

Durability of thermal insulating bio-based lightweight concrete: understanding of heat treatment on bio-aggregates

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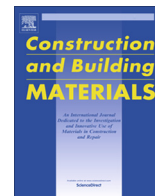
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Durability of thermal insulating bio-based lightweight concrete: Understanding of heat treatment on bio-aggregates

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HIGHLIGHTS

- Effect of heat-treated bio-aggregate on the hydration of cement is investigated.
- Thermal insulation and durability of bio-based concrete are investigated.
- Heat-treated bio-aggregates significantly improve the mechanical strength.
- Heat-treated bio-aggregates have positive effects on the durability.

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ABSTRACT

The organic matter, surface properties and biodegradation of bio-based aggregates are the main factors for their poor performance of bio-based lightweight concrete. In the present study, heat-treatment is applied to bio-aggregates for reducing their negative impacts on cement hydration and performance of thermal insulating bio-based lightweight concrete. The results show that heat-treated bio-aggregates have reduced negative impacts on cement hydration by the decomposition of organic matter and increase of the pH of the leachate, and significantly improves the mechanical strength of concrete. The 28-day compressive strength and flexural strength of heat-treated apricot shell (HAS) concrete increase by 50.2% and 87.7%, respectively, compared to the untreated apricot shell (AS) concrete. The bio-based lightweight concrete in this study has an excellent thermal insulation property, and the thermal conductivity varies from 0.56 W/m-K and 1.25 W/m-K. Moreover, the heat-treated bio-based aggregate significantly reduces the drying shrinkage of concrete. At 108 days, the drying shrinkage of concrete containing heat-treated aggregates reduces by 29.2%–36.1%. Besides, the heat-treated bio-based aggregate enhances the resistance to freeze–thaw cycles, attributed to the reduced micro-cracks and porosity of concrete. Therefore, heat treatment can improve the properties of bio-based aggregates and significantly increase the durability of thermal insulating bio-based lightweight concrete.

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1. Introduction

In the past decades, bio-based concrete has gained increasing attention, especially considering the sustainable development of the concrete industry [1]. Compared to normal-weight concrete, bio-based concrete generally has lightweight characteristics and associated better thermal insulation and sound, attributed to the micropores of bio-aggregates [2]. Besides, bio-aggregates are renewable and possess great potential in saving natural aggregates

and decreasing construction costs [3]. Currently, various bio-based aggregates have been successfully applied to concrete structures, such as oil palm shell [4], coconut shell [5], bamboo [6] and apricot shell [7], etc.

Peach (*Prunus Persica* L.) is a fruit that is originated from China [8]. Peach trees are widely cultivated around the world, mainly in China, Italy, Greece, Spain and the United States [9]. In 2017, the planting area of peach trees around the world was about 1.4×10^6 ha, and the annual output was about 2.4×10^7 tons [10]. Apricot (*Prunus armeniaca* L.) is also one of the main fruits in the world. In 2017, the planting area of apricot trees around the world was about 5.4×10^5 ha, with an annual output of about 4.26×10^6 tons

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[10]. In addition to directly used as a fruit, most peaches and apricots are processed into juices, canned foods, preserves, etc. Consequently, a large number of peach shell (PS) and apricot shell (AS) are discarded, for instance, the annual amount of combined PS and AS exceeds 8×10^5 tons per year in China [11]. In previous researches, PS and AS have been successfully used as soil conditioners [12], fuels [13] and activated carbon [14]. More recently, they are utilized for bio-based lightweight concrete due to the stiffness and lightweight properties [7,11].

Current researches on bio-based lightweight concrete mainly focus on physical properties (workability, density and porosity, etc.) and mechanical properties (compressive strength, tensile strength and modulus of elasticity, etc.). However, limited work on functional properties (thermal insulation, sound absorption) and durability (drying shrinkage, resistance to freeze–thaw), especially peach shell concrete and apricot shell concrete have not been reported. Generally, the bio-aggregates possess high porosity and lightweight properties, which is an advantage that significantly differs from normal-weight aggregates, contributing to the special functional properties of bio-based lightweight concrete such as thermal insulation and sound absorption properties. However, very limited work on the thermal insulation mechanism of thermal insulating bio-based lightweight concrete is available.

Typically, bio-based lightweight aggregates are carbohydrates and porous, which are particularly sensitive to changes in humidity in the environment, and consequently, show a negative impact on the durability of thermal insulating bio-based lightweight concrete, especially water-related properties such as drying shrinkage and resistance to freeze–thaw. Previous studies show that thermal insulating bio-based lightweight concrete usually has a higher drying shrinkage than normal-weight concrete due to the high water absorption and porosity of bio-aggregates. Aslam et al. [15] reported that the oil palm shell concrete has a drying shrinkage of 405–614 μm at 234 days. Bederina et al. [16] investigated that the drying shrinkage of wood sand concrete at 90 days is about 1200–1900 μm , and pretreated wood can significantly reduce the shrinkage of up to 43.6% at 180 days. Since the microstructure, porosity and permeability of bio-based concrete are higher than those of normal weight concrete, the resistance to freeze–thaw cycles is significantly lower than normal-weight concrete. Nguyen et al. [17] reported that the freeze–thaw performance of seashell pervious concrete is considerably lower than the ordinary pervious concrete due to the dissociation of calcium carbonate in the seashell. Therefore, the drying shrinkage and freeze–thaw properties of thermal insulating bio-based lightweight concrete should be investigated and improved prior to its application in practical structure [18].

Similar to palm shell, coconut shell and other bio-materials [19], the PS and AS are mainly composed of cellulose, hemicellulose and lignin [14], and the chemical components are mainly composed of C, O, H, N and O [20]. The sugar released from the organic matter of the bio-based materials can lower the pH value of the cement paste and affect the hydration of the cement, resulting in low mechanical properties of bio-based lightweight concrete [21,22]. Therefore, heat treatment may be an effective method to reduce the impact of the bio-aggregates on cement hydration and improve the physical and mechanical properties and durability of bio-based lightweight concrete by reducing or eliminating the organic matter in bio-aggregates.

Surface treatments of bio-aggregates by chemical solutions such as alkaline solution [23], polyvinyl alcohol [24] and sodium silicate [25], etc., and heat treatment [26] have been studied for reducing the negative impact of organic matter on bio-based lightweight concrete [1]. However, the surface treatment on the performance improvement of bio-based lightweight concrete is limited because organic matter still exists in the bio-aggregates. Moreover, some surface treatments such as wood oil [1] can weaken the bond

between the bio-aggregate and the mortar. Compared to surface treatment, heat treatment can completely decompose the organic matter of bio-aggregates [27] and eliminate the negative effects of biomass on bio-based lightweight concrete. Gupta et al. [28,29] reported that the incorporation of heat-treated wood biochar in concrete offers significantly higher mechanical strength, for example, adding pre-soaked wood biochar with a pyrolysis temperature of 300–500 °C in the mortar increases the compressive strength by 40%–50% compared to plain mortar. Wu et al. [11] investigated that heat-treated bio-aggregates increase the compressive strength of concrete by 33.8–53.6% compared to untreated aggregates. Moreover, Heat treatment can improve the surface quality of bio-aggregates and enhance its adhesion to the mortar and significantly improve the mechanical strength of bio-based lightweight concrete [11]. In addition, heat treatment kills microbial communities and decomposes nutrients for fungal growth, making bio-aggregates possess excellent resistance to biodegradation [30]. Because during the pyrolysis process, the main components of hemicellulose, cellulose and lignin of bio-based materials are gradually decomposed and converted into biochar phase at various temperature ranges. Pyrolysis temperature is associated with physical phenomena such as pore formation, carbonization of char skeleton and the release of volatiles, etc. [31], and significantly influences the physico-chemical properties of bio-based materials [32]. For most bio-based materials, the hemicellulose breaks down when temperature varies from 220 to 315 °C and that for cellulose and lignin is in the range of 315–400 °C and 160–900 °C, respectively [33]. The increase in pyrolysis temperature not only reduces the pore size of bio-based materials but also increases the dimensional stability and pozzolanic activity [34,35], contributes to the improvement in the mechanical properties of bio-based concrete.

Energy consumption is generally related to the temperature and duration of heat treatment. Although heat treatment requires energy consumption to provide temperature (<500 °C) [36], heat-treated bio-aggregates are still relatively energy-saving compared to sintered artificial lightweight aggregates for which a high temperature of more than 1000–1250 °C is often required [37]. Moreover, the energy consumption of the heat-treated bio-aggregates can be minimized by using the latest pyrolysis technology. For example, hydrothermal treatment can save energy by over 50% than dry thermal treatment [38], and the hot gas generated by the low-temperature (200–220 °C) can provide an energy source for the high-temperature process (400–500 °C) [39]. Therefore, heat treatment is still a relatively sustainable and energy-saving method for the treatment of bio-aggregates.

