Optimum conditions for unit processing of artificial lightweight aggregates using the Taguchi method

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ABSTRACT
The purpose of this study was to investigate the unit processing parameters of a drying and preheating, calcination and sintering process for optimal bloating of artificial lightweight aggregates. Each process was divided into the room temperature ranges of up to 300 °C, 300 to 600 °C, 600 to 900 °C, and 900 to 1200 °C, with soaking at 1200 °C. The times allotted for the drying and preheating (room temperature ~ 600 °C) and calcination processes (600 ~ 1200 °C) were 10 to 40 minutes, and the time for the sintering process was 0 to 15 minutes. The experiment was designed according to the Taguchi method. The sintered samples were measured to determine the density and pore size. The processing time in the drying and preheating zone (room temperature ~ 600 °C) did not affect the bloating of the aggregates. The density of the aggregates decreased as the calcination time (900 °C ~ 1200 °C) was shortened and the soaking time (1200 °C) was lengthened. The soaking temperature and time had the greatest influence on the density of lightweight aggregates. The results predicted using the Taguchi method corresponded to the actual measurement results.

1. Introduction
Aggregates are defined as natural or artificially produced stone or sand and are used as basic materials in construction work. Among these, artificial lightweight aggregates refers to an aggregate that is lightened in weight by sintering expanded shale, clay, slate, and/or coal fly ash [1–3]. Recently, the production of lightweight aggregates using various types of waste has been studied [4–8]. The development of lightweight aggregates using waste materials is extremely promising in terms of waste recycling. There are two broad categories of variables that can affect the properties of lightweight aggregates. The first is the chemical properties of the raw materials as thermodynamic behavior. Chemical compositions suitable to the production of lightweight aggregates were defined by Riley [1] and Cougny [2]. According to their findings, there is a proper chemical composition for production of lightweight aggregates and the Fe₂O₃ content is important. Secondly, dynamic parameters are variables produced by forming and sintering. Kang [9] reported that the higher the density of the green aggregates, the better the bloating. Studies of sintering variables have been conducted worked various methods. Different studies have investigated various sintering methods, including rapid sintering [10,11], two-step sintering [12], and normal sintering [1,13]. Riley [1] reported that most clays were expanded by rapid sintering, and defined the chemical composition for the production of lightweight aggregates through the normal sintering process. Li et al. [12] undertook two-stage sintering by dividing the dry section and the sintering section and then established the bloating conditions of lightweight aggregates with a bulk density of less than 1.0. Kang et al. [13] obtained a lightweight aggregate with a low water absorption ratio and density level through normal sintering, and used the rapid sintering method in a study of lightweight aggregates with many types of waste added [10,11]. According to a report by Kaz‘mina et al. [14], it was confirmed that glass becomes bloated at a specific viscosity during the production of foamed glass, revealing that viscosity is an important aspect of bloating. Adell et al. [15] suggested that rapid sintering is more effective than normal sintering for the expansion of aggregates. In our previous study [16], we discovered a bloating activation zone where viscous behavior of aggregates and an increase in high internal pressure coexist under normal sintering conditions. According to Lee et al., bloating occurs in different ways depending on the sintering conditions of the aggregates [17].

In recent years, extensive research on lightweight aggregate production processes using the Taguchi method has been conducted. Chen et al. [18] describe a method for producing lightweight aggregates using reservoir sediments as the main raw materials.

In earlier studies, it was suggested that rapid sintering is effective when used to produce lightweight aggregates. However, the effects of the various temperature
ranges on the density of the aggregates, such as the drying and preheating (room temperature ~ 600 °C), calcination (600 ~ 1,200 °C), and bloating start and activation temperatures, were not specifically mentioned. Li et al. [12] provided the parameters for designing optimum sintering conditions. However, their study does not discuss which temperature ranges are important in normal sintering conditions. In a large-scale rotary kiln for producing lightweight aggregates, it is difficult to realize two-stage or rapid sintering conditions; moreover, a heating process is generally required. Clarifying the effects of unit processes such as drying and preheating calcination and bloating activation on the physical properties of aggregates, therefore making it would be possible to establish the proper production conditions for lightweight aggregates.

