Lightweight Ceramsite based on sewage sludge made by Muffle furnace

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Abstract: It took SS (sewage sludge), PFA (pulverized fuel ash) and BS (building spoil) as raw materials to carry out experiments. An orthogonal experimental design was used to arrange experiments on the mixture ratio and processing parameters to find out a better ratio of green pellets and the right parameters of firing process. In accordance with the right ratio and processing parameters, experiments were carried out by MF (Muffle furnace) to produce lightweight aggregates. The characteristics of products such as 1h water absorption rate, packing density, particle strength were tested. According to the results of orthogonal experiment and range analysis, the optimized parameters suitable for different product requirements were selected. The repeatability of the optimized parameters is proved by the repeated experiments of the same batch, and the corresponding product performance is stable.

1. Introduction
In recent years, with the improvement of China's sewage treatment capacity, more and more urban sewage treatment plants have been established. With the improvement of sewage treatment rate, the output of sewage sludge is increasing day by day. Located in the middle and upper reaches of the Yangtze River, Chongqing is the political, economic and cultural center of the Three Gorges Reservoir. According to the 2015 Chongqing environmental quality bulletin learned: the city's sewage treatment scale was 3 million 780 thousand tons per-day. According to incomplete statistics, the annual sewage sludge (moisture content 80%) production in Chongqing was more than 1 million tons. How to properly dispose of sewage sludge has been urgent.

Generally, conventional sludge disposal technologies include landfill, incineration, agricultural and sea dumping. The high cost of the traditional way of disposal, easy to create secondary pollution, not a sustainable environmental protection strategy. Therefore, the utilization of sludge will be the ultimate way of sludge disposal. It has been reported that the domestic and foreign scholars have made the research on the manufacture of ceramsite by using sewage sludge as the main raw material. Chongqing is located in hilly areas; building spoil (mineral composition similar to clay) can be used as the auxiliary material for the production of sludge ceramsite. The output of fly ash from coal-fired power plant (PFA) is stable and easy to obtain, and it can be used as the skeleton material of ceramic production. Therefore, combined with the actual situation in Chongqing, we choose SS, PFA, BS as raw materials to manufacturing ceramsite.

2. Experiment
2.1 Materials and characters
It took SS, PFA and BS as raw materials to carry out experiments. Especially, SS came from a municipal sewage treatment plant of Chongqing city. PFA came from a coal power plant of
Chongqing city, and BS came from a stone factory. The characteristics of raw materials were shown in Table 1.

| Table 1. Chemical analysis of raw materials (mass percentage) |
|------------------------------------------------------------|
| Category | SiO₂ | Fe₂O₃ | Al₂O₃ | CaO | MgO | Na₂O | K₂O | SO₃ |
| SS | 32.1 | 9.23 | 11.2 | 3.68 | 2.2 | 0.75 | 1.7 | 2.03 |
| PFA(II) | 44.91 | 16.1 | 24.26 | 3.82 | 0.87 | 0.85 | 1.46 | - |
| BS | 59.84 | 4.59 | 12.1 | 1.33 | 1.46 | 1.7 | 1.97 | 0.23 |

2.2 Experimental equipment
A MF was the key experimental equipment with model SX12-2.5. The heating range of the equipment is from room temperature to 1300 degrees Celsius. The auxiliary equipment included mixer, granulating equipment, constant temperature drying box and particle strength tester.

2.3 Experimental process
The raw materials aforementioned were all sieved to the particle diameter ranging from 0.10 mm to 0.20 mm. It took the selected raw materials to make pellets with some certain mixture ratio. Experimentally, it needs moisture content of mixture to be about 20% to make green pellets keep in spherical shape. It also needs to dry green pellets before the sintered process to avoid aggregates breaking due to the extravasations of free water in the pellets when firing them. After the pretreatment process, it was time for feeding pellets into the MF for preheating at some certain temperature for several minutes. When the preheat process finished, it should set a certain temperature of the MF for sintering the pretreated pellets. When the MF reached the presetting temperature, the sintering process finished. But, it did not mean the pellets turned out to be ceramsite at that moment. Usually, it needs to hold the final temperature a few minutes to keep pellets burning completely for bloating them. Finally, it’s time for turning off the MF to let aggregates cool naturally. Then, it could carry out some physicochemical characteristic tests of the ceramsite.