This study aims at reducing the negative impact of organic matter and biodegradation characteristics of bio-based aggregates on the performance of thermal insulating bio-based lightweight concrete, especially for durability. The effect of the heat-treated peach shell (HPS) and heat-treated apricot shell (HAS) powder and their leachates on cement hydration are analyzed and the thermal insulation, drying shrinkage and resistance to freeze–thaw of thermal insulating bio-based lightweight concrete are investigated. Furthermore, the thermal mechanism, drying shrinkage mechanism and freeze–thaw failure mechanism of untreated bio-aggregates concrete and heat-treated bio-aggregates concrete are explored.

2. Materials and methods

2.1. Materials

Peach shell (PS), heat-treated peach shell (HPS), apricot shells (AS) and heat-treated apricot shell (HAS) are used as bio-based aggregates in this study. They are supplied and processed by a

water purification material company (Henan, China), as shown in Fig. 1. The original seed of the PS and the AS is crushed by a crusher and then sieved to obtain the bio-based aggregates. Due to the particle size of the PS is usually larger than that of the AS, they are difficult to obtain the same size. Therefore, the PS with a particle size of 4.75–9.6 mm and the AS with a particle size of 2.36–4.75 mm are used as replacement of total volume of coarse aggregate in the present study, as shown in Table 1. Before heat treatment, all these bio-based aggregates are soaked in water for 24 h, the residual pulp or dust and other wastes are removed and finally air-dried until an internal saturated surface dry state [3,11]. After that, the bio-based aggregates are fed into a rotary kiln to pre-heat at 200 °C for 0.5–1 h, and then the temperature is increased to the pyrolysis temperature of lignin (350–550 °C) for 4 h. Finally, the rotary kiln is cooled down to ambient temperature and the HPS and the HAS are obtained. The detailed heat treatment methodology of the HPS and the HAS are reported in previous studies [11].

The physical and mechanical properties of raw materials are presented in Table 1. The crushing strength and specific gravity of the HPS and the HAS are decreased after heat treatment and have an increase in water absorption. This phenomenon is in agreement with the pyrolysis results of coconut shell [40], miscanthus [35] and other bio-materials [33]. This is due to the decomposition of hemicellulose, cellulose and lignin and the formation of biochar phase lead to an increase in micropores of bio-based aggregates [31]. For artificial lightweight aggregates with a bulk density of 400 kg/m³ and 500–600 kg/m³, their minimum crushing strength requirements 1.5 MPa and 2.0 MPa, respectively (GB/T 17431.1-2010 [41]). The crushing strength of the HPS and the HAS are 2.6 MPa and 3.2 MPa, respectively, which meets the strength requirements of lightweight aggregates.

CEN-NORM sand satisfying European standards (EN 196-1) is used as fine aggregates. CEM I 52.5 R Portland cement is used as the binder (ENCI, the Netherlands) and commercial fly ash and silica fume are applied as supplementary cementitious materials. The polycarboxylate ether superplasticizer (SP) is added to adjust the workability of the fresh mixture.

2.2. Cement hydration test

Generally, sugars leaching from bio-based materials have an adverse effect on cement hydration [21]. Furthermore, when

bio-based materials contact with saturated fresh concrete, leachates from bio-based materials may also affect the properties of cement hydration. The effects of PS powder, AS powder and their leachates on cement hydration are also investigated in the present study.

The preparation process of the leachate of bio-aggregates for cement hydration test is shown in Fig. 2. Firstly, the bio-based aggregates including the PS, HPS, AS and HAS are oven-dried at 105 °C for 24 h, and then milled and sieved through a 500 µm sieve for cement hydration tests; After that, the materials are soaked in distilled water with a solid-liquid ratio of 1:5 [21] and then shaken using a shaker for 24 h with a speed of 250 rpm/min; finally, filtrate and powder are separated with 0.45 µm filter paper, and the pH value of the filtrate is determined by a pH meter.

The TAM Air Isothermal calorimeter is used to measure the hydration heat of cement, a total of three groups of tests are carried out, as shown in Table 2. In Group A, 0.2 g of different bio-based aggregate powders are added to 40 g of cement, the dosage is 0.5% of cement by mass, and then mixed with 20 g of distilled water, and the water-cement ratio is 0.5; in group B, 20 g of distilled water is replaced by the leachates of different bio-based aggregates, and other conditions are kept the same; in group C, pure cement paste is used for the control.

2.3. Mix proportions of concrete

A concrete containing 373.5 kg/m³ of the PS is used as a control mix, the HPS, the AS and the HAS is used to replace the PS by volume, respectively, and the other parameters are kept constant. The mix proportions of the concrete mixes are shown in Table 3. Due to the high water absorption of bio-based aggregates, all coarse aggregates are immersed in water for 24 h and kept a saturated surface dry condition before application to concrete. The detailed mixing methods of the sample are reported in our previous work [1].

2.4. Testing methods of concrete

The slump test of fresh mixture is carried out according to EN 12350-2. The density, 24-hour water absorption and porosity of concrete with a curing age of 28-day are determined according to ASTM C642-13. 40 × 40 × 160 mm³ and 40 × 40 × 40 mm³ samples with a curing age of 28 days are used for determining flexural

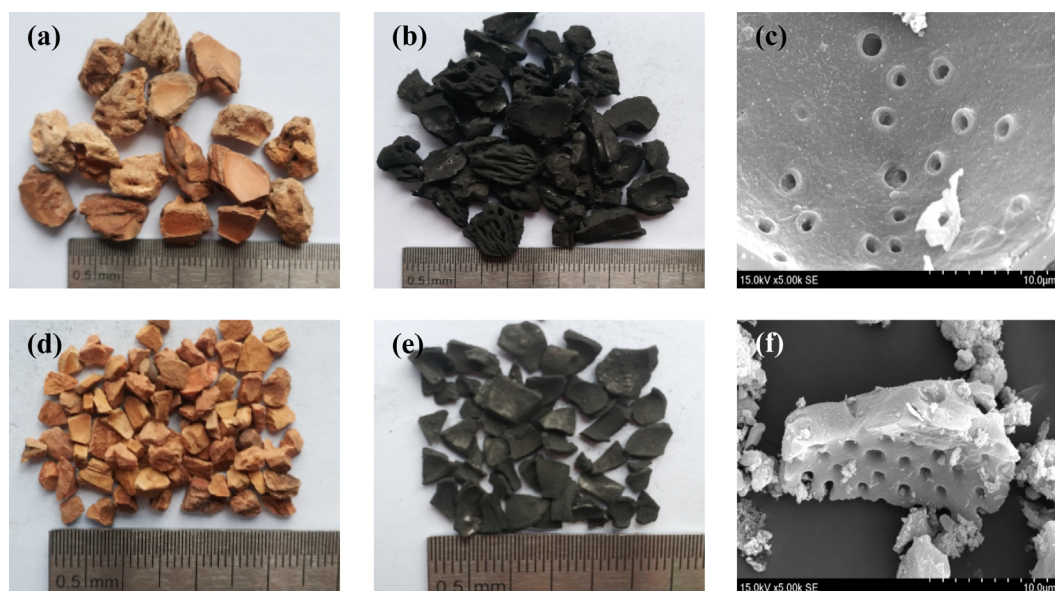


Fig. 1. (a) Peach shell, (b) Heat-treated peach shell and (c) its SEM image, (d) Apricot shell, (e) Heat-treated apricot shell and (f) its SEM image.

Table 1
The physical and mechanical properties of bio-aggregates and sand.

Materials	PS	HPS	AS	HAS	Sand
Particle size (mm)	4.75–9.6	4.75–9.6	2.36–4.75	2.36–4.75	0.08–2
Specific gravity	1.33	1.19	1.47	1.28	2.64
Bulk density (kg/m ³)	536	495	610	575	–
24-h water absorption (%)	16.7	19.4	12.5	14.2	0.2
Fineness modulus	–	–	–	–	2.65
Crushing strength (MPa)	3.4	2.6	8.5	3.2	–

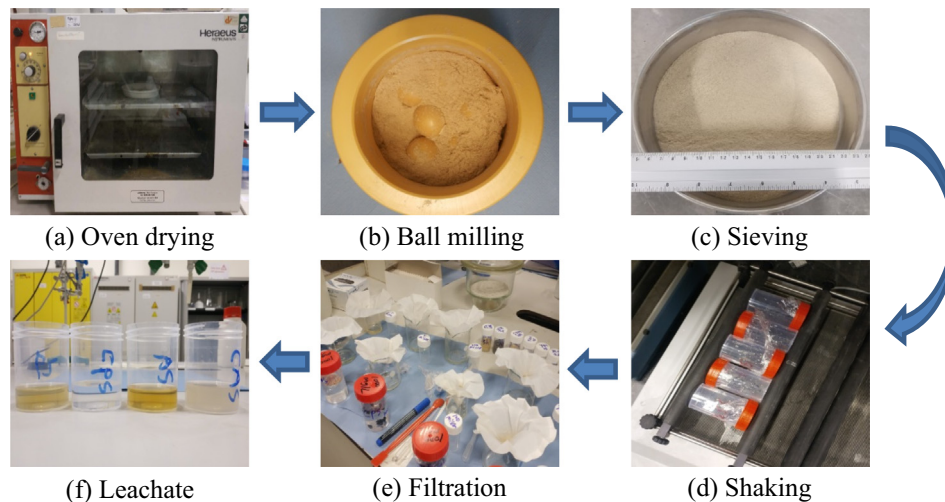


Fig. 2. Preparation process of the leachate of bio-aggregates for cement hydration test.

