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Effect of Potassium Feldspar Sodium-feldspar Composite Flux on The Sintering Performance of Fly Ash-coal Gangue-phosphogypsum Based Wall Insulation Materials

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Abstract: In this paper, fly ash and coal gangue as the main raw materials, potassium feldspar-albite as the composite flux, phosphogypsum as the bonding agent, the semi-melting method was used to prepare fly ash-coal gangue-phosphogypsum based wall insulation material, the effect of composite flux ratio and the amount of coal gangue added on the melting temperature of wall insulation material was studied. Through the visual of high-temperature deformation analyzer to observe the volume contraction, expansion, passivation, and globalization of different formulations of specimens at high temperatures, and record the corresponding temperatures of the occurrence of various situations, to study the composite flux ratio and gangue addition on the fly ash-gangue-phosphogypsum based wall insulation material melting temperature of the impact, to determine the appropriate ratio of the flux and the gangue addition for the solid waste-based will provide a theoretical basis for the preparation of solid waste-based wall insulation materials. The research results indicate that with the increase of coal gangue content, the initial melting temperature, spheroidization temperature, and passivation temperature of the sample all sharply decrease. With the increase of potassium feldspar: albite, the melting temperature range will significantly increase. When the additional amount of fly ash is 50%, the additional amount of coal gangue is 12%, the additional amount of phosphogypsum is 5%, and the ratio of potassium feldspar to albite is 1:1, the sintering temperature of the fly ash- gangue -phosphogypsum based wall insulation material is the lowest.

Keywords: Solid waste, Composite flux, Sinterability, Melting temperature.

1. Introduction

Energy is closely related to the development of human society, and sustainable development is the common goal of all human beings [1]. In the 21st century, all countries in the world have included energy strategy as an important content in their national development plans, and our government has identified it as a major national development strategy. According to the latest statistical data, China's building energy consumption has accounted for about 30% of the total energy consumption of the whole society, and continues to rise with the acceleration of urbanization [2-3] Coal accounts for about 67% of China's energy structure, and thermal power generation is the main power supply. A large amount of coal burning causes increasingly serious air pollution and a large amount of solid waste discharge. Therefore, high and new technologies focused on solving energy and environmental problems have been widely considered and will play an important role in the sustainable development of nature and society [4]. Wall insulation is an important means of building energy efficiency, external wall insulation is currently the most important technical form of energy-saving insulation of the building envelope, and the insulation material is one of the key factors affecting the effect of energy-saving insulation of building walls [5]. Therefore, how to improve the insulation performance of walls and reduce energy and resource consumption has become a top priority. Organic insulation materials are widely used due to their advantages such as good insulation performance, light texture, and mature application technology. However, in recent years, the frequent occurrence of fire accidents caused by the combustion of organic insulation materials has limited their further promotion and application, making inorganic insulation materials gradually become a new favorite in the insulation material market [6-7]。Compared with organic insulation materials, inorganic insulation materials have advantages such as high-temperature resistance and good durability, and have broad prospects for application and development. As a common industrial solid waste, fly ash is mainly derived from the coal waste of thermal power plants. Coal combustion is ground into pulverized coal with a particle size of less than 100 μm through a coal mill, and then sprayed into the coal-fired furnace through a preheated air unit for suspended combustion. The combustible components of the coal are burned and then collected by a dust collector, and the waste discharged from the dust collector is called fly ash. Fly ash is composed of highly dispersed aggregates of fine particles and is a volcanic ash material derived from inorganic substances in coal. The chemical composition and properties of fly ash are related to the type of coal-fired, boiler type, combustion conditions, etc. in thermal power plants. The main components are SiO_2 and Al_2O_3, while other components include Fe_2O_3, CaO, TiO_2, and unburned organic matter [8]. Along with the development of China's thermal power generation industry, the total amount of fly ash emission has been a rapidly growing trend, and by the end of 2017 it has reached 686 million tonnes [9], which is equivalent to more than three times of the amount of municipal domestic waste removal in that year, and its volume can reach 776 million cubic meters, which is equivalent to pouring a standard swimming pool every 1 min, or 2 water cubes per day, and if this fly ashes are not effectively utilized, they will not only If these fly ash is not used effectively, it will not only occupy a large amount of arable land, but also pollute the environment. The resource utilization of fly ash in China has long been highly valued by the government, and the utilization rate has gradually increased in recent years [10]. According to relevant statistics [9], the comprehensive utilization rate of fly ash in China increased from 35% in 1997 to 72% in 2017, which has reached the level of developed countries in terms of utilization rate, but the overall utilization mechanism is not perfect. At present, fly ash in China is mainly used for extensive and simple reuse such as building materials, construction, road construction, backfilling, etc. [11-15], and there are few industrial applications with fine and high added value. Therefore, it is of great significance to promote the industrialization process of fine utilization of fly ash based on improving the comprehensive utilization rate of fly ash.

