Experimental investigation on the deterioration of the physical and mechanical properties of autoclaved aerated concrete at elevated temperatures

Abstract: Autoclaved aerated concrete (AAC) has been extensively studied and applied in the past decades because of its excellent thermal insulation and acoustic performance, energy efficiency, and outstanding structural performance. To investigate the deterioration characteristics of AAC under high temperatures, the physico-mechanical properties of AAC at different temperatures were tested by mass loss, wave velocity, and compressive tests, and the deterioration mechanism was discussed by X-ray diffraction (XRD), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM) tests. The results showed that after exposure to elevated temperatures, the parameters of apparent form, mass loss, wave velocity, and compressive strength underwent remarkable conversions. It was observed that the ultrasonic behavior of AAC was affected at high temperatures as it increased at 100°C and decreased beyond 100°C. In addition, the compressive strength exhibited a two-stage transformation, slightly increased from ambient temperature to 300°C, and exhibited a rapid reduction beyond 300°C. At 900°C, the specimen lost its strength. By XRD, TGA, and SEM, it was confirmed that a series of physicochemical changes in AAC, such as the water escape and evaporation, decomposition of calcium silicate hydrate and calcium carbonate, and structural damage, were the primary reasons for the deterioration of the physical and mechanical properties at elevated temperatures.

Keywords: autoclaved aerated concrete, temperature, compressive strength, microstructure, deterioration mechanism

1 Introduction

Autoclaved aerated concrete (AAC) is a porous silicate concrete comprising silicon and calcareous agents as the main raw material and aluminum powder as the aerating agent through multichannel processes, such as pouring, forming, static stopping, cutting, and autoclaving [1,2]. Owing to its excellent thermal insulation and acoustic performance, energy efficiency, and outstanding structural performance [2], it has been widely applied in various architectural projects since the last century [3]. China, Southeast Asia, India, and the Middle East have emerged as significant regions for AAC manufacturing and utilization [4]. Its low density and high porosity are key factors contributing to excellent thermal insulation, enabling buildings to maintain consistent temperatures throughout hot and cold seasons. Moreover, AAC’s porous structure facilitates effective sound absorption and isolation, resulting in outstanding acoustic performance. These qualities make it a prime choice for applications such as soundproof rooms and residential buildings.

Compared with ordinary concrete, AAC is lightweight and contains more pores, which significantly impact its properties. Thus, many studies on the physical, mechanical, mineralogical, and microstructural properties of AAC have focused on the effect of density or porosity [5–8]. Petrov and Schlegel [7] pointed out that the growth in compressive strength was always linked to an increase in bulk density and a reduction in porosity. Another report stated [6] that the thermal conductivity of AAC showed a correlation with the constituents of concrete under a similar porosity and pore-size distribution. In addition, the outstanding ability of water absorption and moisture
diffusivity of AAC was observed to be related to its low bulk density [9]. Hu et al. [10] conducted a series of physical and mechanical tests to confirm that the compressive strength and elastic modulus were mathematically related to density. Certain scholars focused on the properties of AAC from other perspectives. Huang et al. [11] and Fazel et al. [12] studied the microstructure of AAC to summarize the controlling factors of properties. Wang et al. [13] compared X-ray diffraction (XRD) patterns prior to and after autoclaving and concluded that autoclaving can facilitate the formation of tobermorite to improve the compressive strength of the matrix. Zafar et al. [14] analyzed the calcium silica ratios of waste granite dust-added AAC and proposed that the AAC microstructure was enhanced by the waste granite dust, which was reflected by an increase in compressive strength with an increase in waste granite dust. Through testing, high-reactivity materials such as rice husk ash [15] and air-cooled slag [16] could accelerate reactions for the formation of hydration products to enhance the properties of AAC. Cong et al. [17] tested AAC for 50 freeze–thaw cycles, conducted appearance inspections, quality measurements, and compression tests, and recorded the experimental data of the deterioration of properties.

