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Preparation and Performance Optimization of Organosilicon Slag Exothermic Insulating Riser

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Abstract

The exothermic insulating riser played an important role in the solidification process of metal liquid for the improvement of casting quality. This paper focused on the use of organosilicon slag to replace part of the aluminum powder as an exothermic agent for the riser, to reduce production costs and turn waste into treasure. The experiments firstly studied the effect of organosilicon slag content on the combustion temperature and holding time and determined the components of the riser exothermic agent and organosilicon slag. On this basis, the effects of the content of Na_3AlF_6 flux and alkali phenolic resin binder on the combustion heating time and strength properties of the riser were studied. And the ratio of mixed oxidants was determined by single-factor orthogonal experiments to optimize the addition of three oxidants, Fe_3O_4, MnO_2, and KNO_3. Finally, the performance of the riser prepared after optimization was compared with that of the riser prepared with general aluminum powder. The results showed that with the mixture of 21% organosilicon slag and 14% aluminum powder as the exothermic agent, the highest combustion temperature of the prepared exothermic insulating riser was 1451℃ and the holding time was 193 s; the optimal content of Na_3AlF_6 flux was 4%, and the best addition alkali phenolic resin binder was 12%; the optimized mixing ratio of three oxidants was 12% for Fe_3O_4, 6% for MnO_2, and 6% for KNO_3. Under the optimized ratio, the maximum combustion temperature of the homemade riser was 52℃ and the heat preservation time was 14% longer compared with the conventional exothermic insulating riser with 25-35% aluminum powder.

Keywords: Exothermic insulating riser, Organosilicon slag, Orthogonal optimization

1. Introduction

In foundry practice, the exothermic insulating riser could increase the temperature of the metal liquid through its heat combustion and delay the solidification of the metal liquid, so that the efficiency of feeding could be increased during casting. Some existing studies had improved the performance or defects of the riser by modeling and computer simulation [1-3]. The change in the composition and ratio of the different materials of the riser also affected the casting effect most directly [4]. In addition, the size and structure of the riser, the thickness of the casing, and the location of the sprue would affect the shrinkage efficiency [5, 6]. However, the current exothermic insulating riser with aluminum thermite as the main heat-generating material had the disadvantages of difficult processing, high cost, and flammability, which limited the popularization and application of the risers [7-10]. In this paper, based on the previous research, we used organosilicon slag as a new heat-generating material to replace part of the aluminum powder to make the riser [4]. And the ratio of various materials in the riser was optimized.

The organosilicon slag discussed in this paper originates from the large amount of silicone slurry generated during the synthesis of silicone monomer (especially methylchlorosilane monomer) [11, 12]. Such slurries could pollute the environment if they were discharged directly. Currently, the main treatment method was to reduce slurry toxicity by hydrolysis, and some studies had been conducted to retain the added value of organosilicon slag residue through distillation and catalytic deposition for resource utilization [13, 14]. For the organosilicon slag obtained from separation treatment, there were fewer ways to resource utilization [15]. The use of organosilicon slag for making risers dealt with the problem of organosilicon solid waste and reduced the production cost of fever insulation risers at the same time.

This paper focused on the development of an exothermic insulating riser based on organosilicon slag. The combustion and thermal insulation performance of the riser were balanced by adjusting the content of the exothermic agent and the ratio of aluminum powder to organosilicon slag. In addition, the thermophysical properties such as combustion heating time and strength of the riser were analyzed when the heating agent was determined [16]. Single-factor experiments were used to select the range of various additives and orthogonal experiments were used to optimize the optimal ratios of the three oxidizers. Finally, the performance of the risers made with the optimized ratios was compared with that of the general aluminum powder risers.

2. Experimental

2.1. Materials and instruments

Main materials: Organosilicon slag with a particle size of 75-150 µm was obtained from Yichang Xingrui Company; Aluminum powder particle size not less than 75 µm and purity of more than 99 percent; Fe_3O_4, MnO_2, KNO_3 and Na_3AlF_6 powder, all four powder reagents were analytically pure; Alkali Phenolic Resin; Hollow drift beads produced in Hebei, with a particle size of 150-380 µm.

