

# Reaction Mechanism and Kinetic Analysis of the Decomposition of Phosphogypsum via a Solid-State Reaction

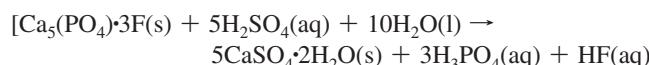
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Phosphogypsum decomposition is very complex because of its complicated compounds. In this study, using high-sulfur-concentration coal as a reducer, thermogravimetric analysis, differential thermal analysis, scanning electron microscopy, and X-ray diffraction were used to characterize the decomposition process of phosphogypsum in a nitrogen atmosphere at different conditions. Mechanism analysis and experiment results showed that the amount of the intermediate production of CaS depended on the heating rate and the size of coal used, which was produced mainly through the reaction between  $\text{CaSO}_4$  and C, and production would decrease with an increase of the coal size. 60 mesh was a suitable size of coal for phosphogypsum decomposition to get high recovery of the main production of  $\text{CaO}$ . Using Kissinger, Flynn–Wall–Ozawa, and Coats–Redfem methods, the mechanism model of phosphogypsum decomposition was confirmed, the decomposition process belonged to the core-form and growing mechanism ( $n = 2$ ), and the kinetic model was  $d\alpha/dt = 1.6 \times 10^{16} e^{-475.99 \times 10^3/RT} \times 2(1 - \alpha)[-ln(1 - \alpha)]^{1/2}$ , where  $\alpha$  is conversion.

## 1. Introduction

Phosphogypsum is the gypsum formed as a byproduct of the processing of phosphate ore into fertilizer with sulfuric acid using a one- (dihydrate) or two-step (hemihydrate–dihydrate) process.



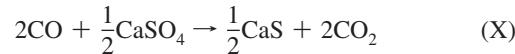
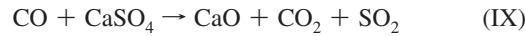
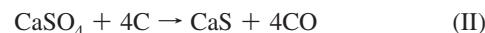
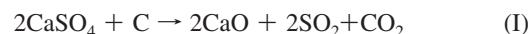
Over 30 million tons of phosphogypsum is produced per annum in China, and less than 10% has been reused.<sup>1,2</sup> By the end of 2005, the stock pile of phosphogypsum was almost 100 Mtons,<sup>3</sup> which caused various environmental and storage problems and had become a urgent task for the government to reuse or recycle this solid waste for the purpose of environment protection. The main components in phosphogypsum are  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (calcium sulfate dihydrate) or  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$  (calcium sulfate hemihydrate) and  $\text{CaSO}_4$  (calcium sulfate anhydrite). One of the recycling methods for this solid waste is thermal decomposition to recycle the sulfur source and produce cement with the residual solid material.<sup>4–6</sup> Phosphogypsum decomposition has been studied under various experimental conditions, such as using reducing gases CO,  $\text{H}_2\text{S}$ ,  $\text{H}_2$ , and  $\text{CS}_2$  and reacting with different types of carbon such as coke, coal, charcoal, etc.<sup>7,8</sup> However, there is still a debate on the precise mechanism and on the elementary step of controlling the decomposition. The nature of the solid and gaseous products varies depending on the reaction conditions and reducing agents, making the process difficult to control.<sup>9–13</sup>

In this study, the kinetic characteristics of phosphogypsum decomposition were analyzed via thermogravimetric analysis (TGA) and a tube reactor by heating the mixture with high-sulfur-concentration coal at elevated temperatures in a nitrogen atmosphere. The purpose of this study is to elucidate the reaction mechanism and reaction kinetics of the decomposition of phosphogypsum in a solid-state reaction. The solid raw and production material were examined using scanning electron

microscopy (SEM) and X-ray diffraction (XRD) analysis to deduce the decomposition reaction process and mechanism. A kinetic model was proposed, and experimental data were adopted to validate the reaction mechanism and kinetic analysis.

