Experimental investigations on water absorption and mechanical properties of expanded perlite mortar under accelerated and natural aging conditions

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Abstract
The objective of this paper is to study the variation in the physical and mechanical properties of expanded perlite mortar with environmental factors and analyze its deterioration mechanism. In this paper, the influence of the heat–rain cyclic and natural aging on the physical and mechanical properties of expanded perlite mortar has been studied. The expanded perlite mortar specimens were subjected to 80 heat–rain cycles (the maximum temperature rises to 70 °C, and the spray volume reaches 1 l/(m² min)) and 60-day natural aging tests (the daily average temperature is 23 °C, with the maximum temperature reaching 36 °C). The corresponding mass loss rate, water absorption, and compressive strength of the specimens at every 10 heat–rain cycles and 10 days were determined. The results show that the mass loss rate gradually increases with the increase in heat–rain cycles and the natural aging days. The variation range was found as about 0.2%∼0.7% and −0.4%∼0.2%, respectively. The water absorption rate gradually decreases with the number of heat–rain cycles and natural aging days; however, the decay rate of the water absorption rate differs by orders of magnitude in the two weathering conditions. The compressive strength does not have an obvious change with the number of heat rain cycles, but it first increases and then decreases with the number of natural aging days. Under artificial and natural aging, the deterioration mechanism and aging speed of expanded perlite mortar are much different. The comparative analysis of the physical and mechanical properties of expanded perlite mortar under artificially accelerated and natural aging conditions can further reveal its evolution model and the corresponding relationships under the two conditions and provide a theoretical basis for establishing a more scientific and reasonable aging system.

1. Introduction
As one of the three areas with the highest carbon emissions [1], the energy consumption and carbon emissions in the building sector are huge [2]. Therefore, achieving energy conservation and emission reduction in this field is of great significance. External thermal insulation composite systems (ETICS) involve fixing a thermal insulation material of a certain thickness on the exterior of the building envelope by pasting or anchoring [3]. It relies on the low thermal conductivity of the thermal insulation layer to achieve thermal insulation performance and has been widely used [4, 5]. The envelope materials encounter the influence of the natural environment or human factors for a long time during the service life, which will deteriorate their physical and mechanical properties and aesthetics, leading to durability issues [6]. Carrying out the performance aging tests and analyzing the aging and deterioration evolution model of thermal insulation materials play an essential role in scientifically and rationally designing ETICS and improving the system durability.
There are two common ways of aging of building materials—artificial accelerated aging (hereinafter referred to as ‘artificial aging’) and natural environmental aging (hereinafter termed as ‘natural aging’) [7]—with various specific methods and standards. Artificial aging involves simulating a certain temperature [8, 9], humidity [10, 11], drying-wetting changes [12, 13], freeze-thaw cycles [14], ultraviolet radiations [15], and other conditions in the aging test box based on the characteristics of the service life environment of the studied material and the characteristics of the material itself. Natural aging is mostly to place the test specimen in a typical service life environment for a certain period to allow it to accept the aging effect of the environment that is most in line with the actual state to evaluate the material durability [16]. Natural aging tests often take a long time and cannot strictly control environmental factors during the natural aging process. Therefore, the results of the natural aging test and the artificial aging test often have certain differences [16–18].

At present, indoor accelerated aging is mainly carried out following the requirements of ETAG 004 [19] to study the durability of insulation materials. The aging test is usually carried out according to the indoor accelerated environmental conditions required. Subsequently, the changes in the microstructure and physical or mechanical properties of the external wall insulation materials or test specimens after aging are evaluated [20–23]. Xiong et al [24] carried out 160 heat-rain cycles on expanded perlite mortar and analyzed the pore structure evolution, water absorption, and moisture absorption characteristics. However, in this study, the aging of expanded perlite mortar in the natural environment was not investigated. Thus, it is not possible to compare and analyze the effects of artificially accelerated aging and natural aging. Petrella et al [25] evaluated the acoustic, thermal, optical, and mechanical properties of mortar with different perlite content. However, the aging trend of expanded perlite mortar performance was not determined. The relationship between artificial aging (according to ETAG 004) and the actual/natural aging results has not yet been clearly explained.

In order to analyze the variation trend and mechanism of physical and mechanical properties of expanded perlite mortar under artificial aging and natural aging conditions, the heat-rain cycle artificial aging test and the natural aging test were conducted in this study. The corresponding mass loss, water absorption, and mechanical properties of the mortar under these two aging modes were compared. Comparing the two results shows the clear difference between the artificially accelerated and natural aging and provides a basis for analyzing the real process and mechanism of material aging.