In this study therefore, we confirmed the effects of the retention time per unit process on the final properties of lightweight aggregates using the Taguchi method. The accuracy of each value predicted by the Taguchi method was verified by comparing the predicted value with the actual value. Optimum processing conditions for the production of artificial lightweight aggregates were designed by optimizing the unit processing variables.

2. Experimental method

2.1. Raw materials

The acid clay used in the experiment was the same as that used in our previous experiments, which was produced by Donghae Chemical Industrial Co., Ltd. (Seoul, Korea) [19]. Table 1 shows the results of an XRF (ZSR-100e, Rigaku, Tokyo, Japan) analysis of the raw materials used in the experimental. Accordance with our previous study [16], the crystal phase of the raw material was composed of materials including montmorillonite, albite, and quartz, and the chemical composition was consistent with the bloating region discussed by Riley [1], although in a Cougny diagram [2], it was found to be close to the bloat limit region due to the lack of FeO3. In our previous study [16], we found that acid clay can form when a certain amount of water is contained, but that the plasticity is low.

2.2. Taguchi experimental design

The aggregates were spherically shaped and 10mm in size. The water content of the aggregate molding was 50 to 55%. The density of the molded bodies was measured at be 1.20 g/cm³. The formed aggregates were put into an electric furnace without drying, and the molded bodies were fired according to the designed conditions. The experiment was designed as follows: The maximum temperature was fixed at 1200 °C and divided into 300 °C intervals for each temperature-rise section. The temperature-rise time was 10 to 40 minutes, and the soaking time was 0 to 15 minutes. An orthogonal array (OA), L₁₆(4⁵) and five controllable four-level factors were adopted. The designed experimental conditions are shown in Table 2. The density of the sintered aggregate was then measured. In our earlier study [16], rapid sintering tests of acid clay were carried out. As the sintering temperature increased, the density of the acid clay increased to 1175 °C, although the density decreased rapidly at 1200 °C. Based on this data, we interpreted the bloating activation start temperature to be 1175 °C and the bloating activation temperature to be 1200 °C. In this study therefore, the bloating start temperature was set at 1175 °C and the bloating activation temperature range was set at 1175 to 1200 °C. The aggregate began to bloat at the bloating activation temperature, with no bloat occurring at temperatures lower than the bloating start temperature. In this study, the sintering temperature was set at 1200 °C, as this temperature is within the bloating activation temperature range and bloating was confirmed by the rapid sintering test. In order to define each temperature range as a unit process, the processes were defined as drying and preheating process at room temperature to 600 °C, calcination processing in a temperature range of 600 to 1200°C, and bloating activation processing at 1200 °C. After confirming the tendency of each unit process, the experiment was designed again using the Taguchi method to confirm the temperature variables (1180, 1200, 1220 °C) in the bloating activation temperature range. Each experimental variable is shown in Table 3. In this experiment an orthogonal array (OA), L₁₆(3⁶) and seven controllable mix-level factors were adopted. The density was measured according to KS F 2503 [20], and the average pore size and pore volume were measured.

2.3. Experimental prediction using the Taguchi method

In order to investigate the feasibility of using the Taguchi method for prediction of the physical properties, we compared the predicted and measured values based on Taguchi test results. Predictions were made with Minitab (Minitab v1.4, Minitab Inc., Pennsylvania, U.S.A.).

Predictions by the Taguchi method are conducted as follows:

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

|       | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO | Na₂O | K₂O | TiO₂ | P₂O₅ | Ig-Loss | total |
|-------|------|-------|-------|-----|-----|------|-----|------|------|---------|-------|
| Acid clay | 67.3 | 12.9  | 2.9   | 2.8 | 2.4 | 1.6  | 1.8 | 0.6  | 0    | 7.7      | 100   |
Table 2. Progress times by temperature interval.

| Process                        | Processing temp. (°C) | Time |
|--------------------------------|-----------------------|------|
| Drying and preheating          | 25°C ~ 300°C          | 10 min, 20 min, 30 min, 40 min |
| Calcination                    | 300°C ~ 600°C         | 10 min, 20 min, 30 min, 40 min |
| Calcination                    | 600°C ~ 900°C         | 10 min, 20 min, 30 min, 40 min |
| Calcination                    | 900°C ~ 1200°C        | 10 min, 20 min, 30 min, 40 min |
| Bloating and activation (B.S.A.)| 1200°C soaking        | 0 min, 5 min, 10 min, 15 min |

\[
D_{\text{pred.}} = D_{\text{tot}} + (D_i - D_{\text{tot}}) + (D_j - D_{\text{tot}}) + (D_k - D_{\text{tot}}) + (D_l - D_{\text{tot}})
\]