Instruments in the experiment: Sand Mixer (JJ-5), SAC Hammer Type Proofer, Electric Blast Dryer(XGQ-2000), High-temperature Resistance Furnace (SRJX-4-13), Paperless Recorder for temperature measurement(SIN-R5000C) with S-type platinum-rhodium thermocouple connected, Strength Tester(SWX), and Environmental Scanning Electron Microscope (GSM-7800).

2.2. Sample preparation and measurement

Firstly, put the organosilicon slag into the Electric Blast Dryer oven at 80°C and dried it to constant weight. Second, according to the experimental recipe, put all the heating and insulation materials into the Sand Mixer and mixed at low speed for about 5 min. Then added a certain proportion of binder according to the total weight of the material and mixed at high speed for about 10 min. After mixing the well, took about 75 g of mixed material each time and put it into the mold to make a 50×50 mm cylindrical sample by hammering the SAC Hammer Type Proofer. Finally, the demolded samples were put into the Electric Blast Dryer oven at 150°C for 30-45 min to heat up and harden.

In order to measure the burning effect of the specimen during combustion, a hole ϕ10×30 mm was drilled at the top midpoint of the cylindrical sample for placing the thermocouple (except for the compressive strength test). The sample was placed in a high-temperature resistance furnace, ignited at 800°C, and removed. Using the Paperless recorder for temperature measurement to determine the riser samples' heat generation, insulation, and combustion properties. These properties specifically included maximum temperature, holding time (sample temperature above 1200°C duration), ignition time (sample into the furnace from the time the sample began to burn violently), and secondary combustion time (sample began to burn violently to the end of the duration of combustion). The compressive strength of the sample was measured with the Strength Tester.

2.3 Experimental methods

Experiment on the effect of organosilicon slag on heat insulation performance: In this experiment, the microscopic morphology and composition of the treated organosilicon slag were first analyzed using environmental scanning electron microscopy. Through preliminary experimental studies, a part of aluminum powder was replaced by the organosilicon slag as the exothermic agent to configure the riser samples. This paper discussed the combustion effect of riser by changing the ratio of organosilicon slag and aluminum powder in exothermic agent under different total amount of exothermic agent. Their content variation ranges were shown in Table 1. Other materials remain unchanged and cylindrical samples of risers were prepared. Determined the effect of organosilicon slag on the maximum temperature and holding time of the riser sample to determine the optimal content of the exothermic agent and organosilicon slag.

Table 1.
Variation range of total exothermic agent, organosilicon slag and aluminum powder

The total amount of exothermic agent (%)		30			35		40	
Ratio of exothermic agent	Organosilicon slag (%)	20	30	40	50	60	70	80
	Aluminum powder (%)	80	70	60	50	40	30	20

Single-factor experiment of riser additives: After determining the components of the exothermic agent in the riser, the riser samples were prepared by changing the addition amounts of different additives in turn through single-factor experiments. The variation range of different auxiliary content was shown in Table 2. Among them, the flux was Na_3AlF_6, and the sample was analyzed for the combustion supporting effect; the binder was alkaline phenolic resin, and the sample was analyzed for the strength performance; the oxidizer includes three kinds: Fe_3O_4, MnO_2 and KNO_3. The effect of oxidants on the ignition time and secondary heat generation time of the samples was analyzed.

Table 2.
Variation range of different additives

Additives		Content variation range (%)					
Flux	Na_3AlF_6	0	2	4	6	8	
Binder	Alkaline Phenolic Resin	8	10	12	14	16	18
Oxidizer	Fe_3O_4	0	3	6	9	12	15
	MnO_2	0	3	6	9	12	15
	KNO_3	0	2	4	6	8	10

Oxidizer orthogonal optimization experiment: Based on the single-factor experiments, the orthogonal experiments were done with the content of oxidants as three factors with three levels per factor, and the orthogonal table L9 (3^3) (scheme shown in Table 4) was selected. Other materials were fixed, and the riser samples were prepared. The samples' ignition time and secondary heat generation time were used as the analysis index. Data analysis was performed using Minitab 18 software.