2. Materials and test methods

2.1. Specimen preparation

In this experiment, the expanded perlite mortar specimen was made with composition 1:1:1.2 (mortar: expanded perlite: water). The mortar is a Type A mortar produced by Zhejiang Jiaxing Hualiang Building Materials Engineering Co., Ltd (China). It has a dry density of 291 kg m$^{-3}$ and thermal conductivity of ≤0.068 W/(m·K), the fire resistance class is A, and the final setting time is ≤2 h. The mortar uses expanded perlite particles as the aggregate, cement as the main cementing material, and fly ash as the auxiliary cementing material, which has good soundness. Among them, the cement is 42.5 ordinary Portland cement. The expanded perlite (particle size: 0.5–1.5 mm; bulk density: 118 kg m$^{-3}$; thermal conductivity: 0.041 W/(m·K); floating rate ≥98%; water absorption rate: 35%; able to withstand the high temperature of 1280°C–1360°C) produced by Henan Xinyang Zhongsen Perlite Application Co., Ltd (China) was used in the study.

An acrylic cylinder with a diameter and height of 5 cm was used as a mold. The expanded perlite, thermal insulation mortar, and other materials were mixed evenly and cast into the mold. The specimen was cured for 24 h in a standard curing room. Once it hardens to a certain degree, the mold can be removed. 138 specimens of expanded perlite mortar were produced in this experiment; the height of each specimen was 5 cm. After the mold was removed, the specimen was cured for 28 days. Then it was placed in an artificial aging test box for the artificial aging test of 80 heat and rain cycles and placed in an outdoor field for 60 days of aging under natural conditions.

2.2. Artificial aging test

The artificial aging test was carried out by a self-developed aging test box. As shown in figure 1, the aging test box is a cubic chamber in which five sides are surrounded by steel plates with thermal insulation materials, and one side is closed by a movable concrete wall. The space inside the box is 1 m × 1 m × 1 m. After the prepared expanded perlite mortar specimens were placed in the box, the closing bolts were tightened to ensure the box sealing. Using industrial compressors and evaporators, a reliable temperature could be provided for the test chamber following the requirements of the test design. Five sprinklers connected to the flow controller were arranged on the top of the box for water spraying on the test specimens according to the design flow to simulate the severe weather conditions of rain after high temperature and perform the heat rain cycle to accelerate the aging of the test specimen artificially.
For the temperature and rain conditions during the artificial aging process, the high-temperature rain artificial aging requires 80 heat-rain cycle tests following the requirements of ETAG 004 [19]. The steps of each heat-rain cycle test are as follows.

1. Heating to 70 °C (temperature rise within 1 h), and then keeping it at (70 ± 5) °C and 10%—30% RH for 2 h (total 3 h);
2. Spraying water for 1 h—the water temperature and volume being (15 ± 5) °C and 1 l/(m² min), respectively;
3. Leaving for 2 h (drainage).

In this experiment, the 6-h heat-rain cycle was divided into four stages—heating, maintaining, raining, and resting. The design temperature in the test box at each stage of 7–10 heat-rain cycles is shown in figure 2. Due to a certain error in the temperature control of the aging test box, there is a specific error between the actual temperature in the box and the design temperature. It can be seen from figure 2 that the actual temperature in the box during the maintaining stage is about 80 °C, which is almost 10 °C higher than the design temperature. In addition, the humidity in the aging test chamber was constantly changing during the heat-rain cycle. Generally, the humidity in the chamber is low during the heating and maintaining phases, while the humidity in the chamber increases sharply after the rain starts. Figure 2 also illustrates the humidity changes in the test chamber during 7–10 heat-rain cycles.
The artificial aging test started at 9:00 on April 6, 2021, and ended at 21:00 on May 26, 2021. During the aging test, at the end of the 10th, 20th, 30th, 40th, 50th, 60th, 70th, and 80th heat-rain cycles, a certain number of expanded perlite mortar specimens were taken out to determine mass loss rate, water absorption, and mechanical properties.

2.3. Natural aging test
An undisturbed outdoor place was selected for the natural aging test. As shown in figure 3, the test specimens were placed on the test platform to allow them to accept the effects of ambient temperature, humidity, rainfall, and wind under natural conditions. The expanded perlite mortar specimens were aged under natural conditions for 60 days in this test. A certain number of specimens were taken out on the 10th, 20th, 30th, 40th, 50th, and 60th days to determine mass loss rate, water absorption, and mechanical properties.

