



Preparation and properties of autoclaved aerated concrete using coal gangue and iron ore tailings



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HIGHLIGHTS

- The optimum activation temperature for coal gangue found in this study is 600 °C.
- A new type of autoclaved aerated concrete is developed using the coal gangue and iron ore tailings.

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ABSTRACT

In this paper a new type of autoclaved aerated concrete (ACC) was developed using coal gangue (CGC) and iron ore tailings (ITOs). The material compositions, calcination temperatures of the coal gangue, and the composition of the hydration products were analysed using differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), X-ray diffraction (XRD), and scanning electron microscope (SEM). The optimal thermal activation temperatures and the activation mechanisms of the calcined coal gangue were also studied. The results indicated that the optimal calcination temperature of coal gangue was approximately 600 °C. The bulk density and compressive strength of AAC samples were approximately 609 kg m⁻³ and 3.68 MPa, respectively, which passed the requirements of A3.5, B06 level of AAC sample regulated by GB/T 11969-2008. Before the autoclaving, the hydration products in ACC containing CGC and ITOs were tobermorite, hibschite, ettringite, and C–S–H gel; during the autoclaving, ettringite was decomposed and more tobermorite formed coupled with C–S–H gel and hibschite.

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1. Introduction

Coal gangue is one type of industrial solid waste that is discharged when raw coal is excavated and washed after mining. Based on statistics [1], every ton of coal production will cause 0.15–0.2 metric tons of coal gangue to be generated. In 2012, the total of coal gangue stockpiled in China reached 5 billion metric tons and was increasing at a rate of 0.15–0.2 billion metric tons per year [2,3]. Coal gangue has become one of the major industrial solid wastes in China. The primary disposal method of the coal gangue waste is storage, which can easily cause the coal gangue to weather and spontaneously combust during long periods of exposure, resulting in environmental problems, such as dust

generation, gas emissions (CO, CO₂, SO₂, H₂S, NO_x) and heavy metal contamination [4]. However, coal gangue may constitute a useful by-product that can be used to generate electricity [4,5], used to produce cement materials [6–8] and glass-ceramics [9] and used to backfill mines [10–12], although the rate of reuse is lower than 15%. The primary chemical constituents of coal gangue are SiO₂, Al₂O₃ and similar mineral oxides, and its primary mineralogical phases are feldspar, quartz and similar silicates [13]. Therefore, reactions between coal gangue and water are very slow [14], and the cementitious ability of the original coal gangue is very low. Kim and Hong reported that calcination was an effective method for stimulating the activity of coal gangue [15]. At high temperatures, the atomic movements in the coal gangue were significantly enhanced, resulting in the rapid removal of bonded water from the minerals and the substitution of magnesium, calcium, iron and other cationic atoms in interstitial positions. Calcination may also

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cause insufficient bonding between the silica and alumina octahedra, thus generating a great deal of bond breakage at the free ends. Research has shown that coal gangue calcined at 700 °C exhibited greater activity [16]. Therefore, it could be expected that the activity of coal gangue would be improved by calcination. After calcination or spontaneous combustion, the broken Al–O and Si–O bonds will improve the chemical activity of the material, resulting in higher cementitious activity.

Iron ore tailings are also an industrial solid waste; these mostly result from the process of iron production. In recent years, the amount of the iron ore tailings has grown rapidly due to rapid development of the steel industry in China. From 2007 to 2011, the stockpiles of iron ore tailings increased by 2.899 billion metric tons; in 2011 in particular, approximately 806 million metric tons were generated. However, the comprehensive recycling of these tailings totalled only 307 million metric tons. To reduce the amount of this waste, a new technology has been applied in recent years in which iron ore tailings are used as the primary siliceous raw material to produce autoclaved aerated concrete (ACC). Mechanical grinding of the iron ore tailings has been used to reduce their grain size and increase their chemical activity when they are used as siliceous materials in AAC [17–19].

In view of the energy savings and waste stream reduction via recycling, a new type of AAC material was developed using waste coal gangue and iron ore tailings materials. In this study, coal gangue first underwent a low-temperature activation treatment and was then crushed and added to the iron ore tailings to produce the AAC. The coal gangue calcination temperature, phase transformation process and activation reaction mechanism were also investigated. Meanwhile, the mechanical and physical properties of the AAC were also analysed in detail. This technology represents a new process for the use of coal gangue and iron ore tailings for the purpose of comprehensive utilisation of solid waste in China.

2. Materials and methods

2.1. Experimental materials

The AAC samples were prepared using the following raw materials: coal gangue, autoclaved coal, iron ore tailings, lime, P.O 42.5 Portland cement and desulphurisation gypsum. The chemical compositions of the raw materials are listed in Table 1.

2.1.1. Portland cement

The initial and final setting times of the P.O 42.5 Portland cement were 118 min and 190 min, respectively.

