



Microstructure of cement paste with natural pozzolanic volcanic ash and Portland cement at different stages of curing



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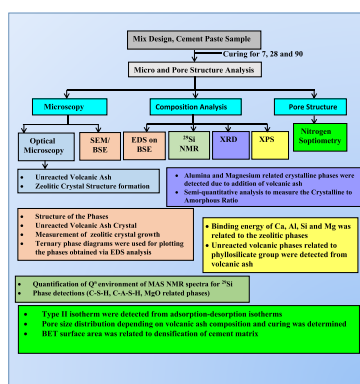
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HIGHLIGHTS

- Replacement of Portland cement with volcanic ash.
- Curing of cement paste at different ages.
- Volcanic ash can be viable substitute to Portland cement.

GRAPHICAL ABSTRACT



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ABSTRACT

The use of volcanic ash as a partial substitute to Portland cement can be a viable alternative for producing sustainable and durable cementitious materials. This study investigates the effect of early and late age curing of hardened cement pastes made with volcanic ash and Ordinary Portland Cement (OPC). Pore and microstructure studies were performed on hardened cement pastes prepared with 10% incremental substitution of volcanic ash up to 50% substitution of OPC. Densification in hardened cement pastes was attributed to formation of Calcium Silicate Hydrate (C-S-H) and Calcium-Alumino-Silicate-Hydrate (C-A-S-H) gels, while the development of Magnesium-Silicate-Hydrates (M-S-H) led to decalcification of C-S-H and C-A-S-H gels which resulted in an increase in porosity of the cementitious matrix. A combination of bulk and surface characterization techniques was used to facilitate effective usage of volcanic ash as a potential substitute for Portland cement that provides a sustainable construction material, and environmentally friendly solution to volcanic ash disposal.

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1. Introduction

As early as the second century BCE the Romans began to use volcanic ash as an additive in blended cements to build highly durable structures [1,2]. For example, pumiceous volcanic ash was used in preparing Roman hydraulic pozzolan concrete that was

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Table 1
Mix proportions and samples cured at 7, 28 and 90 days with 0.25 water to cement (w/c) ratio.

Sample ID	Curing Age (Days)	Composition (%)
IP0-7	7	OPC
IP10-7	7	VA10-OPC90
IP20-7	7	VA20-OPC80
IP30-7	7	VA30-OPC70
IP40-7	7	VA40-OPC60
IP50-7	7	VA50-OPC50
IP0-28	28	OPC
IP10-28	28	VA10-OPC90
IP20-28	28	VA20-OPC80
IP30-28	28	VA30-OPC70
IP40-28	28	VA40-OPC60
IP50-28	28	VA50-OPC50
IP0-90	90	OPC
IP10-90	90	VA10-OPC90
IP20-90	90	VA20-OPC80
IP30-90	90	VA30-OPC70
IP40-90	90	VA40-OPC60
IP50-90	90	VA50-OPC50

used to build the Bay of Naples [2,3]. It is known that the combination of hydraulic lime and pumiceous volcanic ash acts as a “glue” or binding agent in hydraulic pozzolan cement [4]. The volcanic ash used in the preparation of this concrete consisted of alkalis, and when the material was examined later (approximately 2000 years) showed the presence of zeolitic minerals such as analcime, phillipsite, leucite, and chabazite [4–6]. These phases are present as active constituents of zeolites and usually contribute to a high specific surface and may contain some chemically bound water that forms during the hydration of cementitious gels. Most of the volcanic ashes have some amount of inactive minerals such as feldspars, magnetite, quartz and mica which are innocuous in nature and are sometimes not involved in the hydration process when mixed with Portland Cements [5]. Despite these findings, there is a significant challenge associated with deciphering the reacted and unreacted phases during and after the hydration process [7].

Pyroclastic rocks, commonly known as volcanic rocks, originate from the eruption of magma which usually include fragments of rocks, sand and volcanic ash [1,2,5]. Studies indicate that only the volcanic rocks/ash that are acidic and possess high zeolitic contents demonstrate pozzolanic properties. Not all properties of pozzolan components are useful in terms of long term durability. Deleterious zeolitic phases such as montmorillonite and pholopite can cause swelling and expansion leading to internal sulfate attack if the volcanic ashes are used with aggregates that are vulnerable to alkali silica reaction (ASR), contributing to significant damage of the microstructures [8,9]. For these reasons, it is important to understand the proportion of advantageous and deleterious hydration products and the morphological development when natural

pozzolans like volcanic ashes are used as an additive or partial substitute to Portland Cement.