(1)

(D_{\text{pred}}: Predicted value of density, \(D_{\text{tot}}\): Total average value of density, \(D_{i,j,k,l}\): Average value of each variable)

The effect of each variable on the physical properties is reflected in the predicted value.

2.4. Pore observation and pore distribution measurements

In order to observe the porosity, representative samples were selected from the measured samples and sections were observed. A cross-section of the aggregate in each case was observed using an optical microscope (Dr. CAMSCOPE, Somotech, Seoul, Korea) and the micropores were observed by SEM (S-4800, Hitachi, Tokyo, Japan). In order to measure the pore distribution, mercury intrusion porosimetry (PoreMaster-60GT, Quantachrome, Florida, U.S.A.) was utilized. Mercury intrusion porosimetry involves immersing an object in mercury and measuring the distribution of the pores by applying pressure. It is abbreviated as MIP.

2.5. Bloating index (BI) and single aggregate crushing strength

The bloating index and the single aggregate crushing strength of the representative samples prepared with the optimal parameters obtained by the Taguchi method were measured.

The expansion index (BI) has been referenced by previous studies [21,22] and was calculated by the following equation:

\[
\text{BI} = 100 \times \frac{(d_2 - d_1)}{d_1}
\]

(2)

(BI: Bloating index, \(d_1\): Diameter before sintering, \(d_2\): Diameter after sintering)

The single particle breaking strength was measured with reference to previous studies [23,24]. The single aggregate crushing strength was measured for 15 granules using a universal testing machine (DS-001, Daeshin, Namyangju, Korea). The equation used was as follows:

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

(3)

(S: Single aggregate crushing strength, \(P_c\): Crushing load, X: Diameter of the aggregate)

3. Results and discussion

3.1. Density variations according to the processing variables

Artificial lightweight aggregates are mainly produced using a rotary kiln. According to Cougny [2], various types of kilns, such as those with two or three stages, are used for the production of these aggregates. In order to bloat the aggregates, it is necessary to adjust the drying and preheating, calcination, and sintering times, not just the sintering time.

The Taguchi method, also referred to as the orthogonal array method, divides the experimental variables equally according to an orthogonal array table and then statistically outputs the results for each variable to quantify the effect on each variable. Experiments designed according to orthogonal array tables, and the measured densities are shown in Table 4. Figure 1 shows the results of the designed experiments.

3.1.1. Drying and preheating processes

The drying and preheating time had little effect on the densities of the aggregate. The temperature ranges from room temperature to 300 °C and from 300 °C to 600 °C did not affect the densities. This means that the gas affecting the bloating of aggregates is not related to the evaporation of the interlayer water and free water at low temperatures, and the processing time in this section has no a significant effect on the densities of the aggregates. If the drying step in the ceramic process is too short, cracks may occur in the body; thus, it is preferable for the temperature interval to be sufficiently long.

3.1.2. Calcination process

Internal moisture in clay minerals is divided into interlayer water and crystalline water. The process of eliminating the interlayer water is called the drying and preheating process, and the process of eliminating the crystalline water and generating other gases is called the calcination process. In the case of montmorillonite, which is a major component of acid clay, the interlayer

Table 3. Processing variables by item.

| Process                           | Processing temp. (°C) | Variable                  |
|-----------------------------------|-----------------------|---------------------------|
| Drying                            | 25°C ~ 600°C          | 80 min Fixing             |
| Calcination                       | 600°C ~ B.S.A. temp.  | 20, 40, 60 min            |
| Bloating and activation (B.S.A.)   | B.S.A. temp           | B.S.A. time 0, 5, 10, 15, 20, 40 min |
|                                   |                       | B.S.A. temp. 1180, 1200, 1220 °C |
water evaporates at 100 to 200 °C, and the crystalline water begins to be eliminated at about 450 to 550 °C, with the loss of weight continuing slowly. In our previous study \cite{15}, weight loss of acid clay was observed continuously up to 1000 °C. At a temperature of 1100 °C or higher, a high-temperature crystalline phase such as mullite, spinel, or $\beta$-quartz and a glass phase are formed \cite{25}. Riley \cite{1} suggested that montmorillonite-based minerals are more advantageous for lightweight aggregate bloating because the elimination of crystalline water is relatively slow, unlike that of kaolin-based minerals.

