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# Effect of $SiO_2$–$Al_2O_3$–flux ratio change on the bloating characteristics of lightweight aggregate material produced from recycled sewage sludge

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**Abstract**

This study investigates the characteristics of lightweight aggregates sintered from sewage sludge ash by modifying the proportion of the main components ($SiO_2$–$Al_2O_3$–flux). The ash of incinerated sludge from a municipal sewage treatment plant (STP) was used as the tested material and sintering temperature ranged from 1050 to 1100 °C within a time span of 10–30 min. The sludge ash appeared to have a high proportion of $SiO_2$ (44.89%), $Al_2O_3$ (11.62%) and $Fe_2O_3$ (6.81%) resembling the dilatable shale. When the sintering temperature was raised to above 1060 °C, the blowing phenomenon appeared. The aggregates become lighter in weight by prolonging the sintering time and raising the temperature. Cullet powder (amorphous $SiO_2$), $Al_2O_3$, and fly ash were added to sludge ash to analyse the characteristic changes of the aggregates. The results showed that amorphous $SiO_2$ lowered the melting point and increased foaming; $Al_2O_3$ raised the compression resistance; fly ash lowered the sintering temperature required. However, the composition of fly ash can vary dramatically, resulting in a less predictable characteristic of aggregates.


*Keywords:* Incinerated sludge ash; Lightweight aggregates; Sintering techniques; Bloating effect

## 1. Introduction

Treatment of sewage sludge from municipal wastewater treatment plants is one of the important environmental issues in Taiwan. Such sludge can be used as fertilizers or as organic soil because it contains valuable components [1,2]. However, the accumulation of high concentration of heavy metals could result in new environmental considerations when recycled sludge ash had been used as alternative building materials [3,4,19]. However, previous research and development on new building materials or admixture in mortars using sludge ash have shown that it is a very complex task [5,17,18].

Conversion of sludge into lightweight aggregates for concrete production has been investigated by several researchers. Lightweight fine aggregates can be produced from pulverized sludge ash [6]. In the study, sludge ash was pulverized and mixed with water and waste liquor from alcohol plants, and was pelletized before been heated to 1050 °C. The resulting residues were of lower specific gravity and slightly lower strength than the conventional artificial fine lightweight aggregates [7].

Recycling sewage sludge as artificial lightweight aggregates (ALA) involves mechanisms of bloating and sintering of the sewage sludge ash (SSA) during heat treatment. The characteristics of thermal bloating of sewage sludge is an important factor in producing lightweight aggregates from the sludge ash. In general, two essential conditions are considered to be co-existed in the ash at elevated temperatures when gaseous bubbles are generated and trapped inside the pyro-plastic mass; i.e. the development of a glassy phase, and the evolution of gases from the dissociation of mineral components [8].

Typical bloating materials such as clay, shale and slate, and to some extent treated coal shale are known to contain natural gas producing compositions, generating gas at different levels of temperature. Accordingly, this bloating mechanism is sensitive to the variation in ash components and operational temperature. Therefore, major components in sludge ash, such as $Al_2O_3$,

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$SiO_2$, flux (i.e. the mixture of $Fe_2O_3$, FeO, CaO, MgO, $Na_2O$ and $K_2O$), and typical gas-producing materials such as carbonates, oxides, hydrates and sulfates, will profoundly affect the bloating behavior of the sewage ash during heat treatment process.

Due to the heavy metal content, fly ash from municipal solid waste incineration (MSWI) has been classified as a hazardous waste by some countries [9,10]. Reusing this waste as a "recycled aggregate" for concrete production is very appealing since it reduces the use of natural aggregates [11]. Since fly ash usually contains relative rich flux ingredients such as CaO, MgO, $Na_2O$, $Fe_2O_3$, MnO, it can be considered as additives for adjusting flux composition during the sintering process.

Properties of lightweight sludge ash aggregates were also investigated by Bhatty [12,13], who reported the lightweight coarse aggregates made from pelletized or slabbed sludge ash incinerated at between 1050 and 1110 °C could produce moderate strength concrete. In a study by Yip and Tay [14], sludge incinerated in a brick-heating kiln at 1050 °C was used in the production of lightweight coarse and fine aggregates. Khanbilvardi and Afshari [15] also investigated the potential for using sludge ash as fine aggregate in concrete.

