Chemical design of lightweight aggregate to prevent adhesion at bloating activation temperature

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1. Introduction

Recently, research for recycling waste materials into ceramic materials has been actively conducted \cite{1,2}. Artificial light aggregates are one of the promising areas for waste recycling. Artificial lightweight aggregates are building materials that are widely used in the construction industry, where lightweight materials and good heat insulation are required \cite{3–6}. Lightweight aggregates are mainly manufactured by the bloating and sintering of expanded clay or slate in a rotary kiln \cite{7,8}. In recent years, research has focused on the production of aggregates using waste materials \cite{9–15}. The production of lightweight aggregates using waste is a highly promising area due to the ability of this process to recycle a wide range of waste and realize their environmental usefulness. While active research on the production of lightweight aggregates using waste is ongoing, fewer studies have focused on the associated bloating mechanisms. According to Dondi et al. \cite{16}, it is not possible to explain the mechanism of the bloating of lightweight aggregates using waste as a conventional chemical-based bloating mechanism. Therefore, in order to explain the mechanism of bloating of lightweight aggregates using waste, it is necessary to consider not only the chemical composition but also a range of variables, such as the viscous behavior at high temperatures, gas generation, and process variables.

In various studies, the bloating of materials was explained in terms of the viscous behavior and internal pressure. Köse \cite{17} explained the bloating of foamed glass by pressure and viscous behavior. According to Köse, the bloating takes place in three stages, gas generation in the first stage, pore generation by the viscous behavior and internal pressure in the second stage, and pore growth in the third stage. Kaz'mina et al. \cite{18} studied the viscosity for the start of bloating, and de Gennaro et al. \cite{19} presented various gases that could occur during the production of lightweight aggregates. In a previous study by the authors \cite{20}, we found that the production of lightweight aggregates should be done in the optimum bloating zone, where the inner pressure and the viscous behavior of the material are appropriately formed. We also established an optimal unit process for the foaming of lightweight aggregates \cite{21}. Lee et al. \cite{22} found that the rate of the temperature rise is important for the aggregates to bloat and that the bloating mechanism developed depends on this rate. They reported a micropore bloating mechanism that occurs due to viscous behavior and an expansion of the internal pores under normal sintering conditions. With regard to the acid clay used in this study, bloating occurred due to the detachment of crystalline water and the viscous behavior at the optimum bloating activation temperature, and the pores were uniformly distributed throughout. However, using raw materials consisting of 100% acid clay can lead to the kiln outlet clogging problem before the bloating stages in a pilot rotary kiln sintering test. In this study, the reduction of Fe\textsubscript{2}O\textsubscript{3} was...
induced using Fe₂O₃ and carbon as additives to solve this problem.

The bloating phenomenon of lightweight aggregates by the reduction of Fe₂O₃ is widely known and is also referred to as the black core bloating phenomenon because the center part becomes black due to the reduction of Fe₂O₃ [23]. The existing form of lightweight aggregates was made of expanded shale and clay, and it has been the subject of various studies. Riley reviewed various clays and found a chemical composition suitable for the production of lightweight aggregates, after which identifying certain chemical compositions that could be used to form lightweight aggregates [24]. Cougney [25] redefined the four-component state diagram including Fe₂O₃ because Riley’s figure does not adequately show the effect of Fe₂O₃ on aggregates bloating. Kim et al. [26] reported that the area of reduction of Fe₂O₃ was affected by external oxygen partial pressures, and the lower the oxygen partial pressure is, the more active the reduction of Fe₂O₃ in the core becomes. Bernhardt et al. [27] noted that the shape of iron oxides is important to reduce iron oxide in aggregates. Lee [28] reported that Fe₂O₃ reduction occurs more readily in large-sized aggregates. Kang et al. [29] reported the optimum contents of Fe₂O₃ and carbon during the production of lightweight aggregates using coal bottom ash.

In this study, a pilot rotary kiln firing test was conducted to find the optimum bloating activation condition of acid clay. In order to solve the kiln outlet clogging problem occurring during this process, a bloating mechanism involving the reduction of Fe₂O₃ was combined with the bloating mechanism of the existing acid clay. For this purpose, the optimum contents of Fe₂O₃ and carbon were derived. The pilot rotary kiln firing test was carried out to confirm the bloating and adhesion problem of the derived optimum batch, and chemical conditions suitable for mass production were suggested.