## 2. Mechanism Analysis and Kinetic Model

The main components in phosphogypsum are  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (calcium sulfate dihydrate) or  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$  (calcium sulfate hemihydrate) and  $\text{CaSO}_4$  (calcium sulfate anhydrite). The following reactions could take place in a reduction environment:



Besides these, there are many components contained in phosphogypsum (Table 1). Because of its complex components, the reaction process is uncertain<sup>14,15</sup> and depends on the reaction conditions and atmosphere. It is very important to clearly state the decomposition of the reaction mechanism for its design in industry use.

According to the nonisothermal dynamic analysis of solid-state reactions, the basic model for solid decomposition could be expressed as follows:<sup>16</sup>

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**Table 1.** Chemical Components of Phosphogypsum

	composition							
	SO <sub>3</sub>	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	total P <sub>2</sub> O <sub>5</sub>	hydrotropic P <sub>2</sub> O <sub>5</sub>
content (%)	40.86	29.82	9.43	0.236	0.132	0.055	1.17	0.87
	composition							
	total F	hydrotropic F	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	free water	crystal water	acid-insoluble material
content (%)	0.52	0.12	0.043	0.086	0.002	5.38	4.27	8.42

$$\frac{d\alpha}{dt} = Ae^{-E/RT}f(\alpha) \quad (1)$$

where  $\alpha$  is the conversion of phosphogypsum,  $t$  is time,  $A$  is a constant,  $E$  is the reaction active energy, and  $R$  is the gas constant.

The reaction active energy could be calculated by the Kissinger<sup>16</sup> (eq 2) and Flynn–Wall–Ozawa (FWO)<sup>17</sup> (eq 3) multiheating rate methods, respectively.

$$\ln(\beta/T^2) = \ln(AR/E) - E/RT \quad (2)$$

$$\log \beta = \log[AE/RG(\alpha)] - 0.4567E/RT - 2.315 \quad (3)$$

where  $\beta$  is the rate of temperature increase and  $G(\alpha)$  is defined as follows:

$$G(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} \quad (4)$$

Therefore,  $f(\alpha)$  is the differential form of the reaction mechanism function, and  $G(\alpha)$  is the integral form.

The advantage of the FWO method is that the active energy  $E$  could be calculated directly without using the reaction mechanism function. It could be used to test the results calculated by the Kissinger method.

In order to confirm the mechanism function  $G(\alpha)$ , a constant heating rate method was used:

$$\ln[G(\alpha)/T^2] = \ln[AR/\beta E(1 - 2RT/E)] - E/RT \quad (5)$$

If the mechanism function is fitted,  $\ln[G(\alpha)/T^2]$  is linear with  $1/T$  and  $G(\alpha)$  will be the most probable mechanism function. Combined with the active energy  $E$  calculated by the Kissinger and FWO methods, the most probable mechanism function could be confirmed.

### 3. Experimental Section

**3.1. Sample Prepared.** The phosphogypsum sample used in this study came from Yunnan Natural Gas and Chemical Engineering Company; the compounds are listed in Table 1. After drying and filtration, the size of the phosphogypsum

sample was about 0.074 mm. SEM analysis of the phosphogypsum sample is shown in Figure 1, from which it is clear to see the sliced crystal structure of phosphogypsum. The high-sulfur-concentration coal used in the experiment came from Yunnan Chuxiong; the compounds are listed in Table 2. The coal sample also was broken and filtered before experiments.

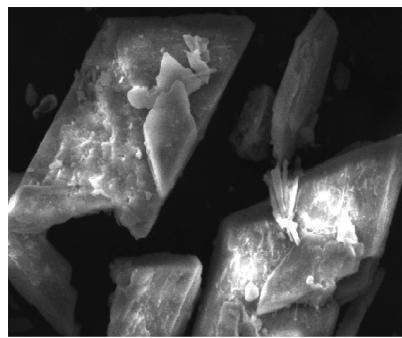
**3.2. Experiment Equipment.** The experiment and analysis equipment used in this study are as follows: XXWRT-2C thermogravimetric analyzer (Beijing Optician Plant); KM9106 complex fuel gas analyzer (KANE Company); D/max-3BPEX-P96 powder X-ray diffractometer (Japan); SK<sub>2</sub> tube resistance stove (Tianjing Zhonghuan Experiment Stove Company, Ltd.).