The aim of this study is to determine the effects of sludge ash composition and heating temperature on properties related to bloating effect, as well as to establish effective parameters for evaluation. The SSA was collected from a secondary sewage treatment plant, and various compositions of sludge ash were tested at 1050–1100 °C, by adding cullet powder, aluminum oxide ($Al_2O_3$), and fly ash from a municipal incinerator. The results of this study provide useful information on co-treating and recycling SSA as lightweight aggregates that effectively account for the sludge ash.

## 2. Materials and methods

### 2.1. Materials

The SSA was taken from a STP, which contained an activated sludge reactor and a rotating biological contactor capable of treating 15,500 tons/day of municipal sewage. The mixed sludge generated from the sedimentation tank, activated sludge reactor, and rotating biological contactor is aerobically digested and dewatered after polymer conditioning.

The sludge ash used in this study was prepared firstly by burning the digested sludge cakes from the primary and secondary combustion chambers at 900 °C for 1 h to obtain incineration residues. The residues were then pulverized with a ball mill and oven-dried at 105 °C until a constant weight was reached. The oven-dried sewage sludge was stored in a desiccator for the preparation as test pellets. Fly ash (cyclone ash) was taken from a mass-burning MSW incinerator located in the northern part of Taiwan for experiments of composition modification. It was oven-dried and desiccated likewise. The test SSA, cullet powder and fly ash are characterized in Table 1 and all three contain relative high proportion of $SiO_2$.

The analysis results show the main components of sludge ash including $SiO_2$, $Al_2O_3$, CaO and $P_2O_5$. The X-ray diffraction (XRD) testing justified the high proportion of quartz in the sludge ash (Fig. 1). This figure indicates that other crystal phase substances include $Al_2O_3$, $Fe_2O_3$, and $P_2O_5$ as well. The heavy metal concentration and leaching tests showed a high content of Pb, Cu, and Zn (Table 2), but was still within the acceptable level of environmental regulations. Table 2 also shows a high proportion of metals, mainly Pb, Cu, Zn, in fly ash. The TCLP test shows that Zn has the highest leaching concentration of 16.2 mg/l. However, Zinc has not been regulated by Taiwan EPA. The TCLP test showed that Cd content exceeded the regulatory standards of Taiwan EPA, therefore was classified as hazardous waste. It will need additional permission from Taiwan EPA before it can be reused.

Table 1
Chemical composition of the sludge ash, fly ash and cullet powder (w/w,%)

| Component | Sludge ash | Fly ash | Cullet powder |
|---|---|---|---|
| $SiO_2$ | 44.89 ± 4.32 | 35.81 ± 3.44 | 92.14 ± 4.60 |
| $Al_2O_3$ | 11.62 ± 0.60 | 9.81 ± 0.24 | 0.84 ± 0.04 |
| $Fe_2O_3$ | 6.81 ± 0.57 | 4.91 ± 0.41 | 0.37 ± 0.02 |
| CaO | 6.49 ± 0.38 | 14.71 ± 0.52 | 0.06 ± 0.01 |
| MgO | 0.10 ± 0.02 | 0.83 ± 0.02 | 0.01 ± 0.00 |
| $Na_2O$ | 0.04 ± 0.01 | 5.98 ± 0.36 | 2.05 ± 0.02 |
| $K_2O$ | 2.93 ± 0.31 | 5.37 ± 0.57 | 0.97 ± 0.12 |
| $P_2O_5$ | 18.68 ± 1.85 | – | – |

### 2.2. Methods

This study covers two parts. The first part observed the bloating effect and diameter of aperture caused by bubbles generated at various heating temperatures, between 1050 and 1100 °C. The second part studied the effects of the main ingredients, i.e. $SiO_2$, $Al_2O_3$ and flux, to the bloating of SSA. The various compositions of SSA were then modified by adding cullet powder (amorphous $SiO_2$), aluminum oxide ($Al_2O_3$), and MSW fly ash (flux materials) in the range from 5 to 20%, respectively, with 5% increments. The chemical compositions of all tested sludge ash and their modifications are shown in Table 3. The tested sludge ash was pelletized to form cylindrical samples with a 1.2 cm in diameter and 1.3 cm in height. The compact SSA pellets without composition modification were heated at temperatures ranging

