



Properties of alkali-activated slag with addition of cation exchange material



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HIGHLIGHTS

- Cation exchange materials drastically reduced mobile alkali metal cations in AAS.
- The type of exchanged cation crucially influenced the properties of AAS.
- Vermiculite was preferable to improve the comprehensive performance of AAS.

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ABSTRACT

The massive mobile alkali metal cations in alkali-activated slag (AAS) cements are significantly connected to the issues such as efflorescence, carbonation and potential alkali-aggregate reaction, and greatly restrict their extensive application and development. This paper attempts to use cation exchange materials to remove the freely moved alkali cations in AAS cements. Two representative cation exchange materials, vermiculite and 5A zeolite, were added at mass ratio of 5% and 10% to slag activated by sodium hydroxide. 5A zeolite showed a faster and stronger ions exchange capability to vermiculite in alkaline solution. However vermiculite immobilized more Na^+ ions than 5A zeolite in AAS pastes, and the specimens with vermiculite displayed a higher compressive strength, attributed to generate more hydration products which were shown in XRD, TG-DSC and EDS tests, and formed a compact microstructure seen in SEM image. It is concluded that these two cation exchange materials could remarkably reduce the unrestrained alkali ions in the AAS cements, and the properties of AAS cements influenced by cation exchange materials are highly dependent on the category of the exchanged cations.

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1. Introduction

For the purpose of reducing environmental impact of the conventional Portland cement production which contributes about 7% of the total worldwide CO_2 emissions [1], researchers are paying closer attention to the development of ecological low carbon cementitious materials [2]. As a typical kind of environmental friendly alternative binder, Alkali-activated materials (AAMs) attract extensive concerns around the world [3–5].

Alkali-activated slag (AAS) is one of the AAMs using slag as the main activated raw pozzolan materials and exhibits excellent mechanical properties and brilliant chemical erosion resistance

[6,3]. In spite of its promising performance, AAS has not obtained extensive industry application, primarily because of lower workability [7], drying shrinkage [8], carbonation [9], efflorescence [10] and potential alkali-aggregate reaction [11]. Aforementioned problems are closely entwined with massive alkali metal cations in AAS. Therefore, it is worthy to study the existence form of alkali metal cations in AAS and relationships between alkali metal cations and hydrated products.

The primarily hydration products in AAS are calcium aluminosilicate hydrated gels (C-A-S-H) with a low Ca/Si ratio, hydroxalite and other mineral phases [12]. Only a small part of alkalis are taken up by C-A-S-H gels through maintaining charge balance of alumina groups, replacing protons or calcium ions of silanol groups and occupying empty sites in the interlayer structure of C-A-S-H gels [13]. Other mineral hydrates including hydroxalite only take up negligible amounts of alkali cations [14]. Consequently, most alkali ions remain in original state and could freely move in the AAS matrix which is directly responsible for the above

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mentioned issues of AAS. How to reduce these unrestrained alkali metal cations is critically essential to improve the corresponding properties of AAS cements and extraordinarily deserved for intensive study.

Some authors attempt to reduce the excessive mobile alkali cations in AAS cement by drawing into aluminosilicate materials, altering curing methods, and varying species of the alkali ions [10,15], but not achieving the desired results. This paper try to handle with these freely moved alkali ions from a brand new perspective, by introducing cation exchange materials which could exchange and immobilize the vast free alkali ions. There are two categories of cation exchange materials used in this study, including layer structure mineral vermiculite and framework structure mineral 5A zeolite which has been studied as a modifier to AAS cements by many researchers [16–18] and proved to availably exchange Na^+ in geopolymeric material [19]. The ion exchange performance of these two cation exchange materials in alkaline solution and AAS cements is tested respectively, and the hydration and property development mechanisms are compared among the different contents of cation exchange materials.

2. Experiment

2.1. Raw materials and specimen preparation

2.1.1. Raw materials

The slag, vermiculite, 5A zeolite and sodium hydroxide (chemical pure) was purchased from commercial company. The chemical composition of slag, vermiculite and 5A zeolite was measured by X-ray fluorescence (XRF) analysis (PANalytical.B.V, Netherlands), and showed in Table 1. The alkaline solution used as activator in this study was prepared by dissolving sodium hydroxide in deionised water with specified sodium equivalent (5 M) and cooling to room temperature prior to produce AAS samples.