**3.3. TGA.** TGA was performed on a XXWRT-2C thermogravimetric analyzer. The phosphogypsum sample and coal were mixed in definite proportions and used for kinetic analysis with masses between 10 and 20 mg. Nitrogen was used as the dynamic atmosphere, with a flow rate of 10 mL/min at different heating rates  $\beta$  (5, 10, 15, 20, and 40 °C/min, respectively).

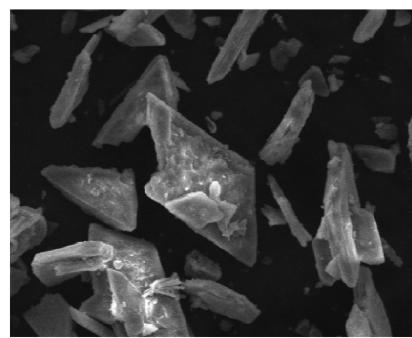
**3.4. Experiments in a Tube Reactor.** A tube reactor (SK<sub>2</sub> tube resistance stove) was used to analyze the decomposition process of phosphogypsum mixed with definite sizes of high-sulfur-concentration coal (200, 160, 100, and 60 mesh, respectively), and data were collected to test the reaction mechanism. The reactor was a quartz tube with 25 cm diameter. The sample was placed in a little porcelain boat; when the temperature was increased about 800 °C, the boat was placed in the middle of reactor. The output gas was analyzed using a KM9106 fuel gas analyzer online.

### 4. Results and Discussion

**4.1. Effect of the Proportions of Phosphogypsum and Coal.** As mentioned in the prework, the proportions of phosphogypsum and coal affect the decomposition reaction,<sup>18</sup> and the mole proportion of C:CaSO<sub>4</sub> = 0.5–1.0 is suitable for the decomposition of phosphogypsum. Figure 2 shows the TGA curve of phosphogypsum mixed with coal at different mass proportions. The mass loss observed between 120 and 130 °C is due to the loss of the hydrated water of phosphogypsum, and the small mass loss between 750 and 800 °C occurs because of

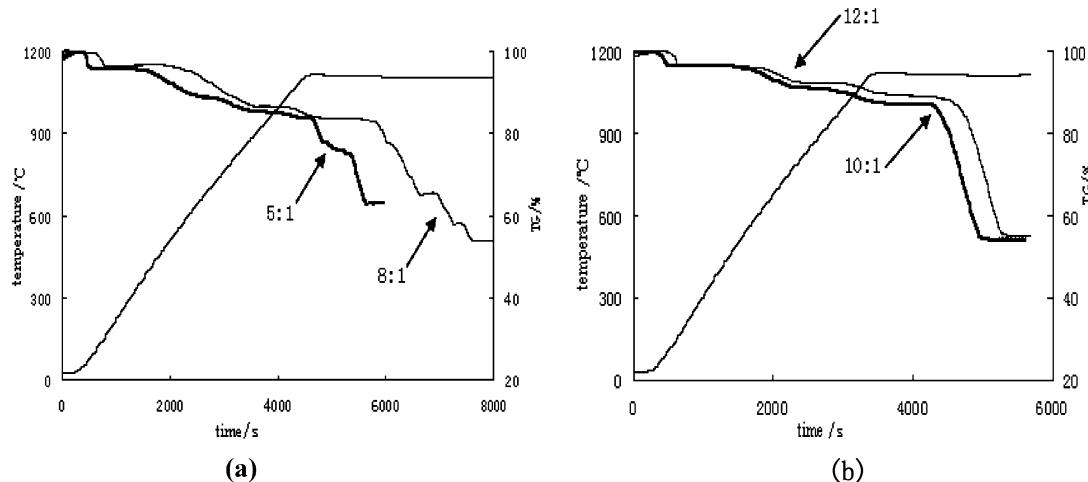


(a) 2000



(b) 1000

**Figure 1.** Phosphogypsum microstructures at different times.



**Figure 2.** TGA and DSC curves of decomposition at different C:CaSO<sub>4</sub> proportions.

**Table 2. Elementary Components of the Coal Sample**

C <sup>r</sup>	H <sup>r</sup>	O <sup>r</sup>	N <sup>r</sup>	S <sup>r</sup>
71.17	3.43	2.73	1.56	1.62