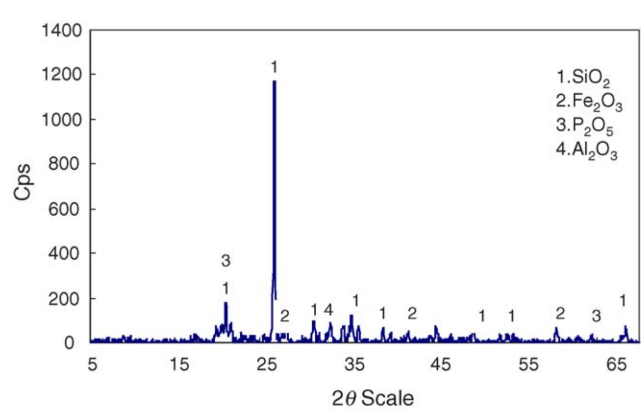



Fig. 1. The species of the sludge ash by XRD.

Table 2
Heavy metal concentrations and leaching test results of the sludge ash and fly ash

| | Pb | Cd | Cr | Cu | Zn |
|---|---|---|---|---|---|
| Concentration of sludge ash (mg/kg) | 201.7 ± 5.2 | 10.3 ± 0.5 | 77.3 ± 2.5 | 420.3 ± 10.2 | 2308.2 ± 16.5 |
| TCLP results of sludge ash (mg/l) | ND | 0.07 ± 0.01 | ND | 0.63 ± 0.01 | 6.05 ± 0.13 |
| Concentration of fly ash (mg/kg) | 1284 ± 14.8 | 80 ± 1.7 | 811 ± 24.6 | 1409 ± 89.1 | 7115 ± 163 |
| TCLP results of fly ash (mg/l) | 0.72 ± 0.022 | 1.82 ± 0.031 | 4.30 ± 0.131 | 0.61 ± 0.056 | 16.20 ± 1.940 |
| Regulatory level of TCLP test by ROC EPA (mg/l) | 5.0 | 1.0 | 5.0 | 15.0 | – |

Table 3
Composition of tested SSA samples

| | Addition rate (%) | Compositions (%) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | SSA (w/w,%) | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $Na_2O$ | $K_2O$ | $P_2O_5$ | Flux[a] |
| SSA[b] | | 44.89 | 11.62 | 6.81 | 6.49 | 0.10 | 0.04 | 2.93 | 18.68 | 35.05 |
| SSA + $SiO_2$ | 5 | 47.51 | 11.07 | 6.49 | 6.18 | 0.10 | 0.04 | 2.79 | 17.79 | 33.39 |
| | 10 | 49.90 | 10.56 | 6.19 | 5.90 | 0.09 | 0.04 | 2.66 | 16.98 | 31.86 |
| | 15 | 52.08 | 10.10 | 5.92 | 5.64 | 0.09 | 0.03 | 2.55 | 16.24 | 30.47 |
| | 20 | 54.08 | 9.68 | 5.68 | 5.41 | 0.08 | 0.03 | 2.44 | 15.57 | 29.21 |
| SSA + $Al_2O_3$ | 5 | 42.75 | 15.83 | 5.88 | 6.18 | 0.10 | 0.04 | 2.79 | 17.72 | 32.71 |
| | 10 | 40.81 | 19.65 | 5.69 | 5.90 | 0.09 | 0.04 | 2.66 | 17.68 | 32.06 |
| | 15 | 39.03 | 23.15 | 5.53 | 5.64 | 0.09 | 0.03 | 2.55 | 17.64 | 31.48 |
| | 20 | 37.41 | 26.35 | 5.39 | 5.41 | 0.08 | 0.03 | 2.44 | 17.59 | 30.94 |
| SSA + fly ash | 5 | 44.46 | 12.14 | 7.62 | 7.29 | 1.42 | 1.11 | 3.75 | 19.61 | 40.80 |
| | 10 | 44.06 | 12.62 | 8.36 | 8.03 | 2.61 | 2.08 | 4.50 | 20.46 | 46.04 |
| | 15 | 43.70 | 13.05 | 9.04 | 8.69 | 3.70 | 2.97 | 5.18 | 21.24 | 50.82 |
| | 20 | 43.37 | 13.45 | 9.66 | 9.31 | 4.70 | 3.79 | 5.80 | 21.95 | 55.21 |

