



Influences of phosphate tailings on hydration and properties of Portland cement



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HIGHLIGHTS

- Influences of phosphate tailings (PTS) mainly arise from the dilution effect.
- The dissolution of phosphorus from PTS has a slight retarding effect on hydration.
- Dedolomitization causes a slight expansion and no significant strength recession.
- The study exhibits the feasibility to utilize PTS in Portland cement as filler.

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ABSTRACT

Phosphate tailings, which mainly consist of dolomite particles, are solid wastes produced from mining and beneficiating operations of phosphate rock. The use of phosphate tailings in Portland cement as filler is helpful to reduce the carbon emission and relieve other environmental problems caused by its large accumulation. Influences of phosphate tailings on hydration, properties of Portland cement and volume stability due to possible dedolomitization are investigated. Phosphate tailings affect setting time, strength and drying shrinkage mainly through the dilution effect. A small part of phosphorus dissolves from the tailings during the early stage of hydration, and the dissolved phosphorus extends the induction period, reduces the main heat peak by the precipitation mechanism. The dedolomitization of phosphate tailings takes place with the presence of alkalis but causes no significant volume expansion and strength recession.

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1. Introduction

Phosphate tailings are solid wastes produced from mining and beneficiating operations of phosphate rock. About 30–40% of the raw ore is rejected as tailings during a typical phosphate beneficiation operation [1–3]. In current practice, the generated tailings are collected and transported in paste or slurry form and disposed of in large tailings impoundments, which is costly and utilizes large areas of land. Disposal of mine tailings in impoundments may also cause environmental and safety problems, including contamination of surface water, groundwater and soils, and failure of tailings dams [4–9]. These problems led to the development of new technologies to dispose of phosphate tailings in a more cost effective and environmentally friendly manner.

Nowadays, limestone is commonly used as filler in cement in order to lower consumption of energy and CO₂ emission associated

with the production of Portland cement. European standard EN 197-1 identified two types of Portland Limestone Cement containing 6–20% limestone and 21–35% limestone, respectively [10]. The modified ASTM C150 standard specification for Portland cement allows the incorporation of up to a 5% mass fraction of limestone in ordinary Portland cements [11].

Phosphate tailings consist of finely ground particles. The average size of phosphate tailings generated from a typical flotation operation is about 30 μm [3]. Phosphate tailings contain high amount of carbonaceous minerals with an assortment of cationic replacement. Dolomite based tailings are most abundant in phosphate tailings. In cement chemistry, phosphate tailings are non-hydraulic particles. Particle properties and inert nature of phosphate tailings make it promising to be used as filler in cement. Compared with limestone, phosphate tailings need less or even no extra energy to reduce their particles size into a desirable level.

The influences of phosphate tailings on the properties of Portland cement need to be understood fully for the potential utilization in cement, especially, those arise from the possible

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dissolution of phosphorus from the tailings for the relatively high P_2O_5 content and the potential reaction between alkali and dolomite contained in phosphate tailings. In the study, the influence of phosphate tailings on hydration kinetics, properties of Portland cement were investigated, alkali-reactivity (dedolomitization) of phosphate tailings was evaluated and its influences on volume stability and strength were also determined by acceleration methods.

2. Materials and methods

2.1. Materials

The cement used was Portland cement (type P-I according to GB 175 [12], similar to CEM I or ASTM type I), with a specific surface area of $348.0 \text{ m}^2/\text{kg}$, an average particle size of $23.8 \mu\text{m}$ and a density of $3.2 \text{ g}/\text{cm}^3$. Its chemical and mineral composition are listed in Table 1. The phosphate tailings contained dolomite ($\text{CaMg}(\text{CO}_3)_2$), apatite ($\text{Ca}_5(\text{PO}_4)_3(\text{OH})$) and a few amount of quartz (SiO_2). The specific surface area of the phosphate tailings was $229.0 \text{ m}^2/\text{kg}$, average particle size of $28.0 \mu\text{m}$, and density of $2.7 \text{ g}/\text{cm}^3$. The chemical and mineral composition of the phosphate tailings are also listed in Table 1. Fine ground quartz (α -Quartz, assumed nonreactive), which has similar particle size distribution to phosphate tailings, was used as reference, so as to study the effect of phosphate tailings on properties of cement at the same dilution effect. The specific surface area of quartz was $240.0 \text{ m}^2/\text{kg}$, with an average grain size of $26.9 \mu\text{m}$. Its chemical composition is reported in Table 1. Fig. 1 shows the particle size distribution of the phosphate tailings, quartz and cement.

2.2. Methods

The rheological properties of cement paste (with a water to solid ratio of 0.5) blended with phosphate tailings were measured with a RheoPlus QC rheometer, and the evolution of hydration heat was monitored by isothermal calorimetry (10 g paste with water to solid of 0.4). In order to analyze the dissolution of phosphorus from phosphate tailings, 200 g of phosphate tailings was mixed with 400 ml of distilled water and calcium hydroxide saturated solution respectively. The mixtures were stirred for 5 min and then waited until precipitation. The supernatant liquid was taken at regular intervals. After dilution (1:500), the phosphorus concentration was measured by means of colorimetric method (phosphate kit). Chemical shrinkage was measured according to ASTM C1608-12 [13]. Strength, water requirement of normal consistency and setting time of Portland cement containing phosphate tailings were measured according to the Chinese standards GB/T1346 [14] (equivalent to ISO 9597:2008) and GB/T17671 [15] (equivalent to ISO 679:1989), respectively. Dry shrinkage of mortar specimens was measured according to JC/T 603 [16] (corresponding to ASTM C596-01).