Table 2
Mix proportions of cement paste for hydration heat test.

Group	Mix code	Materials(g)	Cement(g)	Water(g)	W/C	pH of leachates	Notes
A	PS	0.2	40	20	0.5	–	Distilled water
	HPS	0.2	40	20	0.5	–	
	AS	0.2	40	20	0.5	–	
	HAS	0.2	40	20	0.5	–	
B	PS	–	40	20	0.5	5.6	Leachates
	HPS	–	40	20	0.5	7.8	
	AS	–	40	20	0.5	5	
	HAS	–	40	20	0.5	6.7	
C	Control	–	40	20	0.5	–	Distilled water

Table 3
Mix proportions of concrete (kg/m³).

Mix code	Cement	Fly ash	Silica fume	Water	Sand	SP	Bio-aggregates
PS	440	66	44	192.5	692	5.15	373.5
HPS	440	66	44	192.5	692	5.15	334.2
AS	440	66	44	192.5	692	5.15	412.8
HAS	440	66	44	192.5	692	5.15	359.5

strength and compressive strength, respectively, according to EN 196-1. The loading speed for compressive strength and flexural strength tests are 2400 N/s and 50 N/s, respectively. The microscopic image of concrete is observed by a scanning electron microscope (JOEL JSM-5600).

100 × 100 × 100 mm³ samples are used for the thermal conductivity test by an ISOMET 2104 heat meter (Fig. 3a). The drying shrinkage of 40 × 40 × 160 mm³ samples is determined according to DIN 52450 by using a digital micrometer gauge (Fig. 3b). After demoulding, the samples are stored in a constant temperature room with an ambient temperature of 20 ± 2 °C and a relative humidity of 65 ± 3%. The drying shrinkage is continuously measured in the first month, and then once a week after one month.

The freeze–thaw cycle is determined according to EN 1338 (Fig. 3c). The temperature ranges from –18 °C to 20 °C within 24 h, and 40 × 40 × 160 mm³ samples are completely immersed in water, and the water level is 5 ± 2 mm higher than the upper surface of the samples.

3. Results and discussion

3.1. Effect of heat-treated bio-aggregates on the hydration of cement

3.1.1. Heat-treated peach shell and apricot shell powders

As shown in Fig. 4, although cumulative released heat and heat flow of cement containing bio-based material powders have the

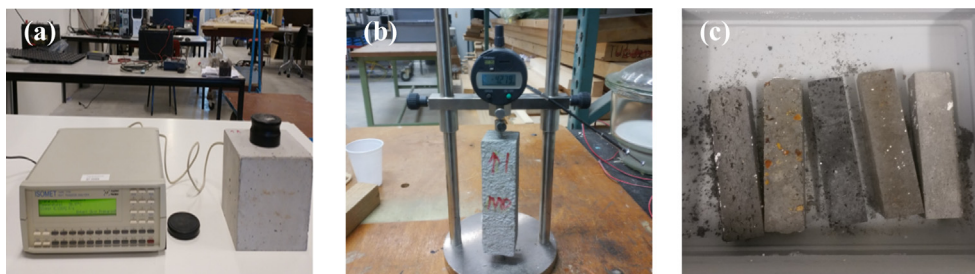


Fig. 3. (a) Thermal conductivity test, (b) Dry shrinkage test and (c) Freeze-thaw test.

same trend as pure cement paste, and almost no retardation is showed, the hydration is significantly weakened. As shown in Table 4, the PS and the AS powders including the heat-treated powders (HPS and HAS) reduce total released heat by approximately 32.4–33.7% compared to plain cement (Table 5).

This may be because the sugar leached from the PS and the AS decreases the concentration of Ca^{2+} during the cement hydration and reduces the formation of hydration products [42]. The same phenomenon is also observed in other bio-based materials, such as spruce [43], coir and bagasse [21]. Besides, the PS and the AS powder have very similar effects on cement hydration, including cumulative released heat and heat flow. This may be due to the similar composition of the PS and the AS, which are composed of C, H and O elements, and their contents are very close as well [13,44].

3.1.2. Heat-treated peach shell and apricot shell leachates

Bio-based materials contain saccharides with different solubility in water, the leachates from the dissolved bio-based materials have an impact on cement hydration [21]. Generally, organic matter has strong calcium chelating groups that prevent C-S-H gel formation [45]. Moreover, sugars leached from organic matter can form a semipermeable layer on the cement grains through nucleation poisoning/surface adsorption [46]. More importantly, some saccharides are unstable in highly alkaline cementitious materials [45]. Organic acids from the degraded bio-based materials are more effective to suppress cement hydration than bio-based materials themselves [47], even damage cement hydration products [21].

Similar to powders, the leachates of the PS and the AS also reduce the cumulative released heat of cement hydration, as shown in Fig. 5. However, the HPS and the HAS reduce the negative impact on cement hydration, especially the HPS, the released heat of its cement hydration increases by 6.5% compared to the PS.

Generally, five monosaccharide components of gum aldose, galactose, glucose, xylose and mannose are the main hydrolysates of cellulose, hemicellulose and lignin [48], which can interfere with the formation of C-S-H gel during the hydration cement [47]. Moreover, during the preparation of the solution, a large amount of carboxylic acid is also precipitated from cellulose [49]. Galactose and glucuronic acid are the two main types of acids precipitated in plant fibres [50], which result in the decrease in the pH of the leachates and significantly affect the crystallinity, strength and hydration of cement [21,22].

The pH value of cement paste varies between 12 and 13 and the acidic solution can delay the cement hydration [22]. The HPS and HAS have a higher pH value than the PS and AS (Table 2), indicating that less sugar is released from the heat-treated bio-materials. Therefore, heat treatment reduces the release potential of sugar from bio-based materials and results in a reduction in the negative impact on cement hydration.

3.2. Physical and mechanical properties of bio-based lightweight concrete

3.2.1. Slump, water absorption, porosity and density

Generally, the addition of bio-based aggregates will reduce the slump of concrete [7]. This phenomenon has also been observed in mussel shell concrete [51] and plastic aggregate concrete [52]. As shown in Table 6, the heat-treated bio-based aggregates (HPS and HAS) increase the workability of concrete because a decrease in wood's hydrophilic group reduces the water absorption and uptake by the wood cell [53]. Yew et al. [26] reported that the workability of bio-based concrete increases with the increasing temperature and duration of heat treatment time. In this study, the slump value of all concretes varies from 45 mm to 70 mm. It has been reported that lightweight aggregate concrete (LWAC)

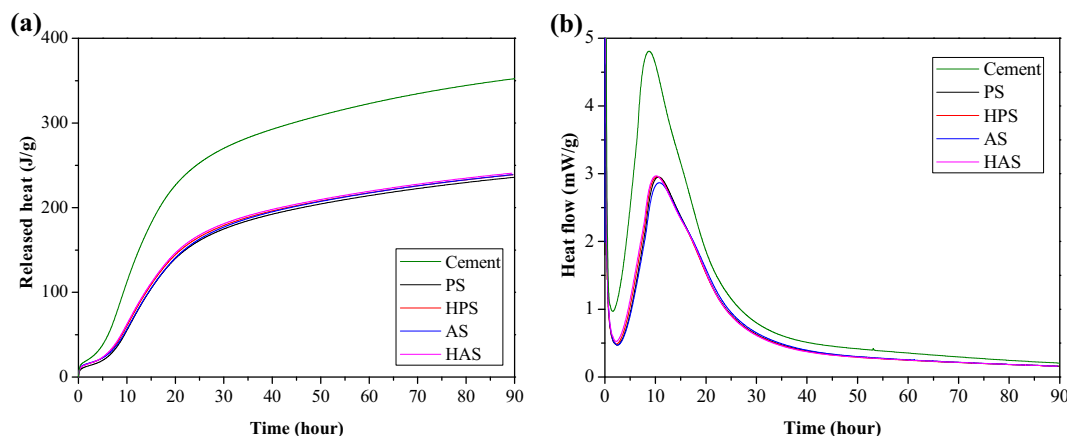


Fig. 4. Effects of bio-based powders on (a) released heat and (b) heat flow of cement hydration.