3. Results and analysis

3.1. Basic composition analysis of organosilicon slag

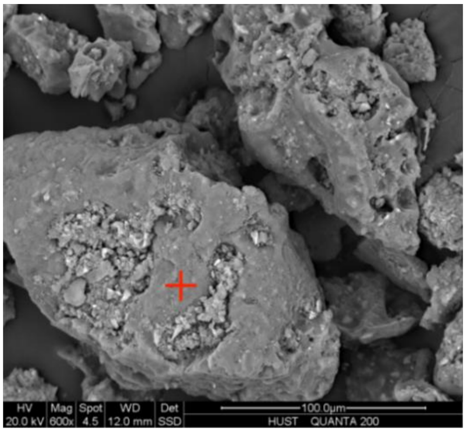


Fig. 1. Organosilicon slag micromorphology

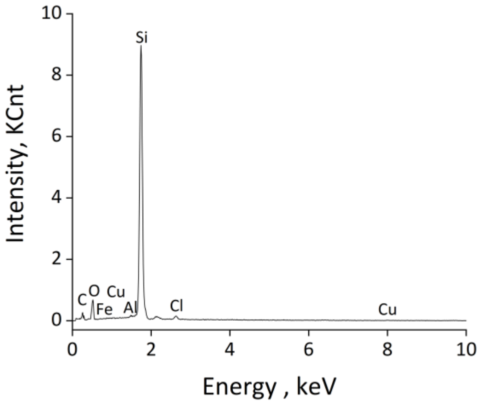


Fig. 2. Energy spectrum curve of organosilicon slag

Table 3.
Elemental content in the area after organosilicon slag treatment

Element	Si	C	O	Cl	Al	Cu	Fe
wt%	82.20	10.31	6.65	0.26	0.23	0.22	0.13

The organosilicon slag microscopic morphology was shown in Fig. 1. The energy spectrum curve diagram and the corresponding mass percentages of each element were shown in Fig. 2 and Table 3, respectively. It could be seen that the mass percentages of the main metallic elements (Al, Cu, Fe) and chlorine elements in the organosilicon slag were less than 1% after the harmless treatment. It indicated that most metal salts of monomeric particles (mainly copper powder) and organic chlorosilanes left in the organosilicon slag by hydrolysis were effectively reduced. The contents of these heavy metals and other impurities were further diluted when organosilicon slag was used as a riser exothermic agent. Therefore, after the riser made of organosilicon slag was cast, the content of harmful substances in the remaining residue would not cause excessive environmental pollution. In addition, the microscopic morphology from Fig. 1 demonstrated that the surface of organosilicon slag would have many irregular pores. This porous structure reduced the heat conduction area, improved the thermal resistance value, and provided better heat insulation and heat preservation effect.

3.2. Contents of exothermic agent and organosilicon slag effect

This experiment was chosen to vary the total content of the exothermic agent and, for each content, to select different proportions of organosilicon slag and aluminum powder for the experiment. The purpose was to select the most suitable ratio of the exothermic to maintain the long time insulation effect while increasing the maximum combustion temperature of the riser. Fig. 3 showed the variation curve of the maximum temperature of the sample. Fig. 4 showed the variation curve of the holding time of the sample.