[a] Summation of $Fe_2O_3$, CaO, MgO, $Na_2O$, $K_2O$ and $P_2O_5$.
[b] Sewage sludge ash from Minsheng STP, in Taiwan.

from 1050 to 1100 °C, with 10 °C increments, for the time span of 10, 20 and 30 min, respectively. The variation of bulk density was measured to benchmark the bloating temperature range. The compact SSA pellets with composition modification were heated at temperatures 1060, 1080 and 1100 °C, respectively, for 20 min. Different engineering properties of the aggregates were then tested after sintering.

### 2.3. Analyses

The characterization of the SSA or ALA was performed according to the following chemical and physical measurement methods:

| | |
|---|---|
| Physical and chemical measurement methods: | |
| Heavy metal content | USEPA SW 846-3050b |
| Speciation | XRD (X-ray diffraction) |
| Chemical composition | ICP-AES (Inductively coupled plasma optical emission spectroscopy) |
| TCLP tests | USEPA SW846-1311 |
| Mechanical tests: | |
| Bulk density | ASTM C127-88 |
| Moisture adsorption | ASTM C556 |
| Porosity | MIP (Mercury intrusion porosimetry) |
| Unconfined compressive strength | NIEA R206.20T (Taiwan) |
| Micro-structural changes | SEM (Scanning electron microscope) |
| Swelling rate | $= \frac{V_1 - V_0}{V_0} \times 100$, $V_0$: Volume before sintering ($cm^3$), $V_1$: Volume after sintering ($cm^3$) |

## 3. Results and discussion

### 3.1. Bloating behavior of SSA

Recycling of sewage sludge ash (SSA) as artificial lightweight aggregates involves mechanisms of bloating and sintering in the heat treatment process. Especially, the thermal bloating effect is a critical factor to be considered when the light-weighting feature is a major consideration. In general, two essential physical–chemical changes in ash at elevated temperature need to exist simultaneously for gaseous bubbles to be generated and trapped inside the pyro-plastic mass, including the development of a glassy phase and evolution of gases from the dissociation of mineral components. The most observed gas-producing reactions of incinerated sludge ash at elevated temperature are:

- Oxidation of sulfide substances at 400 °C to produce $SO_2$.
- Dissociation and evaporation of crystallized water in clay at 600 °C to produce water vapor.
- Oxidation of carbon containing substances at 700 °C to produce $CO_2$.
- Release of $O_2$ from $Fe_2O_3$ at 1000–1100 °C.

The density variation of the resulted SSA caused by gaseous bubbles was examined by changing the heating temperatures and time span. Since the proportion of $SiO_2$, $Al_2O_3$ and flux in SSA was similar to that of the ratio of natural clay to shale, it

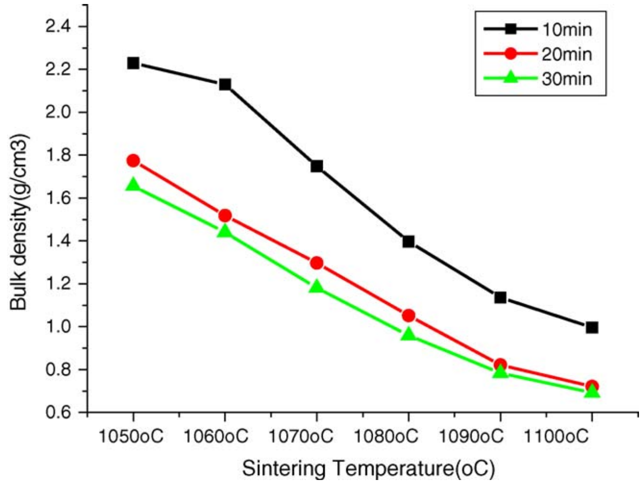



Fig. 2. The bulk density of SSA aggregates at various heating temperatures and heating times.