1.1. Analysis of research status at home and abroad

The total content of SiO_2 and Al_2O_3 in the main components of fly ash is over 60wt%, and it contains a large number of micropores with a high specific surface area, making it suitable for preparing porous materials [16-17]. At present, there has been some progress in the research on the preparation of porous materials using fly ash both domestically and internationally, but there is little research on the preparation of high-content and high closed-cell self insulation wall materials. Bao[18] used 42wt% waste glass, 40wt% fly ash, and 18wt% albite as the main raw materials, selected silicon carbide as the blowing agent, and prepared porous materials under the sintering temperature of 1130 ℃ and held heat for 15 min. The obtained porous material has a pore size of less than 1.5 mm, a uniform distribution of pores, a volume density of 0.39 g/cm^3, a bending strength of 4.60 MPa, and an apparent porosity of 18.6%. Hou[19] prepared porous materials with red mud and fly ash as the main raw materials using starch and manganese dioxide as blowing agents and sodium borate a co-solvent, respectively. The experimental results show that with the increase of foaming agent content, the material exhibits excellent comprehensive properties: bulk density 0.59~0.96 g/cm^3, apparent porosity 41.82%~63.51%, water absorption 3.16%~9.17%, bending strength 2.44~5.82 MPa, and has good acid and alkali resistance. Chen et al. [20] used red mud (40-50wt%), fly ash (25-40wt%), and borax (15-20wt%) as raw materials, added 5% water glass as a foaming agent, and successfully prepared porous materials through dry pressing at 1000 ℃. The volume density was 0.51~0.64 g/cm^3, the porosity was 64.14~74.15%, the water absorption was 2.31~6.02%, and the bending strength was 2.31~8.52 MPa. Li et al.[21] used fly ash as the main raw material, gypsum as the adhesive, and used direct foaming method. After forming, they were sintered at a temperature of 1100 ℃, resulting in the highest open porosity of 94% and the lowest thermal conductivity of 0.042 W/m •K. Zhou [22] and others prepared the fly ash-based phosphate foam material by casting molding process. The amount of fly ash exceeds 90wt%, the flexural strength of the product reaches 6.06MPa, the density is 1.21g/cm^3, and the water absorption rate is 20.1%. Ai Fanrong [23] et al. used the foam impregnation method to prepare the fly ash porous material. When the fly ash content is 70wt%, the porosity of the sample is close to 90%.

