



## One-part alkali activated cement based volcanic pumice



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### HIGHLIGHTS

- Mechanochemical processing of the blend of pumice and other raw materials produced one -part hydraulic cement with modified mineralogy constituents.
- Some of the hydraulic cements exhibited desired strength development characteristics and moisture resistance upon hydration at room temperature.
- The microstructure of hydration products points at the formation of a continuous structure of hydrates embodying interconnected micro-scale pores.
- The hydration rates of the hydraulic cements produced with different sources of alkali and alkaline earth metals vary significantly.

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### ABSTRACT

Pumice, a volcanic tuff, was used as aluminosilicate precursor in production of one-part alkali activated cement. Depending on their cooling rate and aging processes, volcanic tuffs exhibit different degrees of reactivity. The pumice used in this project was subjected to heat treatment followed by mechanochemical processing, together with different naturally occurring sources of alkali or alkaline earth metal cations, into hydraulic (one-part alkali activated) cements. The resultant hydraulic cements were evaluated based on their strength development characteristics via hydration reactions. The particle size distribution and heat of hydration of cement particles, and the chemical composition, mineralogy, bond structure, thermal attributes and morphology of cement particles and hydrated cement paste were also investigated. The results indicated that some of the simple formulations and processing conditions developed in the project produce hydraulic cements with viable structures and strength development qualities.

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### 1. Introduction

Conventional “two-part” alkali activated binders are synthesized via alkali-activation of solid aluminosilicate feedstocks (e.g., metakaolin, coal fly ash, ground granulated blast furnace slag and natural pozzolan) with highly alkaline solutions (e.g., sodium silicate and sodium hydroxide) [1–11]. These two-part alkali activated binders, comprising largely of alkali aluminosilicate hydrates, offer important advantages over alternative cementitious binders in terms of sustainability, thermos-mechanical performance and chemical stability. The two-part nature of alkali activated binders, however, challenges their transition to mainstream construction applications. The need to handle highly caustic solutions, and the requirement to mix these caustic solutions (in lieu of water) with solids constitute setbacks in their large-scale implementation.

Efforts have been undertaken in recent years to develop “one-part” hydraulic cement formulations which, upon addition of water, undergo hydration reactions which yield the desired alkali aluminosilicate hydrate structure of conventional alkali activated binders [12,13]. These efforts can be categorized into two groups: (i) heat treatment of the blend of raw materials (aluminosilicate precursor and alkalis in solid form) to yield a compounded and activated hydraulic cement; and (ii) simple blending of the raw materials in dry form. The second category, however, may not be a viable option because the resultant ‘cements’ embody caustic solids in raw form, which could generate significant heat upon the addition of water. These ‘one-part cements’ could also have limited shelf life due to the affinity of alkalis for the humidity in air. The first group, which seems to be more viable, requires input of relatively high thermal energy to bring about compounding and activation effects; this energy consumption, however, compromises the sustainability of the resultant hydraulic cements. The long-term durability of these “one-part” cements needs to be verified [14–16].

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The primary focus of the work reported herein was on development of one-part alkali activated cements formulated with natural volcanic tuff as aluminosilicate precursor [17]. This work emphasized the sustainability of the resultant hydraulic cement by employing a combination of calcination of volcanic tuff at moderate temperature followed by mechanochemical processing of the blend of volcanic tuff with other raw materials to produce the hydraulic cement.

Volcanic tuffs offer a wide range of reactivity depending upon their degree of crystallinity and mineralogy [18]. Reactive volcanic tuffs have been used for production of historic concrete. Early stone buildings have made use of more reactive volcanic tuffs together with lime to produce relatively strong and durable mortars, some of which have survived few centuries of exposure to weathering and seismic effects [19–21]. Aluminosilicate precursors such as reactive volcanic tuff (or ash) can undergo dissolution and precipitation processes in lime solution [22–26], which yield calcium aluminosilicate hydrate (C-A-S-H) structures with desired binding attributes [27,28]. Considering the variability in the reactivity of different volcanic tuffs, this investigation resorted to thermal activation of the tuff at moderate temperatures in order to achieve viable levels of reactivity prior to processing of the tuff and other raw materials into a hydraulic cement.

## 2. Experimental

### 2.1. Materials

Natural pumice was obtained from HESS Pumice® mining, Idaho (United States). Some properties of this pumice, reported by the manufacturer, are presented in Table 1. The other raw materials used for production of the pumice-based hydraulic cement included lime, Sodium sulfate and Soda Ash ( $\text{Na}_2\text{CO}_3$ ) as chemical reagents, and gypsum as additive. Soda ash as well as sodium sulfate have been shown in past investigations to accelerate the hydration process of lime-aluminosilicate binders [29]. While conventional alkali activated binders are obtained via alkali activation of aluminosilicate precursors without a focus on the addition of lime, the lime constituent of this hydraulic cement produces a hybrid structure of (Ca, Na) aluminosilicate hydrates and probably calcium silicate hydrate which is amenable to room-temperature curing. This hybrid chemistry, when used in the context of a one-part hydraulic cement, also seems to yield hydration products with improved moisture resistance.

