.

Composition	PC	Slag
CaO	63.47	40.89
SiO ₂	20.18	34.94
Al ₂ O ₃	4.83	11.69
MgO	2.47	7.42
Fe ₂ O ₃	3.16	3.32
SO ₃	3.26	1.19
K ₂ O	0.52	0.34
Na ₂ O	0.16	0.16
TiO ₂	0.3	0.49
MnO	0.22	0.27
P ₂ O ₅	0.09	0.01
Loss of ignition	2.18	−0.93
Total	100.34	100.71

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