2. Theoretical background

There are many hypotheses regarding the reaction of lightweight aggregates causing it to expand. It is well known that the reduction reaction of Fe₂O₃ occurs at temperatures above 1000 °C [23]. This is represented as follows:

\[2C + O_2 \rightarrow 2CO\]  
(1)

\[3Fe_2O_3 + CO \rightarrow 2Fe_2O_4 + CO_2\]  
(2)

\[2Fe_3O_4 + CO \rightarrow 3FeO + Fe_3O_4 + CO_2\]  
(3)

The reactions of (1) to (3) occur sequentially according to the partial pressure of the CO gas. The resulting FeO is expected to lower the melting point of the inside of the aggregate through the following reaction:

\[2FeO + SiO_2 \rightarrow 2FeO – SiO_2\text{ melting at 1183 °C}\]  
(4)

\[Na_2O + FeO + SiO_2 \rightarrow Na_2O – FeO – SiO_2\text{ melting at 702 °C}\]  
(5)

FeO reacts with SiO₂ to form fayalite, the melting point of which is 1183 °C. Moreover, when it is reacted with SiO₂-Na₂O, the melting point is lowered to 702 °C. However, Fe₂O₃ reacts with SiO₂ at a temperature of 1200 °C or higher and is present as a crystalline phase without melting. Therefore, when Fe₂O₃ is not reduced to FeO, the melting point is not lowered. Hence, the formation of FeO in the core part lowers the melting point of the aggregate and causes viscous behavior. However, given that the shell part does not undergo such a reduction reaction owing to a sufficient oxygen partial pressure, it has elastic behavior at the same temperature. Figure 1 shows a CT image of a sample bloated by a black core. In this analysis, distribution of the hematite crystal phase of the aggregates was observed through a 3D CT test (Malvern Panalytical CT-soution, Malvern, UK) of the aggregate. CT analysis and XRD analysis were combined. First, the overall crystal phase of the aggregate is analyzed using XRD analysis. Subsequently, the distribution of crystal phases in the cross-section was predicted based on the image data obtained through CT analysis. The software used for crystal phase analysis is HighScore Plus (4.6a) and the software used for CT image analysis is Volume Graphics Studio MAX (3.1). This method has been used in a variety of existing studies [30,31]. According to the Figure 1, the hematite crystal phase is mainly distributed in the shell and scarcely exists in the core. These results are evidence that the reduction of Fe₂O₃ did not occur due to the sufficient oxygen partial pressure in the shell and that the Fe₂O₃ reduction occurred in the core.

3. Experimental method

3.1. Raw materials

The prepared acid clay is identical to that used in our previous study [20]. The purity of the Fe₂O₃ and carbon used as additives was 99.9%, and activated carbon was used as the carbon source. And Fe₂O₃ used was α-Fe₂O₃. Both samples were produced at Reagents Duksan. The chemical composition of the acid clay is shown in Table 1.

3.2. Mixing and molding

The acid clay was compounded according to the desired composition and then mixed with 50 to 55 wt% of water. And it was molded into a spherical shape of 15 mm by hand. The additives were prepared by adding 0–15 wt% of Fe₂O₃ and 0–3 wt% of carbon. Table 2 shows the mixing conditions depending on the change of Fe₂O₃.
As the acid clay contained 2.9 wt% Fe$_2$O$_3$, the content of Fe$_2$O$_3$ was changed according to the amount of Fe$_2$O$_3$ added. At this time, the amount of carbon added was 2 wt %. The mixtures were named ACF0, ACF5, ACF10, and ACF15 according to the Fe$_2$O$_3$ content. Table 3 shows the batch changes with changes in the carbon content. The Fe$_2$O$_3$ content was fixed at 7.7 wt%. The mixtures are termed ACC0, ACC1, ACC2, and ACC3 depending on the carbon content. AC denotes acid clay, and C0 – 3 refer to the amount of carbon added. The pilot rotary kiln experiment was carried out using the optimum mixture derived from the above experiment, and the aggregates used in the experiment were molded by the extruder.