### 2.2. Formulations of raw materials and their processing into hydraulic cements

The formulations of raw materials considered in this investigation for synthesis of hydraulic cements are introduced in Table 2. These formulations were partly based on those developed in the literature on two-part alkali activated binders which employ volcanic tuff in powder form: lime weight ratios in the 0.6–0.75: 0.3–0.23 range [30,31].

Each formulation was blended and heat-treated at 700 °C for four hours (with a heating rate of 10 °C/min) in order to improve its reactivity. This temperature was selected after trial studies involving heat treatment at different temperatures. After heat-treatment, the product was allowed to cool down to room temperature over several hours (with the furnace turned off).

After cooling, the heat-treated blends were ball-milled for the purpose of size reduction and mechanochemical processing. The ball-milling parameters are presented in Table 2. A ceramic jar (5.6 L) ball mill was used in this investigation operated at an optimum rotational speed as a function of the internal diameter of the jar and diameter of the steel balls [32]. This process yielded the hydraulic cements formulated with pumice for construction applications. Transformation of raw materials into hydraulic cement, and the change in crystalline structure caused by ball-milling support the mechanochemical effects rendered by this milling process.

### 2.3. Characterization methods

The hydraulic cement was used to prepare mortar mixtures. The mortar mix designs considered in this investigation are introduced in Table 3. These mixtures

**Table 1**  
Some properties of the pumice used as raw material (aluminosilicate precursor) in synthesis of one-part hydraulic cement.

Hardness (Mohs)	pH	Radioactivity	Crystalline $\text{SiO}_2$	Softening Point	GE Brightness	Specific Gravity
6	7.2	0	None Detected	900 °C	84	2.35

**Table 2**

Mechanochemical processing (milling) conditions of the heat-treated blends of raw materials (three sizes of steel balls were used in equal weights).

Steel Ball Diameter (mm)			Steel Ball-to-Material Weight Ratio	Milling duration (h)
Large	Medium	Small		
76.2	25.4	12.7	10	3

**Table 3**

The formulations of raw materials considered for synthesis hydraulic cements.

Mix	Precursor (pumice) Wt%	Chemical Reagent(s)	Water/binder ratio	Sand ratio
1	60	40 (Sodium Sulfate)	0.6	3
2	65	0.35 (Soda Ash)	0.6	3
3	60	0.2 (lime) + 0.20 (Soda Ash)	0.6	3
4	75	0.25 (lime)	0.6	3
5	75	0.25 (Sodium Sulfate)	0.6	3
6	60	0.2 (Sodium Sulfate) + 0.2 (Gypsum)	0.6	3
7	75	0.20 (lime) + 0.05 (Gypsum)	0.6	3
8	75	0.20 (lime) + 0.05 (soda ash)	0.71	3
9	65	0.35 (lime)	0.71	3
10	20	0.2 (lime) + 0.6 (gypsum)	0.74	3
11	85	0.15 (Sodium Sulfate)	0.6	3
12	50	0.5 (lime)	0.6	3
13	60	0.3 (Sodium sulfate) + 0.1 (lime)	0.6	3

were prepared using a mortar mixer. Fresh mixtures were cast and consolidated in 50-mm cube molds, demolded after 24 h (during which they were stored under wet burlap), sealed and stored at 90% relative humidity and room temperature for compression testing at 7 and 14 days of age. Most mortar mixtures evaluated in this work had a natural sand-to-indigenous hydraulic cement ratio of 2.5, and water/cement ratio of 0.5 (for achieving desired workability). The compressive strength of cube specimens was measured using a LLOYD EZ20 universal testing machine at a crosshead speed of 0.1 mm/min. Ten replicates specimens prepared from each mix were tested in compression, and the mean values are reported as compressive strength.

The chemical composition of raw pumice was determined using X-ray fluorescence (XRF) spectroscopy. Phase compositions of the samples were examined by X-ray diffraction (XRD) spectroscopy. FTIR spectroscopy was conducted in the 4000–400  $\text{cm}^{-1}$  range to investigate the chemical environment (bond structure) of the as-received pumice and the synthesized alkali activated cement based on pumice.

The raw (untreated) pumice and the end products of alkali activation were subjected to SEM observation for evaluating their microstructural features. SEM observations were carried out on a JCM-5000 NeoScope™ at an accelerating voltage of 10–15 kV using a secondary electron (SE) detector. SEM observations were made on the pumice external surface and on the fractured surfaces of hydrated paste at 28 days of age. Samples were sputtered with gold prior to SEM observations.

Thermogravimetric analysis (TGA) was performed by heating the samples at a rate of 10 °C  $\text{min}^{-1}$  in a dry air flow between 50 °C and 950 °C.

The exothermic heat of hydration of the hydraulic cement was measured using a calorimeter (TAM Air Isothermal Calorimeter). In this test, about 25 g of freshly mixed paste was weighed into a flask, which was then capped and placed in the calorimeter. Heat generation caused by exothermic hydration reactions was monitored over 18 h.

## 3. Test results and discussion

### 3.1. Particle size distribution

The visual appearance and particle size distribution (produced via laser granulometry) of the pumice used in this investigation are presented in Fig. 1. The median particle size of the pumice was 9.8  $\mu\text{m}$ , and its specific surface area was 300  $\text{m}^2/\text{kg}$  Fig. 2.

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