In order to understand the effect of phosphate tailings dedolomitization on volume stability and strength, mortar specimens of $25 \times 25 \times 280 \text{ mm}$ (with two copper gauge studs at each end) with 0.4 water/cement ratio, cement-to-sand ratio of 0.5, and specimens of $40 \times 40 \times 160 \text{ mm}$ with a cement-to-sand ratio of 1:3, water-to-cement ratio of 0.5 were prepared respectively. All specimens were cured at $20 \pm 1 \text{ }^\circ\text{C}$ and >95% humidity for 28 days, and then stored in $38 \text{ }^\circ\text{C}$ NaOH solution (0.6 mol/L). The change in length of specimens of $25 \times 25 \times 280 \text{ mm}$ was regularly measured (both the containers and specimens had been cooled for 24 h at $20 \text{ }^\circ\text{C}$ before measurement). The compressive and flexural strength of specimens in size of $40 \times 40 \times 160 \text{ mm}$ were regularly measured. Pastes with 30% cement replacement with phosphate tailings or quartz were cast in glass bottles according to ASTM C1608-12, and a small amount of water was added into the bottles for curing purpose after hardening. After curing for 28 days at $20 \text{ }^\circ\text{C}$, the curing water was replaced with NaOH solution (0.6 mol/L), the change in volume of samples at $38 \text{ }^\circ\text{C}$ were also measured according to ASTM C1608-12. 10 g of phosphate tailings mixed with 100 ml of NaOH solution (0.6 mol/L) was sealed in plastic vital and then stored at $38 \text{ }^\circ\text{C}$ for 28 days. The sample was then washed with isopropanol, vacuum dried and ground. XRD pattern of the treated powder was collected and phase compositions were obtained by Rietveld calculation.

Table 1

The chemical and mineral composition of materials used.

	MgO	CaO	Fe ₂ O ₃	Al ₂ O ₃	P ₂ O ₅	SiO ₂	SO ₃	Na ₂ O _{eq}	f-CaO	L.O.I
PC	2.5	62.5	3.4	4.7	0	21.2	2.8	0.5	0.7	1.7
PTS	18.9	36.8	0.8	0.1	4.4	2.1	1.0	0.1	0	35.8
Quartz	–	–	–	0.1	–	99.8	–	–	–	0.1
	C ₃ S	C ₂ S	C ₃ A	C ₄ AF	Quartz	Dolomite	Apatite			
PC	56.3	20.3	7.0	10.4	–	–	–			
PTS	–	–	–	–	0.9	89.5	9.6			

Ps: PC-Portland cement; PTS-phosphate tailings.

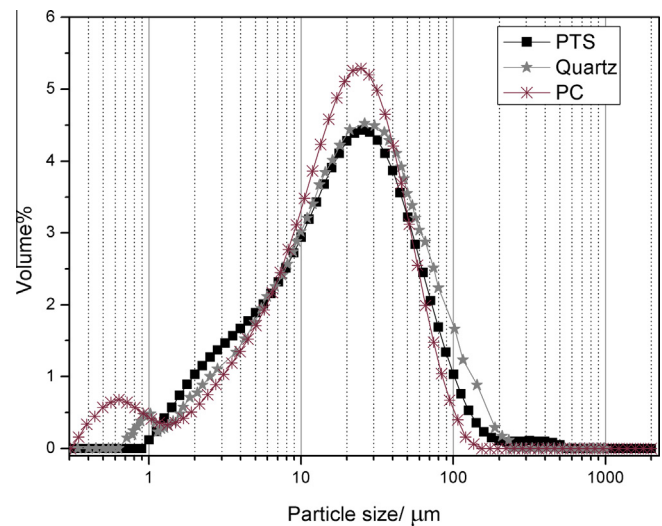


Fig. 1. Particle size distribution of phosphate tailings, quartz and cement.

3. Results and discussion

3.1. Influence on hydration

Many properties of Portland cement, such as strength development, setting and hardening, are associated with the hydration process. Thus, the influence of phosphate tailings on hydration kinetics is discussed first in the section.

At the early stage, chemical shrinkage is directly proportional to the hydration degree of cement [13], so the measurement of chemical shrinkage of cement paste is one of the methods for evaluating the early hydration degree of cement. Power and Jensen's [17,18] research shows that chemical shrinkage value for complete hydration is approximately $0.064 \text{ ml}/\text{g}$. Fig. 2 shows the effect of phosphate tailings and quartz on chemical shrinkage (the right is an enlarged view). In the first 3 h of hydration, the chemical shrinkages of cement with phosphate tailings and quartz were measured roughly as the same. During 4–6 h, the chemical shrinkage of cement with phosphate tailings remains almost constant, which reflects a delay of hydration process.

During the early period of hydration, fast dissolution of anhydrous phases result in a rapid evolution of heat for several minutes. This burst of heat is followed by an induction period (or a dormant period) during which the hydration remains almost at a halt, characterized with continuous low evolution of heat. The induction period may last for several hours and then the hydration accelerates for a few hours until a main peak corresponds to the massive precipitation of the main hydration products of alite is reached. According to the heat flow curve (Fig. 3(a)), the incorporation of phosphate tailings leads to a prolonged induction period and a low main peak.

The measurements of chemical shrinkage and heat evolution rate of blends with phosphate tailings and quartz indicate that

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