As is known, concretes including AAC suffer from fire disasters in engineering structures, causing spalling of components, reduction in building integral rigidity, and eventually, the collapse of structures [18]. The exposure of mineral constituents to high temperatures can induce physicochemical effects. The utilization of AAC products in building structures has recently gained considerable interest worldwide. Therefore, it is important to study AAC properties at high temperatures and evaluate its fire resistance. Although the physical and mechanical properties of AAC at high temperatures have been reported, there is a distinction in the conclusions. For example, Tanaçan et al. [19] explored the effects of elevated temperatures and various cooling regimes on the properties of aerated concrete, and showed that the strength of aerated concrete is gradually lost due to the propagation of the cracks between 600 and 800°C. Sun and Zhang [20] indicated that the compressive strength of lightweight aerated concrete reaches its maximum at 100°C and slightly decreases over 100°C. Nevertheless, Andreini et al. [21] conducted high-temperature tests on a variety of masonry materials, including AAC. The results showed that the maximum compressive strength of AAC is achieved at approximately 250°C.

The above researches show that although the mechanical properties of AAC after high temperature are concerned, relevant research is still insufficient. Due to the different raw materials used in the production of AAC, the evolution of physical and mechanical properties of AAC after high temperature is inconsistent and unable to meet the requirements of current engineering projects. In recent years, the production of AAC from solid waste, such as using coal bottom ash or tailings instead of sand, has been widely accepted. However, the performance of AAC generated from solid waste is also a problem that needs to be studied, and its physical and mechanical properties also need further understanding. In this study, a series of tests were conducted to investigate the physical and mechanical properties of AAC produced from solid waste materials at different temperatures. The effects of temperature on the mass loss, wave velocity, compressive strength, and microstructure of AAC were investigated. The deterioration mechanism of temperature on the performance of AAC was revealed.

2 Materials and methods

2.1 Specimen preparation

AAC with a density grade of B06 was chosen in this experiment. The raw materials of AAC are mainly solid waste materials (52%), lime (10.5%), cement (12.5%), aluminum paste (0.09%), activator (0.06%), and waste mortar (25%). Solid waste materials consist of coal bottom ash and desulfurization gypsum, as well as silicon tailing sand and mud, in a specific ratio of silicon tailings sand:silicon tailings mud:coal bottom ash:desulfurization gypsum = 4:3:3:1.5. The water to solid ratio is 0.61.

The specimens were cut from prefabricated AAC slabs and had a cubic shape with dimensions of 100 × 100 × 100 mm³ (Figure 1), and were measured to ensure experimental accuracy and preserved in a cool place prior to the experiment according to the Chinese standard GB/T 11969-2020 [22]. The test results show that the dry density of the specimen is 598.5 kg·m⁻³, the natural moisture content is 2.0%, the saturated water content is 69.4%, and the porosity is 78%.

2.2 Experimental method

2.2.1 Temperature treatment

The cubic specimens were heated at 100, 300, 500, 700, and 900°C, respectively, at the rate of 10°C·min⁻¹ in a muffle furnace and kept with the temperature maintained for 1 h to ensure a uniform temperature distribution among
Afterward, the muffle furnace was turned off, and the specimens were cooled to ambient temperature and were used for the following tests.

2.2.2 Mass loss

The mass of the specimens was measured using an electronic scale with an accuracy of 0.01 g prior to and after the heating treatment. Thereafter, the obtained data were processed to calculate the mass loss ratio by dividing the change in mass prior to and after the high-temperature heating by the initial mass. The formula is as follows:

\[
W = \frac{M_1 - M_2}{M_1} \times 100\%
\]

where \( W \) (%) is the mass loss rate of AAC, \( M_1 \) (g) is the initial mass of AAC, and \( M_2 \) (g) is the mass after heat treatment.

2.2.3 Wave velocity

The wave velocity of AAC, which could better reflect the internal damage to the AAC specimens, was assessed using a nonmetallic ultrasonic testing analyzer. The specimens prior to and after the heat treatment were prepared for wave velocity tests. During the tests, it was necessary to obtain data perpendicular and parallel to the direction of rise for the following analyses.