It can be seen from the current research that the firing temperature of the fly ash-based porous materials is above 1000 ℃, and the strength is not high. When the fly ash content is high, the pores of the sample may soften and deform due to the high glass phase content, which makes it difficult to control the shape, size, and shape of the foam ceramic pores, resulting in high water absorption. To improve the thermal insulation performance of wall materials, one is to choose substrates with low thermal conductivity, and the other is to reduce the density of the substrate, forming a porous structure within the substrate [24]. However, the open-hole structure of the insulation material is easy to cause water to enter, resulting in an increasing in the water absorption rate of the wall. When water enters, the thermal conductivity of water is higher than that of air, which significantly reduces the insulation performance of wall materials. As a hot summer and cold winter area in Chongqing, the thermal performance requirements of walls are mainly summer thermal insulation and winter thermal insulation [25]. At the same time, Chongqing has abundant precipitation, uneven distribution of time and space, many rainstorms, and the average annual precipitation is up to 1000~1350mm [26]. The average annual relative humidity in Chongqing is mostly between 70% and 80%, which is a high-humidity area in China. Therefore, there is an urgent need for a new type of wall insulation material that can achieve the energy-saving and insulation goals of building envelope structures and solve fire prevention problems under the climatic conditions of the Chongqing region. In this study, fly ash, coal gangue, and phosphogypsum are used to prepare inorganic wall insulation materials, and the structure and performance are designed and adjusted through flux, binder, and preparation technology. This can not only make efficient and large amounts of solid waste use, solve the environmental pollution caused by large amounts of its stacking, but also greatly improve the energy saving and insulation performance of building external walls. Solve the problems of easy aging, poor durability, and easy burning of organic thermal insulation materials, and realize the purpose of building energy conservation.

2. Testing

2.1. Experimental Materials

2.1.1. Substrate

Fly ash is taken from Guodian Chongqing Hengtai Power Generation Co., Ltd., and coal gangue is taken from Weijiagou Coal Mine in Yongchuan District. The XRF test results are shown in Table 1.

Table 1. Matrix chemical composition

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	TiO_2	MgO	K_2O	Na_2O	SO_3	IL
Fly ash	38.93	46.2	3.75	2.77	1.62	0.25	0.53	0.21	1.25	4.26
Coal gangue	60.69	18.81	3.55	0.55	1.18	0.89	3.08	0.16	0.1	18.69

2.1.2. Flux

The function of the flux is to interact with the raw material components during the heating process, thereby reducing the melting point and softening temperature of the raw material and allowing sintering to occur at relatively low temperatures. The lower melting temperature saves energy, shortens the production cycle, and reduces preparation costs. Fly ash and coal gangue are located in the mullite crystal zone in the ternary phase diagram. Due to the high melting point of mullite, the melting temperature of this fly ash is also high. To meet the requirements of preparing foam wall insulation materials, fluxes need to be added to reduce the melting temperature. In the experiment, potassium feldspar and sodium feldspar were used as mixed fluxes, which were provided by a processing plant in Lingshou, and the chemical composition of potassium feldspar and sodium feldspar was examined by XRF, and the results of the examination are shown in Table 2

Table 2. Chemical composition of raw materials

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	LOI
Potassium feldspar	65.8	17.2	0.7	1.2	0.3	12.4	0.7	1.7
Albite	62.2	16.4	1.2	1.6	1.9	1.4	11.8	3.5

2.1.3. Cementitious Material

Phosphogypsum was taken from Sinochem Fuling tailings depot and the XRF test results are shown in Table 3.

Table 3. Chemical composition of binders

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	TiO_2	MgO	K_2O	Na_2O	SO_3	BaO	P_2O_5	IL
Phosphogypsum	3.78	0.47	0.18	29.04	0.07	0.11	0.14	0.05	42.48	0.11	0.95	22.55

2.2. Determination of melting temperature range

2.2.1. The visual of high-temperature deformation analyzer

This instrument is a high-temperature device for measuring the sintering point temperature and fire resistance of inorganic materials, mixtures, and ceramic raw materials, as shown in Figure 1. It allows the tester to see the volume shrinkage, expansion, passivation, and complete spheroidization of the material sample under high-temperature conditions on the screen, and to know the corresponding temperature when various situations occur.

Figure 1. The visual of high-temperature deformation analyzer

2.2.2. Preparation process

After the raw materials used in the experiment were screened, weighed, mixed, and ground evenly, the samples were formed with a specified mold, and the reactions of the samples under high-temperature heating conditions were observed with the visual of high-temperature deformation analyzer The experimental process flow is shown in Figure 2.