**Table 3. TGA Results for the Reaction between Coal and Different Calcium Sulfate Compounds**

phosphogypsum:coal (mass)	beginning time of decomposition (s)	decomposition time (s)	phosphogypsum mass loss (mass %)
5:1	4600	1100	22.2
8:1	5900	1800	29.2
10:1	4300	800	40.1
12:1	4500	900	34.7

**Table 4. Calculated Results of Δ<sup>r</sup>G and Δ<sup>r</sup>H of Reactions**

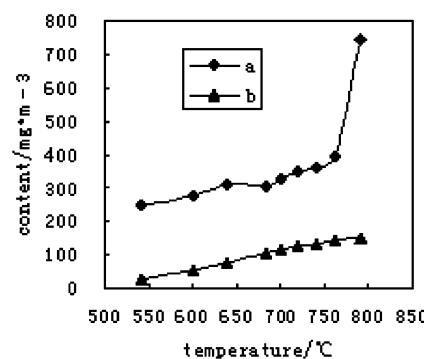
reaction	T (°C)	Δ <sup>r</sup> G (kJ/mol)	Δ <sup>r</sup> H (kJ/mol)
I	900	-26.540	581.000
II	500	-34.385	513.280
III	1200	-14.064	955.949
IV	1500	219.570	331.093
V	300	-155.658	-352.047
VI	300	-300.810	-298.089
IX	1000	-12.879	202.944
X	300	-175.506	-174.127

oxidation of some of the carbon in the mixture. From 950 to 1200 °C, the TGA curve is constantly decreasing because of the decomposition of CaSO<sub>4</sub>. It is clear that the large mass loss appeared at different times, and it increased with an increase of the C:CaSO<sub>4</sub> proportion. The reaction time and mass loss percentages are listed in Table 3.

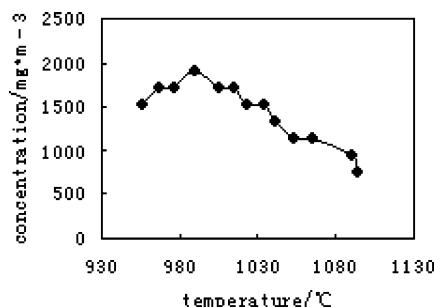
TGA shows that when the mass proportion of phosphogypsum:coal is 10:1, the decomposition is almost complete; therefore, this proportion was chosen as the reactant in the next experiments.

**4.2. Reaction Mechanism Analysis.** The decomposition reaction of phosphogypsum is very complex because of its complicated reaction system. Researchers have given different conjectures,<sup>19–21</sup> and until now, there was no integrated systemic explanation for this process. According to thermodynamic analysis,<sup>18</sup> the beginning temperature of the possible reactions taking place are listed in Table 4. From Figure 2, it is noted that a large mass loss appears at 1000–1150 °C, which means that the main reactions take place in this temperature range.

According to thermodynamic calculations (Table 4), the main reaction I could take place at 900 °C and reaction II could take place at 500 °C. If these reactions took place, there would be a

