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A STUDY ON SINTERING PROCESS OPTIMIZATION OF ALUMINA ATTRACTION FROM FLY ASH

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Abstract

Fly ash is an industrial by-product generated during the combustion of coal in power plant. In Inner-Mongolia, the content of Al2O3 in fly ash can reach as high as 50wt%. In this paper, alumina was attracted through soda-sintering process, the work was based on six sigma approach in order to optimize the sintering variables, analysis of various critical process parameters and the interaction among them was carried out with response surface method- logy (RSM), the optimized parameters obtained using RSM were then tested to meet the predicted parameters.

1. Introduction

Fly ash (FA) is an industrial by-product generated during the combustion of coal in power plant [1].With China's economy developing rapidly, FA is constantly increasing accumulation. According to the report released by resources comprehensive utilization society of China in May 2012, the country fly ash emissions has reached 540 Million tons in the end of 2011 [2], and the amount of FA will increase every year.

The utilization of fly ash has attracted many researchers' intentions [3-9],in this paper, a novel process was developed to extract alumina from the FA, the sintering parameters and the interaction among them was analyzed with response surface method- logy (RSM).

2. Experiment

2.1 Materials

The materials used in the this study are desilicated fly ash (DSFA) that is the residue generated from FA by pre-desilication process. The FA collected from the "Guohua" Power Plant,which located in Erdos, Inner-Mongolia. Analytical grade reagents: NaOH (Sinopharm Chemical Reagent CO., Ltd.), CaO(Tianjin Kemiou Chemical Reagent Co., Ltd.), Soda (Sinopharm Chemical Reagent CO., Ltd.)

The composition of DSFA is listed in Table 1. the content Al2O3 is up to 57.20% and SiO2 is 25.42% after desilication, and the Al/Si ratio increased to 2.25.

Table 1 Chemical Composition of DSFA (wt.%)

SiO_2	Al_2O_3	TiO_2	TFe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	LOI	Tot
25.42	57.20	1.97	1.51	0.013	0.437	2.67	4.19	0.09	0.13	5.93	99.56

The XRD pattern of DSFA is shown in Fig. 1. It indicates that the phases and minerals found in DSFA include corundum and mullite.

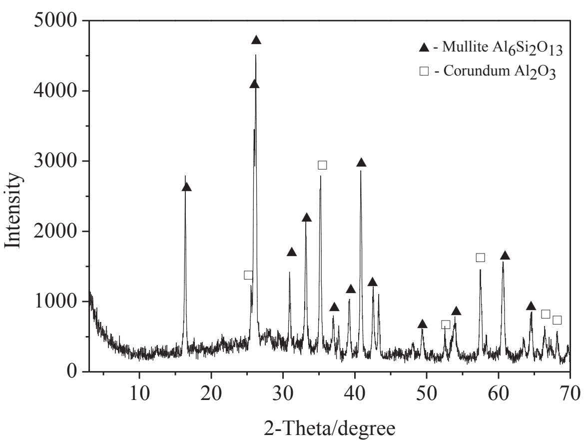

Fig.1 XRD pattern of DSFA

2.2 Experimental Procedure

The response surface methodology (RSM) has been implemented to optimize the sintering parameters. In the research, four main process parameters were studied in the sintering process, Ca ratio, Na ratio, sintering temperature and reaction time. Ca ratio and Na ratio are defined as follows:

$$\text{Ca ratio} = \frac{[\mathrm{CaO}]}{[\mathrm{SiO_2}]+[\mathrm{TiO_2}]} \quad (1)$$

$$\text{Na ratio} = \frac{[\mathrm{Na_2O}]+[\mathrm{K_2O}]}{[\mathrm{Al_2O_3}]+[\mathrm{Fe_2O_3}]+[\mathrm{SiO_2}]} \quad (2)$$

In order to understand the alumina extraction effect of sintering, analysis using thermo-gravimetric and differential scanning calorimetric analysis (TG-DSC) during the sintering process was performed. The clinkers sintered under different temperatures were obtained by XRD diffraction.

The alumina extraction rate is configured as a response, Ca ratio, Na ratio, sintering temperature, and reaction time are configured as response factors. Table 2 depicts the corresponding levels.

Table 2 Sintering process parameters

Ca ratio	Na ratio	Sintering Temperature/℃	Reaction time/min
0.95-1.05	0.95-1.05	950-1250	30-90

3. Results and discussion

3.1 Experimental Results

The DSC result indicated that there were two reactions occurred between 600 ℃ and 1200 ℃, the mass of reaction decrease from 650℃, and became stable at 1100℃.