When the processing time in the calcination zones of 600 ~ 900 °C and 900 ~ 1200 °C was 10 min, the density of the aggregate decreased by 0.1 ~ 0.2, more than the other parameters.

In Riley \cite{1}, most of the aggregates became bloated during the rapid sintering process. This can be interpreted as meaning that rapid sintering is advantageous for bloating because it traps gas generated during the calcination stage. The results of the present experiment also show that low density can be obtained by trapping gas generated during the calcination stage. The experimental results demonstrate that it is advantageous for the bloating of a lightweight aggregate to raise the temperature rapidly in the calcination zone above 600 °C.

### 3.1.3. Bloating activation process

According to Dondi et al. \cite{26}, using the bulk chemical composition reported by Riley et al. \cite{1} is not well suited to prediction of lightweight aggregate bloating. They also discuss other factors, such as viscous behavior and the sintering schedule. According to a study by Kaz’mina \cite{13}, glass does not bloat unless the viscosity exceeds a certain level when bloating is attempted. This means that a certain type of viscous behavior is required during the bloating of aggregates.

Figure 1 shows that the aggregate density decreases as the soaking time at 1200 °C long there. Hence, the temperature of 1200 °C is an appropriate bloating activation temperature, at which, aggregates show appropriate viscous behavior for bloating. The lowering of the density as the soaking time at 1200 °C long there indicates that both the appropriate viscous behavior and an appropriate time for bloating are necessary for the bloating of the aggregate. In Figure 1 confirms that the decreases in the density continued up to a soaking time of 15 minutes.

To extend the results shown in Figure 1, an experiment was designed using the Taguchi method again with the variables in Table 3. These results are shown in Table 5, and an analysis of the experiment is shown in Figure 2.

As seen in Figure 2, the tendency in the calcination section was favorable for bloating when the calcination...
section (600 °C ~ maximum temperature) was shortened. It was found that the density of the aggregate decreases with increases in the temperature in the bloating activation temperature range. This is thought to be related to the viscous behavior of the aggregate; at higher temperatures, viscous behavior increases due to increases in the liquid phase. Lee et al. [10] and Kang et al. [13] also found that the aggregate density was lower at higher temperatures. The density decreased as the soaking time at the bloating activation temperature increased, but the density decreased after 10 ~ 20 minutes, with no significant difference between 20 and 40 minutes. The density of the aggregate is considered to reach saturation when the soaking time at the bloating activation temperature is 20 minutes.

### 3.2. Prediction of aggregate bulk density by the Taguchi method

The aggregates were sintered using the parameters obtained by the Taguchi method. The drying and preheating process time (room temperature ~ 600 °C) was set at 80 min, and the calcination time (600 ~ 1200 °C) was fixed at 20 min. The aggregates were sintered at 1150, 1180, 1200, and 1220 °C. The experimental results are shown in Figure 3. At 1150 °C, the results show a tendency different from those at other temperatures. The temperature of 1150 °C is lower than the bloating activation temperature. In order to bloat lightweight

![Figure 3](image-url)
aggregates, sintering at the bloating activation temperature is required. Even if the soaking time is prolonged at a lower temperature, adequate bloating cannot be achieved.