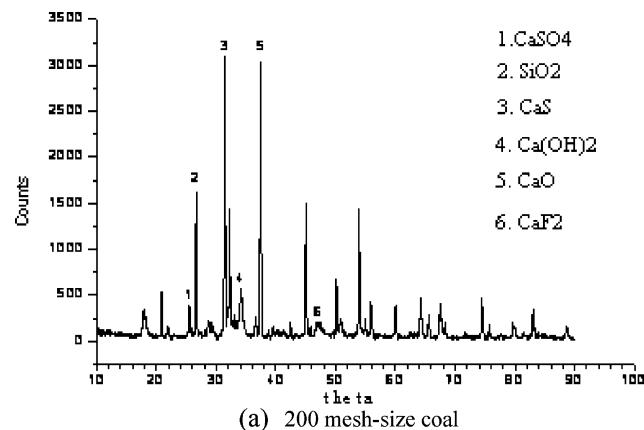


**Figure 3.** Gas production concentration change with the reaction temperature: (a) SO<sub>2</sub>; (b) CO.

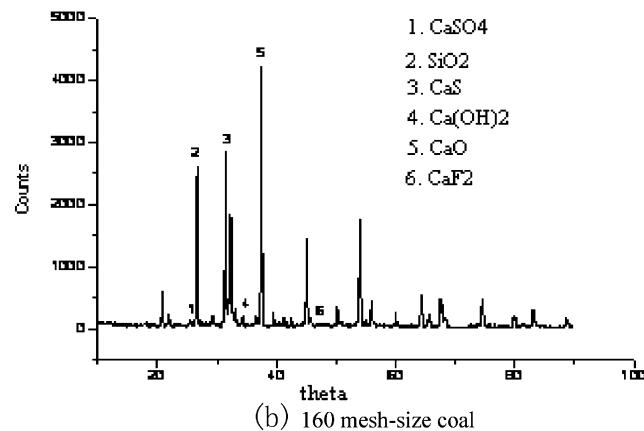


**Figure 4.** Concentration change of SO<sub>2</sub> with the reaction temperature.

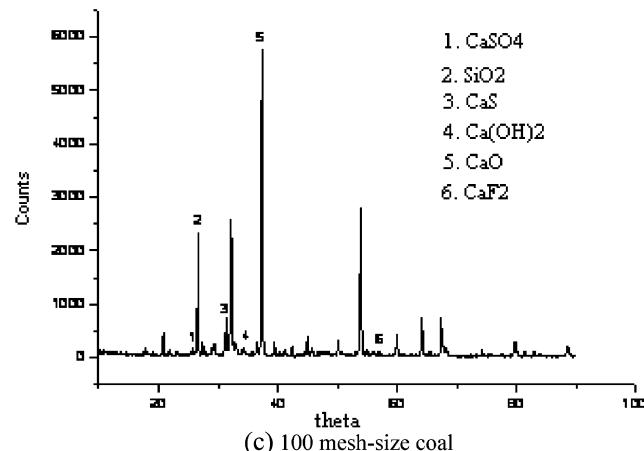
mass loss in the TGA curve. However, there is only a little mass loss between 500 and 850 °C (Figure 2). Except for reaction IV, mass loss would take place from reaction I to reaction X at high temperature (>900 °C). When this is compared with the results by a tube reactor experiment in a N<sub>2</sub> atmosphere (Figure 3), it can be seen that the concentrations of gas productions of SO<sub>2</sub> and CO changed with temperature. The concentrations of both gases are slowly increased with an increase of the temperature; when the temperature was higher than 750 °C, the concentration of SO<sub>2</sub> increased abruptly, while the concentration of CO changed mildly, which indicated that at lower temperature reactions II, V–VII, and X may take place and reactions VI and VII could not take place because of the inert atmosphere. With an increase of the temperature, the decomposition of CaSO<sub>4</sub> began (reaction I), and then the reaction between CaSO<sub>4</sub> and intermediate production of CaS (reaction III) took place. When Figures 2 and 3 are compared, it is clear