### Table 1. Compositions of raw materials (wt%).

|                | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | CaO  | MgO  | Na$_2$O | K$_2$O | TiO$_2$ | P$_2$O$_5$ | Ig-Loss | total |
|----------------|---------|-------------|-------------|------|------|---------|-------|---------|-----------|---------|-------|
| Acidic clay    | 67.3    | 12.9        | 2.9         | 2.8  | 2.4  | 1.6     | 1.8   | 0.6     | 0         | 7.7     | 100   |

### Table 2. Experimental batch according to the amount of Fe$_2$O$_3$ addition amount.

| Batch name | ACF0 | ACF5 | ACF10 | ACF15 |
|------------|------|------|-------|-------|
| Fe$_2$O$_3$ addition amount | 0    | 5    | 10    | 15    |
| Fe$_2$O$_3$ Contents (wt%)  |      |      |       |       |
| Carbon (wt%) |      |      |       |       |

### Table 3. Experimental batch according to the amount of carbon addition amount.

| Batch name | ACC0 | ACC1 | ACC2 | ACC3 |
|------------|------|------|------|------|
| Fe$_2$O$_3$ Contents (wt%)  |      |      |      |      |
| Carbon (wt%) |      |      |      |      |

### 3.3. Sintering

Aggregates were sintered in an electric furnace by a rapid sintering method at the target temperature for ten minutes. The rapid sintering method refers to a method in which a dried shaped body is put into an electric furnace heated to a target temperature and sintered for a predetermined time. The sintering temperature was raised from 1100 to 1200 °C at 25 °C intervals. A pilot-type rotary kiln experiment was carried out to investigate the sintering characteristics as practical work.

### 3.4. Measurement and analysis

The density and water absorption of the sintered samples were measured according to the KS 2503 specification [32], and the cross-sections of the aggregates were observed through an optical microscope (Dr. Camscope, Sometech, Seoul, Korea). The single-particle crush strength was measured using a universal testing machine (DS-001, Daeshin, Namyangju, Korea). The single-particle crush strength value (S) was determined according to the following equation,

\[
S = \frac{2.8P_c}{\pi X^2}
\]

(6)
where $P_c$ is the load at which the rupture occurs and $X$ is the diameter of the aggregate [33,34]. The single-particle crush strength was calculated from tests performed on 15 granules. The loose bulk density was measured according to KS 2505 [35].

4. Result and discussion

4.1. Rotary kiln firing characteristics of the raw acid clay

Figure 2 shows a comparison of the aggregates of acid clay at 100 wt% with rapid sintering at 1200 °C and the aggregates with the black core inside. For the acid clay 100 wt% aggregate (Figure 2(a)), no black core was observed inside. It was also found that the pores were relatively uniformly distributed. However, the aggregate bloated by the black cores shown in other study [22] suggest that the pores are concentrated in the center. (Figure 2(b)) Acid clay 100 wt% is bloated by the elimination of crystalline water at the bloating activation temperature, as described in previous studies [18,19]. The foaming phenomenon caused by the black core stems from the reduction of $\text{Fe}_2\text{O}_3$ inside, where $\text{Fe}_2\text{O}_3$ is reduced to form $\text{FeO}$, with the resulting $\text{FeO}$ reacting with $\text{SiO}_2$ to generate a material with a low melting point, thereby lowering the inner viscosity. The gas produced inside then raises the internal pressure of the aggregates and bloats the aggregates.

Figure 3 shows the results of the particle density and water absorption ratio measurements of the aggregates which had undergone rapid sintering of the 100 wt% acid clay. As a result, the aggregates of 100 wt% acid clay were sintered in the temperature range of 1160–1180 °C and the particle density increased. At 1200 °C, the particle density decreased due to bloating. Based on these results, it can be concluded that the range of 1180–1200 °C is the bloating activation range of acid clay 100 wt%.