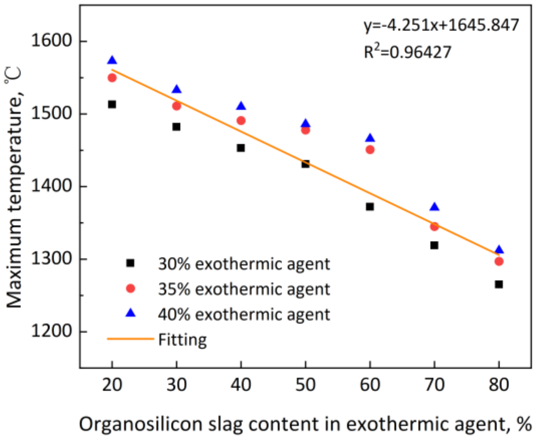


Fig. 3. Relationship between organosilicon slag content and maximum temperature

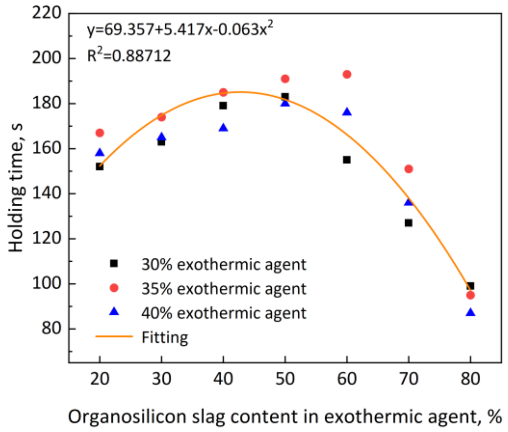


Fig. 4. Relationship between organosilicon slag content and holding time

It was found in Fig. 3 that when the organosilicon slag content was 60%, the maximum temperatures of the three different exothermic agent additions were 1384℃ (30% exothermic agent), 1451℃ (35% exothermic agent), and 1466℃ (40% exothermic agent). It indicated that the higher the overall exothermic agent addition, the higher the maximum temperature of sample combustion. Furthermore, the addition of the exothermic agent had a more significant effect on the heating temperature of the riser when it was lower than 35%. It was shown in Fig. 4 that the general insulation performance of the riser was best when the total addition of the exothermic agent was 35%. When the organosilicon slag content was 20-50%, the lowest holding time was observed for the curve at 40% of the exothermic agent addition. It was because the more the content of the overall exothermic agent in the riser, it would lead to the reduction of the insulation material (hollow drift beads) content, which would affect the insulation performance of the riser. When the content of organosilicon slag was higher than 50%, it shortened the insulation time due to the significant reduction of the maximum temperature. Therefore, the total amount of the exothermic agent was chosen to be 35% best.

It was also shown in Fig. 3 and Fig. 4 that when the organosilicon slag content was 50%, the 30% and 40% exothermic agent content curves reach peak holding times of 187 s and 180 s, respectively. When the organosilicon slag content increased to 60%, the 35% exothermic agent content curve reached a peak holding time of 193 s. However, as the organosilicon slag content increases, the holding time decreases instead. When the organosilicon slag content increased to more than 70%, the highest temperature of the three kinds of exothermic agent content curve was lower than 1450 ℃, while the insulation time was reduced to less than 180 s. It indicated that an appropriate amount of silicone slag content could improve the insulation time. However, too much organosilicon slag content would reduce the content of aluminum powder in the exothermic agent, thus reducing the maximum temperature of the riser. In order to ensure that the riser had sufficient complementary shrinkage effect, its maximum heating temperature reached at least 1450℃, so the organosilicon slag content could not be higher than 60%. Considering the heat preservation performance and the influence of economic factors, the total addition of the exothermic agent was 35%, of which 60% was organosilicon slag, and 40% was aluminum powder. This also meant that the exothermic agent's content in the riser was 21% organosilicon slag and 14% aluminum powder.

3.3. Single-factor experiment of Na_3AlF_6

To reduce the starting temperature at which the riser starts to burn and accelerate the rate of ignition, a small amount of flux was usually added to the exothermic insulating riser. Based on fixed heating material, The ignition rate of the riser was adjusted by changing the addition amounts of Na_3AlF_6 in turn. The samples were ignited in a high-temperature resistance furnace at 800°C, and the ignition time was measured. The experimental results are shown in Fig. 5. When the content of Na_3AlF_6 was higher than 4%, the trend of decreasing ignition time was stable. However, the addition of too much Na_3AlF_6 in the riser would accumulate fluorine elements in the sand. It will cause pollution to the casting or the environment in the long run. Therefore, the amount of Na_3AlF_6 added was 4%.