2.4 Experimental method
In accordance with a theory proposed by Rely and Wilson that suitable chemical composition of clay for sintering ceramsite, some researchers had drawn a conclusion that chemical composition of raw materials on burning high-quality ceramsite, the key chemical composition of raw materials should follow this mass percentage SiO₂ 48%∼79%, Al₂O₃ 8%∼25%, Σ(CaO+MgO+Fe₂O₃+ Na₂O+K₂O) 8%∼24%. It could help us to establish raw material mixture ratio. According to the results of chemical composition analysis of raw materials, the chemical composition of BS could meet the elemental composition range on producing high-quality ceramsite. Experimentally, it tried producing ceramsite just by BS through the MF. But, the results showed that products made by BS appeared light gray color, surface cracks and packing density was greater than 1.2g/cm³. The experimental results showed it could not produce high quality ceramsite by BS separately. The key chemical composition of PFA and SS was not within the scope of making ceramics. Experiments on sintering ceramsite by PFA and SS alone were carried out. The results showed it could not make lightweight ceramsite by PFA and SS alone.

An orthogonal experimental design method was used to arrange experiments. According to the preliminary experiments, it chose the mixture ratio; preheat temperature, sintered temperature and retention time were key factors of the orthogonal experiment table. In accordance with the DPSS statistic software, an orthogonal table such as L₂₅ (₅)⁶ which include five levels of six factors and twenty-five process experimental table was selected to arrange experiments. The experimental factor and levels were detailed in table 2.
Table 2. Level of experimental factors

| Level of factor       | 1   | 2   | 3   | 4   | 5   |
|-----------------------|-----|-----|-----|-----|-----|
| Mass percentage of SS-A(/%) | 10  | 15  | 20  | 25  | 30  |
| Mass percentage of PFA-B(/%) | 30  | 25  | 20  | 15  | 10  |
| Preheat temperature-C/(C °)  | 200 | 250 | 300 | 350 | 400 |
| Sintered temperature-D/(C °) | 1100| 1120| 1140| 1160| 1180|
| retention time-E/(min)  | 1   | 2   | 3   | 4   | 5   |

2.5 Experimental results

The key characters of ceramsite including particle strength, packing density and one hour water absorption rate were selected to identify quality of ceramsite. The experimental process and results were shown in table 3.

Table 3. Experimental process and results

| Num. | A%/ | B%/ | C/° | D/° | E/min | Packing density/kg·m³ | Particle strength/N | 1h water absorption/% |
|------|-----|-----|-----|-----|-------|------------------------|---------------------|-----------------------|
| 1    | 10  | 30  | 200 | 1100| 1     | 731                    | 520                 | 8.12                  |
| 2    | 10  | 25  | 250 | 1120| 2     | 722                    | 741                 | 8.31                  |
| 3    | 10  | 20  | 300 | 1140| 3     | 778                    | 467                 | 13.20                 |
| 4    | 10  | 15  | 350 | 1160| 4     | 713                    | 740                 | 6.90                  |
| 5    | 10  | 10  | 400 | 1180| 5     | 650                    | 747                 | 8.95                  |
| 6    | 15  | 30  | 250 | 1140| 4     | 665                    | 732                 | 9.51                  |
| 7    | 15  | 25  | 300 | 1160| 5     | 666                    | 640                 | 8.13                  |
| 8    | 15  | 20  | 350 | 1180| 1     | 630                    | 678                 | 9.99                  |
| 9    | 15  | 15  | 400 | 1100| 2     | 688                    | 622                 | 9.96                  |
| 10   | 15  | 10  | 200 | 1120| 3     | 679                    | 656                 | 11.33                 |
| 11   | 20  | 30  | 300 | 1180| 2     | 634                    | 608                 | 12.00                 |
| 12   | 20  | 25  | 350 | 1100| 3     | 656                    | 542                 | 11.88                 |
| 13   | 20  | 20  | 400 | 1120| 4     | 623                    | 666                 | 11.08                 |
| 14   | 20  | 15  | 200 | 1140| 5     | 600                    | 712                 | 9.89                  |
| 15   | 20  | 10  | 250 | 1160| 1     | 620                    | 618                 | 10.02                 |
| 16   | 25  | 30  | 350 | 1120| 5     | 611                    | 602                 | 11.38                 |
| 17   | 25  | 25  | 400 | 1140| 1     | 600                    | 636                 | 11.33                 |
| 18   | 25  | 20  | 200 | 1160| 2     | 620                    | 701                 | 12.36                 |
| 19   | 25  | 15  | 250 | 1180| 3     | 558                    | 603                 | 12.11                 |
| 20   | 25  | 10  | 300 | 1100| 4     | 617                    | 555                 | 12.79                 |
| 21   | 30  | 30  | 400 | 1160| 3     | 589                    | 503                 | 12.98                 |
| 22   | 30  | 25  | 200 | 1180| 4     | 576                    | 413                 | 14.33                 |
| 23   | 30  | 20  | 250 | 1100| 5     | 608                    | 489                 | 14.13                 |
| 24   | 30  | 15  | 300 | 1120| 1     | 578                    | 630                 | 13.08                 |
| 25   | 30  | 10  | 350 | 1140| 2     | 540                    | 641                 | 12.22                 |