The acid clay 100 wt% composition was extruded and a pilot rotary kiln experiment was carried out. Figure 4 shows the pilot rotary kiln (a) and aggregates used when clogging occurred (b). The surface of the aggregate of acid clay was fused at the setting temperature of 1130 °C in the kiln. Clogging of the rotary kiln outlet should be avoided during the manufacturing process of lightweight aggregates because it will block the outlet of the sintered aggregates and damage the refractory. In repeated experiments, the sintering condition for mass production of the rotary kiln cannot be secured.

This is believed to be due to the bloating mechanism of the aggregates, as explained above, where the acid clay is bloated by the loss of crystalline water at the bloating activation temperature. During this process, the entire

Figure 2. Cross-section of aggregates foamed by two types of foaming mechanisms (a) Acid clay aggregates sintered at 1200°C (b) Aggregates bloated by black core mechanism [22].

Figure 3. Particle density and water absorption ratio of the sample fired by the rapid sintering method.
amount of aggregates is subjected to viscous behavior, leading to a narrow distribution of the pores. However, due to the viscous behavior throughout the aggregates, the problem of clogging at the rotary kiln during production arises.

4.2. Analysis of raw materials and mixtures

The chemistry for classical lightweight aggregates was defined by Riley [22]. The Riley diagram [24] was used to confirm the chemical properties of the experimental mixture here. In order to show the chemical composition of the mixture in the Riley Diagram, the chemical composition was defined based on the XRF analysis results and the mixing ratio. Where flux is the sum of oxides that lower the melting point of the aggregates, which is the sum of CaO, Fe$_2$O$_3$, MgO, Na$_2$O, K$_2$O. The calculated chemical compositions are shown in Table 4 and plotted in Figure 5. As previously reported [20,21], the acid clay meets the specification of the (ACF0) Riley diagram. The ACF 5 and ACF 10 samples with 5–10 wt % of Fe$_2$O$_3$ added meet the requirements of the foam region according to the Riley diagram. The ACF15 mixture deviated from the Riley bloating area. In our previous study [20], the acid clay (ACF0) used had a content close to the critical Fe$_2$O$_3$ content in the Cougny diagram [25] due to the low amount of Fe$_2$O$_3$. The content of Fe$_2$O$_3$ is considered to be a very important aspect related to bloating by the internal reduction of the aggregates. In the literature, there are many studies dealing with the Fe$_2$O$_3$ content and the phenomenon of bloating due to internal reduction [11,36,37]. In those studies, an appropriate Fe$_2$O$_3$ content is required, and the ACF 5 and ACF 10 mixtures appear to be most appropriate among the mixtures used in this experiment.

Table 4. Chemical composition of each mixture in the ternary diagram (SiO$_2$, Al$_2$O$_3$, flux).

|     | SiO$_2$ | Al$_2$O$_3$ | Flux |
|-----|---------|-------------|------|
| ACF0| 72.9    | 14.0        | 12.5 |
| ACF5| 69.5    | 13.3        | 16.6 |
| ACF10| 66.3    | 12.7        | 20.4 |
| ACF15| 63.4    | 12.2        | 23.8 |

Figure 5. Riley diagram of test samples [24].
4.3. Change of physical properties of aggregates according to additive content

4.3.1. Effect of the Fe₂O₃ content on the physical properties of the aggregates

Green aggregates were fired by a rapid sintering method to produce the aggregates samples. Figure 6 shows the particle density levels and water absorption ratios of the aggregates samples produced. Bloating occurred at a low temperature in the ACF5 and ACF10 compositions, and the particle density levels were also low. The composition of ACF15 showed a particle density drop from a low temperature, but it showed a high density throughout the temperature range used in this study. Figure 7 shows a cross-section of the sintered aggregates. The ACF0 aggregates showed the black core phenomenon due to the reduction of Fe₂O₃ at 1175 °C, but it was not apparent at a lower temperature. The reduction of Fe₂O₃ was observed at 1125 ºC in the ACF5, ACF10, and ACF15 aggregates samples. As a result of the experiment, the proper content of Fe₂O₃ was found to be in the range of 7.5 to 11.7 wt%, and if it exceeds this range, the particle density becomes high. This result is consistent with the findings of Kang et al. [27]. It was observed that when Fe₂O₃ was added in excessive amounts, the internal reducing part leaks out because the excess FeO produced by the reduction of Fe₂O₃ significantly lowers the melting point of the aggregate core portion. This phenomenon appeared at 1150 and 1175 °C with the ACF15 aggregates. If such a phenomenon occurs during mass production, there is a possibility that the aggregates would mutually fuse because the liquid phase would leak from the inside. Therefore, this should be avoided during mass production.

