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Short communication

Effects of heat treatment on the physical properties of lightweight aggregate from water reservoir sediment

Yi-Chong Liao, Chi-Yen Huang [*]

Department of Resources Engineering, National Cheng Kung University, No. 1 University Road, East District, Tainan City 701, Taiwan, ROC

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Abstract

Lightweight aggregates (LWAs) were produced from water reservoir sediment with added calcium oxide by employing four heat treatments, respectively, at temperatures in the range of 1170–1230 °C. The results show that LWAs produced at temperatures above 1200 °C meet European Union regulation EN-13055-1, which states that the unit weight of LWAs should be lower than 2000 kg/m^3. The bulk density was easily lowered by extending the soaking time and increasing the heating rate. The ratio of strength to unit weight of the LWA produced at 1230 °C with a short soaking time and a fast heating rate was near that of a commercial product. The level of water adsorption was below 4%, which increased initially and then decreased due to pore connections and pore sealing. The formation of a glassy phase made the LWAs treated at higher temperature rough and sealed small pores (<0.1 mm) connected to the walls of large pores (>0.2 mm). The mineral phases of the LWAs were quartz, anorthite, and hematite.

Keywords: C. Strength; Bulk density; Water adsorption; Glassy phase; Lightweight aggregates

1. Introduction

Lightweight aggregates (LWAs) are widely applied in the production of lightweight concrete for high-rise buildings due to their low weight. The porous structure of LWAs gives them properties of acoustic and thermal insulation. The formation of a porous structure was inferred by Riley [1], who suggested that bloating behavior has two causes. One is that raw materials have a chemical composition which can form a glassy phase that wraps gas, and the other is that gas is generated as the glassy phase forms. In Riley's study, a diagram composed of SiO_2, Al_2O_3, and flux oxide includes a bloating area where a bloating behavior occurs. Many recycled wastes located in the bloating area are manufactured into LWAs, such as mined residue [2–6], ash [7–11], and sludge [12–16]. Water reservoir sediment also locates in the area.

Water reservoir sediment greatly shortens the usage life of reservoirs. In order to extend the reservoir life and recover water storehouses, dredging operations are performed after the rainy season. The swept sediment is usually disposed of downriver, which can damage the ecology due to silt suspension. According to a government report, an average amount of 14 million metric tonnes of sediment precipitates annually in Taiwan; the total amount of swept sediment in 2009 was 4.33 million metric tons. Recently, Chen et al. [7] and Wei et al. [17] have reported that it is feasible to use water reservoir sediment, which can be mixed with other recycled wastes, to produce LWAs. Tang et al. [18] manufactured LWAs from reservoir sediment for lightweight concrete. Their results showed that the performance of LWAs from reservoir sediment was better than that of commercial LWA. In the process of manufacturing LWAs, heat treatment greatly affects their physical properties because temperature controls bloating behavior, which determines the formation of the porous structure. de'Gennaro et al. [4] used an optical lens to observe the bloating behavior of LWAs manufactured from Neapolitan Yellow Tuff during the heating process. The maximum bloating temperature was determined for LWAs when the bottom of the LWAs was in flat contact with the refractory brick. Pore size was also found to increase gradually with distance away from the refractory brick. Chen et al. [7] mixed water reservoir sediment and various ashes to produce LWAs preheated at

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[*] Corresponding author at: Department of Resources Engineering, National Cheng Kung University, No. 1 University Road, East District, Tainan City 701, Taiwan, ROC. Tel.: +886 06 2757575x62832; fax: +886 06 2380421.
E-mail address: jcyhuang@mail.ncku.edu.tw (C.-Y. Huang).

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500 °C; the LWAs were fired at 1150 °C and 1175 °C with soaking times of 10 min and 15 min, respectively. The longer soaking time increased the particle density of LWAs and reduced the water adsorption.

However, few studies have directly examined how heat treatments including heating rate, duration time, and temperature affect the physical properties of LWAs. In the present study, various heat treatments were employed to produce LWAs from water reservoir sediment with added calcium oxide. In our previous study [19], calcium oxide was found to reduce the water adsorption of LWAs. Since water adsorption affects the setting time of concrete, it is better for using LWAs with low water adsorption in the production of lightweight concrete. After being calcined at various temperatures, the physical properties, macrostructure, and microstructure of LWAs were investigated.