Table 4
Released heat comparison of bio-material powders and cement.

Items	PS	HPS	AS	HAS	Cement
Total heat (J/g)	235.8	238.8	238.9	240.7	355.9
Heat deviation (J/g)	-120.1	-117.1	-117.0	-115.2	-
Reduction (%)	33.7	32.9	32.9	32.4	-

Table 5
Released heat comparison of bio-material leachates and cement.

Items	PS	HPS	AS	HAS	Cement
Total heat (J/g)	237.6	253.0	233.4	235.0	355.9
Heat deviation (J/g)	-118.3	-102.9	-122.5	-120.9	-
Reduction (%)	33.2	28.9	34.4	33.9	-

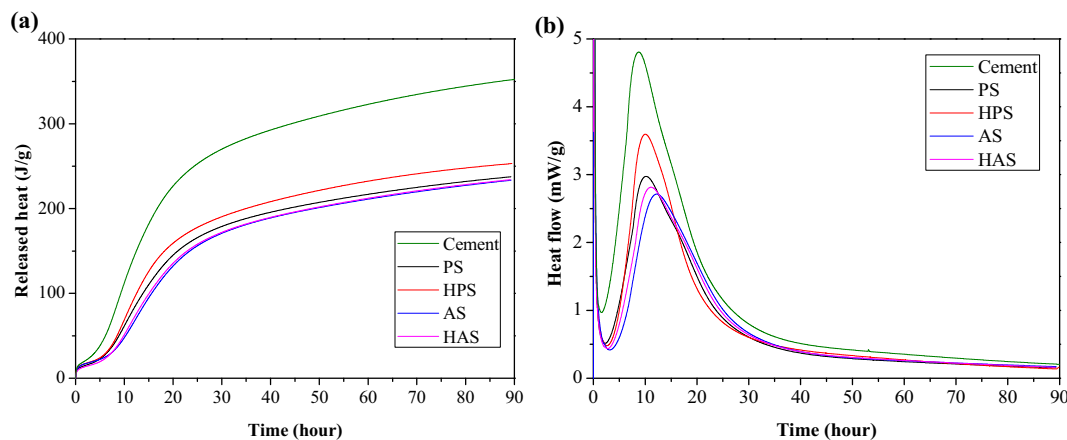


Fig. 5. Effects of bio-based leachates on (a) released heat and (b) heat flow of cement hydration.

with a slump value of 50–75 mm is similar to normal-weight concrete with a slump of 100–125 mm [54]. Therefore, the workability of bio-based concrete manufactured in this study is acceptable.

Lightweight aggregates with high water absorption generally affect the interfacial transition zone (ITZ) of concrete, leading to an increase in the porosity and water absorption of LWAC [55]. The water absorption, porosity and density of concrete is also shown in Table 6. The results show that the heat-treated bio-aggregates (HPS and HAS) decrease the water absorption and porosity of bio-based lightweight concrete, and slightly increase the density. Compared to untreated aggregates, the water absorption of the HPS and HAS decreases by 15.2% and 8.3%, respectively. The decrease in porosity with increasing heat treatment temperature is also observed in oil palm shell concrete [56]. This is because heat treatment improves the surface roughness of bio-based aggregates and reduces the oxygen element content, resulting in a better interfacial adhesion between bio-based aggregates and mortar [11,26].

3.2.2. Compressive strength and flexural strength

The mechanical properties of LWACs depend on the strength of lightweight aggregates and mortar, as well as the bond strength between paste and aggregates [57,58]. As shown in Table 6, the

heat-treated bio-aggregates significantly increase the compressive strength and flexural strength of concrete. Compared to untreated AS aggregates, the compressive strength and flexural strength of the HAS increase by 50.2% and 87.7%, respectively. This is because heat treatment decomposes the organic matter of the bio-based aggregate, reducing the negative effects on cement hydration. Besides, the surface quality of the bio-based aggregate is improved, resulting in a significant increase in the bonding strength between the aggregate and the mortar [11]. Results also show that the AS concrete has better mechanical properties than the PS concrete because the crushing strength of the AS is higher than that of the PS.

Generally, high-strength LWAC is defined as a lightweight concrete achieving a cube compressive strength from 34 MPa to 69 MPa and an oven-dry density not exceeding 2000 kg/m³ [59]. The 28-day compressive strengths of expanded silica concrete [60], coconut shell concrete [61] and oil palm shell concrete [62] are 24–31 MPa, 15–27 MPa and 21–42 MPa, respectively. In this study, the compressive strength of HPS and HAS are 35.8 MPa and 42.5 MPa, respectively. They have better mechanical strengths compared to these bio-based lightweight concretes, especially the HAS which can be used for high-strength bio-based lightweight concrete structures.

Table 6
The physical and mechanical properties of concrete (28 days).

Mix code	Slump (mm)	24-hour water absorption (%)	Porosity (%)	Oven-dry density (kg/m ³)	Compressive strength (MPa)	Flexural strength (MPa)
PS	45	9.2	15.3	1571	23.5	2.54
HPS	55	7.8	13.8	1593	35.8	5.36
AS	50	8.4	14.7	1640	28.3	3.26
HAS	70	7.7	13.3	1659	42.5	6.12

3.2.3. Microstructure

The SEM micrographs of the ITZ of concrete are shown in Fig. 6. Cracks are observed between the untreated PS and AS and the mortar interface due to the organic effect and smooth surface [1,7]. The same phenomenon has also been reported in wood sand concrete [16], bamboo reinforced concrete [6] and oil palm shell concrete [63]. There is a good bond between HPS and HAS and the mortar, indicating that heat-treated bio-based aggregates increase the bond in the ITZ due to the decomposition of organic matter. However, a few cracks are also observed in the HPS because of the increase in brittleness of the heat-treated bio-based aggregates.

3.3. Thermal insulating properties of bio-based lightweight concrete

3.3.1. Thermal conductivity coefficient

Thermal conductivity is an important parameter in the design and application of thermal insulating bio-based LWACs. As shown in Table 7, untreated bio-based lightweight concrete has a lower thermal conductivity due to the porous structure of the bio-based aggregate and the high porosity of the concrete [64]. The thermal conductivity of the HPS and the HAS concrete slightly increases compared to untreated the PS and AS concrete. Generally, low compaction concrete has better thermal insulation properties, because more air bubbles are carried into the concrete during the mixing. Compared to the heat-treated aggregates, the thermal conductivity results also reflect that the untreated PS and AS increase the micropores of the bio-based lightweight concrete and reduce its compaction.

The thermal conductivity of concrete depends on the porosity that determines the mechanical strength. As shown in Fig. 7, in most lightweight concrete, there is a positive correlation between thermal conductivity and compressive strength. Compared to pumice concrete [65], thermal insulating bio-based lightweight concrete in this study has a better compressive strength under the same thermal conductivity, and it has better thermal

conductivity than ferronickel slag mortar [66] with the same compressive strength. Although the thermal conductivity of thermal insulating bio-based lightweight concrete is higher than other lightweight concrete (ultra-lightweight concrete [67], diatomite concrete [65], foam concrete [68] and expanded shale concrete [69]), its compressive strength is significantly better than that of other lightweight concretes, as the compressive strength of other lightweight concretes is usually less than 20 MPa. Therefore, thermal insulating bio-based lightweight concrete in this study shows excellent thermal insulation properties while maintaining high compressive strength.

3.3.2. Thermal insulation mechanism of bio-based concrete

As shown in Fig. 8, for normal-weight concrete, heat transfer mainly occurs through the mortar matrix, the aggregate and the ITZ, because there is no porous thermal insulation material. For untreated bio-aggregate concrete, due to the porous bio-aggregate and the weak bonding interface, heat is mainly transferred through the mortar matrix. The porous bio-aggregate and the weak ITZ act as thermal insulators, which hinder the heat transfer of heat between the two interfaces, thereby reducing the thermal conductivity of the concrete. For heat-treated bio-aggregate concrete, heat is mainly transferred through the mortar matrix and the ITZ, and the heat-treated bio-aggregate has a thermal insulating effect due to the good interfacial bond between the heat-treated bio-aggregate and the mortar. According to the thermal conductivity of individual components of concrete, the thermal conductivity of untreated bio-aggregate concrete can be evaluated by [70]:

$$K = \frac{k_0 v_0 + k_1 v_1 \frac{3k_0}{(2k_0+k_1)} + k_2 v_2 \frac{3k_0}{(2k_0+k_2)}}{v_0 + v_1 \frac{3k_0}{(2k_0+k_1)} + v_2 \frac{3k_0}{(2k_0+k_2)}} \quad (1)$$

Where, K is the thermal conductivity of concrete; k_0 , k_1 , and k_2 are the thermal conductivity of the ITZ, porous aggregate and mortar, respectively; v is the volume fraction of each component.