2.1.2. Specimen preparation

The AAS specimens were manufactured by mixing the slag with 5 M NaOH solution at a constant liquid (deionised water) to solid (including slag and cation exchange materials) ratio of 0.40. Requisite amount of the vermiculite or 5A zeolite was gradually added while mixing until the components were homogenized. The pastes were rapidly casted into cubical steel molds of 40 mm size, and compacted for 5 min on the table vibrator. These specimens were cured at moist room ($\geq 95\%$ RH, 20 ± 1 °C) for 24 ± 2 h before demolding. The demolded specimens were continuingly cured at moist room ($\geq 95\%$ RH, 20 ± 1 °C) for prescribed curing period to following tests. The AAS specimens were synthesized with different vermiculite or 5A zeolite replacement content to slag: 0, 0.05 and 0.10, denoted as C0, V5, V10, Z5 and Z10 respectively.

Select debris from the compression tests and soak the debris in alcohol for three days in order to arrest further reaction. The debris was then put into vacuum drying oven at temperature of 65 °C for at least 24 h. After this, some of the debris was partly ground into fine powder and the particles passed 75 μm sieve were used for further analysis. The rest of the debris was well kept for microstructure analysis. For the BSE and EDX study, the debris

was cut into 1–2 mm thick slices and further impregnated with epoxy resin, polished to 1/4 μm and coated with Pt.

2.2. Analysis techniques

In ions exchange test, the exchange solution was alkaline solution equally to manufacture AAS specimens and sodium chloride solution with the same molar concentration to alkaline solution. 10.8 g cation exchange material was added into 100 g exchange solution in 200 ml Erlenmeyer flask with magnetic stick and sealed by parafilm to prevent evaporation. Place the Erlenmeyer flask on the stirring platform and turn on the magnetic stirrer. After specific time stirring, the mixed solution was filtered to remove solid powder. The filtered solution was given suitable ion concentration for inductive coupled plasma optical emission spectrometer (ICP-OES, Optima4300DV, PerkinElmer) analysis.

Compressive test was performed on WYA-300 Automated Breaking and Compressive Resistance tester (Wuxi Xiyi Building Materials Instrument Factory, China). Six samples of each formulation were tested and the average data were reported.

Thermogravimetric analysis (TG-DSC, STA 449 F3 Jupiter, NETZSCH) was carried out on the sieved AAS sample powders heating from ambient to 1000 °C.

The XRD patterns were obtained with D8 Advance (BRUKER AXS GMBH) using monochromatic $\text{CuK}\alpha$ radiation and running with 20 in the range of 5–70° at a speed of 2°/min.

Microstructural images were obtained using a Scanning Electron Microscope (SEM, Quanta FEG 450, FEI). Backscattered electron images (BSE) coupled with energy-dispersive X-ray spectroscopy microanalysis (EDX) to determine the ratios of the different elements in the matrix on polished samples (more than 100 points were taken for each measurement).

In Na-leachability test, 5.000 g \pm 0.0001 g of powder was added to 50 mL of deionised water in a covered container. After 18 h \pm 2 h vibration in oscillator, the mixed solution was filtered to remove solid powder. The filtered solution was given suitable Na^+ concentrations for inductive coupled plasma optical emission spectrometer (ICP-OES, Optima4300DV, PerkinElmer) analysis.

3. Results and discussion

3.1. Ions exchange test

Vermiculite is a representative 2:1 type layered aluminosilicate mineral, consisting of two tetrahedral layers with a octahedral layer stuck in the middle, and many balance of cation ($\text{Mg}^{2+}/\text{Ca}^{2+}/\text{Fe}^{2+}$) fill the void between layers, as shown in Fig. 1 [20]. For this particular structure, vermiculite possesses the strongest cation exchange capacity (CEC) within natural layered clay minerals seen in Fig. 2 [21], widely applied to remove contaminative metal ions especially heavy metal ions in waste water [22–24].

5A zeolite is an A-type zeolite with pore size of 5 Å and can be obtained by ion exchange method [19], namely replacing Na^+ in 4A zeolite with Ca^{2+} in solution displayed in Fig. 3. The balanced cations (Ca^{2+}) of 5A zeolite could exchange and immobilize massive Na^+ .

Table 1
Chemical composition of slag, vermiculite and 5A zeolite.

Materials	Weight (%)										
	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3	TiO_2	P_2O_5	Loss
Slag	35.37	15.74	0.30	4.70	36.70	0.29	0.45	2.23	3.91	0.11	0.15
Vermiculite	38.22	14.77	4.92	22.88	4.18	/	0.42	0.01	0.71	/	13.82
5A zeolite	34.62	28.39	0.02	/	11.51	3.39	0.06	0.05	/	/	21.91

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