2. Materials and methods

The water reservoir sediment was obtained from Shihmen Reservoir, which is the largest reservoir in north Taiwan. The sediment with a median particle size (d_{50}) of 8.49 μm had a water content of 28% and an ignition loss of 5.57%. The sediment was dried at 120 °C and milled to pass through a No. 100 mesh for further measuring. The chemical composition of the sediment was determined using an X-ray fluorescence spectrometer (XRF, Rigaku). Mineral phases were analyzed using X-ray diffraction (XRD, Siemens D5000).

CaO powders (Shimakyo's Pure Chemicals) were milled to pass through a No. 100 mesh. 5% CaO by weight was directly mixed with the water reservoir sediment by a small cement mixer without drying. The mixture was extruded by a vacuum extrusion machine. The extruded bar was cut into small lumps to be pelletized into 10–20 mm diameter spheres. The spheres were left to stand in the atmosphere at room temperature for one day and then heated in a laboratory-scale furnace. Heat treatment with a soaking time of 30 min and a heating rate of 5 °C/min was employed (denoted as 3005, where first two numbers represent the soaking time and the last two numbers represented the heating rate. Likewise, other treatments were denoted as 3015, 1505, and 1515, respectively. The heating temperature was controlled from 1170 °C to 1230 °C in increments of 15 °C.

The physical properties of the heated LWAs were characterized. The bulk density and water adsorption were measured using the Archimedes method after the LWAs were placed in boiling water for 24 hr. The bulk density and water adsorption were calculated using Eqs. (1) and (2) [20]. The compressive strength was measured using a compression testing system (MTS) with a cross-head speed of 0.1 mm/s. The compressive strength of the heated LWAs was calculated using Eq. (3) [21,22].

$$\text{Bulk density} = \frac{W_\text{D}}{W_\text{S} - W_\text{I}} \tag{1}$$

$$\text{Water adsorption (\%)} = \frac{100(W_\text{S} - W_\text{D})}{W_\text{D}} \tag{2}$$

$$\text{Compressive strength} = \frac{2.8P_\text{c}}{\pi X^2} \tag{3}$$

where W_D is the dry weight of the heated LWAs, W_S is the 24 h saturated surface-dry weight, and W_I is the immersed weight in water. P_c is the fracture load and X is the diameter of the LWAs.

Each recorded testing value was the mean of results obtained from eight samples. Mineral phases of the aggregates were analyzed using XRD after they were crushed into powders through a No. 100 mesh. An optical microscope was employed to observe the appearance and cross-section of samples. Finally, the microstructure of the 3005 and 3015 series of samples was investigated using a scanning electron microscope (SEM).

3. Results and discussion

Table 1 shows the chemical composition of the water reservoir sediment detected by XRF. The sediment was mainly SiO_2 (61.4%), followed by Al_2O_3 (22.5%) and Fe_2O_3 (8.6%). Mineral phases of the sediment with 5 wt% CaO and the raw sediment were analyzed by XRD. Fig. 1 shows the XRD pattern of raw sediment and sediment with added CaO. The mineral phases for the raw sediment and the sediment with added CaO were the same. The mineral phases were quartz (PDF#: 83-2539), albite (PDF#: 41-1480), clinochlore (PDF#: 46-1323), and muscovite (PDF#: 46-1311).

Fig. 2a shows the bulk density of LWAs subjected to various heat treatments. The bulk density decreased with temperature. The 3015 series of samples had the lowest bulk density at various heating temperatures, and the bulk density ranged from 2.02 g/cm^3 to 1.12 g/cm^3. The bulk density of the 1505 and 1515 series of samples were lower than 2 g/cm^3 above 1200 °C. Although the bulk density of the 1505 series of samples was higher than those of other samples below 1215 °C, it was lower than that of the 1515 series of samples above 1215 °C. To lower the bulk density at high temperature, a slow heating rate should be applied for a short duration time. The bulk densities of the 3005 and 3015 series of samples produced at a given soaking time were similar at temperatures above 1200 °C, and different from those of the 1505 and 1515 series of samples. This indicates that a longer soaking time and a faster heating rate easily reduced the bulk density, which decreased at a constant

Table 1
Chemical compositions of Shihmen Reservoir sediment.