The specific process flow of the experiment is as follows:

(1) Passing the fly ash and coal gangue after drying in an electric constant temperature drying oven through a 100 mesh sieve;

(2) Design formulas based on the composition of fly ash, coal gangue, phosphogypsum, potassium feldspar, and sodium feldspar;

(3) According to the designed formula, the electronic balance is used to accurately weigh all kinds of raw materials, and the weighing error is controlled within ±0.01g;

(4) After mixing the weighed raw materials, they are loaded into a ball milling tank for ball milling. The ball milling tank is a resin ball milling tank, and the grinding ball is a zirconia ball;

(5) Take an appropriate amount of evenly ground raw material and press it into cylindrical blocks using a designated mold. The sample size is Φ 6 × 8 mm;

(6) Place the formed green body into the visual of high-temperature deformation analyzer, observe the volume shrinkage, expansion, passivation, and complete spheroidization of the sample under high-temperature conditions through computer images, and obtain the corresponding temperature when various situations occur.

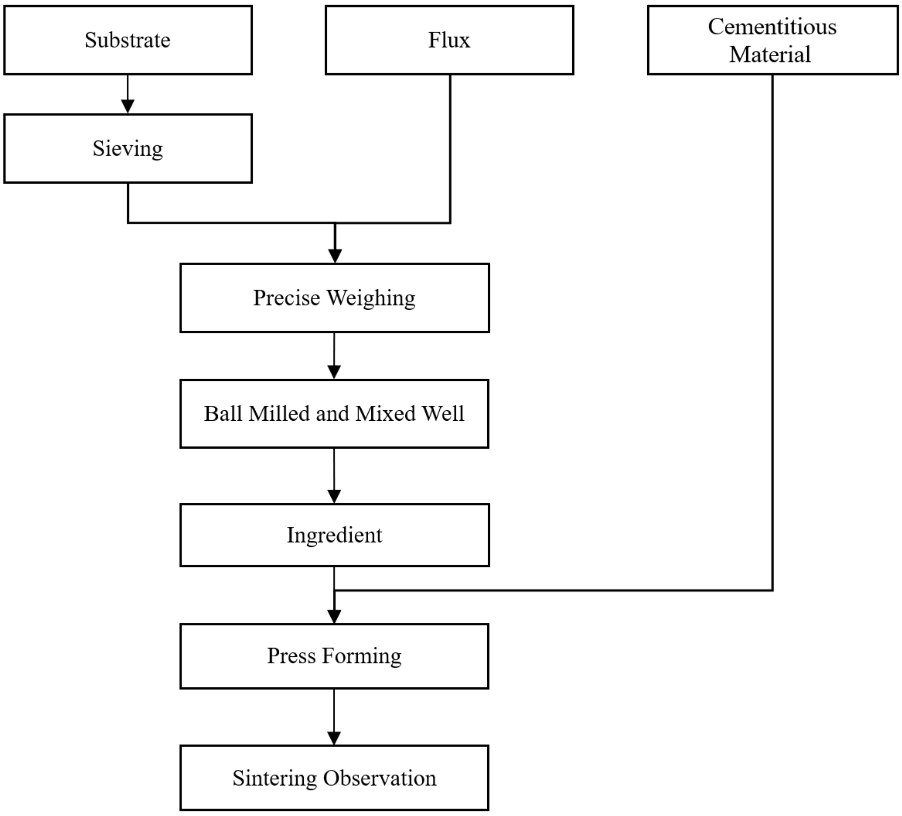

Figure 2. Flow chart of melting temperature measurement research

3. Effect of Flux on The Sintering Performance of Fly Ash Coal Gangue Phosphogypsum-based Wall Materials

The factor levels are shown in Table 3.1. The additional amounts of fly ash are 50%, 60%, and 70%, while the additional amounts of coal gangue are 8%, 10%, and 12%. The additional amount of phosphogypsum remains unchanged at 5%, and the proportions of potassium feldspar to sodium feldspar are 3:1, 1:1, and 1:3, respectively.

Table 4. Table of factor levels

Factor level	Fly ash	Coal gangue	Potassium feldspar: Albite
1	50%	8%	3:1
2	60%	10%	1:1
3	70%	12%	1:3

According to the factor levels in Table 4, an orthogonal experimental scheme was designed, as shown in Table 5.

