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Development of low cost supplementary cementitious materials utilizing thermally activated Pisha sandstone

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HIGHLIGHTS

- A new supplementary cementitious materials produced by utilizing thermally activated PS was reported.
- The effects of temperature on the properties and hydration products were studied.
- A maximum compressive strength of 21.9 MPa of pastes was achieved.
- The main reaction products of modified calcined PS are amorphous aluminosilicate gel.

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ABSTRACT

Pisha sandstone was the main resource of Yellow river sediment mineral with a low pozzolanic activity. This paper reports an investigation of the possibility of development of low cost supplementary cementitious materials using Pisha sandstone via thermally activated. The decomposition of Pisha sandstone when thermally treated at 600 °C and 800 °C and the effect of this treatment on its physicochemical properties, pozzolanic reactivity in cementitious materials were studied. The hydration of the mineral component, reaction products and micromorphology were studied by TG-DTG, XRD and SEM-EDS technique, respectively. Compressive strength test was conducted to assess the mechanical property of modified pastes, and Mercury intrusion porosimetry (MIP) was employed to investigate the pore structure and pore size distribution. This work confirmed that Pisha sandstone could be acted as a kind of low cost supplementary cementitious materials via thermally activated, and the optimal calcination temperature was 600 °C. It was shown that the decomposition of clay mineral of Pisha sandstone via thermal treatment result in an enhance of hydration reaction and mechanical strength, and the sample (calcined at 600 °C) exhibited the highest compressive strength (21.9 MPa). The results of the investigation show that the amorphous aluminosilicate gels (C-A-S-H) were the main hydration products.

1. Introduction

The traditional Portland cement industry consume huge amount of energy and release billions of CO_2 gas which caused the greenhouse effect. In recent years, researchers have been trying to use by-products (such as fly ash, slag) and calcined clay minerals (such as kaolins) form geopolymeric materials as partial replacement of Portland cement, which could meet the engineering demand but with lower CO_2 release [1]. Compared to traditional Portland cement, the geopolymeric materials have more excellent mechanical properties, durability and thermal stability [2–6]. Moreover, the geopolymeric materials would be more resistant to acid erosion than that produced by Portland cement due to their low calcium content [7]. In particular, the geopolymeric materials were more environmental and economic because of the reduced energy requirement for their manufacture and the higher sustainability compared to that of Portland cement.

The use of clay mineral or clay sediments as aluminosilicate source material to form geopolymers has been widely proven. In fact, the clay minerals are widely available all over the world with certain reactivity via a thermal activation process. Generally, the clay was composed of different clay minerals and associated min-

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erals and would be affected by the nature of the parent rocks [8-10]. Pisha sandstone (PS) was a special kind of clay mineral and forms during the Tertiary period [1], it is mainly located in the northwestern region of China with an area of more than 12,000 km^2. It was composed by the amorphous clay minerals (montmorillonite, illite and mica et al.) and crystalline minerals (feldspar, quartz and calcium et al.) [11]. The bonded mechanism of PS was bad and it has an unsatisfactory petrographic structure, PS is very hard like the rock when it is dry, but would collapse into sand within one minute when it is immersed in water due to the expansion of soggy clay minerals. Hence, the value of soil erosion intensity of PS area is very high (more than 20,000 $t \cdot km^{-2} \cdot yr^{-1}$ [12]). PS was broken into sediment by flood and deposited on the Yellow River course and raised the altitude of riverbed.

In Pisha sandstone area, the check dam is the main measure of soil erosion control, in the future, the amount of check dam would reached to 276,5000, thus, the damming materials of check dam amount used is huge. However, due to the serious soil and water loss and the high value of soil erosion intensity, the terrain is fragment and the transport infrastructure is very bad, and the traditional dam materials (such as concrete, stone et al.) are scarce, and the cost of traditional building materials such as concrete (Portland cement) is very high. Compared with traditional damming material, the Pisha sandstone is easy and widely available all over the Pisha sandstone area. Thus, the Pisha sandstone materials have a huge advantage in the economic, available and environmental when compared to the traditional materials of the location check dam.

Therefore, it would be a beneficial exploration to apply PS in the construction industry as a raw material for the production of alkali activated materials. Some studies on the recycling use of Pisha sandstone have been conducted by Changming Li, Lijiu Wang et al. [1,2,13–15], and the alkali activated Pisha sandstone geopolymer of mortar were produced by using Pisha sandstone in their reports. In the published papers, the compressive strength, pore structure, water resistance and hydration products of alkali activated Pisha sandstone geopolymer were also studied. However, the results of the published papers shown that the pozzolanic activity of PS was very low [15], thus, it is significative to study how to improve the pozzolanic activity of PS and development of cementitious materials for concrete utilizing PS. Thermally activated clay minerals have been used as admixture in cements or concrete for many years, and the effect of calcined clay minerals on cement and concrete properties also has been studied [16,17]. Heat treatment could break down the structure of clay minerals and transform into a highly pozzolanic reactive material. The complex amorphous structure of clay minerals would reorganization during the periods of calcinations, and this change would enhance the pozzolanic activity of clay minerals [18-21], and the amorphous phases of clay minerals would react with alkaline in the presence of water producing a cementing materials like C-S-H [22-24].

The purpose of this paper is to development of low cost cementitious materials for concrete utilizing PS via thermally activated, and investigates the effect of mechanical and thermally activated treatment on the alkaline activation reaction of PS, and see how they influence the development of compressive strength, microstructure, hydration process, and reaction products of PS pastes. In addition, excepted that an improved understanding on the potential of calcined PS as low cost cementitious materials could be achieved.

