

Pozzolanic reaction of lightweight fine aggregate and its influence on the hydration of cement



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HIGHLIGHTS

- LWFA was more chemically active with heat-cured.
- LWFA provides large surface for nucleation and growth of hydration products.
- Al released from LWFA, and then take part in the formation of C-A-S-H.

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ABSTRACT

Although lightweight fine aggregate (LWFA) has been used as an internal curing material in concrete for several decades, its chemical activity is not well estimated. In this research, LWFA is incorporated into cement paste to investigate the pozzolanic reaction and its influence on the hydration of cement. The results show that LWFA is more chemically active under heat-cured conditions and continuously releases Al and Si, which increases the Al/Ca and Si/Ca ratios of hydration products. Moreover, LWFA provides large surface for nucleation and the growth of hydration products and promotes the formation of C-A-S-H. The microstructural characterization demonstrates that the reaction products of LWFA are very similar to those of metakaolinite by-products and kaolinite-based clay waste.

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1. Introduction

Lightweight fine aggregate (LWFA) has been used widely in concrete for several decades. Due to the porous structure features, it can be used as the lightweight aggregate for low density concrete and as internal curing material for high performance concrete [1–6]. On the other hand, some LWFAs based on expanded clay or shale and sintered fly ash are mainly composed of SiO₂ and Al₂O₃ and treated by rapid cooling at high temperatures. Based on the principle of chemical reactions, LWFA was estimated to have the same ability of pozzolanic reaction, which has been proven by researchers in recent years [7–9]. The pozzolanic reaction of LWFA has its unique advantages. First, the pozzolanic reaction requires water. Internal curing water absorbed by the LWFA can efficiently promote the reaction, which is more significant in the low water/cement ratio in concrete. Second, the ions released from

the LWFA can migrate with the internal curing water to modify the microstructure of the hydration products in the interfacial transition zone (ITZ), which may be another reason for a denser ITZ. However, LWFA is significantly restricted compared with the pozzolanic reaction of fly ash. The reason for this phenomenon might be that LWFA is larger, resulting in its lower chemical reactivity. In addition, the dense sintered shells on the surface of the LWFA hinder its reaction [7]. To investigate the pozzolanic reaction of LWFA, researchers ground the LWFA into a fine powder and immersed it in the pore solution at 70 °C, which indicated that heat curing can improve the pozzolanic reactivity of LWFA [7–9]. However, few works have focused on the process or the morphology of the pozzolanic reaction products for LWFA. The in-depth analysis of hydration product, morphology, and Ca/Si, Al/Ca and similar ratios of cement hydration products influenced by the pozzolanic reaction of LWFA were also rarely reported. In our previous work, LWFA was used as the internal curing material to improve the performance of heat-cured concrete, and denser ITZ between LWFA and cement paste was observed [10]. In this work, the ions release characters, and the processes and products of the pozzolanic

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reaction of the LWFA are studied. In particular, the hydration product, morphology, and variation of ratios of various elements with respect to calcium are investigated. The results provide a more in-depth understanding and more detailed information about the pozzolanic reaction of LWFA.

2. Materials and methods

2.1. Materials and mixture proportions

LWFA, ASTM type I Portland cement, deionized water and ISO sand were used in this study. The chemical compositions of the cement and LWFA are listed in Table 1. The Bogue method was used to calculate the mass proportions of the four main minerals in the cement: C₃S, 48.5%; C₂S, 25.8%; C₃A, 8.0%; and C₄AF, 9.2%.

LWFA was produced in Yichang of Hubei Province, China. LWFA was produced from the expanded shale rock heated in a rotary kiln from 300 °C up to 1250 °C for 40 min, and then cooled from 1250 °C to 200 °C in 5 min. The bulk density of the LWFA is 928 kg/m³, and the porosity is 43.4%. The LWFA has a 24-h water absorption rate of 24.67% by dry mass (according to ASTM C128–07a) [11].

The X-Ray Diffraction (XRD) data from the LWFA is shown in Fig. 1. The amorphous hump in the XRD curve shows that the LWFA contains some glass phase, and the mineralogical composition of LWFA is quartz and ringwoodite. The content of the glass phase of LWFA was determined by a selective chemical treatment with 20% HF. The procedure described by Fernández-Jiménez [12] and Li [13] is as follows: 5 g of LWFA was added to 100 mL of 20% HF in a plastic beaker, and then sealed and stirred for 6 h at room temperature followed by vacuum filtration. The filter paper containing the insoluble residue was then washed to a neutral pH and dried for 2 h at 105 °C to a constant weight, consistent with the starting mass. The glass phase content of the LWFA is 79.1%, which is determined by mass loss. The filtrate was also collected, and the concentration of dissolved Si and Al was determined by induction-coupled plasma atomic emission spectrometry (ICP-AES). Thus, the mass content of the dissolved Si and Al were calculated. The composition of the LWFA is shown in Table 2.

The mix proportions and gradation of the ISO sand and LWFA are listed in Table 3 and Table 4, respectively, with the water/cement (w/c) ratio fixed to 0.35. The amount of LWFA was calculated based on Eq. (1), which was developed by Bentz et al. [14]. It is found that 19.39% of the ISO sand (on a volume basis) was replaced by saturated LWFA.

$$C_f \times CS \times \alpha_{\max} = S \times \Phi_{LWFA} \times M_{LWFA} \quad (1)$$

where C_f (=560 kg/m³) is the cement content of the mixture, CS (=0.07 ml water/g cement [14]) is the chemical shrinkage of the cement, $\alpha_{\max}$ (=0.97) is the expected maximum degree of hydration, Φ_{LWFA} (=24.67% kg water/kg dry LWFA) is the absorption capacity of the LWFA used (24-h absorption value), $S = 1$ is the expected degree of saturation of the LWFA expressed as a function of the assumed absorption value, and M_{LWFA} (kg/m³) is the mass of the LWFA.