4.3.2. Effect of the carbon additive content on the physical properties of the aggregates

As described in the previous section, the amounts of organic matter and carbon contained in the aggregates were varied. Figure 6 shows the change of physical properties of aggregates according to Fe₂O₃ content. Figure 7 shows the change of aggregates cross section according to Fe₂O₃ content and firing temperature.
aggregates directly affect the amount of reduced Fe$_2$O$_3$ in the aggregates. Kim et al. [26] reported that the partial pressure of CO is important for the internal reduction reaction of aggregates. When the amount of carbon is insufficient, the CO partial pressure becomes low. Therefore, the content of carbon in the aggregates is a very important variable.

In order to investigate the effect of the carbon content on the internal reduction reaction of lightweight aggregates, the Fe$_2$O$_3$ content was fixed at 7.7 wt%, the carbon was adjusted to 1–3 wt% and the mixtures were prepared and the aggregates were formed using the mixtures. The green aggregates were then fired by a rapid sintering method in each case. Figure 8 shows the particle density levels and water absorption ratios of the aggregates samples produced. When the carbon content was 0% by weight (ACC0), the particle density of the aggregates decreased from 1150 °C, but the decrease was not large. If the carbon content is 1% by weight (ACC1), the particle density continued to increase up to 1175 °C. When the carbon content was 2–3 wt% (ACC2, ACC3), the density gradually decreased due to bloating at 1125 °C. The trends of the change in the water absorption ratio according to the temperature of ACC0 and ACC1 were similar. The higher the sintering temperature was, the lower the water absorption ratio became. It was observed that the water absorption ratio of ACC0 overall was lower than that of ACC1 at the same temperature. The water absorption ratios of ACC2 and ACC3 decreased rapidly until 1125 °C. However, the decrease in the water absorption ratio at temperatures higher than 1125 °C was minor.

To investigate the effects of the internal reduction of Fe$_2$O$_3$ depending on the amount of carbon added, the cross-section was observed with an optical microscope. Figure 9 shows the cross-section outcomes. The internal Fe$_2$O$_3$ reduction phenomenon did not arise in the ACC0 samples. In the ACC1 samples, a black core was formed by the reduction of Fe$_2$O$_3$ despite the fact that this sample reached 1175 °C. In the ACC2 and ACC3 samples, reduction of Fe$_2$O$_3$ was observed from 1125 °C. It is known that the added amount of carbon affects the bloating temperature and physical properties of the aggregates [29]. The density levels of ACC0 and ACC1 did not decrease at 1125 and 1150 °C, and no reduction of the internal Fe$_2$O$_3$ was observed. At a temperature of 1175 °C, a small area of a black core was observed in ACC1, but the particle density was increased by sintering. In the ACC0 aggregates, a decrease in the density was observed at 1175 °C, thought to be due to the detachment of crystalline water at the bloating activation temperature. In the ACC1 case, the content of carbon is not sufficient, and it is considered that the internal reduction phenomenon does not occur actively. When Fe$_2$O$_3$ is reduced to FeO, the internal melting point can be lowered. However, the reduction reaction of Fe$_2$O$_3$ becoming FeO occurs through the intermediate stage of Fe$_3$O$_4$. When the amount of carbon added is insufficient, a considerable amount of Fe$_2$O$_3$ will not become FeO but will exist as Fe$_3$O$_4$ phase. Fe$_3$O$_4$, an intermediate product of the Fe$_2$O$_3$ reduction reaction, causes the interior of the aggregates to develop a black color but does not lower the internal melting point. However, in the ACC2 and ACC3 aggregates samples, bloating was observed due to the reduction of Fe$_2$O$_3$ in the temperature range of 1125−1175 °C, indicating that the added amount of carbon significantly affects foaming due to the reduction of Fe$_2$O$_3$. Experimental results show that a carbon content of 2–3 wt% is required for bloating by the reduction of Fe$_2$O$_3$ in the rapid sintering condition.