The results were based on the data obtained using the Taguchi method estimated. Each predicted value is a value considering the influence of each experimental condition, and the predicted values are compared with the measured values. These results are shown in Figure 4. This was done to verify application of the Taguchi method to experimental predictions. The density variation tendency was found to be generally similar. The density was lowered at a soaking time up to 10 to 20 minutes, after which the sample became saturated and the density was scarcely lowered. The pressure created by the production of gas creates and expands the pores, and this phenomenon lowers the density of the aggregate. The density of the aggregate decreased as the soaking time increased. The reason for this can be explained by the viscous behavior of the aggregate and the increased internal pressure due to gas production. In order to bloat the aggregate, it is necessary to maintain proper viscous behavior during aggregate bloating, and the longer the time, the more the internal pressure is relieved and more pores are generated. The saturation time of the density change of the aggregate decreased with increases in the sintering temperature. This had two causes. First, the higher the sintering temperature, the earlier the bloating is activated. Second, at higher temperatures, more vigorous active viscous behavior and gas evolution occur. The saturation time of the density change of the aggregate decreased with increases in the soaking temperature.

As a result gas is generated and discharged at a high rate leading to an earlier release of the pressure. It is therefore considered that the time required to reach saturation density is shortened. The mean densities were similar when the soaking times were 20 minutes and 40 minutes. The longer the soaking time at the bloating activation temperature, the less the density is lowered as the internal pressure is relieved. This phenomenon was common within the bloating activation temperature range. Hence, 20 minutes is judged to be a sufficient time to reach saturation.

3.3. Observation of the pore structure

3.3.1. Observation of the cross-section

According to Köse et al. [27], the generation and development of pores can be divided into three stages.

1. Gas is generated inside the body.
2. The generated gas increases in pressure and breaks the internal pore walls, merging the pores.
3. The pores grow due to a difference in pressure between small pores and large pores.

At this time, processes 1 and 2 take place in the beginning, and process 3 takes place afterwards. A cross-section of the aggregate was observed with an optical microscope. The changes in the density of the aggregate were monitored. This observation showed the development of the pore structure. The changes in the density of the aggregate were monitored. This observation showed the development of the pore structure.
aggregate according to the soaking time are shown in Figure 5, and the changes with the soaking temperature are shown in Figure 6. In order to observe the changes in the pore size more closely, we utilized SEM, as shown in Figure 7. It was found that the longer the soaking time at the bloating activation temperature of the aggregate and the higher the soaking temperature, the larger the pore size. This can be explained by the porosity growth theory of Köse et al. [27].

The higher the soaking temperature, the larger the pore size observed because of the viscous behavior of the aggregate increases with increases in the temperature of the aggregate increases. Even if the same amount of gas is generated moreover, the formation and binding of the pores become more active due to the highly viscous behavior of the aggregate. The pores of the aggregate, therefore, grow larger when the soaking temperature is high. It was also observed that the pore size gradually increases according to the soaking time. When the viscous behavior of the aggregate is identical at the same temperature, a decrease in the density and expansion of the pores occur simultaneously for the first 10 to 20 minutes. In this section, it is assumed that bloating takes place due to processes 1 and 2. At 40 minutes, the porosity expanded but the density of the aggregate was not reduced. It was determined that the generation of gas was terminated at a soaking time of 40 minutes because the density was not lowered over time. Due to the expansion of pores through process 3, the pores continued to grow.

3.3.2. Mercury intrusion porosimetry (MIP) measurements

A MIP analysis was conducted to measure the pore distribution of the samples. These results are shown in Figure 8, and the total volume of the pores analyzed based on this data is shown in Figure 9.

In the previous section (3.3.1), we confirmed that the structure of the large pores varies with the soaking time and temperature of the aggregate. Because it is difficult to judge the micropores by observing cross-section, however, the changes in the micropores were quantified through MIP measurements. In the MIP measurement method, the sizes and volumes of the pores are calculated by measuring changes in the mercury input under pressure. Owing to this method, measuring of micropores is easy, but measurement of pores 200 μm or measuring greater in size is impossible. The results in Figure 7 (a) show that when the soaking time is 0 min, the micropores are most widely distributed and the volume of large pores is low. It

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**Figure 5.** Cross-section of aggregates (soaking temperature-time): (a) 1200°C-0, (b) 1200°C-10, (c) 1200°C-20, and (d) 1200°C-40.