2.2.4 Compressive strength

The compressive strength of AAC was examined using a Uniaxial Compression Test System. The loading was controlled by displacement at a rate of 0.2 mm/min\(^{-1}\). Prior to loading, a linear variable differential transducer was applied to measure the compression of the specimens, and the measured values were used to evaluate the axial strain. The compressive direction of AAC was qualified as parallel and perpendicular to the direction of rise (Figure 2). Three valid datasets were obtained for each experimental group to eliminate system errors and ensure the repeatability of the test results.

Figure 1: AAC specimens: (a) AAC slab and (b) specimens of 100 × 100 × 100 mm\(^3\).

Figure 2: Direction of loading for compression test of specimen.
2.2.5 XRD

The chemical composition and phase identification of AAC at different temperatures were investigated by XRD. The specimens were ground into powder, and the XRD patterns were recorded using the diffractometer.

2.2.6 Thermogravimetric analysis (TGA)

The conversion of mineral constituents was analyzed by TGA. The specimens were ground into powder and tested with a TGA8000 thermogravimetric analyzer. Approximately 10 mg of the specimen was placed in a ceramic crucible and analyzed in a flowing air atmosphere at a heating rate of 10°C·min⁻¹, from 25 to 1,000°C.

2.2.7 Scanning electron microscopy (SEM)

The microstructure of the AAC specimens was characterized by SEM. The AAC specimens were cut into pieces with thicknesses in the range of 1–2 mm. Prior to the test, the specimens were coated with gold to obtain good SEM images.

3 Results and analyses

3.1 Appearance inspection

The images of the surface appearance, after exposure to different temperatures, are shown in Figure 3. The surface color of the specimens constantly deepened, and cracks developed inch-by-inch with an increase in temperature. No apparent cracks were detected at 300°C. At 700°C, fine cracks were intertwined and formed a reticular structure, which covered the entire cross-section of the specimens. At 900°C, crocodile cracks were observed, and the load-bearing capacity of the specimens was practically lost. Therefore, it was not necessary to perform the compression test on this group.

Figure 3: Pictures of specimens after temperature treatment: (a) ambient, (b) 100°C, (c) 300°C, (d) 500°C, (e) 700°C, and (f) 900°C.
3.2 Mass loss

The mass loss of the specimens exposed to different temperatures is shown in Figure 4 in terms of percentage. The mass loss continuously increased with an increase in temperature, which might have been related to the physico-chemical reactions and evaporation of water in AAC. After exposure to multiple temperatures, the mass loss of AAC with temperature increased by 0.76, 4.39, 7.05, 8.97, and 10.77% at 100, 300, 500, 700, and 900°C, respectively. The percentage increase values in adjacent temperature were 3.63, 2.65, 1.92, and 1.80%, respectively. This shows that the rate of mass loss was gradually slowing down.

3.3 Wave velocity

The wave velocity results at different temperatures are shown in Figure 5. The statistics in perpendicular groups were observed to be always higher than those in parallel groups, which confirmed that the compactness of AAC was better exhibited perpendicular to the direction of rise. A slight increase occurred for all groups from ambient temperature to 100°C. When the temperature exceeded 100°C, the specimens underwent a drop in wave velocity. It was concluded that the wave velocity perpendicular to the direction of rise would be more affected than that parallel to the direction of rise. The gap between the two directions decreased as the temperature increased. At 700°C, the percentage wave velocity of the perpendicular and parallel groups decreased by 27.84 and 23.11%, respectively.

3.4 Compressive strength

The stress–strain curves of some representative AAC specimens are shown in Figure 6. The evolution of compressive strength as a function of temperature is shown in Figure 7 and Table 1.

With the increase of temperature, the compressive strength of AAC first increased and then decreased, and the compressive strength of the specimens reached the maximum at 300°C. The average compressive strengths were 3.98 and 5.39 MPa for the parallel and perpendicular groups at 300°C, respectively. They increased by 17.06 and 21.67% compared to those at ambient temperature. As the temperature increased, particularly after 300°C, an apparent reduction in strength was observed for the specimens. The minimum compressive strengths (3.18 and 4.14 MPa, respectively) were obtained at 700°C.

In addition, it is worth noting that the loading direction had a major impact on the compressive strength of AAC as the values of the perpendicular groups always exceeded those of the parallel groups. The temperature had a strong influence on the strength of the specimen in the direction perpendicular to the rising direction.