4.4. Characteristics of aggregates sintered in a rotary kiln

We investigated the optimum amounts of Fe$_2$O$_3$ and carbon for the production of aggregates in the previous section. As a result, we undertook extrusion molding to produce a rotary-kiln-fired aggregates using the ACC2 mixture. The ACC2 mixture contains acid clay 100 + Fe$_2$O$_3$ 5 + Carbon 2. The green aggregates were dried at

Figure 8. Change of physical properties of aggregates according to carbon content (a) Particle density (b) Water absorption rate.
100 °C for 24 hours and then fired in a pilot rotary kiln. Figure 10 shows the sample surface and its cross-section after firing in a rotary kiln. In the AC (raw) case, a mass production condition could not be obtained because this sample became fused at the rotary kiln setting temperature of 1160 °C. However, the ACC2 mixture aggregates did not fuse, and mass production was therefore deemed possible. The rotary kiln firing of ACC2 was performed at 1130 °C. Hence, we were able to identify two important aspects.

First, in the laboratory rapid sintering test, samples with bloating due to the overall viscous behavior of the ceramic body may cause clogging problems in a rotary kiln. Such clogging problems become a major problem during mass production.

Secondly, vitrification of the aggregate surface is required to bloom a lightweight aggregate [38–40]. However, the vitrified surface accelerates the adhesion of the aggregates and confounds mass production. However, when the reduction of Fe₂O₃ was induced in the aggregates, the adhesion phenomenon could be solved in the rotary kiln. Because FeO lowers the melting point of the core portion, the process can be performed at a temperature lower than the viscous behavior temperature of the raw material. Therefore, we could reduce the process temperature by more than 20 °C by inducing the Fe₂O₃ reduction phenomenon in the aggregates while also preventing surface adhesion, making the material feasible for mass productivity.

Table 5 shows the single-particle crushing strength, loose bulk density and particle density of the aggregates produced using the rotary kiln. The single-particle crushing strength was 4.1 MPa. Generally, commercialized lightweight aggregates are known to have a single-particle crushing strength of 2 to 3 MPa [33,34]. The samples produced with the pilot rotary kiln here had better strength than commercialized lightweight aggregates. The loose bulk density of the produced
aggregates was 810\text{kg/m}^3, which satisfied the structural lightweight aggregates standard KS F 2527 stipulation [41]. The particle density was 1.30 g/cm\(^3\). The particle density of the structural lightweight aggregates as specified by ASTM 330 C [42] is less than 1.50 g/cm\(^3\), and the aggregates samples produced here met this standard. The aggregates prepared using the ACC2 mixture satisfied the structural lightweight aggregate standard and showed better single-particle crushing strengths than a commercialized aggregate.

5. Conclusion

The experimental results of a novel chemical design method to prevent the adhesion phenomenon at the bloating activation temperature are presented below.

(1) In the condition of 7.5–11.5 wt% of Fe\(_2\)O\(_3\) and 2–3 wt% of carbon, the bloating phenomenon caused by the reduction of Fe\(_2\)O\(_3\) could be successfully induced in aggregates using acid clay.

(2) The bloating activation temperature was lowered due to the increased viscous behavior caused by the FeO generated in the aggregates, which occurred during the reduction phenomenon of Fe\(_2\)O\(_3\).

(3) Due to the reduction of Fe\(_2\)O\(_3\) inside the aggregates, the viscous behavior of the core and the shell differed, which prevents the adhesion of the aggregates.

(4) The raw acid clay underwent clogging due to adhesion before bloating under rotary kiln firing condition. The ACC2 mixture was prevented from clogging and was able to produce aggregates at lower temperature.

(5) The aggregates produced by a pilot rotary kiln with the ACC2 mixture satisfied the KS F 2527 specifications [41] and showed properties similar to those of commercial lightweight aggregates.