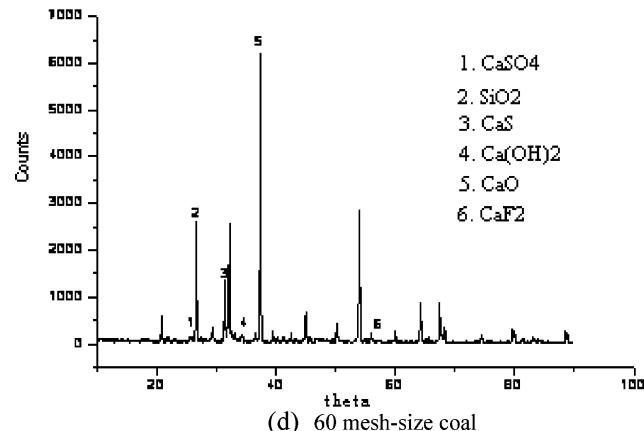
(a) 200 mesh-size coal



(b) 160 mesh-size coal



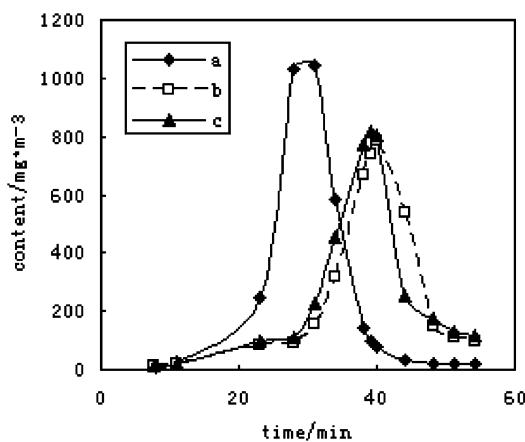
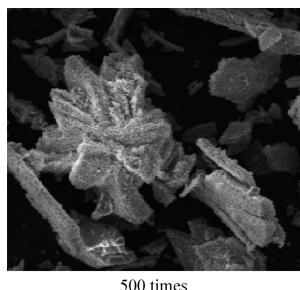
(c) 100 mesh-size coal



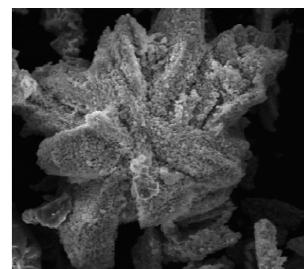
(d) 60 mesh-size coal

**Figure 5.** XRD characterization of the solid production.

that the decomposition temperature is lower than that of theoretical calculations (Table 4); this may be due to the

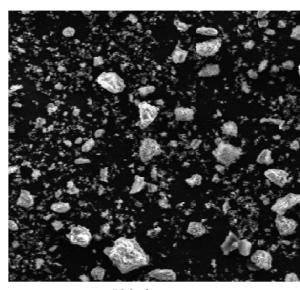
**Figure 6.** Concentration change of CO with the reaction time at different coal sizes: (a) 200 mesh; (b) 100 mesh; (c) 60 mesh.

500 times

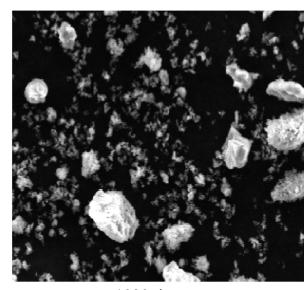


1000 times

(a) 200 mesh number

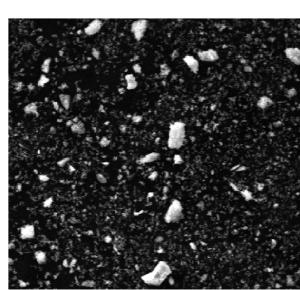


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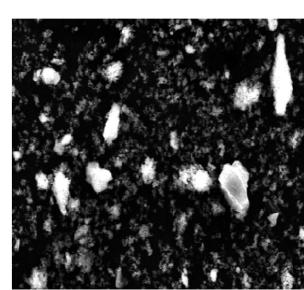