Table 5. Orthogonal test plan

Number	Fly ash (%）	Coal gangue (%)	Potassium feldspar: Albite
z1	50	8	3:1
z2	50	10	1:1
z3	50	12	1:3
z4	60	8	1:1
z5	60	10	1:3
z6	60	12	3:1
z7	70	8	1:3
z8	70	10	3:1
z9	70	12	1:1

According to Table 5 of the orthogonal experimental plan, experiments were conducted separately, and the results are shown in Table 6.

Table 6. Orthogonal experimental results

Number	Initial melting temperature (℃)	Passivation temperature (℃)	Spheroidization temperature (℃)	Melting temperature range width (℃)
z1	990	1120	1185	195
z2	940	1105	1165	225
z3	950	1095	1110	160
z4	1025	1150	1208	183
z5	1035	1130	1185	149
z6	900	1060	1145	245
z7	1055	1185	1230	175
z8	980	1105	1200	220
z9	935	1070	1135	200

Analyze the experimental results as shown in Table 7.

Table 7. Analysis of orthogonal design experimental results

Index		Fly ash	Coal gangue	Potassium feldspar: Albite
Initial melting temperature	Range R	30	95	56.7
	Factor primary→secondary	Coal gangue→Potassium feldspar: Albite→Fly ash		
Passivation temperature	Range R	13.3	76.7	41.7
	Factor primary→secondary	Coal gangue→Potassium feldspar: Albite→Fly ash		
Spheroidization temperature	Range R	35	77.7	7.3
	Factor primary→secondary	Coal gangue→Potassium feldspar: Albite→Fly ash		
Melting temperature range width	Range R	6	17.3	58.7
	Factor primary→secondary	Coal gangue→Potassium feldspar: Albite→Fly ash		

3.1. The influence of various factor levels on the initial melting temperature and passivation temperature

The range analysis method was used to analyze the effects of various factors on the initial melting temperature and passivation temperature of the sample, as shown in Figures 3 and 4

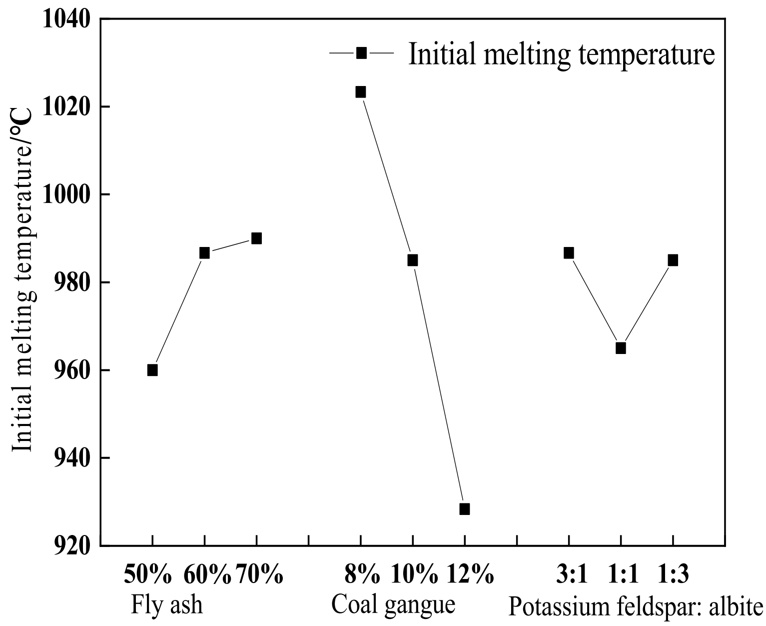

Figure 3. Relationship between initial melting temperature and various factor levels