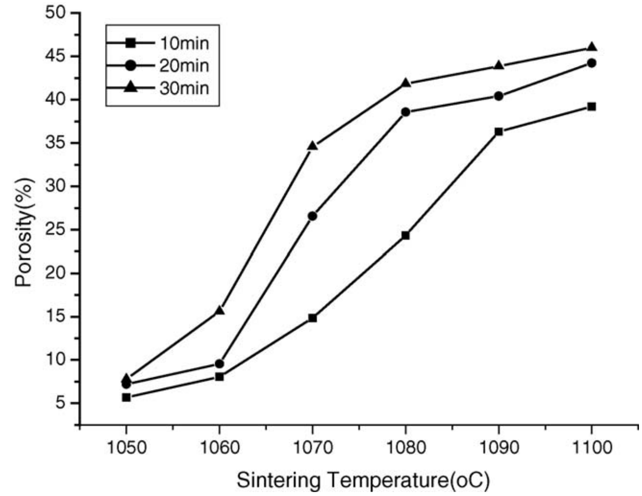



Fig. 3. The porosity of SSA aggregates at various heating temperatures and heating times.

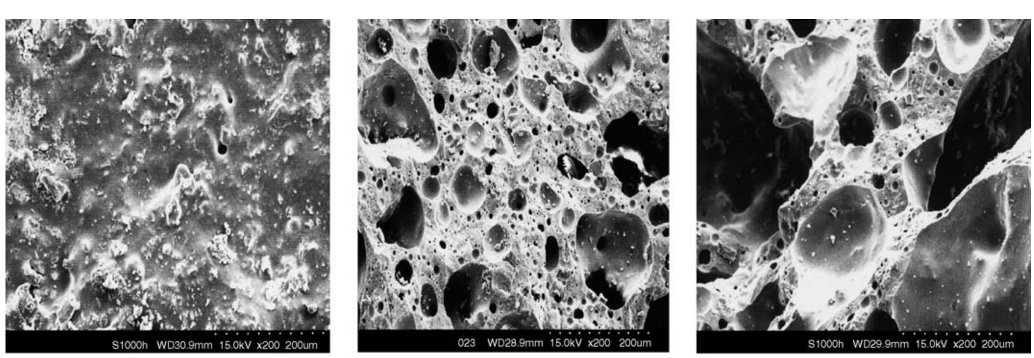
has the similar sintering and bloating capabilities during high temperature heating. Fig. 2 shows that the SSA particles started to sinter at 1050 °C, and with the increase in temperature, the surface of the SSA started to melt into glass phase and bloat. The density decreased with the raise of temperature, which fell between 0.69 and 2.23 g/cm$^3$. When sintering below 1100 °C, the longer time span resulted in lighter aggregates. The density for sintering at the 20–30 min range was observed to be significantly lower than that of sintering for 10 min, to be as low as 0.69–0.72 g/cm$^3$. Fig. 2 also shows the boating effect at various heating times. The density on the 10 min curve was higher than that of the 20 and 30 min curves. This indicates the overall bloating effect would require 20 min to come to a relative thermo stable state. The reduction of density on the 30 min curve was not significantly different from that of the 20 min curve, meaning the overall thermal reaction had been finished after 20 min heating to reach the desired density of the aggregate.

The trend of porosity variation based on the heating temperature variation indicates that the increase of sintering temperature resulted in the formation of a viscous flow, in turn reduced the pore volume, and therefore decreases the porosity of the pellets.

Fig. 3 shows a positive correlation between the degree of porosity and the bloating effect, by increasing the temperature when heating it for a 10 min duration, the porosity could be increased dramatically from 5.68 to 39%. Prolonging the heating time could also increase the degree of porosity. The SEM observation also shows that only the aggregate produced at 1050 °C does not have any signs of bloating. For sintering temperatures above 1060 °C, vigorous formation of apertures and pathways to the surface indicates the ending of bloating effect occurred, resulting in the decrease of density likewise.