1000 times

(b) 100 mesh number



500 times



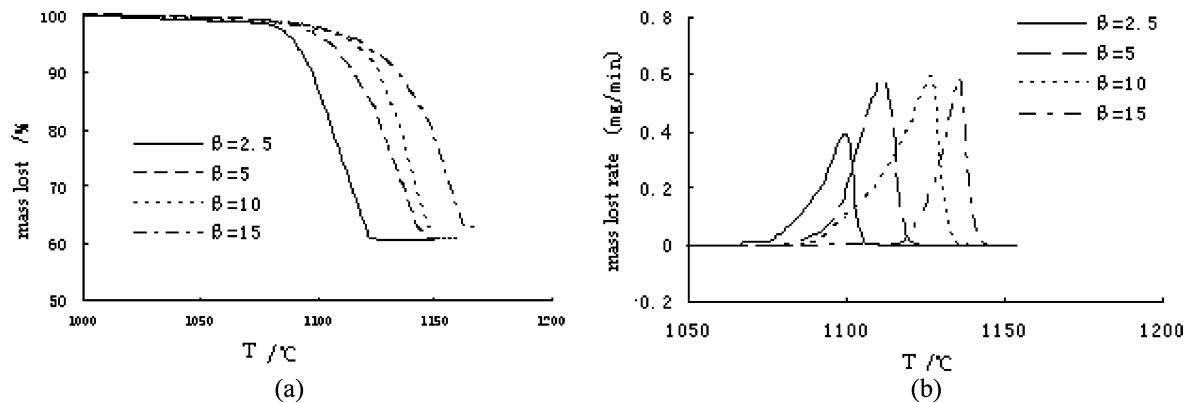
1000 times

(c) 60 mesh number

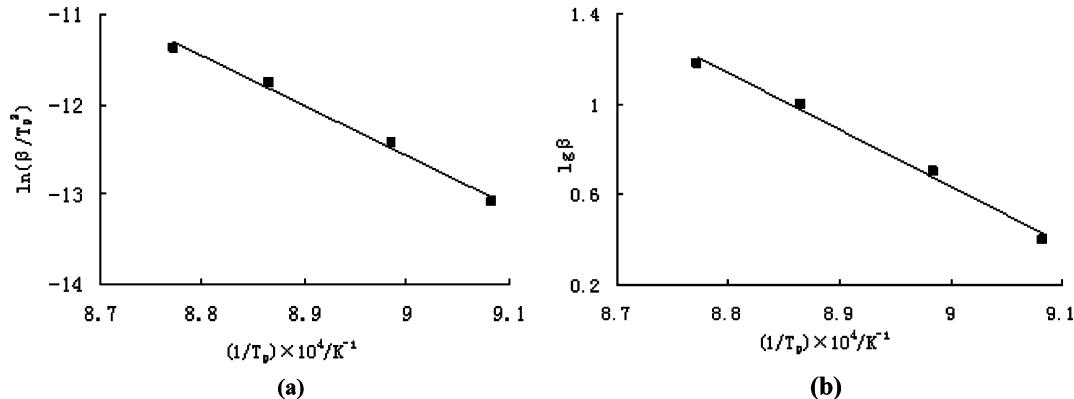
**Figure 7.** Microstructures of solid production at different coal sizes.

impurity compounds contained in phosphogypsum. From reactions IV and V, there will be a production of S; however, reaction IV will not take place at this temperature range according to thermodynamic analysis, and from XRD analysis of the decomposition production (Figure 5), the elementary S could not be found in the production; therefore, the reaction rate of reaction V is very slow at this condition.

In order to further improve the reaction mechanism, CO was used as the reduction reactant instead of coal in the experiment in a tube reactor; the results are shown in Figure 4. It is clear