In order to obtain specific aging environmental data during the natural aging process, a small climate observatory was set up near the specimen placement platform to collect relevant data in this experiment. The climate observatory was equipped with a temperature and humidity sensor, a rain gauge, and an anemometer. The photovoltaic solar power supply system (35W solar panel and 38AH battery) was used to supply the required electric energy, which recorded the collected data every hour. The natural aging test of expanded perlite mortar started on April 28, 2021, and ended on June 27, 2021. The temperature, relative humidity, wind speed, and rainfall information during the period are shown in figure 4. During this period, the outdoor natural environment temperature gradually increased, and the maximum temperature was close to 36 °C, and the average temperature was 23 °C. At the same time, there was sufficient rainfall. It was similar to the artificial accelerated aging test environment and had certain effects on the natural aging of the expanded perlite mortar.

2.4. Performance test on the aging specimens
Performance testing included assessing the mass loss rate, water absorption, and compressive strength changes of the expanded perlite mortar specimens with different aging degrees. The mass loss rate test needed the specimens to be dried (temperature 105 °C) to constant weight and weighed before the aging test. Then artificial aging and natural aging were performed on these specimens. The specimens were then taken out and repeated the same procedure at different aging times. The specimens were dried to a constant weight and test mass, and the mass loss rate was determined by comparing the difference between the two specimens. The mass loss was measured by UW6200H electronic balance, Shimadzu, with an accuracy of 0.01 g.

The compressive performance of the expanded perlite mortar was tested using the SANS-CMT-5205 universal testing machine. Three specimens were tested for compressive strength for each artificial aging and natural aging time mode (The average value was taken as the representative compressive strength.). The load was controlled by displacement during the test. The loading speed was set as 3 mm min⁻¹, and the force measurement range was 0–300 kN.

According to ASTM C1585–13 [26], the test steps for the water absorption determination of expanded perlite mortar are as follows.

(1) The test specimens were dried to a constant weight and weighed (temperature 105 °C) (mass recorded as \( m_0 \));
The test specimens were sealed using a waterproof material and silicone rubber, as shown in figure 5. It was ensured to spare 1 cm from the bottom surface of the test specimens not to be sealed to prevent the waterproof material from absorbing water at the contact point of the side of the test specimen [27]. In addition, two open-air holes at the sealing material on the top surface of the test specimen were made to ensure proper exhaust. The mass of the test specimen after sealing was recorded as \( m_1 \);

(3) The ambient temperature was kept at a temperature of 18 °C–28 °C (allowable fluctuation ±2 °C) and relative humidity of 40%–60% (allowable fluctuation ±5%). The sealed test specimens were put into the aluminum alloy pan with the bottom surface (unsealed surface) facing down. Water was poured into the pan to make the test specimens immersed in water depth of 5 ± 2 mm;
The samples were taken out at time intervals of 5 min, 20 min, 1 h, 2 h, 4 h, 8 h, 12 h, and 24 h. The surfaces were wiped clean with a slightly damp sponge within 1 min, and the mass at that moment was determined, which was recorded as $m_t$;

The specimens were tested once every 24 h for more than 24 h until the water came out on the upper surface of the test specimen or the mass became constant;

The mass variation per unit area, $\Delta m_t$ (g cm$^{-2}$) of the specimen at each moment was calculated according to equation (1).

$$\Delta m_t = \frac{m_t - m_1}{A}$$

3. Results and analysis

3.1. Mass loss analysis

In this paper, the mass loss rate is used to characterize the mass change of the expanded perlite mortar specimens during the aging process. The meaning of this index is the percentage increase or decrease of the drying mass of the specimens after aging compared to the drying mass before aging. In order to adapt to the ‘mass loss’, this article defines the mass loss rate of the specimen under the condition of reduced mass after aging as a positive value, whereas a negative mass loss rate means that the mass of the specimen increased after aging. According to the definition, the mass loss rate of each specimen under artificial and natural aging is shown in figures 6(a) and (b), respectively. It can be seen that during the 10 to 80 heat-rain cycles of artificial aging, the mass loss rate of each specimen is positive, i.e., the mass of the specimen is reduced. The variation range of the mass loss rate is about 0.1%—1.0%, and the variation range of the average value of the mass loss rate of the three specimens at each aging stage is about 0.2%—0.7%. During the 60 days natural aging process, the mass loss rate of each test specimen within 10—30 days is negative, and the positive value appears after the 40th day. The average range of the mass loss rate of the three test specimens at each aging stage changes is about −0.4%—0.2%. This phenomenon indicates that the mass of the specimens of initial natural aging has increased, and the mass begins to decrease after the aging time reaches a certain level. Vyšvařil et al [28] demonstrated that incorporating expanded perlite significantly increases the porosity of the mortar. It was shown that the porosity of the expanded perlite mortar first decreased and then increased with the aging process [24], indicating that the substances contained in the mortar would first increase or decrease after subtraction, which is similar to the trend in the natural aging environment in this study. During the early stage of natural aging, the specimen mass is higher than that at the initial time, which may be attributed to the formation of new products due to the pozzolanic reaction between the vitreous aluminosilicate component of perlite and calcium hydroxide released from cement clinker [29]. Since then, as the degree of aging increases, the mass loss due to aging exceeds the mass of the newly formed substance, resulting in a gradual decrease in the mass of the test specimens.