2.2. Specimen preparation and curing regimes

The materials were mixed according to the mix proportions in Table 3, and specimens cast (4 × 4 × 16 cm³) at room temperature (25 °C). During the delay period before heat curing, the specimens were covered with polyethylene sheets and kept at 25 °C at a relative humidity (RH) >95% for 3 h. The curing regime is shown in Fig. 2. The heat curing was performed from room temperature to the maximum temperature at 55 °C with a heating rate of 15 °C/h, and held at peak for 4 h. Then, the temperature was reduced to room temperature at a cooling rate of 15 °C/h. The total time of heat curing is 8 h, after heat-cured, the specimens were removed from the molds and stored indoors under a fixed temperature and relative humidity (T: 25 ± 3 °C, RH: 70% ± 5%).

To observe the pozzolanic reaction of LWFA, 5 g of LWFA were immersed in 40 mL of saturated limewater in plastic bottles. As the pozzolanic reaction of the LWFA would consume calcium hydroxide (CH) and decrease the pH value of the saturated limewater, 1 g of CH was added in the saturated limewater to guarantee saturation. Then, the plastic bottles with LWFA and saturated limewater were sealed and cured with the same curing regime as the mortar specimens. At 1, 7, 28, and 56 days, the plastic bottles with the LWFA and saturated limewater were centrifuged, and the LWFA was separated from its leaching solution. The LWFA was dried to observe its morphology by SEM, and the Al and Si elemental concentrations of the leaching solution were measured by ICP-AES.

Table 1
Chemical compositions of the cement and LWFA used in this study (% by weight).

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	Loss on Ignition
Cement	21.72	4.92	3.01	62.14	2.27	0.65	0.22	1.78
LWFA	65.46	16.75	7.63	1.08	2.43	3.82	0.94	0.27

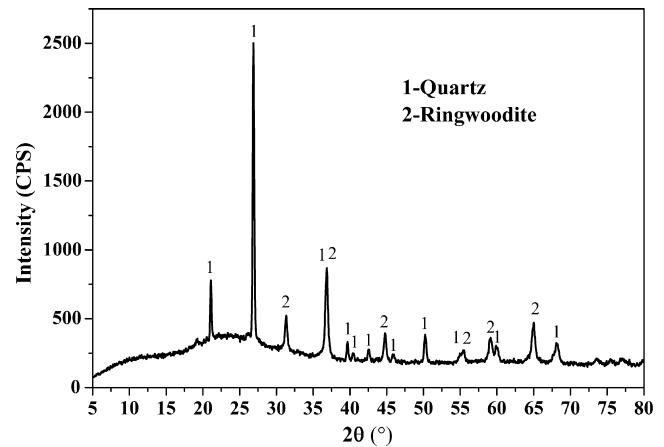


Fig. 1. XRD data from the LWFA.

Table 2
Composition and phase of the LWFA.

crystalline content	glass phase content	glass composition of LWFA (g/100 g)	
20.9%	79.1%	SiO ₂	Al ₂ O ₃
		45.5	18.2

2.3. Morphology and energy dispersive X-ray (EDX) analysis

After 28 and 56 days of hydration, slices of ~1 cm² cross section and 5 mm thick were cut from the centers of the prisms, and immersed in alcohol (volume concentration >99.7%) to stop the hydration process. At specified ages, the samples were dried in a vacuum oven at 40 °C for 6 h. Then, the slices were vacuum-impregnated with a low viscosity epoxy, and ground using silicon carbide papers with a grit size of 220, 500, 1000 and 1200. The slices were then polished with diamond abrasives of sizes 9, 6, 3, 1 and 0.25 μm. An FEI QUANTA FEG 450 ESEM and a JEM-2100F STEM with energy dispersive X-ray analysis systems were used for image acquisition and EDX analysis. Backscattered electron (BSE) images and EDX analysis were performed at low vacuum (60 Pa), so no sample coating was required to avoid charging effects. This can increase the accuracy of the EDX analysis as the coating would be a barrier for the signal from the samples [15]. The BSE images were captured at an accelerating voltage of 20 kV, while the secondary electron images were captured at 5 kV and the transmission electron microscope (TEM) was operated at 200 kV. The accelerating voltage was 20 kV and the spot size was set to 5.0 to obtain approximately 5000 counts/s, while the measuring time was 60 s in the SEM-EDX analysis. The elements analyzed were Ca, Si, Al, S, Fe, K, Na, and Mg. Oxygen was calculated by stoichiometry and microanalysis with totals >80% being rejected [16]. Spot data were collected from 100 points (5 areas × 20 points/area) of each specimen, and the distance between the points and the LWFA or anhydrous cement were approximately 3 μm [17]. The influence region of electron beam is approximately 1 μm at the 20 kV accelerating voltage, so the influence of the LWFA or anhydrous cement on the analysis results was relatively weak [16].

The various test zones were labelled as follows:

C/L-Op/lp/Ap-28/56: Control mortar / LWFA mortar - Outer product/Inner product/Around LWFA product - 28 days/56 days.g. C-Op-28: the test zone is the outer product in the control mortar at 28 days.

L-AP-56: the test zone is the product around the LWFA in the mortar at 56 days.

2.4. Nuclear magnetic resonance (NMR) test

To investigate the silicate anion structure of the C-S-H gel, an NMR test was carried out. Since both the ISO sand and the LWFA would interfere with the NMR signal, the cement paste was used. The cement was mixed with the LWFA leaching solution (1 day) and saturated limewater respectively. The water to cement ratios

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