3. Discussion

3.1 Statistical analysis

The range analysis of key factors affecting the particle strength, packing density and 1h water absorption rate of ceramsite was shown in table 4.

Table 4. Range analysis of key factors

| Num. | Packing density /kg·m³ | Particle strength /N | 1h water absorption/% |
|------|------------------------|----------------------|-----------------------|
| A    | B          | C      | D          | E | A        | B        | C        | D          | E | A        | B        | C        | D          | E |
| K1   | 719        | 624    | 645        | 645 | 639 | 643 | 620 | 617        | 617 | 618 | 9.10 | 11.30 | 10.90 | 10.90 | 11.03 |
| k2   | 666        | 628    | 641        | 638 | 634 | 666 | 623 | 621        | 622 | 622 | 9.78 | 11.15 | 11.03 | 11.11 | 11.16 |
| k3   | 627        | 637    | 635        | 630 | 642 | 629 | 615 | 616        | 617 | 633 | 10.97 | 11.15 | 11.24 | 11.28 | 10.83 |
| k4   | 601        | 643    | 624        | 634 | 631 | 619 | 627 | 624        | 642 | 630 | 11.99 | 10.88 | 11.20 | 10.70 | 10.89 |
It is well known that typical characters of ceramsite were hard, lightweight and low water absorption rate. According to the range analysis of key factors, it made it clear that the project A5B1C4D5E5 was suitable for achieving lower packing density; project A2B5C5D4E3 was suitable for acquiring higher particle strength and project A1B5C1D4E3 fit for getting lower water absorption rate. How to determine the best baking parameters depends on the performance requirements of ceramsite products.

Meanwhile, the repeatability of the optimal scheme needs further study. In this paper, further experiments according to the optimized projects were carried out to verify combined effects about the key factors on the products. Under the same experimental conditions, the experimental scheme of each group was repeated 3 times.

### 3.2 Experimental repeatability

#### 3.2.1 Project one (A5B1C4D5E5)

Experiments with project one was carried out in MF. Through these experiments, several ceramsites were randomly selected to take scanning electron micrographs. As fig.1 (I) showed, the internal structure of cross section and appearance reveals the reason of ceramsite lightweight. It’s easy to find out the porous structure is the main cause leading to ceramsite lightweight. The porous structure is an important characteristic of ceramsite products. In these pore structures, a part of micropores are interconnected. The penetration of micropore is the main factor that results in the increase of water absorption rate. According to fig.1(A), we can see some larger black holes in the micrograph. This is due to the loss of the larger particles during the firing process.

It is generally believed that the gas produced in the process of sintering ceramsite could make the porous structure more suitable for the operation of sintering ceramsite. The honeycomb structure of uniform distribution is due to the composition of raw materials to produce gas overflow generated at high temperature. The gas composition of the raw material mainly includes the evaporation of the water in the pellet, the gas produced by the combustion of carbon particles and the decomposition of salts. The ceramsite appearance was brown. Some ceramsite surface is similar to enamel material. The surface touch feeling was not smooth, some products appeared chapped cracks. The particle strength, packing density and 1h water absorption rate of project one were tested. The results were showed in table 5.