Using the least-squares method, the average values of the mass loss rate at each aging stage were linearly fitted, as shown in figures 6(c) and (d). Although the correlation coefficient between the mass loss rate and the number of heat-rain cycles in the artificial aging process is low ($R^2$ is 0.54), the measured average data points are included in the 95% confidence interval of the fitted straight line. As a general trend, it can be considered that the mass loss rate gradually increases with the number of heat-rain cycles, and the aging of the specimen is getting
more and more severe. Under natural aging, the mass loss rate of the expanded perlite specimens linearly changes with the aging time, and the fitting correlation is better ($R^2 = 0.77$), which also indicates the degree of aging of the expanded perlite mortar under natural conditions is getting increasingly severe.

### 3.2. Water absorption

According to equation (1), the water absorption per unit area can be calculated at different times. In this paper, three specimens were taken from artificial and natural aging to test the water absorption. The average value is used as the water absorption performance index under artificial aging or natural aging. It can be seen from figure 7 that the water absorption per unit area of the expanded perlite mortar specimen increased sharply in the first two hours. The slope of the water absorption curve was very steep, and then the water absorption curve began to slow down within 2–4 h gradually. Then the water absorption increases very slowly and gradually stabilizes.

The water absorption of expanded perlite mortar is a capillary water absorption process, which is greatly affected by the microscopic pore structure of the mortar. In the initial stages of water absorption, the capillary water is quickly lifted into the pores. Afterward, the smaller capillary pores are filled with water, and the weaker capillary suction in the larger pores causes the water absorption to slow down [30]. Therefore, it can be considered that the pore structure of the expanded perlite mortar, especially the structural characteristics of the capillary pores, greatly influences the water absorption rate at the initial stage of the water absorption. The water absorption rate at the initial stage of the water absorption test also reflects the condition of the capillary pores of the expanded perlite mortar. Combined with the previous analysis results, this study takes the slope of the water absorption per unit area curve in the 0–2 h period to characterize the water absorption rate at the initial stage of the water absorption test.

During artificial and natural aging, the change in the water absorption rate of expanded perlite mortar is shown in figure 8. It should be noted that the water absorption rate at the 30th and 60th cycles in the artificial aging test is apparently unreasonable and unrealistic, which is recorded as invalid test data in this article. The linear fitting analysis of other data shows that the water absorption rate of the expanded perlite mortar gradually decreases with the increase of the number of heat-rain cycles, and its $R^2$ is 0.82. This phenomenon is related to the slow pozzolanic reaction of expanded perlite [28]. Expanded perlite has a high water absorption rate, and the amorphous SiO$_2$ contained in it continues to react with Ca(OH)$_2$ under artificial aging conditions [31]. The resulting product fills the pores and seals the expanded perlite particles, gradually slowing the water absorption rate of the expanded perlite mortar. It can further be seen from figure 8(b) that the water absorption rate of expanded perlite mortar has certain fluctuation during the natural aging process. However, the overall trend is decreasing, while $R^2$ is merely 0.54 when linearly fitted. From the fitting results, it is inferred that the water absorption rate of the expanded perlite mortar with artificial aging declines, i.e., the slope of the fitting curve is $-0.00065$, whereas the slope of the decay rate fitting curve during natural aging is $-0.0011$, the difference between the two is an order of magnitude, which indicates that when the expanded perlite mortar is artificially aged, its influence on the water absorption rate is different from that of the natural aging environment. Since artificial aging and natural aging respectively use the number of heat-rain cycles and the number of aging days to represent the aging time, it is impossible to establish a direct correlation between the decay rate of the water absorption under the two aging conditions. When designing an indoor artificial aging test for the water absorption rate of expanded perlite mortar, a more reasonable indoor aging system should be considered.