3.5 Composition variation

The XRD spectra of AAC exposed to different temperatures are shown in Figure 8. The mineral constituents of AAC at ambient temperature were silica, tobermorite, calcium silicate hydrate (C–S–H), calcium sulfate, and calcium
carbonate. Silica and tobermorite, the main constituents of AAC, constitute the compact structural framework of AAC and endow it with strong compressive properties. Other constituents contributed more or less to the strength of AAC. Throughout the entire heating process, the distinct diffraction peaks of silica, tobermorite, and calcium sulfate were identified in all the temperature groups. The values of the silica peaks formed the characteristics of three stages as the height of the diffraction peak decreased, increased, and decreased again. However, the values of the tobermorite and calcium sulfate peaks were relatively

Table 1: Compressive strength of specimens

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Compressive strength (MPa)</th>
<th>Ambient</th>
<th>100°C</th>
<th>300°C</th>
<th>500°C</th>
<th>700°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Parallel to the direction of rise</td>
<td>3.29</td>
<td>3.69</td>
<td>4.22</td>
<td>3.58</td>
<td>3.18</td>
<td></td>
</tr>
<tr>
<td>Perpendicular to the direction of rise</td>
<td>3.34</td>
<td>3.54</td>
<td>4.07</td>
<td>3.52</td>
<td>3.23</td>
<td></td>
</tr>
</tbody>
</table>

Figure 6: Stress–strain curves of specimens: (a) perpendicular to the direction of rise and (b) parallel to the direction of rise.

Figure 7: Compressive strength of AAC after different temperatures.

Figure 8: XRD spectra.
stable. The diffraction peaks of C–S–H only existed from ambient temperature to 300°C, and the diffraction peaks disappeared after 500°C, demonstrating that the substance had been completely decomposed at 500°C. From 500 to 700°C, the diffraction peaks of calcium carbonate progressively decreased. At 900°C, the diffraction peaks disappeared, and the calcium carbonate content dropped to zero. At this point, the remaining components were silica, tobermorite, and calcium sulfate. This showed that some components in AAC underwent chemical changes at high temperatures.

3.6 TGA

The thermogravimetric curves of AAC are shown in Figure 9. AAC continually lost mass during the heating process. In general, two endothermic peaks could be observed on the derivative thermogravimetric (DTG) curve. The first endothermic peak occurs between 25 and 300°C, which was attributable to the evaporable water and decomposable C–S–H [23]. When the temperature rose to 700–760°C, another endothermic peak was observed due to the decarburization reaction of calcium carbonate. When the temperature rose further, the TG and DTG curves remained approximately horizontal, and the AAC constituents were relatively fixed. Afterward, the mass loss had practically stopped. At 900°C, the specimen had lost 11.78% of its total mass. The mass loss of AAC was related to its endothermic and thermal decomposition behavior [24].

3.7 Micromorphology

The SEM images of AAC at high temperatures are shown in Figure 10. As shown in Figure 10(a), tobermorite and silica formed a compact structural framework with uniform distribution. Other constituents were attached to the surface of the structural framework. At the ambient temperature (Figure 10(a)), crystallized mineral constituents were observed on the surface of the structural framework. Many well-crystallized speculars, flake tobermorite, and flocculated C–S–H were evenly spread across the surface of the framework, forming a solid dense microscopic reticular structure. No apparent cracks are observed in the SEM images. When the temperature changed from ambient temperature to 100°C (Figure 10(b)), no significant change was observed in the surface crack condition. The distribution of constituents was relatively uniform such that it was similar to that at ambient temperature. At 300°C (Figure 10(c)), the amount of the flocculated C–S–H reduced. Accompanied by the decomposition of mineral constituents, fine pores were observed. At 500°C, fine cracks were observed (Figure 10(d)), and the structure of the matrix was transformed. There was no C–S–H distribution attached to the matrix. When the temperature rose from 500°C to 900°C (Figure 10(d)–(f)), the overall integrity of AAC was reduced because of the development of cracks. The initial shape of the cracks was slender/needle-like and gradually extended. Therefore, the SEM images with the same magnification showed more cracks. At 900°C (Figure 10(f)), the surface was covered with crocodile cracks, and the structural framework was destroyed by these cracks.