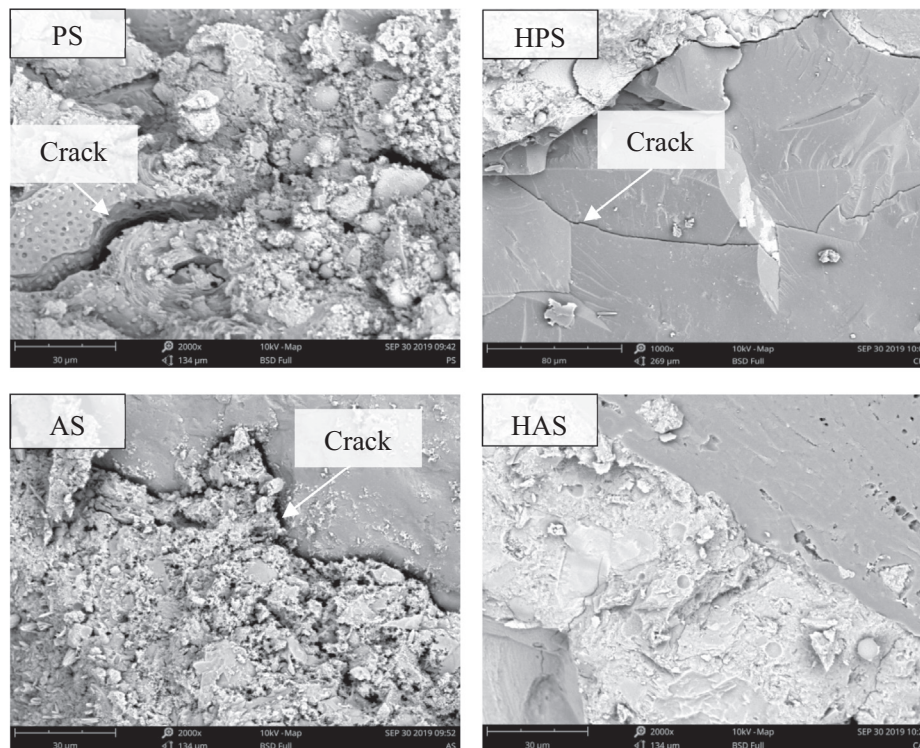


Fig. 6. SEM micrographs of the interfacial transition zone of concrete.

Table 7
Thermal insulating properties of concretes.

Mix code	Thermal conductivity coefficient (W/m·K)	Volumetric heat capacity (MJ/m ³ ·K)	Thermal diffusivity (10 ⁻⁶ m ² /s)
PS	0.56	1.55	0.40
HPS	0.94	1.79	0.70
AS	0.62	1.66	0.34
HAS	1.25	1.73	0.55

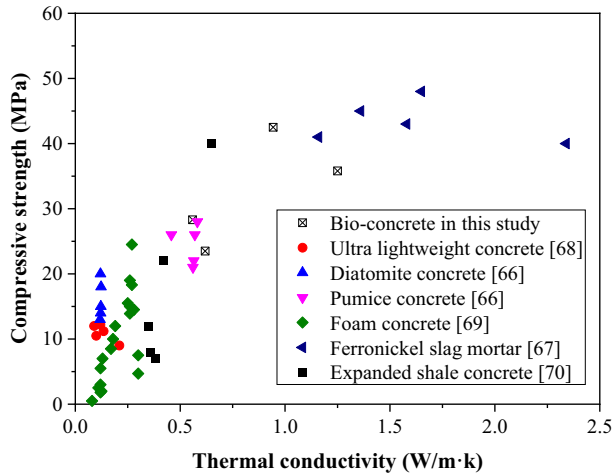


Fig. 7. Relationship between thermal conductivity and compressive strength of concretes.

When the thermal insulation of the ITZ is not considered, only the mortar and aggregate are considered, the thermal conductivity of heat-treated bio-aggregate concrete is calculated by:

$$K = (312_1 - 1)k_1 + (312_2 - 1)k_2 + \left\{ [(312_1 - 1)k_1 + (312_2 - 1)k_2]^2 + 8k_1k_2 \right\}^{1/2} \quad (2)$$

Where, the sign's letters are the same as Eq. (1).

3.4. Durability of thermal insulating bio-based lightweight concrete

3.4.1. Drying shrinkage properties of concrete

(1) Drying shrinkage and mass loss

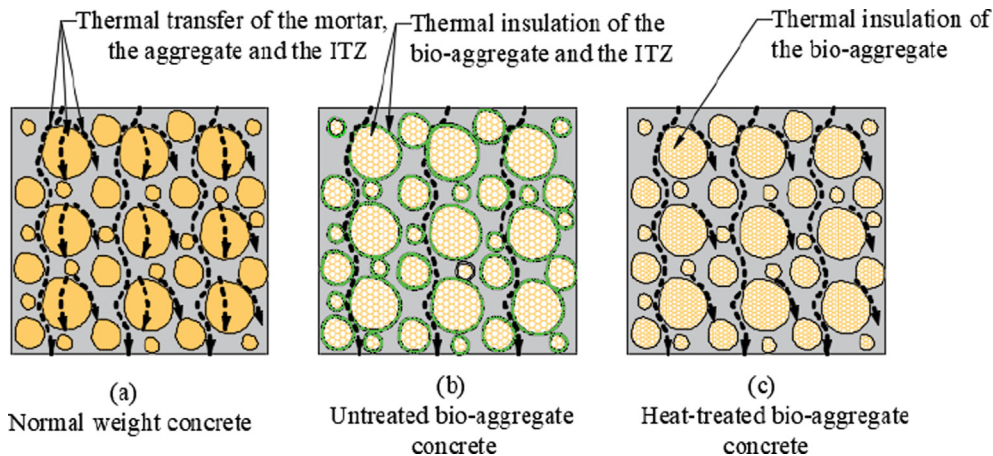


Fig. 8. Thermal insulation mechanism of bio-based concrete.

As shown in Fig. 9, in the first month of the shrinkage test, the drying shrinkage and mass loss of concrete increase rapidly due to the sharp decrease of the relative humidity inside the concrete [71]. After that, the drying shrinkage and mass loss of concrete continue, but with a lower rate, and at 108 days, the drying shrinkage of HPS and HAS concrete are 1343.8 με and 993.8 με, respectively. Compared to untreated bio-aggregates, heat-treated aggregates reduce the drying shrinkage by 29.2% and 36.1%, respectively. Generally, LWACs have a higher drying shrinkage value than normal-weight concrete. Previous studies showed that the drying shrinkage of oil palm shell concrete [15], wood sand concrete [16] and foamed concrete [72] at 90 days are approximately 450–550 με, 1200–1900 με, and 2250–3000 με, respectively. For normal-weight concrete, its drying shrinkage value should not exceed 1000 με for the safety design of concrete components [73]. Therefore, the drying shrinkage of the HAS in this study is acceptable for the manufacture of structural bio-based LWAC.

Previous studies show that the drying shrinkage of concrete is caused by the loss of moisture in the capillary pores [74], which depends on the relative humidity [75] and porosity inside the concrete [76]. Therefore, low-porosity concrete usually has a low drying shrinkage [77]. Moreover, lightweight aggregates with large size have a higher drying shrinkage than the aggregates with smaller size [78]. Apricot shell concrete has a lower shrinkage than peach shell concrete because the PS particles are larger than the AS in this study.

The drying shrinkage ratios of heat-treated aggregates (HPS and HAS) to untreated aggregates (PS and AS) can be divided into a variable stage and a stable stage, as shown in Fig. 10. The drying shrinkage ratio first decreases rapidly, and then gradually reaches a plateau. After the dry shrinkage ratio reaches a stable stage, the dry shrinkage ratios of the HPS and PS, the HAS and AS are 0.65 and 0.74, respectively, indicating that heat-treated bio-aggregates can significantly reduce the drying shrinkage of bio-based LWACs. This may be because heat treatment increases the bond between bio-aggregate and mortar, reduces the porosity of concrete, and decreases the loss of water from inside the concrete.