Component	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O
Composition (wt%)	61.4	22.5	8.6	0.7	2.0	1.3	3.4

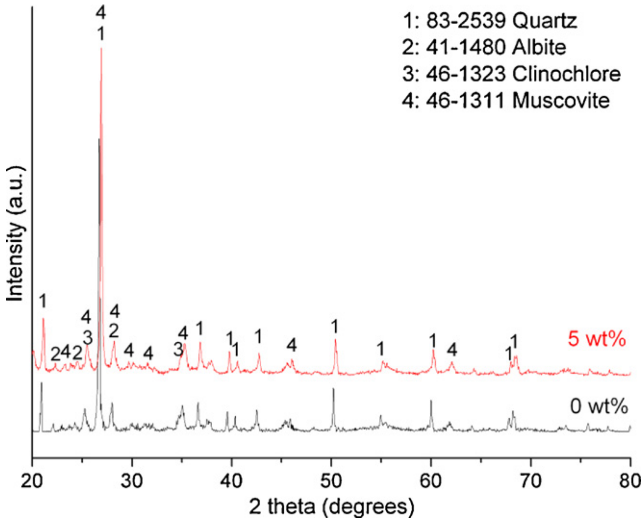

Fig. 1. Mineral phases of Shihmen Reservoir sediment.

rate due to the eventual bloating behavior. In previous studies [3,10,23], a reduction reaction of Fe_2O_3 was found to promote bloating behavior. This may be due to a faster heating rate resulting in more unburned carbon from the organic matter in the sediment and a longer duration time making LWAs bloat sufficiently. The bulk density of the 1515 series of samples produced at 1230 °C was the highest since its heating time is the shortest, leading to an insignificant bloating behavior. This result is different from that obtained by Chen et al. [7], who mentioned that longer soaking time increased bulk density. The reason is they had different heat treatment. In the article, the spheres were left to stand in the atmosphere at room temperature for one day and then heated in a laboratory-scale furnace. Heat treatment with a soaking time of 15 or 30 min. and a heating rate of 5 or 15 °C/min was employed. While, the heat treatment adopted by Chen et al. consisted of pre-sintering and sintering. In fact, the dried pellets were placed in the preheating chamber of the kiln and heated at 500 °C for different duration. Then the preheated pellets were placed in sintering chamber of the kiln and heated at several maximum temperatures for different duration. According to European Union regulation EN-13055-1, the unit weight of LWAs should be lower than 2000 kg/m^3. LWAs produced by employing a heat treatment of a long soaking time and a fast heating rate (3015) easily meet the above regulation at 1170 °C, and LWAs produced by employing the other heat treatments meeting the regulation at temperatures above 1200 °C.