The reduction of Fe\(_2\)O\(_3\) inside the aggregates was found to lower the bloating activation temperature during the sintering process. In addition, because it causes a difference in the viscous behavior between the core and the shell, it has the effect of preventing the adhesion phenomenon in mass production with a rotary kiln. Therefore, it can be expected that the reduction of Fe\(_2\)O\(_3\) during the mass production of aggregates will reduce the fuel cost during the production of aggregates and solve the clogging problem during mass production.

Table 5. Physical properties of aggregates produced in pilot rotary kiln.

| Single Strength (MPa) | Crushing | Loose Bulk Density (kg/m\(^3\)) | Particle Density (g/cm\(^3\)) |
|----------------------|----------|---------------------------------|-------------------------------|
| 4.1                  | 810      | 1.30                            |

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Disclosure statement

No potential conflict of interest was reported by the authors.

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References

[1] Seo SK, Park JW, Cho HK, et al. Physical properties of cement system insulation using blast furnace slag. J Kor Ceram Soc. 2018;55(1):61–66.
[2] Lee KG, Bae SJ. Carbonation of circulating fluidized bed combustion fly ash with hybrid reaction. J Kor Ceram Soc. 2018;55(2):160–165.
[3] Aslam M, Shafigh F, Jumaat MZ, et al. Benefits of using blended waste coarse lightweight aggregates in structural lightweight aggregate concrete. J Clean Prod. 2016;119:108–117.
[4] Lo TY, Tang WC, Cui HZ. The effects of aggregate properties on lightweight concrete. Build Environ. 2007;42:3025–3029.
[5] Joseph G, Ramamurthy K. Influence of fly ash on strength and sorption characteristics of cold-bonded fly ash aggregate concrete. Constr Build Mater. 2009;23:1862–1870.
[6] Kumar VRP, Anandh K, Kumar M. An experimental study on partial replacement of natural coarse aggregate with fly ash coarse aggregate (FACA). Res Appl Sci Eng Tech. 2014;2:212–223.
[7] Decleer J, Viaene W. Rupelian boom clay as raw material for expanded clay manufacturing. Appl Clay Sci. 1993;8:111–128.
[8] [cited 2019.Aug.05]. Available from: https://www.leca.com
[9] Ayati B, Molineux C, Newport D, et al. Manufacture and performance of lightweight aggregate from waste drill cuttings. J Cleaner Prod. 2019;208(20):252–260.
[10] Piszcz-Karas K, Klein M, Hupka J, et al. Utilization of shale cuttings in production of lightweight aggregates. J Environ Manage. 2019;231:232–240.
[11] Yang C, Cui C, Qin J. Recycling of low-silicon iron tailings in the production of lightweight aggregates. Caram Int. 2015;41:1213–1221.
[12] Laursen K, White JJ, Cresswell DJF, et al. Recycling of an industrial sludge and marine clay as light-weight aggregate. J Environ Manage. 2006;80:208–213.
[13] Moreno-Maroto JM, Gonzalez-Corrochano B, Alonso-Azcarte J, et al. Manufacturing of lightweight aggregates with carbon fiber and mineral wastes. Cem Concr Compos. 2017;83:333–348.
[14] Cioffi R, Colangelo F, Montagnaro F, et al. Manufacture of artificial aggregate using MSWI bottom ash. Waste Manag. 2011;31:281–288.
[15] Abd EL-Raoof F, Soltan AMM, Kahl W-A, et al. Lightweight aggregates from mixtures of granite wastes with clay. J Clin Prod. 2016;117:139–149.
[16] Dondi M, Cappelletti P, D’Amore M, et al. Lightweight aggregates from waste materials: reappraisal of expansion behavior and prediction schemes for bloating. Constr Build Mater. 2016;127:394–409.

[17] Köse S, Bayer G. Schaumbildung im system alglas-SiC und die eigenschaften derartiger schaumgläser. Glas Ber. 1982;55:151–160.

[18] Kaz’mina OV, Vereshchagin VI, Abiyaka AN, et al. Viscosity evaluation for glass and glass crystal compositions in their foaming temperature range. Glas Ceram. 2009;66(7–8):236–239.