**Table 5.** Range analysis of key factors

| Num. | Packing density | Particle strength | 1h water absorption |
|------|-----------------|-------------------|---------------------|
| 1    | 620/(kg·m⁻³)   | 566/(N)           | 11.74%(%)           |
| 2    | 611/(kg·m⁻³)   | 601/(N)           | 9.9%(%)             |
| 3    | 628/(kg·m⁻³)   | 593/(N)           | 10.64%(%)           |
| Relative change index* | 2.74% | 5.97% | 16.77% |

*The formula for calculating the relative change index: range/average value.

The table 5 showed: (i) the relative change index of packing density is very small; (ii) the relative change index of 1h water absorption rate is relatively high. The experimental results show that the repeatability of the experimental results is normal, and the control of bulk density is more successful.

#### 3.2.2 Project two (A2B5C5D4E3)

Same as the previous experimental method, experiments with project two was carried out in MF. As shown in fig.1 (II), the internal structure and appearance of ceramsite reveals the reason of ceramsite high strength. The surface of the shell is brown ceramic support structure of ceramsite high particle strength. Acicular mullite is widely distributed in the glass phase, the glass phase plays a supporting, strengthening and enhancing the skeleton effect, and the ceramsite has high strength.

The particle strength, packing density and 1h water absorption rate of project two were tested. The results were showed in table 6.
Table 6. Experimental results

| Num. | Packing density | Particle strength | 1h water absorption |
|------|----------------|-------------------|---------------------|
| 1    | 651/(kg·m⁻³)   | 623/(N)           | 12.1/(%)            |
| 2    | 667/(kg·m⁻³)   | 655/(N)           | 10.3/(%)            |
| 3    | 639/(kg·m⁻³)   | 672/(N)           | 11.0/(%)            |
|      | Relative change index |         |                     |
|      | 4.29%          | 7.54%            | 16.07%              |

The table 6 showed: (i) the relative change index of particle strength is small; (ii) the relative change index of 1h water absorption rate is relatively high. The variation of particle strength is mainly due to the randomness of test samples. The experimental results showed that the repeatability of the experimental results is normal, and the control of particle strength is good.

3.2.3 Project three (A1B5C1D4E3). Same as the previous experimental method, experiments with project three was carried out in MF. As shown in fig.1 (III), the dense outer enamel structure of ceramsite reveals the reason of lower water absorption. Quartz and silicate minerals (kyanite) have a dense structure, widely distributed in the ceramic surface, which is a very important factor of ceramsite low water absorption rate [13].

![SEM and appearance of ceramsite](image)

Figure 1. SEM and appearance of ceramsite

The particle strength, packing density and 1h water absorption rate of project three were tested. The results were showed in table 7.

Table 7. Experimental results

| Num. | Packing density | Particle strength | 1h water absorption |
|------|----------------|-------------------|---------------------|
| 1    | 640/(kg·m⁻³)   | 590/(N)           | 8.9/(%)             |
| 2    | 626/(kg·m⁻³)   | 633/(N)           | 9.4/(%)             |
| 3    | 661/(kg·m⁻³)   | 650/(N)           | 9.0/(%)             |
|      | Relative change index |         |                     |
|      | 5.45%          | 9.61%            | 5.49%              |

The table 7 showed: (i) the relative change index of 1h water absorption rate is small; (ii) the relative change index of particle strength is relatively high. The variation of particle strength is mainly due to the randomness of selected samples. The experimental results showed that the repeatability of the experimental results is normal, and the control of particle strength is well.

4. Conclusion

The main conclusions of this study are as follows:

(i). In order to achieve a low packing density, the experimental parameters should meet: mass percentage SS/PFA/BS=30%/30%/40%, preheat temperature 350°C, sintered temperature 1180°C and retention time 5 min.

(ii). In order to obtain high strength ceramsite, the experimental parameters should meet: mass percentage SS/PFA/BS=15%/10%/75%, preheat temperature 400°C, sintered temperature 1160°C and retention time 3 min.
In order to obtain low water absorption ceramsite, the experimental parameters should meet: mass percentage SS/PFA/BS=10%/10%/80%, the preheat temperature 200 °C, sintered temperature 1160 °C and retention time 3 min.

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