Since the relative humidity inside the concrete causes the drying shrinkage of the concrete [76], the mass loss can be used to reflect the relative humidity inside the concrete [74]. As shown in Fig. 11, the relationship between the mass loss and drying shrinkage of bio-based concrete can be divided into three stages: the first stage comprises the accelerated water loss, which explains the low increase in drying shrinkage because the free water is quickly lost from the concrete surface and internal pores of the

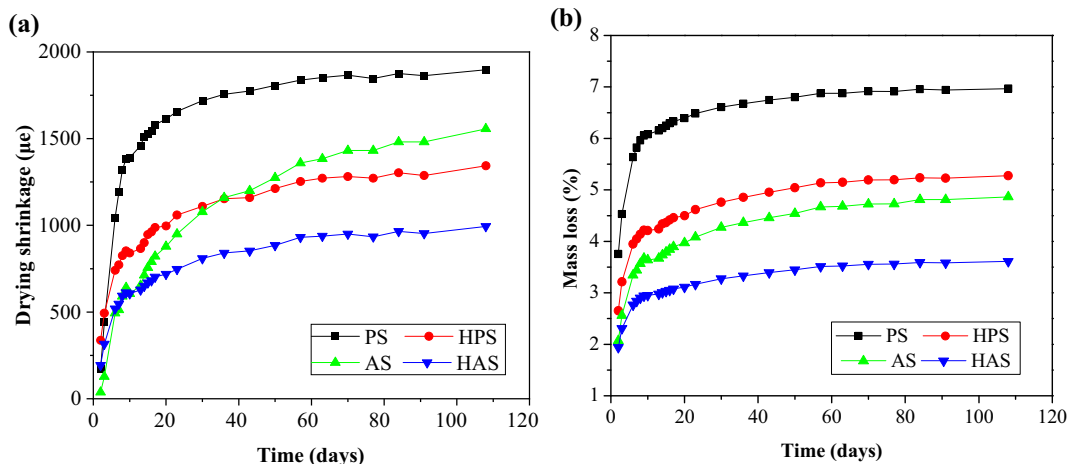


Fig. 9. (a) Drying shrinkage and (b) Mass loss of concretes.

concrete, the mass loss increases rapidly. The second stage features a linear increase between mass loss and drying shrinkage, i.e., the drying shrinkage of concrete increases linearly with the increase of mass loss. The same phenomenon has also been reported in previous studies [74,75] due to the gradual decrease of the relative humidity in the capillary pores of the concrete, resulting in a gradual increase in drying shrinkage [79]. The third stage is the stable stage, the mass loss of the concrete is stable, although the shrinkage is still increasing with a gradually decreasing rate [46].

(2) Drying shrinkage mechanism of bio-based concrete

The mortar and porous bio-aggregates inside the concrete are at saturated state when concrete is cast, as shown in Fig. 12. For bio-based concrete, mortar and porous bio-aggregates can simultaneously shrink due to the loss of water from saturated porous aggregates and from mortar (Fig. 12a). Because the drying shrinkage of normal weight aggregate is small and can be neglected, drying shrinkage of normal weight concrete is mainly caused by the drying shrinkage of mortar (Fig. 12b). Since bio-based aggregates can absorb more water compared to normal weight aggregates, bio-based concrete generally contains more free water in the initial drying shrinkage stage due to the high porosity and permeability [76]. Therefore, drying shrinkage and mass loss of thermal insulating bio-based lightweight concrete are higher than that of normal weight concrete.

The drying shrinkage of concrete is mainly caused by the water loss inside the C-S-H matrix under dry conditions, resulting in the

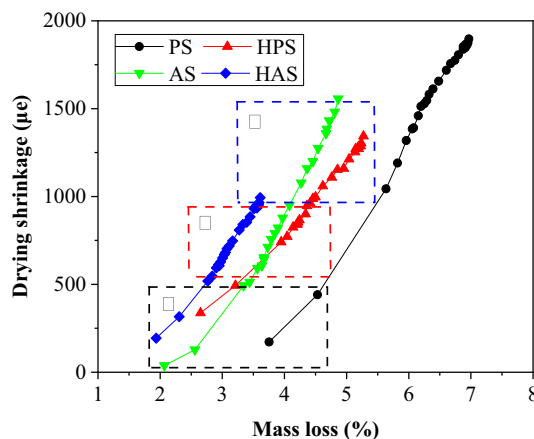


Fig. 11. Relationship between mass loss and drying shrinkage.

drying shrinkage of the mortar, especially the water loss inside the capillary pores below 50 nm, which significantly affects the volume change of the concrete [80]. Moreover, the degree of hydration of cement [81] and the elastic modulus of concrete [82] also affect the drying shrinkage of concrete. Since the bio-based aggregate is very sensitive to changes in moisture in the environment, especially untreated bio-aggregates have a very high shrinkage deformation under dry conditions. After heat treatment, the organic matter inside the bio-based aggregate is decomposed, and the dimensional stability of the aggregate is significantly improved [11]. Moreover, the interface between the heat-treated bio-aggregate and the mortar has better interface bond, which significantly reduces the drying shrinkage of thermal insulating bio-based lightweight concrete.

The micrographs of concrete after drying shrinkage test are shown in Fig. 13. For untreated bio-based aggregates (PS and AS), obvious cracks are observed in the ITZ between the bio-aggregate and the mortar, and drying shrinkage cracks also appear on the surface of the mortar. For the heat-treated bio-based aggregates (HPS and HAS), which have a good bonding interface with the mortar, only some micro-cracks or pores appear in the ITZ. The micro-cracks or pores in the ITZ and the porous aggregates are also the reasons for the drying shrinkage of bio-based concrete. The results show that heat-treating bio-aggregates are effective in improving the dimensional stability of bio-based aggregates, and also reduces the initiation of micropores and cracks in bio-based lightweight concrete.

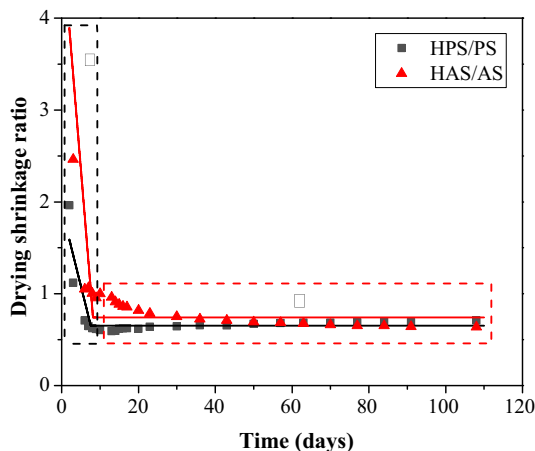


Fig. 10. Dry shrinkage ratio of concretes.

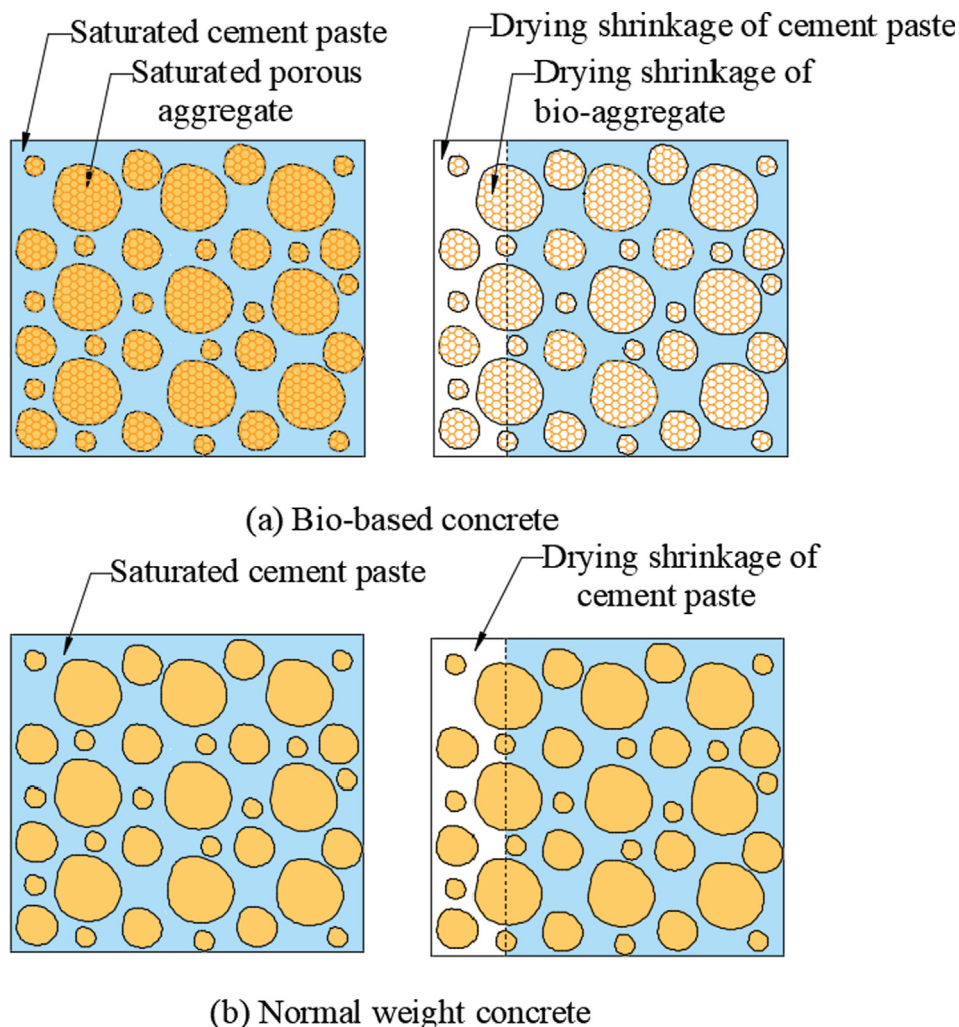


Fig. 12. Schematic diagram of drying shrinkage of concrete.