4 Discussion

AAC underwent a series of physical and chemical reactions at high temperatures, including loss of water, decomposition, and transformation of components, which led to the change in material composition and destruction of AAC structure.

In the range of 25–300°C, the thermogravimetric curve of the sample had the first significant endothermic peak (as shown in Figure 9). At this stage, the main reason of mass loss was the release of adsorbed water and interlayer water in specimens. When the temperature gradually increased, the moisture in AAC was spilled out through the pore–inter-pore pore wall, combined with rapid mass loss [25]. This resulted in an increase in the wave velocity and compressive strength of the specimen, which reached a maximum when the temperature reached 300°C. The conclusion obtained in this study indicated a slightly higher temperature for the maximum compressive strength compared to the highest value reported by Andreini et al. [21].

When the temperature exceeded 300°C, the performance of AAC deteriorated greatly. At 100–300°C, C–S–H
in AAC had a small amount of decomposition. The structure of C–S–H could not remain constant when the temperature was higher than 400°C [26], the dehydration and the recrystallization of C–S–H began. The diffraction peak of C–S–H in the XRD spectra completely disappeared after 500°C (as shown in Figure 8). In addition, quartz (a crystalline form of silica) was converted from α-quartz to β-quartz at 573°C [24,27]. Within the range of 600–800°C, the thermogravimetric curve of the sample had the second significant endothermic peak (as shown in Figure 9). The disintegration of calcium carbonate damaged the matrix of AAC at this range. At approximately 870°C, β-quartz was transformed into β-tridymite [27]. At this point, as the temperature increased, the ability of AAC to withstand external loads gradually decreased, and the compressive strength also decreased gradually, which showed a strong alignment with Tanaçan et al. [19]. Meanwhile, the appearance of cracks reduced the numerical values of the wave velocity.

Along with these variations, the microstructure of AAC also changed. At 100°C, no apparent cracks were observed, and the integrity of the matrix was relatively excellent and the compressive strength slightly increased. When
the temperature exceeded 300°C, defects such as pores and cracks in AAC caused by material composition decomposition and shrinkage increased gradually (as shown in Figure 10). After 300°C, the decomposition of the C-S-H gel promoted the evolution of the pore structure. In particular, the C-S-H gels suffered from continuous decomposition and shrinkage after 300°C [28,29]. This led to further reduction of wave velocity and compressive strength of AAC specimens. Especially after 900°C, the framework of AAC was totally destroyed by crocodile cracks, and the specimens could no longer bear the external load.

The results above suggested that with the increase of temperature, the materials composition, and microstructure of AAC have changed in varying degrees, leading to changes in its macro-physical and mechanical properties.

5 Conclusions

The physico-mechanical properties of AAC were investigated through an experiment after exposure to elevated temperatures, and the deterioration mechanism was analyzed. The following conclusions were made:

1. The surface morphology of the AAC transformed at a high temperature. The specimens, originally grayish-white, gradually deepened with an increase in temperature. Cracks started to occur when the temperature exceeded 300°C and continued to grow until penetration cracks were formed, which destroyed the structure.

2. The mass loss increased as the temperature rose. It was related to the evaporation and decomposition reactions in the AAC.

3. The wave velocity exhibited a distinct two-stage characteristic when exposed to different temperatures. The maximum value was reached at 100°C and then dropped rapidly.

4. Temperature was a vital factor affecting the mechanical properties of AAC. Within 300°C, the compressive strength gradually increased with temperature. When the temperature exceeds 300°C, the compressive strength continued to decrease. Whatever the temperature, the compressive strength perpendicular to the direction of rise was higher than that parallel to the direction of rise.

5. The deterioration mechanism of AAC was attributed to a series of physicochemical reactions at high temperatures. In the lower temperature range, the compressive strength was slightly increased due to water escape and evaporation. With the increase of temperature, the pore formed by the decomposition and transformation of material components, as well as the cracks formed by thermal stress, led to the deterioration of AAC performance.

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References