### *3.2. Micro-structural variation of SSA aggregates by SEM*

Fig. 4 shows the surface of ALA after heating at 1050, 1080 and 1100 °C, respectively, for 20 min under SEM. At 1050 °C, the surface of ALA appeared to be in the glass phase, with few openings caused by the penetration of bubbles. When the temperature was raised to 1080 °C, the bubbles started to burst and form apertures with average diameters of 20–150 μm. Such bloating effects cause the lightweight property of the aggregates. At 1100 °C, more bubbles generated and busted more vigorously, which marked the surface of the ALA with prominent apertures (>70 μm).




1,050°C-20min(x200) 1,080°C-20min(x200) 1,100°C-20min(x200)

Fig. 4. Difference sintering temperatures vs. bloating effect by SEM.

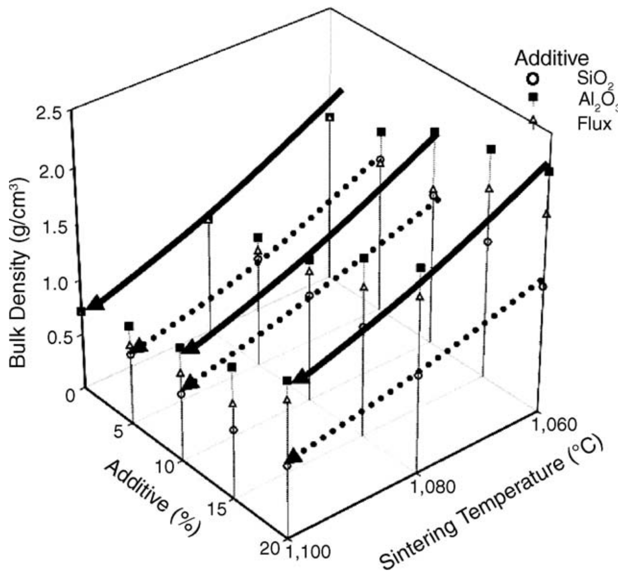



Fig. 5. The bulk density vs. different additive ratio for SSA as modified ash pellets.

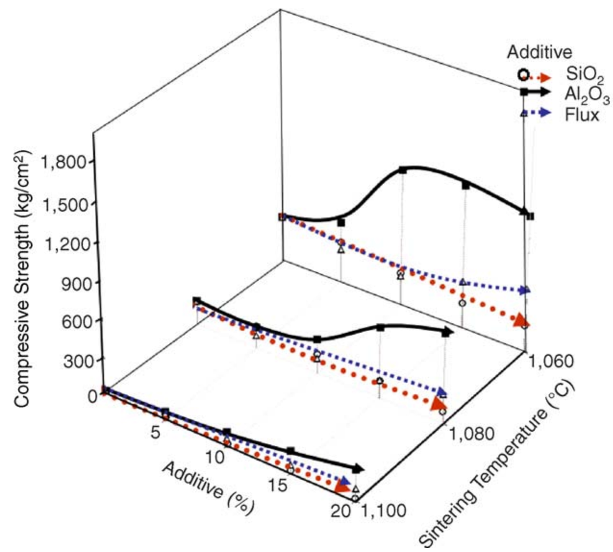



Fig. 6. The compressive strength vs. different additive ratio for SSA as modified ash pellets.

### 3.3. Bloating as additive variation of $SiO_2$/$Al_2O_3$/flux and heating temperature

We have shown that any composition modification of SSA by the addition of cullet powder, aluminum oxide or fly ash of any amount within 5–20% in weight resulted in bloating effects. The major factors can be discussed as the following.

#### 3.3.1. Bulk density

Fig. 5 shows the density changes of ALA by the addition of various quantities of additives. The density dropped significantly with the increase of addition of cullet powder (main composition is $SiO_2$), which was caused by the increase of low melting point additives. The more sticky glassy materials formed at the surface, the more gas would be wrapped up, which made the aggregates lighter. The strength of the aggregates can be increased by adding $Al_2O_3$, which has a higher melting point at around 2030 °C. However, the rate of density increase would slow down, especially when the adding of $Al_2O_3$ exceeded 15%. The density was 1.05 g/cm$^3$ with 5% addition and increase to 1.75 g/cm$^3$ with 15% addition at the fixed temperature of 1100 °C.

Overall, the density of aggregates from testing materials with high sintering temperature was lower than that with low sintering temperatures. Increasing the cullet powder to the aggregates can decrease the density to below 0.64 g/cm$^3$. This change in density was more significant when the temperature was increased. Moreover, the density changes by adding fly ashes and $Al_2O_3$ were quite similar to that of adding cullet powder.