In this study, micro and pore structures were examined for five different combinations of hardened cement pastes prepared with volcanic ash and OPC after 7, 28 and 90 days of curing. We utilize a variety of techniques consisting of X-ray Diffraction (XRD), Magic Angle Spinning Nuclear Magnetic Resonance (MAS NMR), X-ray Photoelectron Spectroscopy (XPS), Back Scattered Electron Microscopy (BSE), Energy Dispersive Spectrum (EDS), optical microscopy and nitrogen adsorption. As a result, we have furthered the understanding of how to effectively substitute volcanic ash into Portland cement by considering various microstructural product formation along with the influence on pore structure.

2. Experimental procedure

2.1. Materials

An experimental evaluation was performed to investigate the effect of curing at early (7 days), 28 days and late age (90 days) by partial replacement of Portland cement with volcanic ash. Mixes were prepared by substituting 10, 20, 30, 40 and 50% of Portland cement with volcanic ash (See Table 1). For sample nomenclature, we refer to each mix combination based on the percent replacement of OPC with volcanic ash and age of hydration. For example, 10% substitution of volcanic ash with OPC which was cured for 7 days was designated as IP10-7, while 50% substitution of Portland cement that was cured for 28 days was designated as IP50-28. The samples were prepared as per ASTM C 305 [10]. The samples were cured for 7, 28 or 90 days and were inserted in acetone to retard the hydration after each curing period.

2.2. Methods

Surface and bulk structure/composition characterization was performed using X-ray Florescence (XRF) spectroscopy, Particle Size Analyzer, X-ray Photon-electron spectroscopy (XPS), ²⁹Si Magic Angle Spinning (MAS) Nuclear Magnetic Spectroscopy (NMR), X-ray Diffraction, Scanning Electron Microscopy (SEM), Energy Dispersive Spectra (EDS) and nitrogen sorptiometry. The list of experimental techniques that were used to study the surface and bulk structure of cementitious material is shown in Table 2.

2.2.1. Particle size distribution

Particle size distribution (PSD) was conducted on volcanic ash and OPC by suspending them in isopropyl alcohol using a Laser Light Scattering technique with a particle size analyzer. The mean particle size for volcanic ash and OPC was found to be 17.14 μm and 12.73 μm, respectively (See Fig. 1, Table 3).

2.2.2. X-ray Florescence Spectroscopy (XRF)

The chemical composition of OPC and volcanic ash was measured using X-ray Florescence (XRF) spectroscopy and the results are shown in Table 4. The sum of silicon oxide (SiO₂), aluminum oxide (Al₂O₃) and ferric oxide (Fe₂O₃) components for the raw volcanic ash is 64.3%, indicating that this material is a Class C type of ash according to ASTM C 618 [11].

2.2.3. ²⁹Si NMR analysis

Solid-state ²⁹Si MAS NMR spectra were collected at 119.22 MHz on an NMR spectrometer using a probe for 4 mm MAS BB/1H outer diameter zirconia rotors and a spinning speed of 10 kHz. The ²⁹Si MAS NMR experiments employed a pulse width of 5 μs, a relaxation delay of 20 s and 4141 scans. All spectra were collected

Table 2
Experimental Techniques for examining the surface and bulk structure/composition of cementitious materials.

Characterization Technique	Sample Type	Parameters Measured
Qualitative and Semi-quantitative XRD	Powder	Chemical Phases (Crystalline to amorphous ratio)
Surface Roughness using a Stylus Profiling System	Solid (Polished)	Surface roughness on the polished samples before SEM/BSE imaging
Scanning Electron Microscopy (SEM), Energy Dispersive Spectra (EDS)	Solid (Polished)	Details in microstructure and element mapping using energy dispersive spectrum (EDS)
Optical Microscopy	Solid (Polished)	Microstructure
²⁹ Si Magic Angle Spinning (MAS) Nuclear Magnetic Spectroscopy (NMR)	Powder	Quantification of amorphous phases such as C-S-H and C-A-S-H gels
N ₂ sorptiometry	Solid	Pore structure and BET surface area
X-ray Photoelectron Spectroscopy (XPS)	Powder	Quantitative surface elemental analysis

BET: Brunauer-Emmett-Teller.

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