[19] de Gennaro R, Cappelletti P, Cerri G, et al. Zeolitic tufts as raw materials for lightweight aggregates. Appl Clay Sci. 2004;25:71–81.

[20] Wie YM, Lee KG. Optimum bloating-activation zone of artificial lightweight aggregate by dynamic parameters. Materials. 2019;12:267.

[21] Wie YM, Lee KG, Lee KH. Optimum condition for the unit process of an artificial lightweight aggregate using the taguchi method. J Asian Ceram Soc. 2019;7(3):331–341.

[22] Lee KH, Lee JH, Wie YM, et al. Bloating mechanism of lightweight aggregates due to ramping rate. Adv Mater Sci Eng. 2019. Article ID 2647391. doi:10.1155/2019/2647391

[23] Park JY, Kim YT, Lee KG, et al. The mechanism of black core formation. J Kor Cryst Grow Cryst Tech. 2005;15(5):208–215.

[24] Riley CM. Relation of chemical properties to the bloating of clays. J Am Ceram Soc. 1951;34(4):121–128.

[25] Cougny G. Specifications for clayey raw materials used to produce expanded lightweight aggregates. Bull Int Ass Eng Geol. 1990;41:47–55.

[26] Kim YT, Ryu YG, Jang CS, et al. A study on the black core formation of artificial lightweight aggregates at various sintering atmospheres. J Kor Cryst Grow Cryst Tech. 2009;19(6):318–323.

[27] Bernhardt M, Justnes H, Tellesbø H, et al. The effect of additives on the properties of lightweight aggregates produced from clay. Cem Concra Compos. 2014;53:233–238.

[28] Lee KG. Bloating mechanism of lightweight aggregate with the size. J Kor Ceram Soc. 2016;53(2):241–245.

[29] Kang SH, Lee KG, Kim YT, et al. Effects of chemophysical properties of carbon on bloating characteristics of artificial lightweight aggregates using coal ash. Ceram Trans. 2012;232(12):35–42.

[30] Takahashi H, Sugiyama T. Application of non-destructive integrated CT-XRD method to investigate alteration of cementitious materials subjected to high temperature and pure water. Constr Build Mater. 2019;203:579–588.

[31] Chung SY, Kim JS, Stephan D, et al. Overview of the use of micro-computed tomography (micro-CT) to investigate the relation between the materials characteristics and properties of cement-based materials. Constr Build Mater. 2019;229:116843.

[32] KS F. 2503: testing method for density and absorption of coarse aggregate. 2007.

[33] Yoshima S, Kanda Y, Sano S. Relationships between particle size and fracture energy or impact velocity required to fracture as estimated from single particle crushing. Powder Technol. 1987;51:277–282.

[34] Li Y, Wu D, Zhang J, et al. Measurement and statistics of single pellet mechanical strength of differently shaped catalysts. Powder Technol. 2000;113:176–184.

[35] KS F. 2505: standard test method for bulk density and solid contents in aggregates. 2017.

[36] Lee KG. Bloating mechanism for coal ash with iron oxide. J Kor Cryst Growth Cryst Technol. 2014;24:77–83.

[37] Gredmaier L, Banks CJ, Pearce RB. Calcium and sulphur distribution in fired clay brick in the presence of a black reduction core using micro X-ray fluorescence mapping. Constr Build Mater. 2011;25:4477–4486.

[38] Tsai CC, Wang KS, Chiou IJ. Effect of SiO₂-Al₂O₃-flux ratio change on the bloating characteristics of lightweight aggregate material produced from recycled sewage sludge. J Hazard Mater. 2006;134(1–3):87–93.

[39] Han MC, Han D, Shin JK. Use of bottom ash and stone dust to make lightweight aggregate. Constr Build Mater. 2015;99:192–199.

[40] Kang SH, Lee KG. Bloating mechanism of artificial lightweight aggregate for recycling the waste glass. J Kor Ceram Soc. 2010;47(5):445–449.

[41] KS F. 2527: concrete aggregate. 2018.

[42] ASTM. C330: standard specification for lightweight aggregates for structural concrete. 2017.