The XRD spectra of sintered mixture as shown in fig. 3 indicated that the mullite was decomposed, and new phases of $NaAlO_2$ and Na_2CaSiO_4 were formed, and the $NaAlO_2$ was soluble.

The following reactions are proposed according to above discussion:

$$3Al_6Si2O_{13} + 3Na_2CO_3 = 2NaAlSiO_4 + 4NaAlO_2 + 3CO_2\uparrow \quad (3)$$

$$Al_2O_3 + Na_2CO_3 = 2NaAlO_2 + CO_2\uparrow \quad (4)$$

$$Na_2CO_3 + CaO + NaAlSiO_4 = Na_2CaSiO_4 + NaAlO_2 + CO_2\uparrow \quad (5)$$

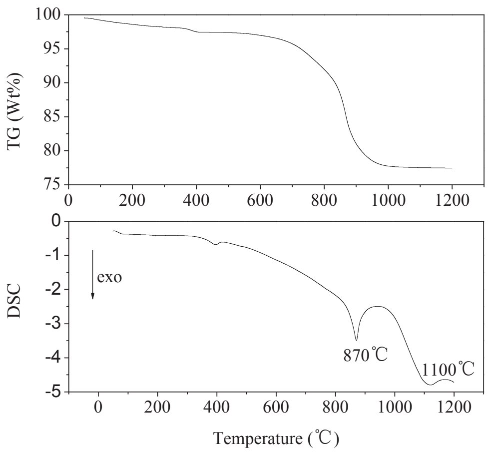

Fig. 2 TG-DSC curves of delisilicated FA, soda and CaO

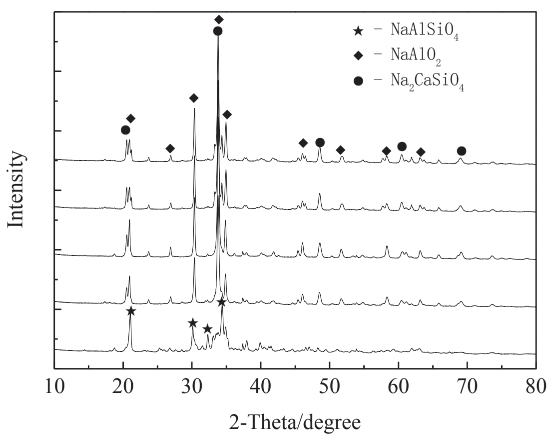

Fig. 3 XRD spectra of sintered mixture

The experimental results are given in the table 3.

Table 3 the results of experiment

No.	Na Ratio	Ca Ratio	Temperature (℃)	Time (min)	Alumina extraction rate(%)
1	1.05	1.05	950	90	94.99
2	0.95	0.95	950	90	93.43
3	1.05	0.95	950	30	92.95
4	0.95	1.05	950	30	92.38
5	1.00	1.00	1100	60	96.71
6	1.00	1.00	1100	60	95.89
7	0.95	0.95	1250	30	93.76
8	0.95	1.05	1250	90	92.66
9	1.05	0.95	1250	90	94.94
10	1.05	1.05	1250	30	93.46
11	0.95	0.95	950	30	92.10
12	0.95	1.05	950	90	94.00
13	1.05	1.05	950	30	93.66
14	1.05	0.95	950	90	95.27
15	1.00	1.00	1100	60	95.54
16	1.00	1.00	1100	60	96.78
17	0.95	1.05	1250	30	95.40
18	0.95	0.95	1250	90	92.71
19	1.05	0.95	1250	30	95.32
20	1.05	1.05	1250	90	94.90
21	1.00	1.00	950	60	94.44
22	1.00	0.95	1100	60	96.03
23	1.00	1.05	1100	60	96.06
24	1.00	1.00	1100	60	96.94
25	1.05	1.00	1100	60	96.58
26	1.00	1.00	1100	60	96.89
27	1.00	1.00	1100	90	96.78
28	1.00	1.00	1100	30	95.91
29	0.95	1.00	1100	60	96.12
30	1.00	1.00	1250	60	96.99

3.2 Analysis of Model

The model of alumina extraction rate and various critical process parameters was fitted with response surface methodology (RSM). model can be obtained in the equation 3. The adjusted R2 for fitted regression line is 0.79. It indicates that the linear metamodel provides a good fit over the ranges of process parameters.

$$Y_{Al2O3}=81.52+10.57Na+1.11Ca+0.002563T+0.008778t-452.2(Ca-1)^2-0.000065(T-1100)^2+0.2313(Na-1)*((t-60)-0.00012(T1100)*((t-60) \quad (4)$$

3.3 Contour Analysis

In order to determine the optimal response point and the variables impact on the alumina extraction rate, the contour analysis is developed with the contour profiling.

