



Influence of mechanical and chemical activation on the hydraulic properties of gamma dicalcium silicate



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ABSTRACT

The aim of this work is to clarify the effect of various activation methods on the hydraulic properties of γ -C₂S, one of the main phases in stainless steel slags. Synthetic C₂S, sintered at 1450 °C for 20 h, consisted of 88 wt.% γ -C₂S and 12 wt.% β -C₂S. Two activation paths were followed: mechanical activation, by means of high energy milling, and chemical activation, by means of NaOH and Na₂CO₃ addition. Results indicated that both mechanically and chemically activated γ -C₂S hydrated at ambient temperature. XRD analysis showed that the amount of amorphous phase increased during hydration, whereas TGA/DTG data revealed weight loss in the decomposition region of C–S–H. Analyses by FTIR and SEM also support that C–S–H is one of the main hydration products. Mortars prepared with mechanically and chemically activated γ -C₂S developed mechanical strength, reaching the values of 9.1 MPa and 10.3 MPa at 90 days, respectively.

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1. Introduction

The production of stainless steel is reported to be one of the fastest-growing sectors of the manufacturing industry globally [1]. In the steelmaking process, slags are used to prevent oxidation of the steel through contact with air, to limit heat losses through radiation and to remove impurities from the molten steel [2]. Approximately one tonne of stainless steel slag is generated per three tonnes of stainless steel [3].

Stainless steel slags are generated in the Electric Arc Furnace (EAF) operation as well as in the Argon Oxygen Decarburisation (AOD) and Ladle Metallurgy (LM) processes. Unlike EAF slag, AOD and LM slags consist mainly of Ca, Si and Mg with very low amounts of Cr (<1 wt.% in Cr₂O₃). The main minerals typically found in these slags are dicalcium silicate (Ca₂SiO₄), merwinite (Ca₃MgSi₂O₈), bredigite (Ca₇MgSi₄O₁₆) and periclase (MgO) [4]. The conventional method for stainless steel slag management is landfilling. However, this should only be a temporary solution [5]. If the stainless steel slags are valorised, they are typically used as aggregate and fertilizer [6]. Some higher value applications, such as the use of stainless steel slag as a hydraulic binder, have also been proposed [7–10].

Dicalcium silicate (C₂S) occurs in several polymorphic forms: α , α' , α'_H , α'_L , β and γ [11–15]. Most of them are stable in the pure state only at

elevated temperatures and it is merely the γ form that is thermodynamically stable at room temperature [13,16]. Each polymorph shows different reactivity with water. The α and α' phases stabilised by Al₂O₃, Na₂O, MgO and K₂O show considerable reactivity with water [13]. The β -C₂S, which is stabilised usually in the presence of some impurity ions, is one of the principal components of Portland cement clinker [17,18]. It is known as belite in cement chemistry, and compared to the other polymorphs it reacts rapidly with water [19].

The γ polymorph forms at temperatures below 500 °C through the $\beta \rightarrow \gamma$ conversion [17]. This conversion involves a rotation of the SiO₄ tetrahedra and large movements of calcium atoms [20] with an accompanied volume increase of 12% causing a break up into small particles, a phenomenon known as “dusting” [17,21,22]. It is known that the $\beta \rightarrow \gamma$ conversion can happen during or long after the cooling [22], as well as that it can be stress-induced e.g. by grinding [17]. It is also known that the $\beta \rightarrow \gamma$ conversion can be suppressed by rapid cooling and/or by addition of suitable dopant ions such as boron [7].

Statements on the reactivity of the γ polymorph of C₂S in the presence of water vary between “non hydraulic” and “essentially non-hydraulic” [12,18,23,24] to “slowly hydraulic” [13,25]. As a result, most of the studies focus on the prevention of β to γ transformation [26] and the ions that can stabilise β , rather than the improvement of γ -C₂S hydraulic properties. Nonetheless, some papers have already addressed this issue. Trettin et al. [27], studying the mechanism of γ -C₂S hydration, suggested that γ -C₂S hydrated via a topochemical mechanism, similarly to C₃S. This is contradicting an earlier study by Ghosh et al., who suggested its hydration via a “through solution” mechanism [13]. With

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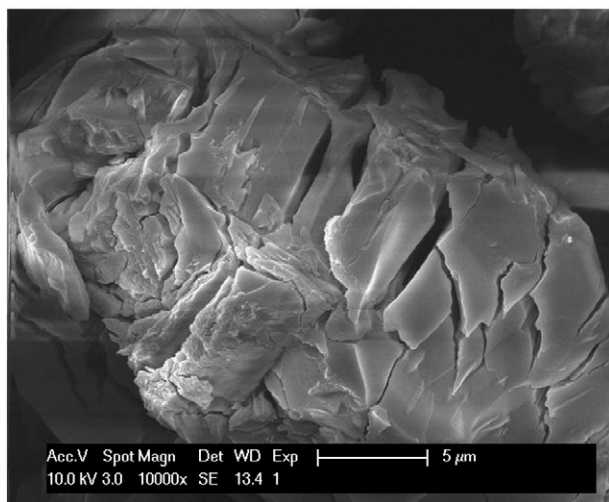


Fig. 1. SEM image of the original γ -C₂S powder.

