



Impact of superplasticizer on the hardening of slag Portland cement blended with red mud



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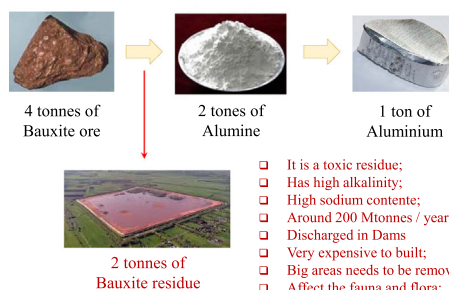
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HIGHLIGHTS

- The association of BR and cement may be an alternative to a large-scale application.
- BR improve the cohesion of suspensions, needing more water content to mix.
- A good particle dispersion (using superplasticizer) is need.
- The use of superplasticizer affects the hardening in a different way of BR.
- So, the system BR–cement–superplasticizer needs to be evaluated.

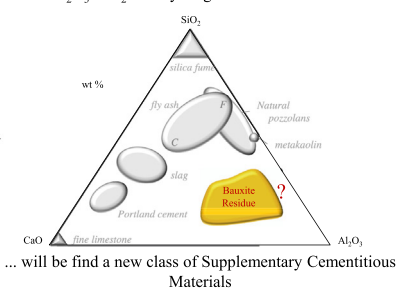
GRAPHICAL ABSTRACT

- Primary Aluminium is obtained from the bauxite ore;



- If confirmed its association with cement...

CaO–Al₂O₃–SiO₂ ternary diagram of cementitious materials



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ABSTRACT

Although the use of slag Portland cement has many benefits, its chemical reaction is slow in comparison with binders of high clinker content. At the same time, the search for uses of bauxite residue (BR), or red mud as is commonly known, in cementitious compositions has been quickening in recent years due to the need for large-scale applications of this waste. It is known that the interaction Portland cement–BR, improves certain hardened-state properties, but its transition from fluid-to-solid behavior is still not well-understood and has great practical importance. By associating slag Portland cement and BR, the large amount of sodium content in the BR can help the slag reaction, which changes rheological properties over time. Therefore, in this work, superplasticizers, based on sulfonated melamine and/or sodium polycarboxylate molecules were used to minimize these effects, and their influence on the fluid-to-solid transition behavior was monitored through isothermal conduction calorimetry and oscillatory rheometry. The results showed that the BR delays the induction period, increases the reaction rate in the acceleration period, and accentuates the Aft formation and particles' agglomeration. It was also found that sodium polycarboxylate influences hardening significantly, delaying hydration reactions and stabilizing particles in suspension for a longer period of time.

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1. Introduction

The hardening of cement pastes occurs due to two different yet complementary factors: coagulation/flocculation and hydration

reactions [1–3]. While the reactions refer to the chemical contribution of the process and can be determined as a function of heat release, the coagulation/flocculation represents the physical contribution and can be compared continuously by means of a rheological property: the storage modulus (G') variation.

In this way, different correlations between the agglomeration and chemical reactions can be observed, depending on the physical

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and chemical characteristics of the powder. The coagulation/flocculation forces are intensified over time due to hydration reactions, resulting in an increase in ionic concentration and formation of hydrated compounds.

When bauxite residue is used to partially substitute for a portion of Portland cement, the mean distance among particles as well as their mobility are reduced. The tendency for agglomeration and the particle specific surface area are increased, and the particle surface charge changes [1,3]. This can bring about an unfavorable effect on its rheological properties, due to an increase on the kneading water demand. Thus, works in which the consistency is kept constant, commonly use a higher quantity of water for the mix, even though this can affect the hardening properties, performance and durability of the cementitious materials in use [4].

In order to find an alternative to reduce the water demand in the mix without changing the rheological properties of the suspensions or the kinetics of the cement hydration reaction, the residue calcination was evaluated [4]. However, despite the sodium content decrease from 13% to 7% in the suspension, it was observed that this process did not reduce the water content required: even after lowering the BR specific surface area, the electrophoretic mobility of particles was also changed with the thermal treatment, resulting in lower suspension stability. In addition, when sodium aluminate $[\text{NaAl}(\text{OH})_4]$ comes into contact with Ca^{2+} -ions from cement dissociation, calcium aluminate precipitation begins, and accelerates the cement hydration reaction.

Other alternatives, presented in this work, include the use of a superplasticizer and blast furnace slag cement. While the former may improve the particle stabilization, the use of this kind of cement may fix the free Na^+ from BR due to a reaction with blast furnace slag (BFS) [5,6].

Thus, this is not an innovative strategy. Pourcher et al. [5] had already evaluated the influence of three types of superplasticizers on the C_3A hydration in the presence of gypsum. They observed that polycarboxylate and naphthalene based admixtures retard the ettringite formation due to the deceleration of the C_3A dissolution rate. Winnefeld and Figi [6] and Zingg et al. [7] also evaluated the interaction between polycarboxylate-based superplasticizer and cements comparing how the polymer structure affects the stability of cements with different C_3A content.