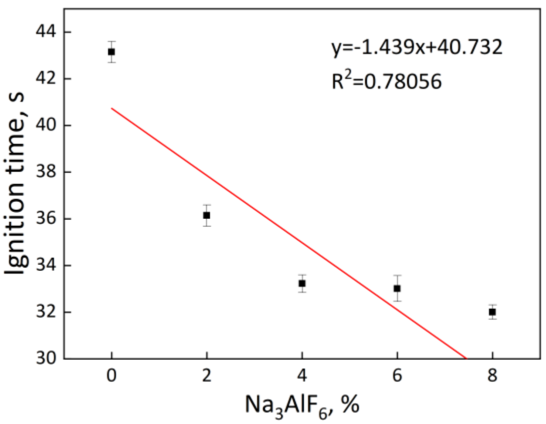


Fig. 5. Relationship between Na_3AlF_6 content and ignition time

3.4. Single-factor experiment of phenolic resin

The heating and insulation materials in the riser did not have bonding properties of their own, so a binder needed to be added to ensure that the riser had a certain strength. In this paper, alkali phenolic resin was chosen as the binder. The compressive strength of the samples was measured with the strength tester. Fig. 6 showed the experimental results of strength performance. With the increase of alkali phenolic resin addition, the compressive strength of the samples increased continuously. The combustion test found that the thermal strength of the riser sample with a compressive strength lower than 4 MPa was not high, and it was easy to be deformed at high temperature, which would affect the shrinkage effect of the riser when casting. However, the compressive strength was too higher that would also affect the permeability of the riser, and the oxygen decomposed by the oxidizer could not fully contact with the exothermic agent, making the burning performance of the riser lower. Therefore, the amount of alkali phenolic resin added should be selected at 12%.

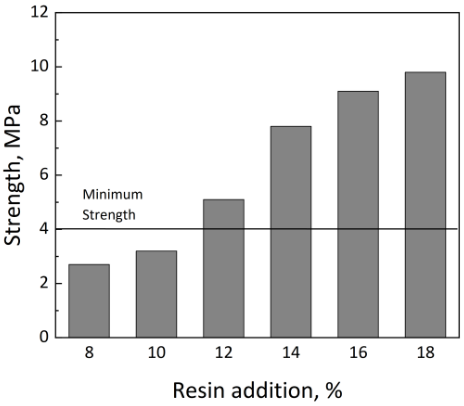
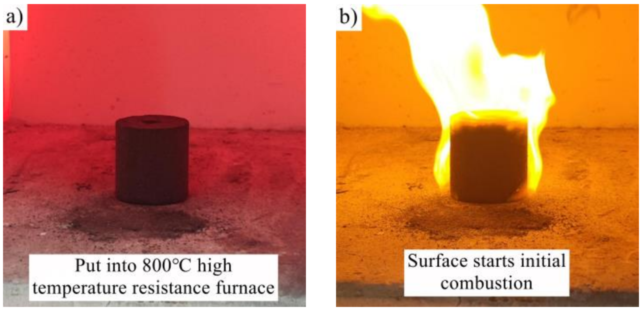


Fig. 6. Relationship between phenolic resin and compressive strength

3.5. Single-factor experiment of oxidizer

The riser's ignition time and secondary combustion time could be adjusted by changing the content of different oxidants in the heating material. The less the ignition time, the faster the riser could provide heat to the metal liquid; the longer the secondary combustion time, the longer the reaction time between the exothermic agent and the oxidizing agent after ignition, and the longer the solidification time of the metal liquid. The process of the riser sample from the beginning of ignition to complete combustion was shown in Fig. 7. The temperature of high temperature resistance furnace was set to 800°C in the experiment, but the temperature of metal liquid was higher than the temperature of resistance furnace in the actual casting process. This experiment simulated the casting environment at a fixed temperature and measures the ignition time and secondary heating time of the riser sample.

When the riser was first put into the high temperature resistance furnace, its surface would burn rapidly, but the ignition stage was not completed at this time. It ended only when a bright flame was emitted from the burning area and the secondary heating phase was entered. At this point, the sample was removed and placed at room temperature, and the flame gradually spread throughout the sample.