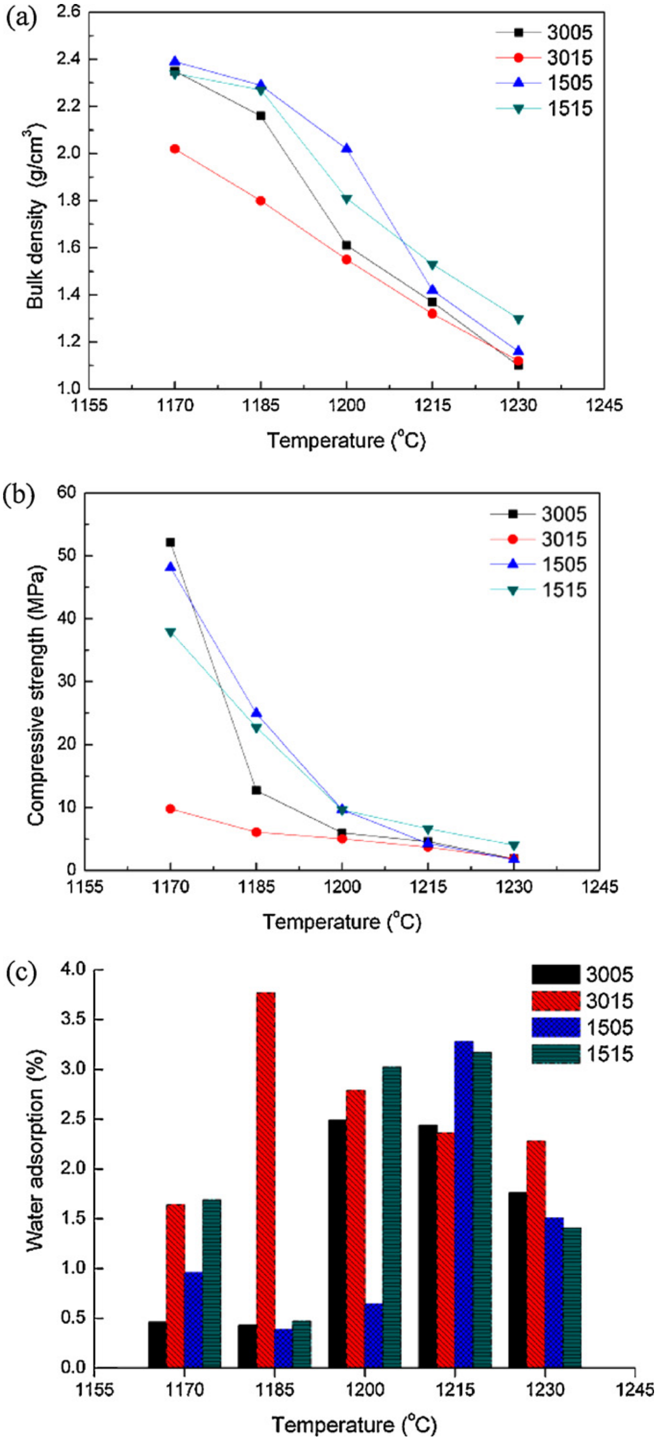

Fig. 2. Physical properties of LWAs subjected to four heat treatments, respectively, at various temperatures. (a) Bulk density, (b) compressive strength, and (c) water adsorption.

The compressive strength of each series of LWAs is shown in Fig. 2b. Compressive strength was positively related to the bulk density; LWAs with low bulk density had low compressive strength. The 1515 series of samples produced at 1230 °C had the highest compressive strength (4.08 MPa). In a previous study [6], LWAs were produced and compared to commercial LWA Leca Strutturale, which has a bulk density of 1.3 g/cm^3, a water adsorption of >7%, a compressive strength of 4.5 MPa, and a ratio of strength to unit weight of 3.46. Their results showed that LWAs with a bulk density lower than 2 g/cm^3 and a compressive strength higher than 1 MPa were suitable for lightweight concrete. The ratio of strength to unit weight is an index for engineering and that of the LWAs produced from the sediment with added CaO is shown in Table 2. The LWAs produced at 1230 °C were compared to a commercial product because their bulk densities were lower than 1.3 g/cm^3. The strength to unit weight ratio of the 1515 series of samples (3.14) was close to that of the commercial product; those of the other

Table 2
Ratios of strength to unit weight of particles for each series of samples.

Series	Temperature				
	1170 °C	1185 °C	1200 °C	1215 °C	1230 °C
3005	22.18	5.90	3.74	3.37	1.70
3015	4.87	3.39	3.27	2.83	1.71
1505	20.15	10.89	4.81	3.04	1.52
1515	16.22	10.00	5.35	4.36	3.14

samples were much lower. Although the strength performance did not reach that of Leca Strutturale, LWAs produced at temperatures above 1200 °C can be applied to lightweight concrete production.