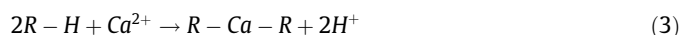
3.4.2. Freeze-thaw characteristics of concrete

(1) Mass loss and surface changes

In the cold environment, the pore water inside the concrete becomes ice and then melts into water during the freeze-thaw cycles, once the freeze-thaw included expansion force exceeds the maximum value that the pore can withstand, crack occurs in concrete. With the gradual accumulation of such internal damage, the concrete will show a gradual freeze-thaw failure [83]. As shown in Fig. 14, the mass loss of all concretes increases with time during the freeze-thaw test. After 30 freeze-thaw cycles, only the HAS concrete still maintains an intact shape, and the other concretes are broken into particles. The results also show that most freeze-thaw failures develop particularly rapidly. Once obvious cracks are formed on the surface of concrete, the bio-based concrete is destroyed within the next 7 freeze-thaw cycles, and the exposed single-particle aggregates can be seen (Fig. 15b). This is because the development of micro-cracks causes more water to enter the concrete, and larger cracks will form during the next freeze-thaw cycle and accelerate freeze-thaw damage [83].

The concrete surfaces after freeze-thaw cycles are shown in Fig. 15. The results show that some bio-based aggregates on the surface of the concrete are exposed after 7 freeze-thaw cycles, the peach shell concretes (PS and HPS) are completely broken after 21 freeze-thaw cycles when obvious freeze-thaw damage occurs on corners of the apricot shell concrete (AS and HAS). This may

be attributed to the following reasons: (1) the tensile strength of bio-based concrete is relatively low, and the generated freeze-thaw expansion force is more prone to cause the expansion and damage of micropores or cracks of the bio-based concrete; (2) the degradation and decomposition of the bio-aggregates after immersing into water lead to the reduction in the pH of the solution and weaken the performance of the concrete, and the reaction formula is as follows [17]:



(2) Failure development process and freeze-thaw mechanism of bio-based concrete

The development process of freeze-thaw failure of thermal insulating bio-based lightweight concrete is shown in Fig. 16. Firstly, the peeling of the bio-aggregate occurs on the concrete surface during the initial freeze-thaw cycle stage; after that, the macro-crack appears and the freeze-thaw damage is observed at the corners of the concrete; finally, the expansion force generated from the repetition process of turning water into ice gradually causes the complete failure of the concrete. The location with the worst compaction is generally the main location where freeze-thaw failures occur because of the presence of more macropores [17].

The freeze-thaw failure of concrete is due to the thermodynamic changes in the process of water into ice and then the melting of ice into water. Water penetrates the micro-cracks or pores of

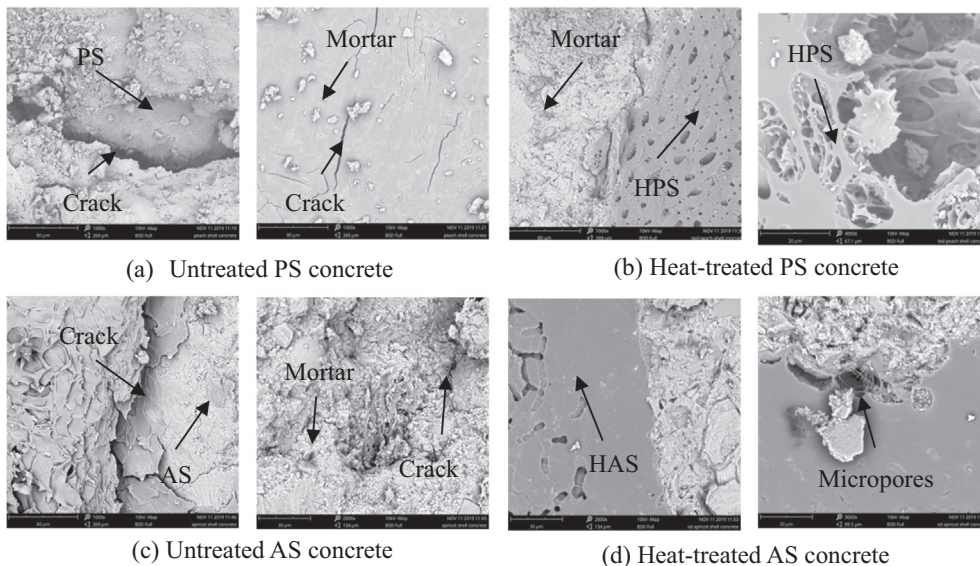


Fig. 13. Micrographs of concrete after drying shrinkage test.

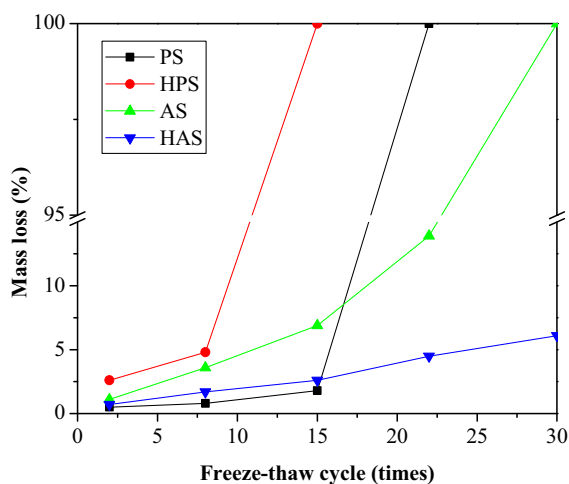


Fig. 14. Mass loss of concrete during the freeze-thaw test.

concrete through capillaries, when the temperature is below 0 °C, the water in the micropores becomes ice, which gradually generates an expansion force during the formation of ice, and consequently results in the increases in the size of the micro-cracks or

pores. After that, more water enters the micropores during the next freeze-thaw cycle, and the concrete gradually undergoes freeze-thaw damage due to the continuous growth of the micropores [83].

The freeze-thaw failure mechanism of thermal insulating bio-based lightweight concrete is shown in Fig. 17. Due to the very low permeability of normal weight aggregates, water cannot enter the interior of normal weight aggregates during freeze-thaw cycles, only the ITZ and the mortar are subject to freeze-thaw damage (Fig. 17a). For the untreated bio-based aggregates, water can easily penetrate the ITZ and the micropores of the bio-based aggregates during the freeze-thaw cycle. When the saturated water turns into ice, the micropores or cracks in the mortar and the ITZ will generate expansion force, resulting in freeze-thaw damage. Furthermore, due to the significant water absorption of porous bio-based aggregates, the expansion force also appears inside the bio-based aggregate, resulting in accelerated degradation of bio-based concrete (Fig. 17b). However, due to the increased interfacial adhesion between the heat-treated aggregate and the mortar, water penetrating the ITZ is decreased, which leads to less damage of concrete caused by freeze-thaw cycles (Fig. 17c). However, once water enters the inside of the porous bio-based aggregate, the expansion force is generated inside the heat-treated aggregates that accelerate the freeze-thaw damage process, which explains why the HPS concrete is rapidly destroyed. Therefore, reducing the permeability of thermal insulating

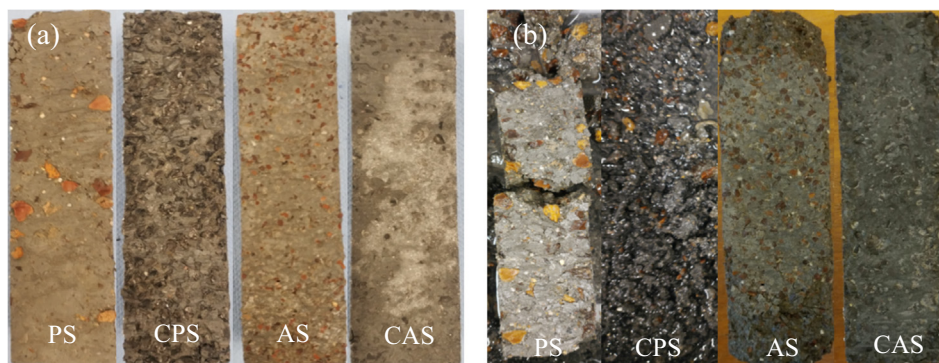


Fig. 15. Concrete surfaces after (a) 7 and (b) 21 freeze-thaw cycles.