2.1.2. Iron ore tailings

The iron ore tailings were from Beijing and its mineralogic phases were identified by XRD, as shown in Fig. 1. Table 1 shows that the amount of SiO₂ in the iron ore tailings was approximately 68.96%, and the level of residual iron in the tailing, which was present in the form of magnetite, was approximately 5.2%. The iron ore tailings thus were of a high-silicon low-iron magnetite–quartzite type.

2.1.3. Coal gangue

The coal gangue used in this study was from Heilongjiang. The mineral phases in the coal gangue are shown in Fig. 2, which indicates quartz and clay minerals (kaolinite, montmorillonite, illite, and muscovite) as the main mineral phases,

and siderite and anorthite as the minor phases. Quartz and anorthite are highly inert phases as they belong to the trigonal and triclinic crystal systems. In contrast, kaolinite, illite and montmorillonite are layered silicates which are easily altered with increased levels of chemical activity. Hence, it may be inferred that the high activity of the coal gangue was primarily due to the activation of clay minerals at relatively low temperatures.

2.1.4. Lime

The digestion time of the lime was 13 min, and the digestion temperature was 66 °C.

2.1.5. Desulphurisation gypsum

The primary mineral phase of desulphurisation gypsum is CaSO₄·2H₂O.

2.1.6. Aluminium powder

Aluminium powder was used as a gas-producing agent for foaming of the slurry.

2.2. Material preparation

First, the coal gangue was crushed to a particle size of less than 2 mm. Then, the powder was dried and further crushed to a fine powder with a specific surface area of 321 m² kg⁻¹ using a ball mill. Finally, the coal gangue samples were calcined in a CD-1400X muffle furnace for 4 h at five temperatures (500 °C, 600 °C, 700 °C, 800 °C, 900 °C) and then water quenched. The iron ore tailings were first dried and then crushed using a ball mill to yield a specific surface area of 317 m² kg⁻¹.

2.2.1. Preparation of AAC

The mass proportions of CGC in the dry mixtures were 40%, 38%, 35%, 33%, 30%, 28%, 25%, 23%, 20%, 18%, 15%, 13%, 10%, 8%, 5%, 3% and 1%, respectively. The corresponding mass proportions of crushed iron ore tailings were 20%, 22%, 25%, 27%, 30%, 32%, 35%, 37%, 40%, 42%, 45%, 47%, 50%, 52%, 55%, 57% and 59%, respectively. The amounts of lime, cement and gypsum in each dry mixture were 25%, 10%, and 5%, respectively, and those of aluminium powder and water were 0.06% and 60% by mass of dry mixtures. The AAC samples with these various amounts of CGC were denoted as CT1–CT17.

Warm water (55 ± 1 °C) was added into the well mixed raw material powders and the slurries were stirred for 90 s. Then aluminium powder was added into the slurries with additional 40 s stirring. The obtained slurries were cast into pre-heated steel moulds with dimensions of 100 mm × 100 mm × 100 mm, and they were allowed to expand and harden at 55 ± 1 °C for 3 h in a chamber. After removing the expanded parts above the top surface of the moulds, the trimmed samples were de-molded and placed in an industrial autoclave for 8 h at 185 °C with a pressure of 12.5 bars.

2.3. Sample characterisation

The bulk density and compressive strength of the AAC samples were tested according to GB/T 11968-2008. For bulk density measurements, three samples of each AAC mixture were oven dried at 60 ± 5 °C for 24 h, then at 80 ± 5 °C for 24 h, and finally at 105 ± 5 °C for 24 h, consecutively. The mass of the oven dried samples were used to calculate the bulk density. The compressive strength were performed on three samples of each AAC mixture with a moisture content of 8–12% at a loading rate of 2.0 ± 0.5 kN/s.

The X-ray diffraction (XRD) spectra of the coal gangue, iron ore tailings and AAC samples were performed using a D/Max-RC diffractometer (Japan) with Cu K α radiation, voltage of 40 kV, current of 150 mA and 2 θ scanning ranging between 5° and 90°. The DSC–TG analysis of the coal gangue was performed from 20 to 1000 °C at a rate of 10 °C/min using a Netzsch STA 449C thermal analyser with dry air as the stripping gas. FESEM observation was performed to analyse the hydration products of the AAC samples using a Zeiss SUPRA™55 scanning electron microscope coupled with a Be4-U92 energy spectrum.

Table 1
Chemical composition of raw materials (wt.%).

Material	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	FeO	MgO	CaO	Na ₂ O	K ₂ O	SO ₂	Loss
Coal gangue	34.05	26.00	0.49	1.70	0.61	0.67	0.00	0.16	0.28	32.76
Autoclaved coal	51.05	36.71	3.12	0.42	0.92	1.05	0.00	0.23	0	0.72
Iron ore tailings	68.96	7.68	2.32	4.47	3.64	4.35	1.41	1.85	0.024	2.49
Lime	5.45	3.85	1.68	0.08	3.56	78.76	–	1.25	0.45	3.93
Cement	25.06	6.10	3.31	0.21	3.87	55.56	0.23	0.95	–	4.16
Desulphurisation gypsum	2.84	0.78	0.25	0.03	0.47	40.13	0.14	0.12	33.21	–

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