Fig. 2c shows the water adsorption of LWAs at various temperatures. The water adsorptions were all below 4%, which is beneficial for lightweight concrete. Except for the 1505 and 1515 series of samples, whose water adsorptions did not increase at the initial heating temperature, the water adsorption of each series of samples increased and then decreased with temperature. The samples bloated to form LWAs during the heating process. Since the 1505 and 1515 series of samples had shorter heating times than those of the 3005 and 3015 series of samples, the glassy phase did not completely form at the bottom of the LWAs due to the thermal conductivity of the crucible lowering the temperature, as can be seen in the cross-sections of samples. The level of water adsorption mainly depends on the open pores of LWAs. Pores are mainly formed by a glassy phase wrapping gas. The glassy phase flows at high temperature and seals smaller pores, decreasing water adsorption. Since the shape of LWAs affects the fluidity of lightweight concrete, the LWAs with good roundness can avoid the choke of concrete while pumping. A higher amount of the glassy phase cannot maintain the roundness of LWAs due to its flowing. When the water adsorption of LWAs started to decrease, the glassy phase started to flow to seal open pores, changing the shape of the LWAs. This indicates that the appropriate amount of glassy phase is the amount at the top of water adsorption level for each series of samples; at 1200 °C for the 3005 series, at 1185 °C for the 3015 series, at 1215 °C for the 1505 series, and at 1200 °C for 1515 series. After the corresponding temperature was reached, the glassy phase started to flow and sealed small pores, decreasing water adsorption.