#### 3.3.2. Compressive strength

The measurement of the compressive strength of the ALA pellets can be extrapolated to assess the strength characteristics of the resultant concretes. In general, the compressive strength of SSA aggregates was affected by a number of inter-related factors, such as the porosity, the pore size and distribution, the mineral species in the SSA, speciation variation during heating, densification effects by sintering, bloating of the SSA, and even pellet surface cracking or fracturing by thermo-stress.

Compressive resistance of the ALA is usually considered to be an important index for on-site estimation of the strength of the basic structure. Fig. 6 showed the decrease of compressive resistance with the addition of $SiO_2$. The more prominent bloating took place, the more porous the ALA would be. Although the strength of the ALA was consequently decreased, the relative percentage of the decrease is not significant. The compressive resistance increased when adding more $Al_2O_3$, which could be observed from the figures obtained by heating at 1060 and 1080 °C by adding more than 10% of $Al_2O_3$. The compressive resistance was directly proportional to the density of the ALA produced, which meant the lower the density, the lower the compressive resistance, and vice versa.

#### 3.3.3. Swelling rate

Fig. 7 shows that the addition of $SiO_2$ had obvious effect on the swelling rate of ALA. The swelling rate increases as the sintering temperature increases. On the contrary, the swelling rate decreases with increasing temperature when $Al_2O_3$ and fly ash was added. However, the bloating of ALA did not have a positive effect when adding either component.

#### 3.3.4. Water adsorption

The water absorption of aggregates, commonly used as an index in finding the extent of sintering, was determined by measuring the apparent increase in weight of a dried sample after its immersion in water for 24 h (defined as 24-h adsorption) [7].

The water adsorption property of ALA as showed on Fig. 8, measured by the 24-h water adsorption test, is another important characteristic for the application to construction work. Results show that adsorption rate is increased by incrementally adding cullet powder and fly ash. This is due to an increase in porosity

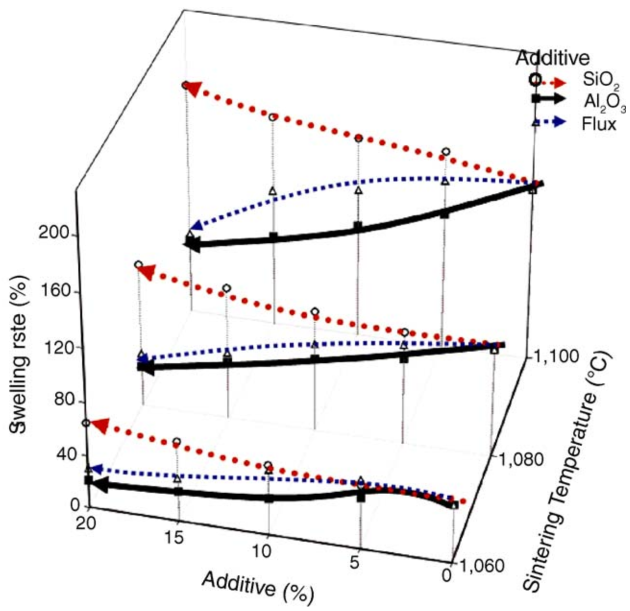



Fig. 7. Swelling rate vs. different additive ratio for SSA as modified ash pellets.

of the aggregates and the ALA became lighter weighted. Meanwhile, the water adsorption ability increased as well. However, the adsorption rate could still be controlled at below 5.84% of ALA. The effect of water adsorption ability by adding $Al_2O_3$ was less prominent in comparison with that by adding cullet powder or fly ash (Fig. 8).

### 3.4. Bloating ranges of SSA in comparison with clay

Our studies further discover that by adjusting the ratio of $SiO_2$–$Al_2O_3$–flux, the temperature and time required for sintering temperature and time could be reduced, and also change the optimal range of bloating effect.