2. Experimental

2.1. Materials

In this work, The studied natural PS was obtained from Loess Plateau in Inner Mongolia and fly ash obtained from Xinjiang province, China. The natural PS was air-dried and pulverized until all solids passed a #18-mesh (1000 μm opening) sieve, then the PS was calcined in an electrical muffle with a heating rate of 10 °C/min. the distinct temperatures were set at 600 °C (CPS1) and 800 °C (CPS2) and the dwell time was fixed at 1 h. Then the natural and calcined PS (CPS) material was dry milled using a porcelain ball mill with alumina milling media for 0.5 h to increase the specific surface area. Table 1 summarizes the chemical compositions of PS and fly ash by X-ray fluorescence (XRF) spectrometry (Germen, Siemens-Bruker, SRS3400). The fly ash used in this work is Class C according to ASTM C618. Fig. 1 and Fig. 2 show the feature of natural PS and the calcined PS, respectively (Fig. 3).

2.2. Sample preparation and characterization

The PS, CPS and fly ash, were mixed with alkaline activator to prepare the pastes, the composition of alkaline activator solution and each paste were summarized in Table 2. NaOH pellets (99% purity quotient, Tianjin Kemiou Chemical Reagent Co., Ltd. China) and water glass (Na_2SiO_3) were dissolved in distilled water to a certain concentration to preparation activator solution. The water glass (molar ratio SiO_2/Na_2O, M_s = 3.0) and NaOH solution were mixed to preparation activator solution with a combined modulus of M_s = 2.0 (SiO_2/Na_2O = 2.0, Na_2O = 13.25%, SiO_2 = 26.5%). In order to maintain constant the workability for all the mixtures, the total alkaline activator solution (including NaOH pellets, water glass and water)/total solid (the mixture of PS, CPS and fly ash) weight ratio was fixed at about 0.35.

To cast a cubic specimen for compressive strength test, a half of the plastic kettle is firstly filled with activator solution, and added the raw materials (PS, CPS or fly ash) into activator solution, the mixture was vibrated for 30 s, and then the mixture was mixed for 10 min by a magnetic stir bar. After vibrating and mixing, the fresh mixtures were poured into a cubic steel mold, and the specimen is pressed by a press machine under a pressure of 3.0 MPa for 5 min. To ensure repeatability, 3 specimens were prepared for each type of paste. Immediately after casting, the steel moulds were covered with wet hessian and sealed in plastic sample bags. All specimens were then stored in laboratory at ambient temperature (25 ± 2 °C). At 24 h, the specimens were removed from the steel moulds. To investigate the effect of curing temperature on strength of PS paste, the specimens were sealed with plastic bags and cured in an oven (80 °C) and the remaining specimens were stored in laboratory at ambient temperature (25 ± 2 °C), respectively.

The compressive strength of samples hydrated for 3, 7, 28 and 90 days was measured in triplicate using a controls multipurpose electronic universal testing machine with a load cell of 100 kN capacity. And a constant displacement rate of 0.5%/min (ASTM C109) was used to test the mechanical properties of the 25 × 25 × 25 mm cubes specimens. In order to minimize the friction between the surfaces of the samples and the steel plates of the machine reducing the shear stresses, the contacted surface ends of each specimen were polished to have flat surfaces in contact with the machine, and both contact surface ends of the specimens were coved by a thin layer of lubricant coating before the compressive strength testing. After that, the debris from the crushed PS pastes specimens was dried in a desiccator under vacuum. Part of the dried samples was ground in an agate mortar.

The porosity and pore structure of thermally PS materials were characterized by mercury intrusion porosimetry (MIP, AutoPore IV) testing, and the measurements were carried out using an automated mercury porosimeter over the pressure range between 0.001 and 400 MPa. Before testing, specimens were cleaned in a microwave bath and were dried at 80 °C until constant weight was achieved. Particles passing an 80 μm sieve were used for X-ray diffraction (XRD), FTIR and thermogravimetric (TGA) analyses, and some pieces of the crushed PS pastes were selected out for the SEM analysis. Mineralogical characterization of raw materials and PS pastes were done using X-ray diffraction (XRD), Siemens-Bruker D5000 using Cu Kα radiation, λ = 1.54 Å and operating at 40 kV and 30 mA was used. Diffractograms were recorded in step-scan mode, in 5–70° 2θ range, with a step size of 0.02° 2θ and rate of 2°/min. A Switzerland Mettler-Toledo simultaneous thermal analyser was employed to measure some physical properties of the material as a function of the temperature change. For thermogravimetric analysis, the samples were heated in an atmosphere of nitrogen at 10 °C·min^{-1} from 40 to 1000 °C. A JEOL JSM 6460 scanning electron microscope (SEM) fitted with a Link EDAX energy dispersive X-ray spectroscope device equipped was used to examine the microstructure and chemical elemental of 90 days of PS pastes samples.

3. Results and discussion

3.1. Effect of calcinations on Pisha sandstone

The thermal treatment would lead to a significant change on the structure of clay mineral, the montmorillonite would dehydroxylation after calcined at high temperature, at the same time, the structure of the aluminum oxide octahedron and silica tetrahedral sheet layer would be changed or destroyed at high temperature [10-12,25,26]. The montmorillonite dehydroxylation temperature depend on the structure of constitution water. Generally, the reac-