Study on Preparation of Ceramsite from Shale Slag and Its Application

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Abstract. This paper studies the proportion of raw materials and process system for firing shale slag ceramsite. The raw materials and raw material ratio of the shale slag ceramsite were determined by the composition analysis of the shale slag; Through basic experiments, it is concluded that when the proportion of raw materials is shale slag: coal gangue: fly ash: expansion agent = 65 : 16 : 9 : 10. Through optimization analysis, when the tube pressure strength is used as the evaluation index, the optimal plan A2B1C2, the bulk density as the evaluation index, the optimal plan A1B3C2, and the water absorption rate as the evaluation index, the optimal plan A3B1C3. Conclusion: The best sintering system for shale slag granule firing is preheating temperature 50°C, heating time 20min, holding time 15min, heating to 1100°C, heating for 40min, and finally holding for 20min. Through the optimization analysis of the analysis and analysis of variance results, it is known that the firing temperature has the most significant effect on the ceramsite performance.

1. Introduction
With the deepening of people's understanding of ceramsite, ceramsite has become more and more widely used as artificial aggregate. Shale slag is a waste residue from shale retorting or combustion. In China, especially in Liaoning, a large amount of shale slag is discharged every year, which occupies land and pollutes the environment. The traditional artificial light aggregates mostly use natural resources such as clay as the main raw materials, and a large amount of mining will destroy the ecological environment. In this test, the shale slag is ground, a certain auxiliary material is added, and
the spherical material is prepared according to the proportion of the designed raw materials, and then fired into ceramsite to study the influence of the sintering system on the performance of the ceramsite.

2. experiment

2.1. Experimental raw materials

The shale slag is from shale slag from the industrial waste shale slag (semi-coke) of Beipiao City, Liaoning Province. The chemical composition is shown in Table 1; the fly ash is used in Shenhai Thermal Power Plant, and the chemical composition is shown in Table 1.

| Table 1 Chemical composition analysis / % |
|------------------------------------------|
| Raw material | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO | K₂O | Na₂O |
| Coal gangue | 51.17 | 30.73 | 9.45 | 1.12 | 1.60 | 1.10 | 0.70 |
| Shale slag | 54.54 | 14.48 | 6.27 | 8.95 | 3.80 | 3.39 | 1.00 |
| Fly ash | 53.47 | 30.48 | 4.73 | 1.12 | 1.40 | 2.52 | 0.90 |
| Quartz sand | 96.89 | 0.61 | 0.87 | 0.19 | 0.07 | 0.08 | 0.04 |

2.2. Experimental methods

The shale slag and coal gangue are ground by a ball mill in the laboratory, and the grinding time is about two hours. Then the ground raw materials are taken out, and the shale residue, coal gangue and fly ash powder are passed through a 200 mesh sieve. Screening, and then selecting the auxiliary materials to be mixed according to the established ratio. During the mixing process, a small amount of water is added (the amount of water is 35%~38% of the mixture) until the mixture becomes sticky and the ball is used. The disc is made into a spherical object of 10 mm to 15 mm. After granulation is completed, it is naturally dried in the curing room for 8 hours, then placed in a blast drying oven and dried at 105°C for 6 hours. Then, it is taken out from the blast drying oven, placed in a muffle furnace, and calcined according to a predetermined sintering system. After the sintering is completed, it is naturally cooled to room temperature in a muffle furnace, and the sintered ceramsite is taken out. The effects of raw material ratio, preheating temperature, holding time and heating rate on the properties of ceramsite were studied.

2.3. Test plan

The proportion of various raw materials in the experiment is shown in Table 2.

| Table 2 Experimental ratio scheme/% |
|------------------------------------|
| Shale slag | Coal gangue | Quartz sand | Fly ash | Toner | Pulverized coal | Sodium manganese dioxide | Sodium carbonate | Sodium bicarbonate | Glass powder |
| 1 | 57 | 38 | - | - | 5 | - | - | - | - |
Results and Discussion

3.1 Optimization scheme of sintering process system

Orthogonal experimental protocol: In addition to the influence of the type of foaming agent, the performance of shale slag ceramsite is also affected by factors such as the amount of blowing agent, firing temperature and sintering time. Therefore, this orthogonal test adopts three-factor and three-level orthogonal test. The foaming agent dosage is selected at three levels of 3%, 5% and 7%. The firing temperature is selected from three levels of 1050°C, 1075°C and 1100°C. The orthogonal experiment was carried out at three levels of 15 min, 20 min and 25 min. The orthogonal table is shown in Table 4. The influence factors of fired shale slag ceramsite were studied by range analysis and analysis of variance.

(1) Evaluation index: 24h water absorption, bulk density, compressive strength.
(2) Influencing factors: A: firing temperature; B: holding time; C: foaming agent dosage
(3) Because this experiment uses the method of blending in the foaming agent, that is, the foaming agent is calculated into the mass ratio, so the method of adding the glass powder can be used to change the blending amount of the foaming agent while ensuring the quality of the remaining raw materials. The proportion remains unchanged.

