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Influence of the activator concentration and calcium hydroxide addition on the properties of alkali-activated porcelain stoneware



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HIGHLIGHTS

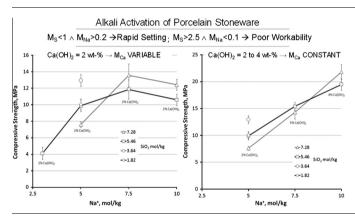
- A new added value reuse application for waste material is demonstrated.
- Keeping the modulus of solution at 1–2.5 is highly recommended in PS mortars.
- The calcium to sodium molar ratio (M_{Ca}) determines the setting time of PS mortars.
- Strength increased linearly with sodium for a constant amount of SiO_2 and $M_{Ca.}$
- Compressive strengths of 36 MPa were obtained in mortars cured at 65 °C for 7 days.

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G R A P H I C A L A B S T R A C T



ABSTRACT

Objective: The aim of this research was to investigate the influence of the alkali activator concentration and calcium dosage on the fresh behavior, compressive strength and microstructure of the binder produced by the alkali activation of porcelain stoneware waste, using NaOH and sodium silicate solution as activators.

Materials: Porcelain stoneware waste, NaOH and sodium silicate.

Method: Tested properties of mortars were the compressive strength and the setting times. Thermogravimetric analyses, microscopic studies, pH measurements, XRD and FTIR tests were used to assess the evolution of the microstructure developed.

Results: The results show that the activator concentration and calcium to sodium molar ratio strongly influence the fresh behavior and the hardening process of mortars. A linear evolution of compressive strength with sodium addition was observed when keeping the SiO_2 concentration constant. Mortar samples with up to 36 MPa under compression were obtained after curing for 7 days at 65 °C.

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1. Introduction

Manufacturing Portland cement (PC) is a resource-exhausting, energy-intensive process that releases huge amounts of greenhouse gas CO_2 into the atmosphere. Indeed, the production of 1 ton of Portland cement requires about 2.8 tons of raw materials, including fuel and other constituents [1]. Efforts have been made

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by the scientific community and the PC industry to reduce this impact. Among the different alternatives proposed, alkali-activated cementitious materials, produced from an alumino–silicate precursor activated in a high alkali solution, have attracted increasing attention as a viable solution to reuse and recycle industrial solid wastes and by-products [2]. When compared to Portland cement, the mortar and concrete made from the alkali activation process has its advantages, such as higher strength (when properly designed), low cost, excellent durability, low energy consumption and reduced greenhouse gas emissions [3–5]. Furthermore, one of the most advantageous aspects it offers is that it allows the development of binders to be made entirely, or almost entirely, from waste materials [6].

Generally, materials containing amorphous silica and alumina can be activated by alkalis. As reviewed by Li et al. [5], two models have been established for alkali-activated binding systems. The first is the case of blast furnace slag activation (GGBFS, Si + Ca) with mild alkaline solutions, where calcium silicate hydrate is the main reaction product. The general example for the second alkali activation model is metakaolin (MK) or Class F fly ash, (Si + Al), with medium to high alkaline solutions, giving rise to zeolite-like geopolymers. The stages that take place to form the alkaline silico aluminate network in the latter system are summarized in [7,8]. Initially, the dissolution of solid species occurs, and the OH⁻ ions in the alkaline medium sever the covalent Si-O-Si, Si-O-Al and Al-O-Al bonds present in the amorphous phase of the solid material. Silicon and aluminum ions are released into the medium, where they form Si-OH and Al-OH groups. This stage is followed by the reorganization and formation of small nuclei that later polycondensate to form an alkaline aluminosilicate gel characterized by its three-dimensional structure. As pointed out by Yip and van Deventer [9], the main difference between the hydration product from GGBFS activation (Case 1) and that from fly ash activation (Case 2) is calcium. Although this element is not essential in any part of a basic geopolymeric structure, it is generally accepted that if sufficient calcium is added to the system, a C-(A)-S-H or (C,N)-A–S–H-based cementitious material may form instead [10–12]. This fact was proved by Alonso and Palomo [7], who mixed highly pure metakaolin with different Ca(OH)₂ solid contents (30–50%). Although the main reaction product of alkaline activation is an amorphous aluminosilicate with a tridimensional network and cementitious properties, addition of calcium hydroxide to the system has been found to provoke the formation of a C-S-H gel type as a secondary reaction product [7]. Moreover, several studies have shown that the addition of moderate amounts of calcium has a positive effect on the mechanical properties of alkali-activated binders [9,10,13].