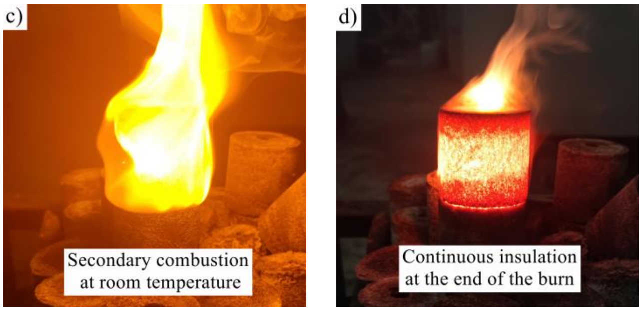


Fig. 7. Different stages of combustion of riser sample: a) Before ignition, b) Ignition stage, c) Secondary combustion stage, d) Continuous insulation stage

The relationship curves between Fe_3O_4 content, ignition time, and secondary combustion time were shown in Fig. 8. As the oxidizer content increased, the ignition time showed a decreasing trend. When the content increased to 9%, the change of ignition time became stable and small, and the ignition time decreased to below 51 s. At this time, compared with no oxidizer, the ignition rate of the riser sample increased by 60%. The secondary combustion time gradually increased under the increase of oxidizer content, reaching a peak of 37 s at 9%, and then slowly decreased. Therefore, the amount of Fe_3O_4 added should be selected at 9%-12%.

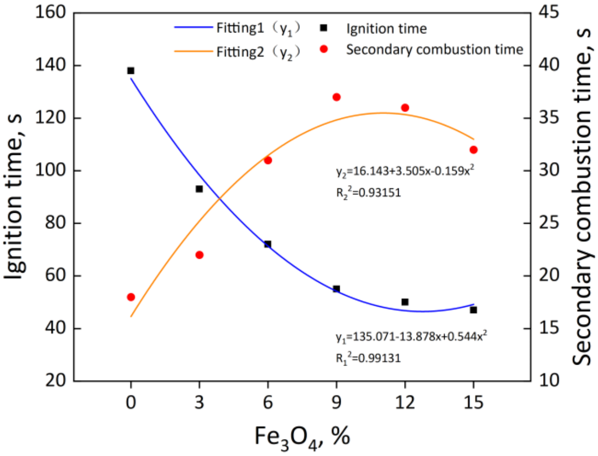


Fig. 8. Relationship between Fe_3O_4 and ignition time and secondary combustion time

The relationship curves between MnO_2 content, ignition time, and secondary combustion time were shown in Fig. 9. With the increase of oxidizer content, the ignition time decreased. When the content increases to 6%, the change of ignition time tends to be stable and small, and the ignition time decreases to below 61 s. At this time, compared with no oxidizer addition, the ignition rate of the riser sample increased by 55%. The secondary combustion time peaks at 34 s when the oxidizer content increases to 6%, and the secondary combustion time gradually decreases as the content increases. Therefore, the amount of oxidizer MnO_2 added should be selected at about 6%.

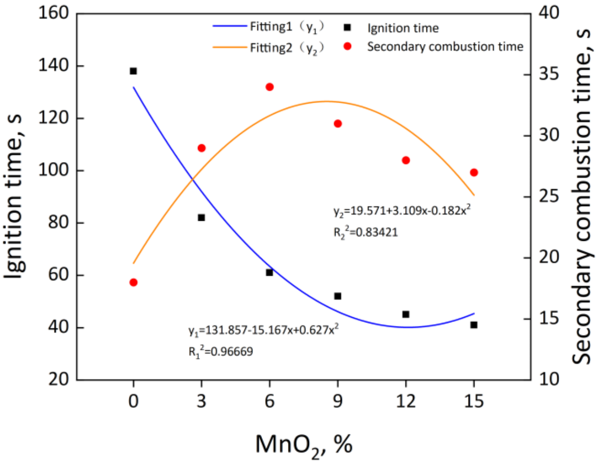


Fig. 9. Relationship between MnO_2 and ignition time and secondary combustion time

The relationship curves between KNO_3 content, ignition time, and secondary combustion time were shown in Fig. 10. As the oxidizer content increases, the ignition time gradually decreases, while the secondary combustion time gradually increases. When the content to 4%, the ignition time and secondary combustion time changes are stabilized, the ignition time is reduced to 59 s, the ignition speed when not adding oxidizer increases by 57%, and the second heating time increases to 28 s. When not adding oxidizer extend by 10 s. Therefore, the amount of KNO_3 added should not be less than 4%.