Table 3 Orthogonal experimental scheme

| Level | Burning temperature / °C | Holding time / min | Foaming agent dosage / % |
|-------|--------------------------|--------------------|-------------------------|
| 1     | 1050                     | 15                 | 3                       |
| 2     | 1075                     | 20                 | 5                       |
| 3     | 1100                     | 25                 | 7                       |

Table 4 Orthogonal experimental data sheet

| Test number | A (factor) | B | C | Evaluation index |
|-------------|------------|---|---|------------------|
| 2           | 56         | 8 | 20| 13               |
| 3           | 56         | 8 | 20| 13               |
| 4           | 65         | 16 | 9 | 10               |
| 5           | 65         | 16 | 9 | 10               |
| 6           | 65         | 16 | 9 | 10               |
| 7           | 65         | 16 | 9 | 10               |
| 8           | 65         | 16 | 9 | 10               |
| 9           | 65         | 16 | 9 | 10               |
Range analysis: This table is based on a water absorption of ceramic as an evaluation index can be seen from Table Factors A very poor R maximum, is 12.24, indicating that the effect of firing temperature on the water absorption ratio of the maximum ceramsite. Under the influence of factor A, the average values of water absorption at three levels were 20.68 %, 13.58 %, and 8.44 %, respectively. Since the water absorption of ceramsite is as small as possible, the third level, i.e., the firing temperature is 1100°C. Time is optimal. Whereas the comparative holding time B and a foaming agent dosage C, both poor R were 4.29 and 9.93, so the dosage of the foaming agent C for the water absorption of the ceramic is greater than the influence of holding time B. In this two factors selected minimum level of water absorption, wherein the dosage of the foaming agent C corresponds to the first three levels of best, while the holding time B corresponds to the first is an optimal level.

| Source of error | SS     | f  | S    | F ratio | Significant |
|-----------------|--------|----|------|---------|-------------|
| A               | 226.776| 2  | 113.388 | 12.920  | * (significant) |
| B               | 27.537 | 2  | 13.769 | 1.569   | O (not significant) |
| C               | 157.868| 2  | 78.934 | 8.994   | O (not significant) |
| error           | 17.553 | 2  | 8.776 | 12.920>F0.1 (2,2) =9.00 |

Analysis of variance: It can be seen from the data table of variance analysis that the effect of firing temperature on water absorption is significant in the three test factors, and the amount of foaming agent and holding time are not significant. From the comparison of F ratio, it can be concluded Effect of water absorption of the order of three factors: a firing temperature A > dosage of the foaming agent
C > holding time B. Corresponds to the results of the range analysis. In summary, the optimal solution for ceramsite water absorption is A3B1C3

### Table 6 Variance experimental data sheet

| Test number | factor | Evaluation index | Bulk density / kg / m³ |
|-------------|--------|-----------------|------------------------|
|             | A      | B               | C                      |                          |
| 1           | 1      | 1               | 1                      | 740                     |
| 2           | 1      | 2               | 2                      | 790                     |
| 3           | 1      | 3               | 3                      | 630                     |
| 4           | 2      | 1               | 2                      | 810                     |
| 5           | 2      | 2               | 3                      | 900                     |
| 6           | 2      | 3               | 1                      | 700                     |
| 7           | 3      | 1               | 3                      | 600                     |
| 8           | 3      | 2               | 1                      | 700                     |
| 9           | 3      | 3               | 2                      | 170                     |
| K1          |        |                 |                        | 716.67                  |
| K2          |        |                 |                        | 713.33                  |
| K3          | 803.33 | 796.67          | 590                    | 6040                    |
| R           | 313.33 | 296.67          | 123.33                 |

This table is based on the bulk density of the ceramic as an evaluation index. From Table 6, factor A is very poor R maximum, is 313.33, indicating the effect of firing temperature on the ratio of the maximum bulk density of ceramic. Under the influence of factor A, the average values of the three levels of bulk density are 720 kg/m³, 803.33 kg/m³, 490 kg/m³, respectively, because the bulk density of ceramsite is as small as possible, so the third level is optimal at 1100°C. Whereas the comparative holding time B and foaming agent dosage C, both poor R are 296.67 and 123.33, the holding time B effect is greater than the bulk density of the foaming agent dosage ceramic C. Among the two factors, the lowest level of bulk density is selected, wherein the second level corresponding to the holding time B is optimal, and the blowing agent amount C corresponds to the first level is optimal. In summary, the order of influence on the bulk density of the three factors is: firing temperature A > holding time B > blowing agent dosage C; the optimal solution is A3B3C2.

### Table 7 Variance Analysis Data Sheet

| Source of error | SS     | f | S         | F ratio | Significant |
|-----------------|--------|---|-----------|---------|-------------|
| A               | 158022.222 | 2 | 79011.111 | 7.493   | O (not significant) |
| B               | 141355.556 | 2 | 70677.778 | 6.703   | O (not significant) |
| C               | 29,622.222  | 2 | 14811.111 | 1.405   | O (not significant) |
Analysis of variance: It can be seen from the data table of variance analysis that the influence of the three test factors on the bulk density is not significant, but from the comparison of the F ratio, the order of influence on the bulk density of the three factors can be obtained: the firing temperature $A >$ holding time $B >$ Volume foaming $C$.