Fig. 3 shows XRD patterns of each series of samples for various temperatures. The mineral phases were quartz (PDF#: 85-0794), anorthite (PDF#: 41-1481), and hematite (PDF#: 87-1165) in each series. The anorthite phase formed mainly due to

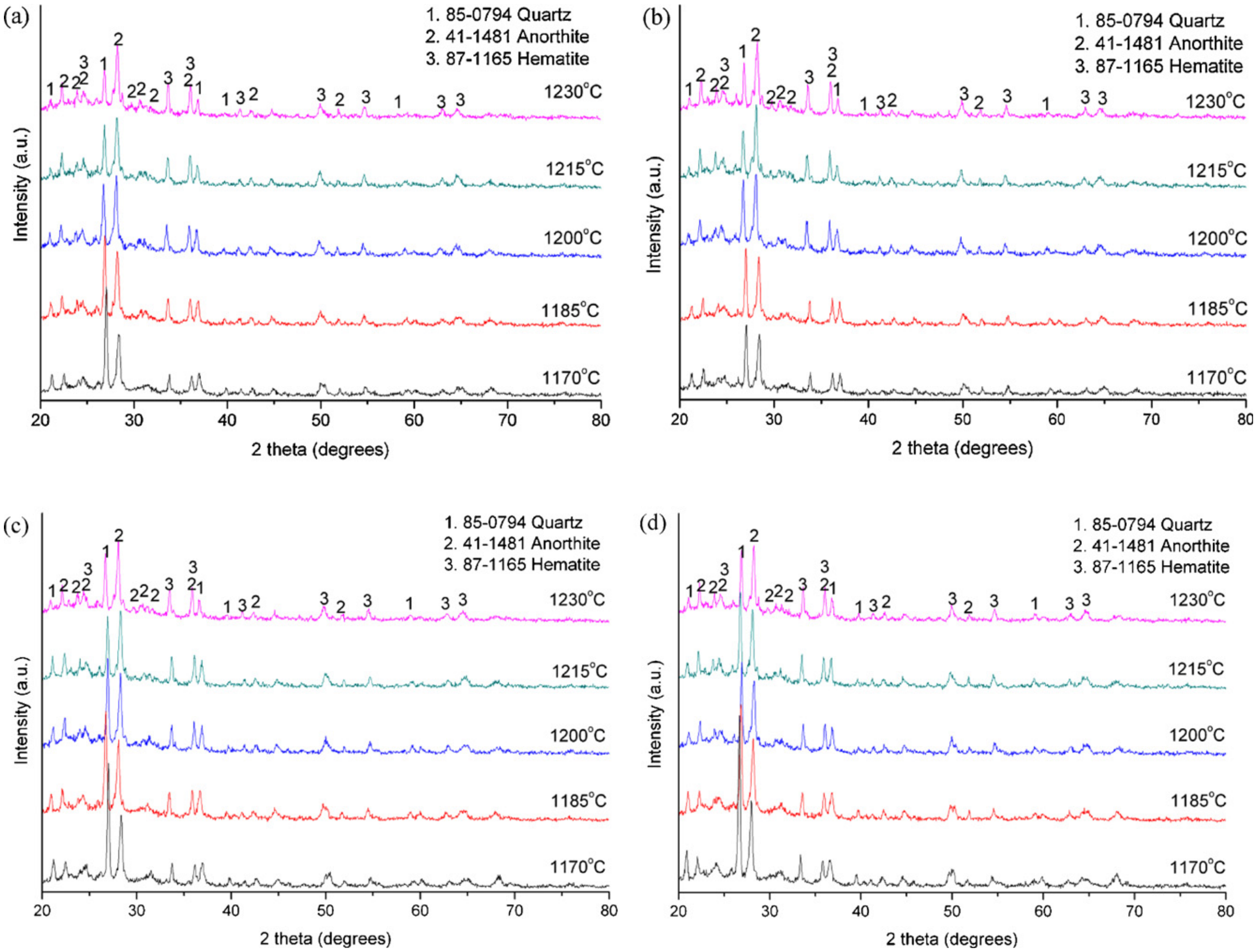

Fig. 3. Mineral phases of LWAs subjected to various heat treatments. (a) 3005, (b) 3015, (c) 1505, and (d) 1515.

Fig. 4. Appearance of LWAs for samples: (a) 3005, (b) 3015, (c) 1505, and (d) 1515.

CaO addition and the hematite phase was segregated from the decomposition of clinochlore during the heating process. The hematite phase can react with unburned carbon to promote bloating behavior by the generation of O_2 gas from the reduction of Fe_2O_3 [3,10,23]. Although Fe_2O_3 can reduce to FeO with unburned carbon and release O_2 gas, no wuestite phase (FeO) was found in the XRD patterns. In a previous study, the change between Fe^{2+} ion and Fe^{3+} ion in LWAs made from reservoir sediment was observed by X-ray absorption near edge structure (XANES) spectra. The result showed that no Fe^{2+} ion was found in LWAs produced at 1150 °C [24]. In this study, fast heating rate may result in a higher amount of unburned carbon. However, the sediment had an ignition loss of only 5.57%, which means that the practical amount of unburned carbon was below 5.57%. This may put the wuestite phase out of the detection range of XRD even if the reduction reaction occurred. The XRD patterns also show that the intensity of the quartz phase decreased with increasing temperature, which is due to the glassy phase forming, or due to a reaction with calcium ions to form anorthite. The glassy phase increased the background of XRD between 20° and 30° due to its amorphous structure. All XRD patterns show this phenomenon with increasing temperature.

The appearance of the LWAs is shown in Fig. 4. The color of the LWAs was green–yellow and the roughness increased with increasing temperature. The 1515 series of samples at 1170 °C was white and had an unreacted state, since the heating time was insufficient for bloating behavior to occur. With increasing temperature, the glassy phase formed and some open pores formed on the surface. The viscosity decreased and the

