



Inexpensive intumescent alkali-activated natural pozzolan pastes

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Received 14 January 2015; received in revised form 7 March 2015; accepted 15 March 2015

Abstract

Perlite, a natural pozzolan, was activated with NaOH to prepare alkali-activated (AA) intumescent pastes. After curing at 100 °C for 3–24 h, the solidified pastes had medium strengths, up to 40 MPa, and low densities, ~1500–2000 kg/m³, compared to Portland cement pastes. Upon heating, volume expansion in the 200–600 °C range resulted in a porous solid. Expansion occurs due to a loss of silanol condensation water, as vapor. Chemical composition of the pozzolan, activator content, and heat-treatment temperature influence the size and amount of pores formed. Mechanical, physical, and microscopic investigations were made. Volume increases up to 225% were measured. Densities decreased to as low as ~450 kg/m³ after heat treatment and cooling. Strengths of porous products were in the 3–7 MPa range. Such materials able to intumesce over specific temperature ranges could be useful in applications where absorption of thermal energy is necessary, such as passive fire protection.

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Keywords: Alkali activation; Porous; Intumescent; Natural pozzolan; Sodium hydroxide

1. Introduction

Porous materials are used in many industrial processes owing to their wide range of properties such as low density, high surface area, low thermal conductivity, high energy absorption, etc. [1–3]. Recently, much research effort has been dedicated to preparing inexpensive porous materials that use common raw materials and that require less energy to process than ceramics. One such method is to introduce porosity into alkali-activated aluminosiliceous materials (AAMs). Some AAMs are also referred to as geopolymers [4–6]. AAMs have been attracting interest mainly as potential alternatives to portland cement binder systems, due mainly to their possibly low CO₂-footprints [7]. Some formulations can also have superior chemical or thermal durability [8–11]. They are typically produced at or slightly above room temperature using alkaline activators and a mostly amorphous aluminosiliceous raw material. The raw material can be natural such as volcanic glasses, artificial such as industrial wastes (slags, fly ash), or a combination (natural but processed) such as dehydroxilated (meta)kaolin [12–15]. The activator,

typically a concentrated solution of NaOH or KOH partly dissolves the raw material to form free SiO₄ and AlO₄ tetrahedral units. The dissolved Al and Si complexes diffuse from the particle surface to the interparticle space inducing the formation of a gel phase and, finally, the gel phase hardens [16,17]. Lightweight AAMs can be made by incorporating lightweight materials such as diatomaceous earth or expanded polystyrene beads into the mixture [18,19]. Porous AAMs of varying densities and pore sizes however, are typically produced using foaming or blowing agents [20,21]. Inorganic blowing agents, like H₂O₂ or Al powder, that evolve a gas are commonly reported [22–26]. In this approach, most of the porosity is formed prior to or shortly after setting and the pores are rather large (roughly mm-sized) and not connected [27]. Another recent method of pore forming is the use of elemental Si metal which oxidizes with water in alkaline environments at 50–100 °C to evolve H₂, forming pores [28–30]. These pores are also rather large [31]. Since Si or Al powder can be expensive, the use of silica fume as a porogen has been suggested. However, the amount of silica fume needed to get a significantly porous material can be rather high, 10–50% [28].

Properties of AAMs are strongly influenced by their Si/Al ratios. While Si/Al of 1–2 gives a product more like cement paste, higher ratios can give less rigid, more polymer-like

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products [6,32] reported for a mixture of aqueous silica, potassium oxide solution and silica powder with Si/Al ~ 27, cured at 80 °C, a mass loss of ~7% and a volume expansion of 400–500%, during heating from 250 to 625 °C. The final product was described as an open cell microcellular, amorphous product, with a pH of ~7.5. The pores were assumed to be formed due to dehydration and escape of gaseous H₂O. Oudadesse et al. [33] reported, for a similar mixture, a greater drop in pH with increasing Si/Al and the formation of >100 μm pores at lower temperatures followed by the formation of smaller pores in the walls of the large pores at higher temperatures. Fletcher et al. [34] prepared mixtures of metakaolin and a NaOH solution, and investigated the effect of increasing Si/Al (up to ~266) by adding silica fume on their behavior when exposed to high temperatures. “Bubble” formation was reported in the 100–225 °C range for mixtures with Si/Al > 20, and the onset temperature of this phenomenon was reported to drop with increasing Si/Al. The rate of growth of the bubbles and the temperature up to which the foaming continued were also related to Si/Al.