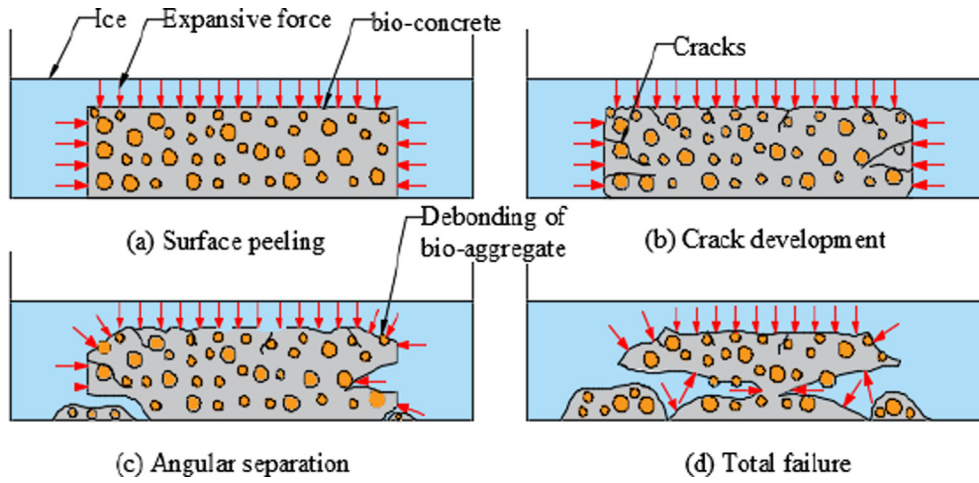


Fig. 16. Development process of freeze-thaw failure of bio-based concrete.

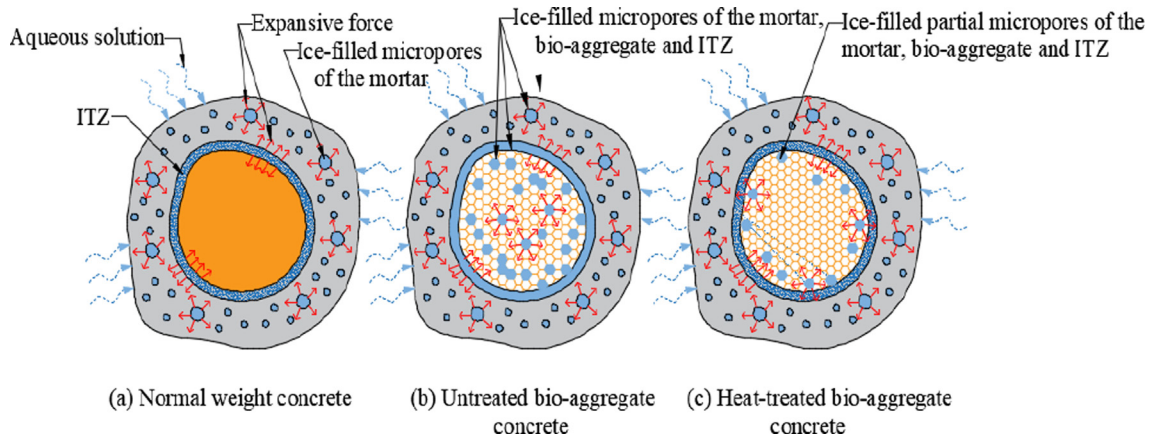


Fig. 17. Freeze-thaw failure mechanism of concretes.

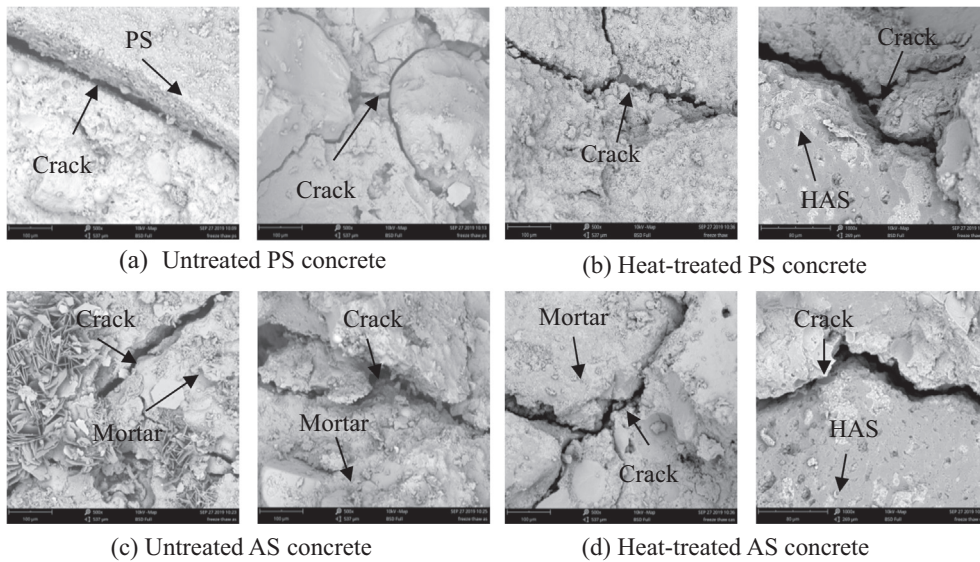


Fig. 18. Micrographs of concrete after the freeze-thaw test.

bio-based lightweight concrete can significantly enhance its resistance to freeze–thaw.

The micrographs of concrete after the freeze–thaw test are shown in Fig. 18. The results show that after the freeze–thaw cycles, micro-cracks appear in the mortar matrix and the ITZ of all concretes, because when the pore water inside the concrete becomes ice, the expansion force is generated, which causes micro freeze–thaw damage of the concrete. Although the heat-treated aggregate has a good interfacial bond with the mortar, microcracks are observed in the ITZ after the freeze–thaw cycle (Fig. 18b, d). Therefore, for the application of thermal insulating bio-based lightweight concrete in cold areas, more treatments should be used to decrease the water absorption of the bio-based aggregates and improve its resistance to the freeze–thaw cycle.

4. Conclusions

In the present study, a heat treatment is applied to reduce the negative impact of organic matter and biodegradation characteristics of bio-based aggregates on cement hydration and performance of thermal insulating bio-based lightweight concrete, including the mechanical strength, thermal insulation, drying shrinkage and freeze–thaw resistance. The following conclusions can be drawn based on the acquired results:

(1) The PS and AS powder and their leachates reduce the degree of hydration of cement, with a reduction in total released heat of 28.9–34.4% compared to plain cement. Heat-treated bio-based aggregates have a clearly reduced negative impact on cement hydration through the decomposition of organic matter and increase of the pH of the leachate. Moreover, heat-treated bio-based aggregates decrease the water absorption and porosity of thermal insulating bio-based lightweight concrete.

(2) The heat-treated bio-aggregates significantly improve the compressive and flexural strength of thermal insulating bio-based lightweight concrete by reducing the negative impact of sugars on cement hydration, improving the surface roughness of the bio-based aggregates and the interface bonding with mortar. The 28-day compressive strength and flexural strength of the HAS are 42.5 MPa and 6.12 MPa, respectively, and increase by 50.2% and 87.7%, respectively, compared to the untreated AS. Therefore, the HAS aggregates can be used for producing high-strength bio-based lightweight concrete.

(3) The developed thermal insulating bio-based lightweight concretes have an excellent thermal insulation property along with high mechanical strength. The thermal conductivity of the bio-based lightweight concretes in this study varies from 0.56 W/m·K and 1.25 W/m·K. The porous bio-aggregates and the weak interfacial transition zone (ITZ) act as insulators, which contribute to the low thermal conductivity of thermal insulating bio-based lightweight concrete.

(4) The heat-treated bio-based aggregate significantly reduces the drying shrinkage of thermal insulating bio-based lightweight. At 108 days, the drying shrinkage of the HPS and HAS concrete reduces by 29.2% and 36.1%, respectively. Moreover, the heat-treated bio-based aggregate enhances its resistance to freeze–thaw cycle because of the reduced micro-cracks and porosity of thermal insulating bio-based lightweight.

CRediT authorship contribution statement

Fan Wu: Methodology, Investigation, Data curation, Formal analysis, Validation, Writing - original draft. **Qingliang Yu:** Conceptualization, Supervision, Project administration. **Changwu Liu:** Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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