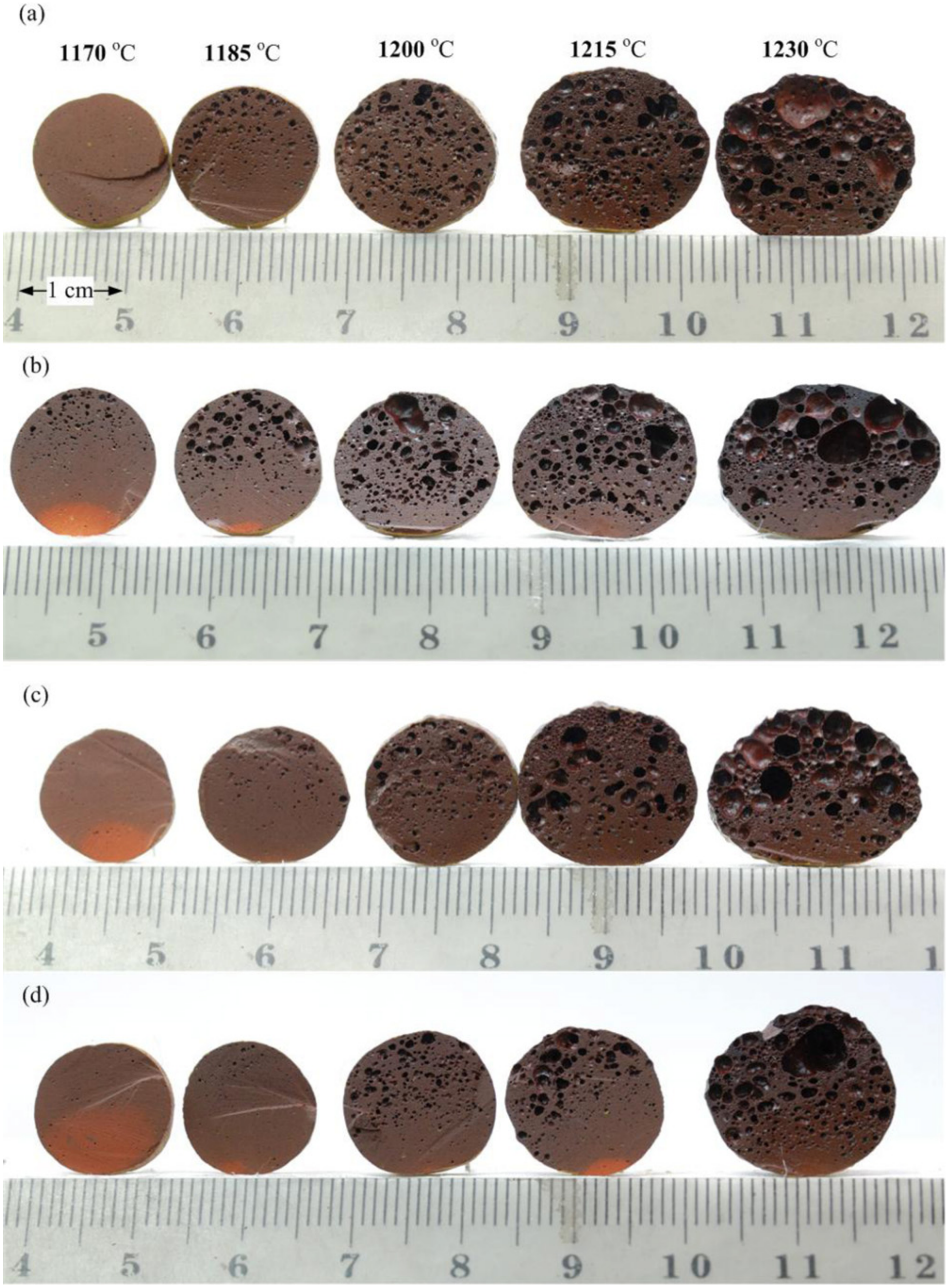

Fig. 5. Cross-section of LWAs for samples: (a) 3005, (b) 3015, (c) 1505, and (d) 1515.

roundness of the LWAs was hard to maintain. Fig. 5 shows the cross-section of samples. The number of pores increased and pores grew with temperature. Few pores formed at the bottom area in contact with the crucible, which was also observed by de'Gennaro et al. [4]. This may be caused by the thermal conductivity of the crucible, which makes the temperature at the bottom lower than that at the top. The heated area at the top was brown and that at the bottom was brick-red. The brick-red area was an unreacted area. Although a longer soaking time and a lower heating rate (3005) resulted in completely heated LWAs, pores easily formed in the brown area of the 3015 series of samples at 1170 °C. Pores were observed using SEM. The microstructure of the 3005 series of samples and the 3015 series of samples is shown, respectively, in Fig. 6a and b. The walls of pores had a smooth surface, which was caused by the glassy phase. At low temperatures, small pores (<0.1 mm) on the walls of large pores (>0.2 mm) connected to each other; however, the small pores were then sealed by glassy phase, which flowed at higher temperatures. This explains why the level of water adsorption initially increased and then decreased with temperature. Moreover, cracks were found between the smooth surface and the matrix. The formation of cracks may be caused by the difference of expansion coefficient between the glassy phase and the matrix during the cooling process. The cooling process can be improved to reduce crack formation.