In summary, the optimal solution for ceramsite bulk density is $A_3B_3C_2$.

### Table 8 Orthogonal experimental data sheet

| Test number | factor | Evaluation index Compressive strength / MPa |
|-------------|--------|-------------------------------------------|
|             | A      | B  | C   |                                      |
| 1           | 1      | 1  | 1   | 5.23                                   |
| 2           | 1      | 2  | 2   | 6.65                                   |
| 3           | 1      | 3  | 3   | 3.75                                   |
| 4           | 2      | 1  | 2   | 10.79                                  |
| 5           | 2      | 2  | 3   | 20.77                                  |
| 6           | 2      | 3  | 1   | 9.44                                   |
| 7           | 3      | 1  | 3   | 7.04                                   |
| 8           | 3      | 2  | 1   | 10.47                                  |
| 9           | 3      | 3  | 2   | 9.22                                   |
| K1          | 5.21   | 7.69 | 8.38 |
| K2          | 13.67  | 12.63 | 8.89 |
| K3          | 8.91   | 7.47 | 10.52 |
| R           | 8.46   | 5.16 | 2.14 |
| R           | 83.36  |     |     |

This table is a single grain of ceramic compressive strength as an evaluation index can be seen from Table Factors A very poor R maximum, is 8.46 , indicating that the effect of firing temperature on compressive strength ratio of the maximum Ceramisite . Under the influence of factor A , the average values of the three levels of compressive strength are 5.21 MPa , 13.67 MPa , 8.91 MPa , respectively. The higher the compressive strength of ceramsite, the better the ceramsite quality, so the second level is burnt. It is optimal when the temperature is $1100^\circ$C. Whereas the comparative holding time B and a foaming agent dosage C , both poor R were 5.16 and 2.14 , so the holding time B impact compressive strength greater than a foaming ceramic Volume C . Among these two factors, the highest level of compressive strength is selected, wherein the second level corresponding to the holding time B is optimal, and the blowing agent amount C corresponds to the third level. The level is optimal. In summary, the order of influence on the bulk density of the three factors is: firing temperature $A >$ holding time $B >$ blowing agent dosage $C$ ; the optimal solution is $A_2B_2C_3$.
Table 9 analysis of variance data

| Source of error | SS      | f | S       | F ratio | Significant       |
|-----------------|---------|---|---------|---------|-------------------|
| A               | 107.831 | 2 | 53.916  | 3.822   | O (not significant) |
| B               | 51.109  | 2 | 25.555  | 1.811   | O (not significant) |
| C               | 7.504   | 2 | 3.752   | 0.266   | O (not significant) |
| error           | 28.216  | 2 | 14.108  |         |                   |

Analysis of variance: It can be seen from the data table of variance analysis that the effects of the three test factors on the compressive strength of single grain are not significant, but the influence of the three factors on the compressive strength of single grain can be obtained from the comparison of F ratio. order: firing temperature A > holding time B > Volume foaming C . In summary, the optimal scheme for the single-grain compressive strength of ceramsite is A2B2C3 , but it can be seen from the analysis of variance that the data error of this group is too large and cannot be used as the main reference data.

4 conclusions

In this stage, six groups of orthogonal experiments were carried out to investigate the effects of firing temperature, holding time and foaming agent dosage on the performance of ceramsite. The water absorption and bulk density of a total of 18 groups of ceramsite were studied. The test of compressive strength finally analyzed the orthogonal experimental data and obtained the following results:

( 1 ) The experiment finally chose sodium carbonate as a blowing agent.
( 2 ) It is concluded by orthogonal experiment that among the three factors of firing temperature, holding time and foaming agent dosage, the most important influence on the performance of ceramsite is the firing temperature.
( 3 ) The optimum firing temperature is 1100°C, the optimum holding time is 15 min, and the blowing agent is added to 7%. At this time, the water absorption rate was 0.23%, the bulk density was 600 kg/m$^3$, and the compressive strength was 7.04 MPa.

references

[ 1 ] Deng Jiaping, Zhang Minghua, Li Zelin, et al. Research and process design of ultralight ceramsite fired from oil shale slag [J].
[ 2 ] Wang Shujuan, Jin Yujie et al. Study on physicochemical properties of oil shale semi-coke from different producing areas. Journal of Jilin Jianzhu University.
[ 3 ] Li Yunfei, Liu Ji, Deng Jiaping. Comprehensive utilization of oil shale ash in building materials. "Brick and Tile" No. 7 of 2018.
[ 4 ] Tan Liquan, Hu Xianghong, Yu Mei, Zhou Jianmin et al. Progress in the application of oil shale ash slag. Chemical engineer.
[ 5 ] Li Zhaofu, Zhao Xin et al. Feasibility study on smelting silicon-aluminum-iron alloy in Fushun shale waste residue. Recycling.
[ 6 ] Zhou Jianmin, Niu Xianchun et al. Source and comprehensive utilization technology of oil shale
ash . Journal of Guangdong Institute of Petrochemical Technology .
[ 7 ] Yang Shi . Discussion on laboratory test and small test operation of ceramsite raw material sample . Brick World .