A large number of minerals and by-products have been alkaliactivated, of which metakaolin [7,13,14], fly ash [1,8,15,16] and ground granulated blast furnace slag [5] have been the most extensively analyzed. Increasing interest in the alkali activation process has led the scientific community to identify other suitable waste materials for the alkali activation process. Among the experimental research performed, Lee et al. [17] combined three different industrial by-products (fly ash, slag and bottom ash) to develop a new low-strength material using NaOH as an activator. He et al. [2] also combined rice husk ash with the major waste by-product that originates when extracting alumina from bauxite ores (red mud) to develop alkali-activated pastes with compressive strengths of up to 20.5 MPa after 49 curing days (room temperature). In the work by Yusuf et al. [18], 28-day compressive strength values of 44 MPa were achieved through the activation of palm oil fuel ash and ground blast-furnace slag combinations with sodium silicate and sodium hydroxide. In the study by Kourti et al. [19], air pollution control residues and glass-forming additives were mixed to obtain a glass using DC plasma technology. High compressive strengths (~130 MPa) were achieved by the alkali activation of this high calcium alumino-silicate glass with a sodium hydroxide solution. Similarly, Sun et al. [20] reported high compressive strength values (a maximum of 71.1 MPa in samples cured for 28 days) and good high-temperature properties (higher strength after heat treatment of 1000 °C) in ceramic waste activated with alkali hydroxides and sodium/potassium silicate solutions. Ceramic materials were also activated in the work by Khater [21], where 23 MPa were achieved under compression in pastes developed by the alkali activation of concrete and demolished walls after 90 days of curing at 40 °C. Hydrated-carbonated Portland cement was also activated by Payá et al. [4], where mortars with compressive strengths over 10 MPa were obtained after curing at 65 °C for 3 days.

Different types of ceramic tiles [22,23] have also been successfully activated using NaOH and sodium silicate solutions. These products are widely used in construction in Spain and, despite the Spanish production rate having lowered by almost 40% from 2006 to 2010 (608 and 366 million m², respectively), it still ranked seventh in 2010, with 3.8% of the total world production [24]. As explained by Puertas et al. [25], although ceramic tiles can be reused for the tile manufacturing process, the cost of the milling required for that purpose is not compensated by the savings generated in raw materials. Ceramic materials have also been used as cement admixtures [25,26] and concrete aggregates [27]. However, only a portion (usually 10–35%) of cement is replaced in these applications.

In the work by Reig et al. [23], porcelain stoneware tile (PS) wastes were activated, and mortars with compressive strengths close to 30 MPa were obtained after 7 curing days at 65 °C. Similarly to the previous work by Granizo et al. [13], which analyzed the structure of metakaolin and Ca(OH)₂ mixes (1:1), a major influence of Ca(OH)₂ addition was noticed in [23]. So, while no activation took place in the absence of calcium hydroxide, rapid setting occurred when 5% of PS was replaced with this material. However, no correlation between the rapid setting of mortars and alkaline concentration or calcium addition was observed.

The study discussed herein is actually a logical extension of the previous work presented in [23], and it aims to understand the influence of the alkali activator concentration and calcium dosage on the fresh behavior and properties of the hardened binder developed by the alkali activation process.

2. Experimental

2.1. Materials

Porcelain stoneware (PS) tiles were broken with a hammer, crushed in a jaw crusher (BB200, Retsch) and dry milled in alumina media for 30 min. Calcium hydroxide (93% purity) was used as a partial replacement of ceramic waste. Sodium hydroxide pellets (98% purity, Panreac), water and sodium silicate (Merck, SiO₂ = 28%, Na₂O = 8%, H₂O = 64%) were used to prepare alkali-activating solutions.

After the milling process, dense irregular particles were obtained. The mean particle diameter, determined through laser granulometry (Mastersizer 2000, by Malvern instruments), was close to 21 μ m, with a d_{90} of 53 μ m (90 vol% with this size). The chemical composition, determined by X-ray fluorescence (XRF), was similar to that presented by metakaolin [5], with SiO₂ (71 wt%) and Al₂O₃ (19.3 wt%) being the major constituents. The other components, such as Na₂O (4.7 wt%), CaO (0.5 wt%) or Fe₂O₃ (1.1 wt%), were not significantly detected.

The amorphous content, determined according to UNE EN 196–2 specifications, was around 46%. The obtained results are consistent with the study by Zanelli et al. [28], where 93 porcelain stoneware samples were analysed, being composed mainly of an amorphous phase (40–80 wt%) containing dispersed crystalline components, such as quartz (10–30 wt%), mullite (4–10 wt%) and feldespars (\approx 15 wt%). As reported by García Ten [29], the amorphous phases in PS come about when sintering tiles (1190–1220 °C) due to the partial fusion of the crystalline phases. As explained by Li et al. [5], the term 'amorphous phase' is more appropriate for PS tiles than 'glassy' because, similarly to MK, the disordered phase is generated when breaking down the crystalline structure while sintering, which generally occurs at temperatures below those required to generate a liquid phase. In contrast, the glassy phase of GGBFS and fly ash is formed by melting and rapid cooling.

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