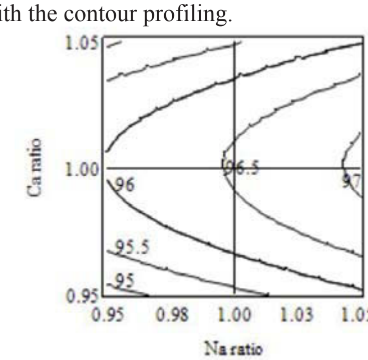

Fig.4 contour of Na ratio and Ca ratio

1) The impact of Na ratio and Ca ratio on the alumina extraction rate was researched, as shown in fig.4, fixing the sintering temperature of 1100℃ and the reaction time of 60 min. The main effect of the Na ratio and Ca ratio changed from 0.95 to 1.05 was given in Fig. 2. With the increase of Na ratio, the extraction of alumina increased, and with the increase of Ca ratio, the extraction of alumina increased initially and reached a top point, then decreased with further increase of Ca ratio.

2) Fig. 5 showed the contour of sintering temperature and reaction time on the alumina extraction rate, fixing the Na ratio and Ca ratio at 1.00. The main effect of the sintering temperature changed from 950℃ to 1250℃ and reaction time changed from 30min to90min. It was indicated that with the increase of sintering temperature, the extraction of alumina increased initially and reached a top point, then decreased with further increase of sintering temperature. The extraction of alumina increased with reaction time.

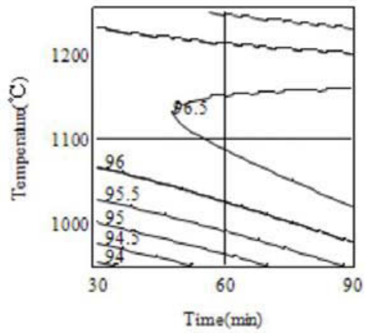

Fig.5 Contour of sintering temperature and reaction time

3.4 Predicting and Validation Experiments

The maximum of the alumina extraction rate was predicted with factor profiler, as shown in the fig. 6, the maximum of alumina extraction rate is 97.68%, under the optimized sintering conditions: Na ratio=1.05, Ca ratio=1, sintering temperature =1100 ℃, and reaction time=90 min.

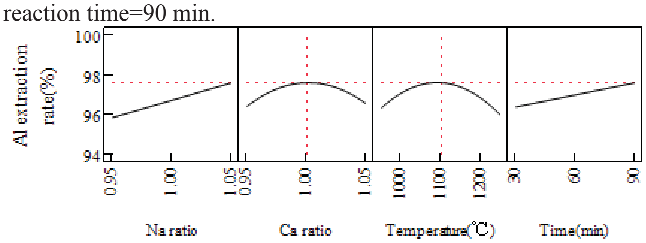

Fig. 6 Optimized parameters of the maximum alumina extraction rate

In order to verify the predicted results, two validation experiments were conducted, as shown in the table 4, the results of validation experiments were very close to the predicting. It indicated that the fixed model matched with the sintering process.

Table 4 Results of validation experiments

Na Ratio	Ca Ratio	Temperature(℃)	Time (min)	Alumina Extraction Rate(%)
1.05	1	1100	90	97.63
1.05	1	1100	90	97.67

4. Conclusions

The mechanisms of sintering process was studied, the mullite decomposed during the sintering process, sodium aluminate and sodium calcium silicate were formed.

With the response surface methodology, the results obtained by the CCD design was analyzed and a fitting model was established. The contour analysis showed that sintering parameters played different roles on the alumina extraction rate. And optimal sintering conditions were obtained by factor profiling.

The maximum of alumina extraction rate was predicted and verified. The alumina recovery rate could reach to as high as 97.68%, under the operation conditions: Na ratio=1.05, Ca ratio=1, sintering temperature =1100 ℃, and reaction time=90 min.

Acknowledgements

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