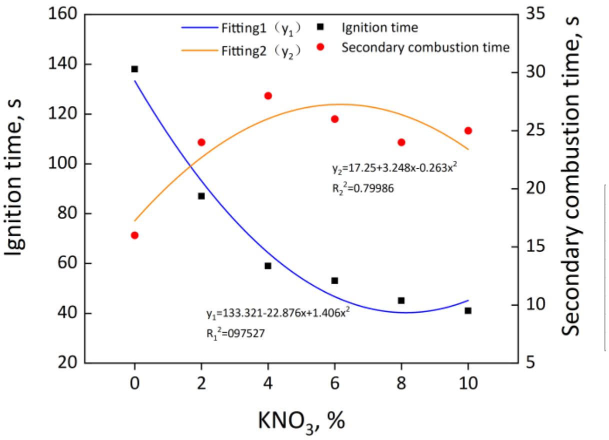


Fig. 10. Relationship between KNO_3 and ignition time and secondary combustion time

3.6. Oxidant orthogonal optimization

The single-factor experimental results showed that the content of each oxidizer increases to a certain extent, the rate of decline in the ignition time of the sample would slow down, and too much oxidizer also reduced the secondary combustion time. The ignition time of the heat insulation riser was generally controlled in the range of 25-45 s. Ignition time was too fast or too slow would affect the effect of shrinkage. It was also necessary to extend the secondary combustion time of the riser as much as possible. The use of a single oxidizer was challenging to make the combustion performance of the riser to meet the requirements. Therefore, a mixture of oxidizers was needed, and the ratio needed to be further optimized orthogonally.

The orthogonal tests and the extreme value analysis were shown in Table 4 and Table 5, respectively. The ratios of different oxidant mixes were analyzed using the polar difference method. The extreme difference between the three factors was used to represent the importance of each factor's effect on the indicator. The greater the value of a factor's extreme difference, the greater the effect of that factor on the response index. When the indicator was the ignition time, the order of the size of the extreme differences between the three factors was $R_A > R_B > R_C$, where R_A and R_B were much larger than R_C. It indicated that KNO_3 content had the slightest effect on the ignition time. There was little difference in the significance of the effects of both Fe_3O_4 and MnO_2. When the index was the secondary combustion time, the order of the size of the polar differences of different factors was $R_B > R_C > R_A$. The influence of MnO_2 content on the secondary combustion time was the largest, followed by KNO_3 and Fe_3O_4. The optimal ratio combination was determined as $A_3B_2C_3$(Fe_3O_4 content was 12%, MnO_2 content was 6%, and KNO_3 content was 6%) according to the average value of each water. The riser samples prepared using the optimized oxidant ratios had an ignition time of 32 s and a secondary combustion time of 44 s.

Table 4.
Orthogonal test protocol and results

No	Factors			Experimental results	
	A: Fe_3O_4 (%)	B: MnO_2 (%)	C: KNO_3 (%)	Ignition time (s)	Secondary combustion time (s)
1	6	4	4	53	38
2	6	6	5	47	45
3	6	8	6	36	42
4	9	4	5	49	37
5	9	6	6	33	45
6	9	8	4	40	46
7	12	4	6	37	41
8	12	6	4	35	43
9	12	8	5	28	40

Table 5.
Means and extreme value of each factor for different indicators

Indicator	Level	A: Fe_3O_4	B: MnO_2	C: KNO_3
Ignition time	1	45.33	46.33	42.67
	2	40.67	38.33	41.33
	3	33.33	34.67	35.33
	R	12.00	11.67	7.33
Secondary combustion time	1	41.67	38.67	42.33
	2	42.67	44.33	40.67
	3	41.33	42.67	42.67
	R	1.33	5.67	2.00

4. Validation of optimization results

In order to verify the results of ratio optimization of the riser, homemade risers of the same size were compared with conventional exothermic insulating risers on the market (The exothermic agent was mainly aluminum powder and its content exceeded 30%). The combustion experiment was shown in Fig. 11. And the two risers' heating and combustion performance and temperature variation curves were measured.