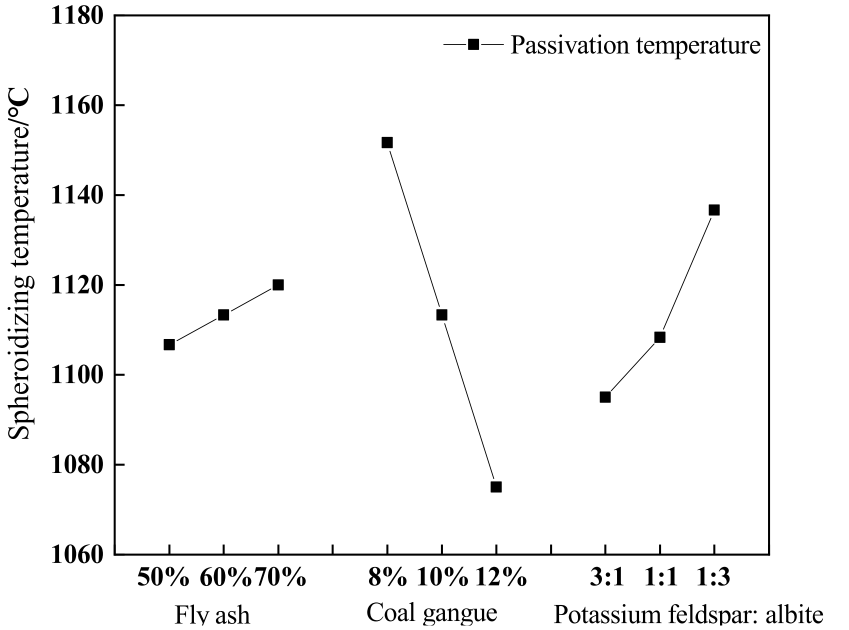

Figure 4. Relationship between passivation temperature and various factor levels

From Figures 4 and 3, it can be seen that the relationship between the initial melting temperature and passivation temperature is consistent with the levels of various factors. The order of influence of each factor on the initial melting temperature and passivation temperature is: Coal gangue>Potassium feldspar: Albite>Fly ash, With the increase of coal gangue content, the initial melting temperature and passivation temperature of the sample sharply decrease; With the increase of fly ash content, some fly ash cannot melt and contract, and the initial melting temperature and passivation temperature show an upward trend, especially when the content increases from 50% to 60%, the initial melting temperature significantly increases; The initial melting temperature of the sample shows a trend of first decreasing and then increasing with the increase of the proportion of albite, while the passivation temperature increases with the increase of the proportion of albite.

3.2. The Influence of Various Factors on Spheroidization Temperature.

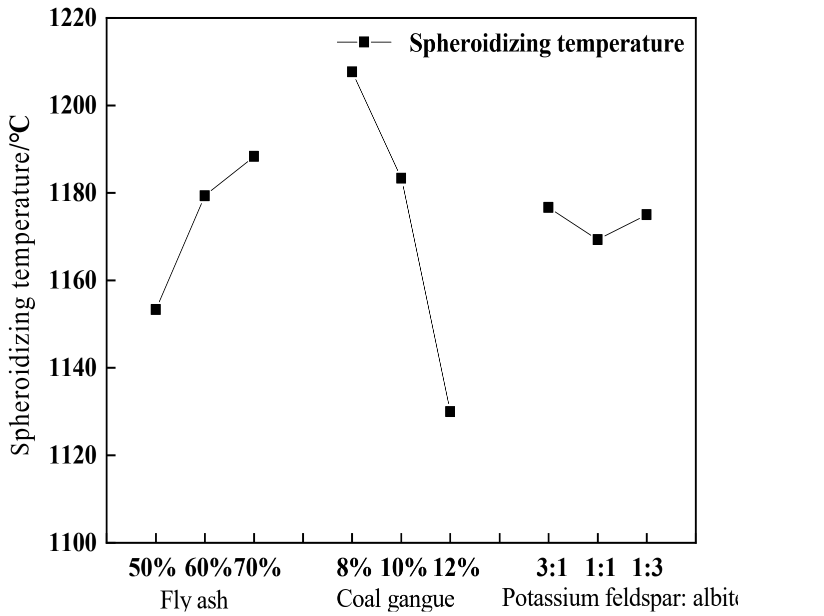

Figure 5. Relationship between spheroidization temperature and various factor levels

From Figure 5, it can be seen that the order of influence of various factors on the spheroidization temperature is: Coal gangue>Potassium feldspar: Albite>Fly ash, and the influence law on the spheroidization temperature of the sample is consistent with the initial melting temperature.