![Figure 7. Water absorption test results: (a) Artificial aging (b) Natural aging.](image-url)
3.3. Compressive strength

As the size of the expanded perlite mortar specimens has certain disparities, in order to unify the comparison standard, this paper uses the stress-strain curve to compare its uniaxial compressive behavior. The strain at any instant is calculated by dividing the corresponding compressive displacement by the specimen height (50 mm). Similarly, the stress is calculated by dividing the corresponding pressure at that instant by the cross-sectional area of the specimen. There are a large number of specimens. The stress-strain curves of each specimen at the 0th, 40th, and 80th heat-rain cycle and the 0th, 30th, and 60th natural aging day, are shown in figure 9(a). It is observed from the stress-strain relationship that regardless of the aging condition, for the expanded perlite mortar specimens, there is an upper concave section at the initial stage of the uniaxial compression test. The main reason for this phenomenon is that the expanded perlite mortar has a high porosity. Under external load, the pore volume will be rapidly compressed, and the mortar will gradually become dense, resulting in a gradual increase in its modulus. Therefore, the stress-strain curve at this stage is concave upward. As the load continues to increase, the stress-strain curve of the expanded perlite mortar shows a nearly linear change. It can be considered that this stage is elastic, the deformation gradually increases, and certain cracks begin to appear in the mortar. Afterward, the stress-strain curve starts transforming into a convex nonlinear change. The deformation of the mortar and the development of the cracks start accelerating. Also, the plastic deformation gradually increases. When the deformation of the specimen reaches the highest point of the stress-strain curve, a macroscopically connected fracture surface is formed, and the internal structure of the thermal insulation mortar suffers significant damage. The deformation increases drastically, and the bearing capacity gradually decreases. Figure 9(b) is the picture of the specimen after the uniaxial compression test in which the longitudinal penetration cracks of the expanded perlite mortar can be seen.

The stress corresponding to the highest point of the stress-strain curve is taken as the compressive strength of the expanded perlite mortar specimen, while the average value of the compressive strength of the three
specimens at each time point of artificial aging and natural aging is taken as the representative compressive strength. The results of compressive strength values under heat-rain cycles or natural aging days are shown in figure 10. The compressive strength fluctuates with the number of heat-rain cycles for the expanded perlite mortar under artificial aging conditions. It does not have a noticeable variation trend. Except for the initial time (0 d), the compressive strength of expanded perlite mortar under natural aging increases first and decreases subsequently with the number of days of natural aging. The variation trend of compressive strength is similar to the change mentioned earlier for mass loss rate. During artificial aging, the deterioration degree of expanded perlite mortar increases, and the porosity increases. However, in the early stage of natural aging, thermal insulation mortar continues to hydrate, resulting in decreased pores and increased mass. Correspondingly, its compressive strength gradually increases [33]. When natural aging progresses to a certain degree, the aging becomes severe, causing the porosity of the expanded perlite mortar to increase, thereby gradually reducing its compressive strength. Expanded perlite mortar cannot be used as load-bearing member material because of its low strength. However, its lightweight and low thermal conductivity make it suitable for thermal insulation layer material on exterior wall surfaces, roofs, and pipes. Overall, there is a significant difference in the compressive strength of expanded perlite mortar under artificial and natural aging conditions. A more reasonable indoor artificial aging system should be designed to simulate the service life environment of expanded perlite mortar.

4. Conclusions

In this paper, artificial and natural aging tests were carried out on the expanded perlite mortar specimens. The corresponding mass loss rate, water absorption, and uniaxial compressive behavior of the specimens under these two different aging environments were compared and analyzed. Following conclusions have been drawn from the analysis.

1. The mass loss rate of expanded perlite mortar gradually increases with the number of heat-rain cycles and natural aging days. The increase rate of the mass loss under the two conditions is between 0.2%~0.7% and −0.4%~0.2%, respectively, with a big difference. The mass increase of the expanded perlite mortar in the early stage of natural aging comes from the pozzolanic reaction of perlite.

2. The water absorption rate of the expanded perlite mortar gradually decreases with the number of artificial aging heat-rain cycles or the natural aging days. The decay rates of the two are very different, about an order of magnitude difference.

3. The compressive strength gradually decreases with the increase of the number of heat rain cycles, increasing first and then decreasing under natural aging conditions. The increase is mainly influenced by the pozzolanic reaction between perlite and calcium hydroxide produced by cement hydration.

Due to the different parameters that characterize the aging time, the deterioration rate of the physical and mechanical properties of the expanded perlite mortar under artificial aging conditions cannot be directly applied to the natural aging conditions. In future research, further attention should be paid to longer-period heat-rain cycles. Also, more performance aging tests of expanded perlite mortar in natural environments should be carried
out to compare and analyze the evolution of various properties under different aging mechanisms and establish the corresponding relationship between the two. Further, an artificial aging system that represents the characteristics of actual aging evolution should be developed for more reliable results.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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