Lee et al. [8] studied composites with blast furnace slag and red mud as a substitute for cement and observed that the fluidity and the mechanical properties are more dependent on the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio than the water/binder ratio. Pan et al. [9], on the other hand, evaluated a new kind of alkali-slag-red mud cementitious material and found that hardened cement paste mostly consisted of C-S-H gel, being very low in the Ca/Si ratio, very fine in size and extremely irregular in shape. Portlandite, Aft or zeolite products have not been detected, and these characteristics are considered the main chemical reasons for the high early and ultimate strength and good resistance to chemical attacks.

Wu [10] evaluated the early age activation of BFS and blends of slag and Portland cement (OPC) using different kinds of sodium compounds as activators. The authors also used a superplasticizer based on sulfonated naphthalene formaldehyde to reduce the water demand and observed higher strengths on the activated BFS-cement mortars than on the OPC, reduction in the setting time and that the effect of blends of additives is better than that of individual ones. Therefore, it is clear that the use of superplasticizer or BSF alkali-activation is not new; but the evaluation of its conjoined contribution to the hardening has still been poorly investigated.

The main purpose of this work was monitoring the changes promoted by the use of different superplasticizers based on sulfonated melamine or polycarboxylate molecules, during early hydration, reactions of blast furnace slag cement blended with bauxite

residue. Isothermal calorimetry, X-ray diffraction and oscillatory rheometry were used to evaluate, respectively, the heat release during the chemical reaction, the formation of mineralogical phases and the rate of particle agglomeration.

2. Experiments

2.1. Physico-chemical characterization of raw materials

2.1.1. Chemical analysis

The determination of chemical composition was performed according to ASTM C114/05 – Standard Test method for the Chemical Analysis of Hydraulic Cement, and NBR 13810/97 – Determination of metals - Guidelines for Atomic Absorption spectroscopy [11].

2.1.2. Laser granulometry

A Malvern Mastersizer S long bed, ver 2.19, with a detection range from 0.05 to 555 μm , was used to perform the particle size distribution.

2.1.3. Helium picnometry

The real powder density was determined using a Quantachrome MVP 5DC Multipicnometer.

2.1.4. Specific surface area (SSA)

This analysis was performed according to the BET method (Braunauer, Emmet and Teller) using a Gemini 2375 Micromeritics machine. Samples were pre-treated at 60 °C and 100 mmHg pressure for 24 h.

2.2. Paste characterizations

2.2.1. Isothermal conduction calorimetry

Heat flow during the hydration reactions was monitored in a TAMAir calorimeter of TA Instruments, maintaining the temperature at 23 °C for 48 h. The powder was added in a glass ampoule, water was injected with a syringe and the suspension was mixed manually for 1 min. All tests started 1.5 min after mixing. The characteristic heat flow of cement is illustrated in Fig. 1, linked with the steps used to compare results [12,13].

In the first stage, the wetting period, occurs the initial dissolution/precipitation of hydrated compounds. In the second stage, the polymerization of hydrated calcium silicate monomers (i.e., C-S-H gel) begins, precipitating a layer around the cement particles, which apparently delays the hydration reactions. During this period, called induction, there is a gradual increase of Ca^{2+} , K^+ , Na^+ , SO_4^{2-} , OH^- ions and silicate ($\text{H}_2\text{SiO}_4^{2-}$), which commonly extends up to 3 h depending on the cement mixture. After oversaturation of calcium ions in the solution has occurred, the third stage begins, accelerating the portlandite (CH) and C-S-H precipitation and ettringite; this is called the reaction acceleration period [1,12,13]. In this work, as it will be presented follows, this was true in the solutions of pure Portland cement, but when the bauxite residue was used, the formation of CH and C-S-H was lower.

2.2.2. X-ray diffraction

X-ray diffraction patterns were obtained using a PANalytical Empyrean diffractometer (PIXcel^{3D} detector) with Cu K α radiation ($\lambda = 1.54 \text{ \AA}$), a divergence slit size of 0.5°, a nickel filter and a spinning frequency of 0.5 rps. All scans were measured over an angular range from 5° to 70° (2θ) with a 0.017° step size; the accumulated time per step was set to 60 s. Because all diffractograms were obtained in the same conditions, the mineralogical phase contents as a function of time to cure was determined by comparing the intensity of the observed peaks (counts). The hydration reaction was stopped after 30 min, 1 h, 2 h, 4 h, 8 h and 24 h of cure by freeze-drying immersing the samples in liquid nitrogen (−186 °C) for 2 min. The adsorbed and interlamellar frozen water was removed by sublimation in a Terroni Fauvel freeze-dryer, model LC1500. Once dried, the samples were ground, sieved through a 75 μm -aperture mesh and stored in a desiccator with silica gel under vacuum until each test.

2.2.3. Oscillatory rheometry

Tests were performed in an AR550 rheometer, TA Instruments, with parallel plate geometry of 40 mm and a gap of 1000 μm , as shown in Fig. 2. The pastes were mixed for 1 min at 900 rpm in a RW 20 mixer (IKA, Labortechnik). After homogenization, the particles were dispersed in an IKA T25 Basic, a high energy mixer, at 9500 rpm for 1.5 min. Paste consolidation was monitored for 4 h at a constant frequency of 1 Hz and a strain of 10^{-4} . During the tests, a solvent trap was used to preserve the humidity. A pre-shear of 200 s^{-1} was applied for 1 min before all tests.

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