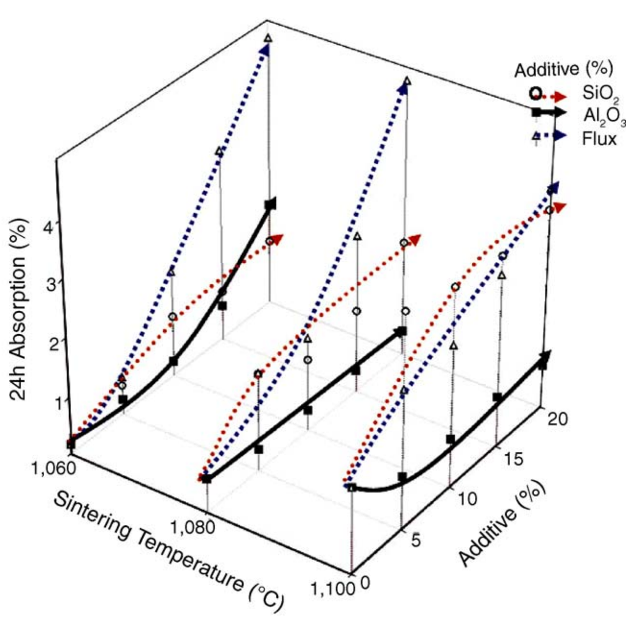



Fig. 8. The water adsorption rate vs. different additive ratio for SSA as modified ash pellets.

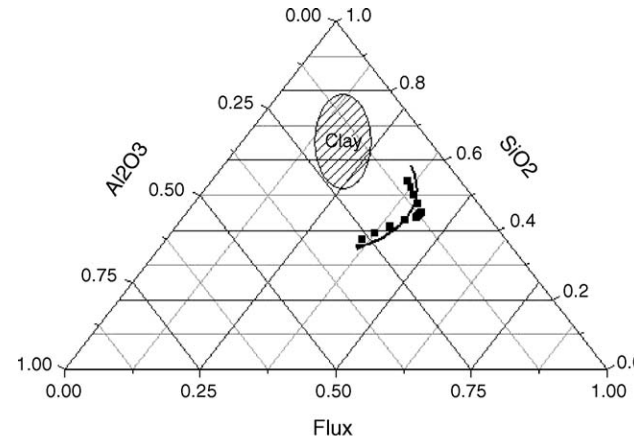



Fig. 9. Bloating effect area of $SiO_2$–$Al_2O_3$–flux.

Jiang and Zen's [16] study indicated the main composition of nature clay are quartz (>67%), $Al_2O_3$ (<17.1%) and flux (<9.68%) with a trace amount of $P_2O_5$ (less than 0.02%). Therefore, the main composition of the SSA used in this study (listed in Table 1) is far different from nature clay. The flux used in all tests of this study had been controlled between 29.21 and 55.21%. Usually, this high flux content will lower the temperature for sintering and change the position of the bloating effect domain area in the $SiO_2$–$Al_2O_3$–flux diagram. Fig. 9 shows the bloating effect in relation with the ratio of $SiO_2$–$Al_2O_3$–flux of natural clay (operated at 1000–1300 °C), which was used to compare the results of our studies. The boating conditions for SSA was obviously different from the nature clay, the ratio of $SiO_2$ could be reduced to 35–44%, the ratio for $Al_2O_3$ was similar to clay at around 10–26%, and the ratio of flux needed to be around 36–44%, that was much higher than the 8–28% for the nature clay. Overall, the bloating effect was shifted to the lower right of the chart for SSA in Fig. 9.

## 4. Conclusions

The results of this study showed that the quantity of secondary sludge ash is an important factor governing the bloating temperature of the ALA. The SSA bloated at temperatures ranging between 1160–1100 °C. Sludge ash compositions modified by adding of cullet powder, aluminum oxide ($Al_2O_3$), and MSW fly ash at ratios ranging between 0–20% by weight showed that MSA itself is generally a good thermal-bloating material for producing lightweight aggregates. Any modification by $Al_2O_3$, or MSW fly ash tends to increase the bulk density of heat-treated sludge ash pellets. However, modification of glass phrase component of the ash by cullet powder enhanced the bloating for SSA, indicating that impurities and amorphousness of the cullet powder might contribute to the bloating effect. The co-treatment of sludge ash with cullet powder is therefore a favorable option for the production of lightweight aggregates from sludge ash.

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