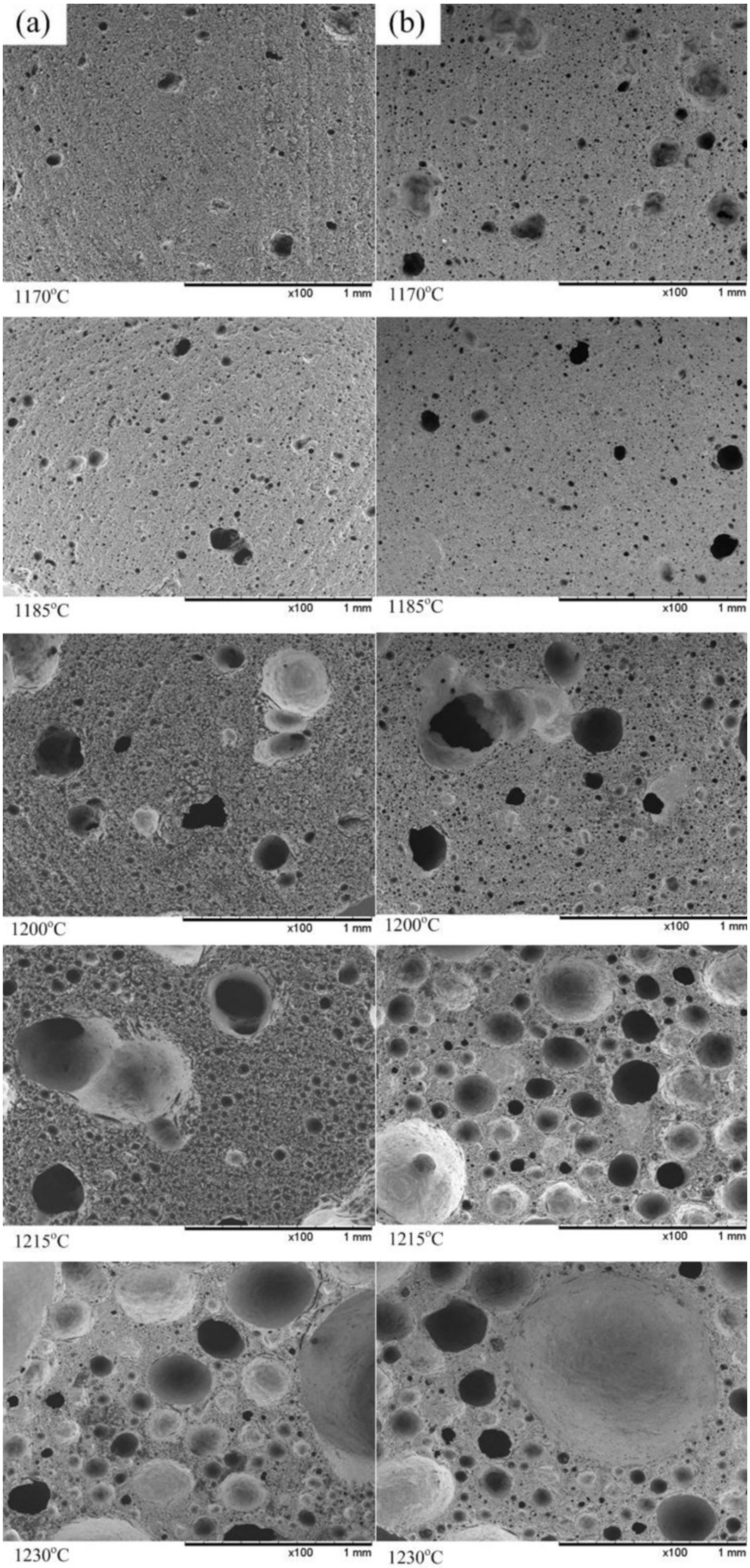

Fig. 6. Microstructure observation by SEM for samples: (a) 3005 and (b) 3015.

4. Conclusion

Four heat treatments were employed to observe their effect on the physical properties of LWAs produced from water reservoir sediment with added calcium oxide. The bulk density of LWAs produced at temperatures above 1200 °C meets European Union regulation EN-13055-1. The compressive strengths of the LWAs were above 1 MPa, making the LWAs suitable for lightweight concrete. It was found that a long soaking time and a high heating rate (3015 series) promoted bloating behavior and thus lowered the bulk density. However, a short soaking time and a high heating rate (1515 series) was better for producing LWAs with good strength performance (ratio of strength to unit weight of 3.14). The level of water adsorption of each series of samples ($<4\%$) initially increased and decreased with temperature due to the formation of the glassy phase, which made the walls of pores smooth and sealed the small pores (<0.1 mm) that were connected to large pores (>0.2 mm). The mineral phases of the LWAs were quartz, anorthite, and hematite. The wuestite phase was not found, possibly due to it being out of the detection range of XRD.

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