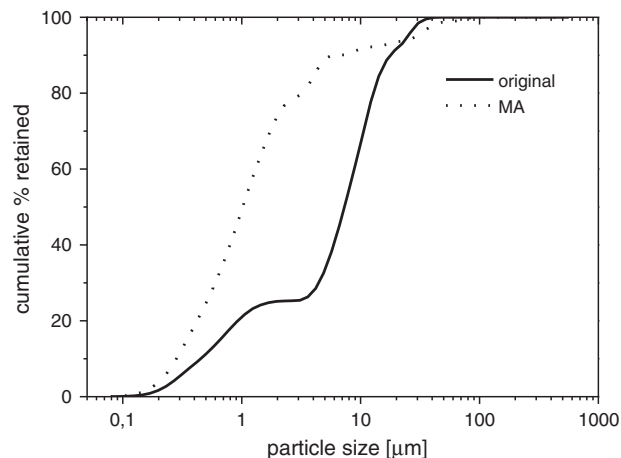


Fig. 2. Particle size distribution of the original and mechanically activated (MA) γ -C₂S powder.

respect to the hydraulic properties of γ -C₂S, it has been mentioned [28] that they could be improved with unspecified accelerating admixtures. A significant rate of γ -C₂S hydration at elevated temperature in the presence of saturated steam was mentioned for instance by Ghosh et al. [13].

Despite the slow hydration kinetics of γ -C₂S in water, the reactivity of this phase could be enhanced through the action of strong alkalis that stimulate its dissolution and result in the formation of binding phases. This method, known as alkali activation, has been receiving substantial attention for a wide range of materials [29], stainless steel slags included. Salman et al. [10] reported that the simultaneous alkali and thermal activation of γ -C₂S containing stainless steel slags led to C–S–H type hydration products and strength development. Improved cementitious properties of γ -C₂S containing steel ladle slag after alkali activation at room temperature were also reported by Shi and Hu [4,30]. On the other hand, mechanical activation applied on various stainless steel slags was also found to enhance the hydraulic properties of γ -C₂S [31]. Still, these works were performed on multiphase (stainless) steel slags and no research on the potential influence of activation processes has been found with respect to pure γ -C₂S. This issue is discussed in the present work, where the hydraulic properties of synthetic γ -C₂S were studied through activation methods, such as mechanical and chemical activation, with the aim to provide a better understanding on the hydration of γ -C₂S and to find a suitable activation method which could be eventually applied on the stainless steel slags. The combination of mechanical and chemical activation is not included in this work. These two processes have been explored in a sequential scheme elsewhere [32] and it was found that the overall effect on hydration was only minimal. The idea of exploring these two activation processes concurrently has not been investigated and it may be an interesting follow up of this work.

Table 1
QXRD analysis of the mechanically activated γ -C₂S.

	Non-hydrated	3d	7d	28d
γ -C ₂ S	49 ± 8	24	17	16
β -C ₂ S	13 ± 1	2	<1	<1
Portlandite		1	1	2
Calcite		1	1	2
Tobermorite		2	5	5
Others/amorphous	37 ± 8	70	75	75

2. Materials and methods

Synthetic γ -C₂S was prepared from analytical grade CaCO₃ and SiO₂. The compounds were mixed and homogenised as an ethanol suspension for at least 12 h (Turbula T2C). After drying, the mixture was sintered in a platinum crucible using a bottom loading furnace (AGNI ELT 160–02). The sintering consisted of 3 steps: heating at 5 °C/min, holding at 1450 °C for 20 h and slow cooling at 1 °C/min. The produced material was milled and sieved below 125 μ m. Mechanical activation (MA) was performed by additional milling in a high energy bead mill (Dispermat SL-12-C1, VMA). Approximately 100 g of powder was milled in 500 ml of ethanol at 5000 rpm for 6 h using 1 mm ZrO₂ milling balls.

The particle size distribution was measured using the laser scattering method (MasterSizer Micro Plus, Malvern) and the surface area was determined by the nitrogen sorption method BET (Micromeritics TriStar 3000 V6.04 A) after drying at 150 °C for 12 h. The mineralogy was determined by X-ray diffraction analysis (Philips, PW 1830) using CuK α radiation of 45 kV and 30 mA. The X-ray patterns were collected with a step size of 0.02° and step time 2 s. Quantitative XRD results were obtained adopting the Rietveld method [33,34]. ZnO was used as an internal standard to determine the amorphous content.

Isothermal calorimetry, carried out at 20 °C, was used to study the early hydration (TAM Air device, TA Instruments). For this purpose, the γ -C₂S powder was mixed with water keeping the liquid to binder (l/b) mass ratio equal to 1. In the case of alkali activation, an alkali

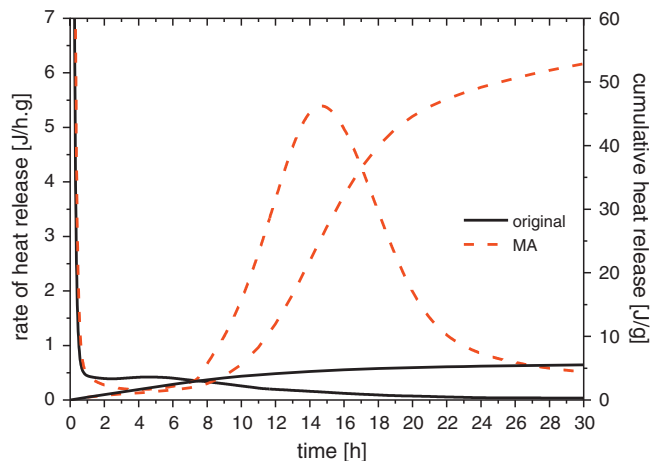


Fig. 3. Calorimetry measurements of the original and mechanically activated (MA) γ -C₂S sample.

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