The optimized ratio of organosilicon slag riser:

- 35% of the exothermic agent content (including 21% of organosilicon slag and 14% of aluminum powder)
- 4% of Na_3AlF_6 chosen as flux
- 12% of alkali phenolic resin added as a binder
- 24% of the oxidizer content (including 12% of Fe_3O_4, 6% of MnO_2, and 6% of KNO_3)
- Hollow drift beads for filling vacant content

Fig. 11. Combustion experiment of riser: a) The organosilicon slag made riser, b) The aluminum powder made riser

The performance comparison between the organosilicon slag made riser (OSR) and the aluminum powder made riser (APR) was shown in Table 6, and the temperature-time curve was shown in Fig.12. There was not much difference in the ignition time between the two types of risers in the ignition stage. By the secondary combustion stage, the riser started to heat up rapidly, and the heating rate of APR was much faster at this stage. In about 70 s, the maximum temperature of the two risers reached the highest, with a difference of 52°C. In the cooling curve stage, the cooling speed of OSR is slower, and when the time was around 140 s, the two temperatures were nearly equal, and then the temperature of OSR was gradually higher than that of APR. In the temperature curve, the insulation time of OSR and APR are 210 s and 184 s, respectively, and the insulation effect of the riser was improved by 14%. Compared with the general aluminum powder prepared exothermic insulating riser, the organosilicon slag homemade riser had little heating performance and better insulation performance.

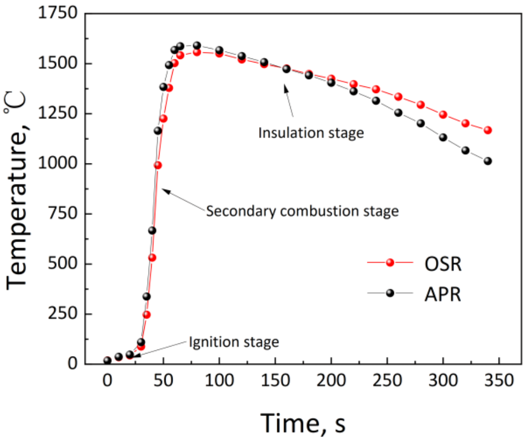


Fig. 12. Temperature-time curves of OSR and APR

Table 6.
Performance comparison of OSR and APR

Riser	Maximum temperature (°C)	Holding time (s)	Ignition time (s)	Secondary combustion time (s)
OSR	1542	210	31	45
APR	1594	184	27	42

5. Conclusion

(1) The test found that after harmless treatment, the organosilicon slag had sound heat generation and heat preservation performance. In this paper, we proposed a method of resourceful application of organosilicon slag, which replaced part of aluminum powder as the primary exothermic agent of the heating and heat preservation riser and made a new type of heating and heat preservation riser by ourselves.

(2) The total content of the exothermic agent and organosilicon slag's content would affect the riser's heating and insulation performance. The total amount of exothermic agent was 35% (21% for organosilicon slag and 14% for aluminum powder), the combustion temperature of the homemade riser could reach more than 1450°C, and the heat preservation time was kept above 180 s.

(3) To improve the combustion and strength performance of the self-made riser, the oxidizer ratio was optimized using the single-factor method and orthogonal method, and the addition of flux and binder was adjusted. The optimized formula was 4% for flux

Na_3AlF_6, 12% for binder furfural modified phenolic resin, 12% for Fe_3O_4, 6% for MnO_2, and 6% for KNO_3.

(4) The heating and burning performance of the homemade exothermic insulating riser using organosilicon slag could reach the general aluminum powder riser level, and the insulation effect was 14% higher than that of the general aluminum powder riser.

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