3.3. The influence of various factor levels on the width of the melting temperature range.

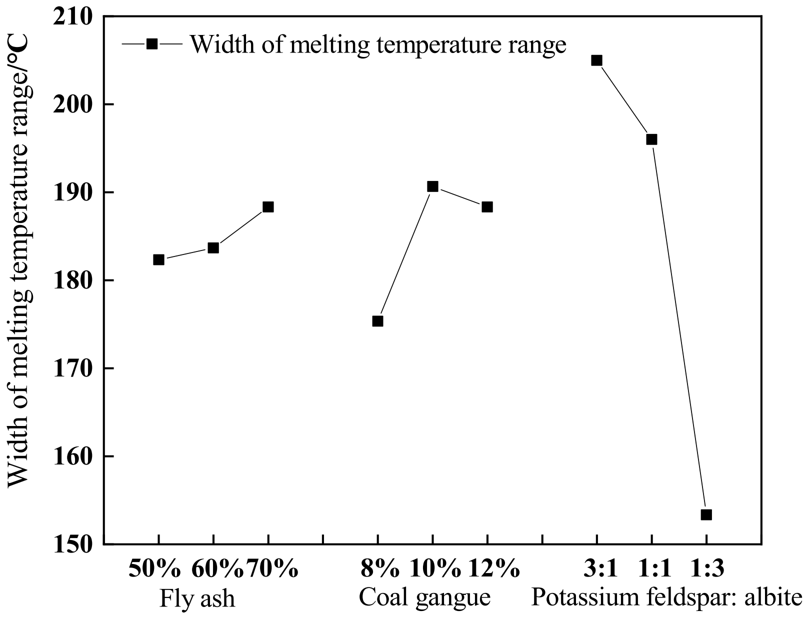

Figure 6. Relationship between spheroidization temperature and various factor levels

The range analysis method is used to analyze the influence of various factors on the width of the sample melting temperature range. The relationship between the width of the melting temperature range and various factors is shown in Figure 6. From Figure 6, it can be seen that the order of influence of various factors on the width of the melting temperature range is: Potassium feldspar: Albite>Coal Gangue>Fly ash. At high temperatures, K^+ and Na^+ introduced by potassium feldspar and sodium feldspar can move more freely compared to the initial melting temperature, weakening the Si-O bond more significantly, thereby reducing the melt viscosity. However, the proportion of K^+ introduced in potassium feldspar is higher than that of sodium feldspar. Therefore, as the proportion of potassium feldspar added increases, the width of the melting temperature range greatly increases; Add an appropriate amount of coal gangue to increase the viscosity of the melt, Therefore, the width of the corresponding melting temperature range will increase, but the introduction of a large amount of coal gangue will reduce viscosity, and the width of the corresponding melting temperature range will also decrease; There are many refractory high melting point substances in the chemical composition of fly ash, so the more fly ash is added, the less likely it is to melt, and the corresponding melting temperature range width will also be correspondingly increased.

4. Conclusion

The preparation of wall insulation materials based on fly ash-coal gangue-phosphogypsum has excellent sintering performance, while the proportion of potassium feldspar sodium feldspar composite flux and the amount of coal gangue added have a significant impact on the melting temperature of wall insulation material samples. With the increase in coal gangue content, the initial melting temperature, spheroidization temperature, and passivation temperature of the sample decreased sharply. With the increase of potassium feldspar: albite, the melting temperature range will be significantly increased. When the additional amount of fly ash is 50%, the additional amount of coal gangue is 12%, the additional amount of phosphogypsum is 5%, and the ratio of potassium feldspar to albite is 1:1, the sintering temperature of the fly ash coal gangue phosphogypsum based wall insulation material is the lowest.

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