**Figure 6.** Cross-sections of aggregates (soaking temperature-time): (a) 1180°C-20, (b) 1200°C-20, and (c) 1220°C-20.
was also found that a soaking time of 10 minutes increases the distribution of pores larger than 100 μm. This occurs because the expansion of the pores due to the generation of gas occurs when the soaking time is prolonged. However, at a soaking time of 10 minutes or more, the number of pores 100 μm in size gradually decreased. This is considered to have occurred because the pores grew beyond the measurement limit of the MIP process and were not reflected in the measurement results. As shown in Figure 6, it is clear that the porosity increases as the soaking time long there. The total volume of pores larger than 100 μm is shown in Figure 8. The total volume of pores less than 100 μm in size did not significantly increase at 1200 °C with a soaking time of 10 min or more because the density of the

Figure 7. Microstructural changes with soaking times (a) and temperatures (b).

Figure 8. Pore distribution changes with soaking time and temperature: (a) soaking time, (b) soaking temperature.
aggregate did not decrease significantly and the pore size grew to a range that could not be measured by MIP even if the soaking time was prolonged.

It was observed that at a soaking temperature of 1220 °C, the number of large pores at 100 μm in size increased abruptly, and the total pore volume was enhanced. This is a phenomenon that occurs because the higher the soaking temperature in the bloating activation range becomes, the greater the intensity of viscosity behavior by the aggregate.

When the soaking temperature was high, the pore volume increased over the entire range. Additionally, as the soaking time became longer, small pores disappeared and larger pores appeared. When the soaking temperature is high, pores are generated and grow relatively quickly due to high-viscosity behavior. As the soaking time increases, the growth of the pore size is considered to be due to the growth of pores caused by Ostwald ripening.

3.4. Bloating index and single aggregate crushing strength

The sintering conditions of the aggregate were optimized by the Taguchi method. The sintering conditions of the aggregate are 80 min for the drying and preheating process (room temperature ~ 600 °C) and 20 min for the calcination process (600 ~ soaking temperature) at soaking temperatures of 1180, 1200, and 1220 °C and a soaking time of 15 min. The BI and single aggregate crushing strength were also measured.

The results for the BI measurement are shown in Figure 9. The BI tended to increase as the temperature increased. The lower the density, the higher the BI became, as in our previous study [16]. At a soaking temperature of 1180 °C, the BI was 8% and the density was 1.38 g/cm³. At 1200 °C, the BI was 18% and the density was 1.10 g/cm³. This satisfied EN 13055–1 the structural lightweight aggregate standard [28]. At 1220 °C, a light aggregate with a particle density of less than 1.0 g/cm³ was obtained.

The crushing strength of a single aggregate was measured, as shown in Figure 11. The single aggregate crushing strength of the prepared aggregate is 1.8 ~ 3.5MPa, while that of a commercially available aggregate is 3 ~ 4MPa [22]. The aggregates calcined at 1180 °C for...
15 min showed properties similar to those of commercially available structural aggregates.

4. Conclusion

In this study, we investigated the effect of various unit processes on the physical properties and pore structures of aggregate materials using the Taguchi method. The following conclusions were obtained.

1. Temperatures below 600 °C did not affect the physical properties of lightweight aggregates. In order to obtain a low density, it is advantageous to make the calcination process (600 ~ soaking temperature) as short as possible.

2. The final density of the aggregate was lowered as the temperature was increased during the bloating activation process, and it was found that as the viscous behavior of the aggregate rose at higher temperatures, bloating of the aggregate became more favorable.

3. When the soaking time is prolonged, there is an inflection point at which the decreases in the aggregate density decelerate. The higher the processing temperature rises, the shorter the time becomes. In this experiment, the inflection point appeared at 5 ~ 15 minutes.

4. Bloating of aggregates occurs in the bloating activation range, and aggregates do not bloat at lower temperatures. For proper bloating, a soaking time of 5 minutes or longer is required at the bloating activation temperature.

5. Using the Taguchi method, changes in the aggregate properties could be predicted. These outcomes are considered to provide good reference materials for the design of aggregate properties.

6. When the soaking time becomes too long, the density of the aggregate does not decrease but the pores become larger. It is therefore desirable to use a proper soaking time during the bloating activation process.

Through this experiment, we were able to understand the effect of each production process on the physical properties of the aggregates, also to design processing variables for the mass production of such aggregate materials. Using the Taguchi method enabled to produce aggregates with various properties.

Disclosure statement

No potential conflict of interest was reported by the authors.

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