Perlite is a volcanic aluminosilicate, commonly used in its expanded form as a lightweight aggregate [35]. Owing to its glassy nature and its chemical composition, ground perlite is an effective natural pozzolan [36,37] and can also be activated with alkalis [38–41]. Si/Al for perlite suggests that upon alkaline activation, it can give a solid able to intumesce, i.e., expand when exposed to heat [42]. Being quite abundant, with a known total worldwide reserve base of ~7 billion tons [43], it can be an economical starting material to produce porous AAMs. In fact, Vaou and Panias [44] already reported on a NaOH-activated “foamy” perlite geopolymer but that paste was expanded while still in the fresh state using H₂O₂ hence despite a reported mass loss from 100 to 600 °C, a volume change was not reported. Since many other natural pozzolans have compositions similar to perlite, they may also be suitable for preparing intumescent solid or porous AAMs, without the use of foaming or blowing agents. This paper discusses a method of preparing inexpensive alkali-activated unexpanded ground perlite pastes that can expand upon exposure to temperatures > 100 °C, and properties of the obtained porous solids.

2. Materials and methods

2.1. Materials

Perlite used was obtained from a quarry in Erzincan, Turkey. Table 1 provides the chemical composition of the perlite used, as determined by X-ray fluorescence.

The perlite was ground in a laboratory ball mill to a Blaine fineness of ~4600 cm²/g. The specific gravity of the ground perlite was determined as 2.43. X-ray diffraction reveals that the perlite is mostly amorphous (>85%), containing some

Table 1
Oxide analysis of the perlite used (%).

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	Loss on ignition
71.1	13.0	1.6	1.6	0.5	1.6	3.8	4.2	3.2

aluminum- and silicon-bearing crystalline phases (anorthite and quartz, and a small amount of mullite). The NaOH used (Merck, USA) was in solid pellet form with >97% purity but was used after dissolving in distilled water to obtain the desired solution concentrations.

2.2. Methods

Three different NaOH solution concentrations were used: 4 M, 8 M, and 16 M. The NaOH solution (S) was mixed at room temperature with the ground perlite (P) at a mass ratio of 1:2. Since S/P (ratio by mass) was kept constant, the water-to-perlite mass ratio changed from 0.28 for the 16 M sample to 0.43 for the 4 M sample. The mixing duration was 2 min. The paste obtained was cast into cubic molds with 5-cm sides. The molds were placed into a dry oven at 100 °C for the necessary duration, typically 24 h, following [41]. Curing at temperatures lower or higher than 100 °C gives AAMs with lower strength, and curing at room temperature does not yield a solid product. This stage will be referred to as “curing”. No pre-oven curing was used. The top surfaces of the samples inside the oven were observed to completely solidify within 1 h. The samples were demolded after the desired oven curing duration. The samples were then placed in a high temperature oven at room temperature and the oven temperature was set to the desired high temperature, typically 300 or 500 °C. This stage will be referred to as “heat treatment,” in order to distinguish it from the curing stage. The rate of temperature increase was ~3 °C/min. The samples were kept at the desired temperature for ~1 h, after which the oven was turned off and the samples were allowed to slowly cool inside the oven. Measurements on the heat-treated expanded samples were made after cooling. Fig. 1 shows a schematic diagram of the process.

As can be seen in Fig. 1, if the process is stopped after curing, an alkali-activated solid material (without any visible pores) is obtained. This solid material has low-to-medium compressive

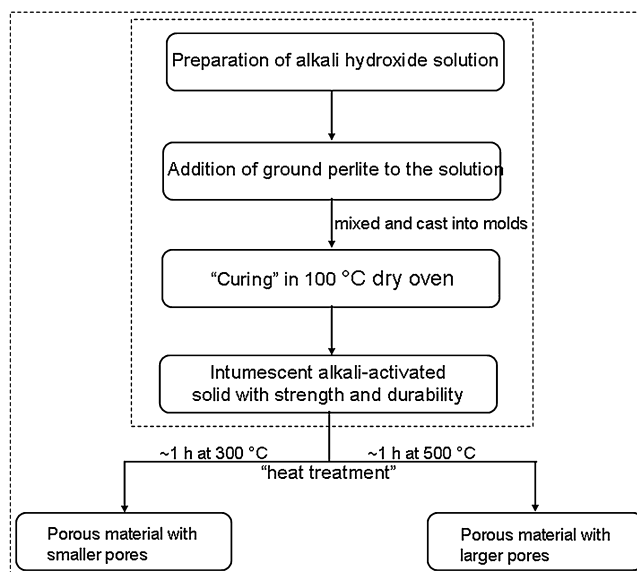


Fig. 1. Schematic diagram of the process for preparing alkali-activated intumescent/porous materials.

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