**Figure 8.** TGA (a) and DTG (b) curves of phosphogypsum decomposition with coal at different heating rates  $\beta$ .



**Figure 9.**  $\ln \beta/T_p^2 - 1/T_p$  of the Kissinger method (a) and  $\log \beta - 1/T_p$  of the FWO method.

**Table 5. Active Energies and Linearities Calculated with Different Mechanism Functions**

$G(\alpha)$	$\beta = 2.5 \text{ } ^\circ\text{C}/\text{min}$		$\beta = 5 \text{ } ^\circ\text{C}/\text{min}$		$\beta = 10 \text{ } ^\circ\text{C}/\text{min}$		$\beta = 15 \text{ } ^\circ\text{C}/\text{min}$	
	$E \text{ (kJ/mol)}$	$r$	$E \text{ (kJ/mol)}$	$r$	$E \text{ (kJ/mol)}$	$r$	$E \text{ (kJ/mol)}$	$r$
$\alpha^2$	1410.55	0.9668	1403.24	0.9734	1396.33	0.9694	1387.11	0.956
$(1 - \alpha) \ln(1 - \alpha) + \alpha$	1561.37	0.9787	1551.72	0.9826	1545.49	0.9804	1538.34	0.9708
$[1 - (1 - \alpha)^{1/3}]^2$	1849.34	0.9893	1829.08	0.9853	1823.76	0.9844	1825.09	0.9852
$-\ln(1 - \alpha)$	971.91	0.9912	963.68	0.9907	962.01	0.9933	961.43	0.9917
$[-\ln(1 - \alpha)]^{1/2}$	479.81	0.9909	475.56	0.9913	474.56	0.9939	474.01	0.9915
$[-\ln(1 - \alpha)]^{1/3}$	312.19	0.9923	308.67	0.9886	308.11	0.9924	308.11	0.9928
$1 - (1 - \alpha)^{1/2}$	820.76	0.9823	813.52	0.9827	811.80	0.9847	809.15	0.9778
$1 - (1 - \alpha)^{1/3}$	913.13	0.9892	902.73	0.9838	899.9	0.9837	900.82	0.9854
$(1 - \alpha)^{-1} - 1$	1358.42	0.9736	1346.35	0.9693	1346.70	0.9786	1351.94	0.9865
$(1 - \alpha)^{-1/2} - 1$	1168.78	0.9888	1155.65	0.9835	1156.56	0.9911	1159.22	0.9953

that, with an increase of the reaction temperature, the production through  $\text{SO}_2$  decreased, which means that the reaction rate of reaction X is faster than that of reaction IX and an amount of  $\text{CaS}$  is produced.

**4.3. XRD–SEM Analysis for the Decomposition Production.** The decomposition production at different coal sizes was analyzed with XRD and is shown in Figure 5. With an increase of the coal size, the compound intensity of  $\text{CaSO}_4$  and  $\text{CaS}$  decreased distinctly and the production of  $\text{CaO}$  increased, which further explained the reaction process analyzed above. A high decomposition recovery at a defined size of coal can be obtained; 60 mesh is suitable for decreasing the intermediate production of  $\text{CaS}$  and increasing the production of  $\text{CaO}$ , while a smaller coal size may helpful for gasification of C to CO and then produced  $\text{CaS}$  (reaction X). The change of the concentration of CO at different coal sizes further attested to this phenomenon (Figure 6).

Figure 7 shows the SEM analysis for the decomposition production from above experiment with different coal sizes.

At 200 mesh size of coal, the crystal structure is floral and coarse on the surface and many intermediate productions such as  $\text{CaS}$  exist, while a small satin particle appears when the coal size is 60 mesh. Therefore, 60 mesh is suitable for phosphogypsum decomposition to obtain the main production of  $\text{CaO}$ .

**4.4. Kinetic Model.** In order to confirm the mechanism function, the TGA data (Figure 8) obtained from different heating rates were used for calculations in eq 5 with different mechanism functions; when the linearities of  $\ln[G(\alpha)/T^2] - 1/T$  are compared and the best linearity mechanism function is chosen as the most probable model for this process, the active energy of the reaction could be calculated. The results are listed

**Table 6. Active Energies Calculated by a Mechanism Function**

	$\beta \text{ (}^\circ\text{C}/\text{min})$				
	2.5	5	10	15	average
$E \text{ (kJ/mol)}$	479.81	475.56	474.56	474.01	475.99
$\ln(A/\text{s}^{-1})$	38.70	37.57	36.75	36.22	37.31

in Tables 5 and 6. From the results, the differential form of the suitable mechanism function could be

$$f(\alpha) = 2(1 - \alpha)[-ln(1 - \alpha)]^{1/2} \quad (6)$$

and the integral form is

$$G(\alpha) = [-ln(1 - \alpha)]^{1/2} \quad (7)$$

The active energies calculated with the Kissinger and FWO methods are shown in Figure 9. It is clear that the results are consistently calculated with these two methods, which means that this mechanism function is suitable for explaining the process of phosphogypsum decomposition. The kinetic equation for phosphogypsum decomposition is as follows:

$$\frac{d\alpha}{dt} = 1.6 \times 10^{16} e^{-475.99 \times 10^3 / RT} \times 2(1 - \alpha)[-ln(1 - \alpha)]^{1/2} \quad (8)$$

## 5. Conclusion

Phosphogypsum decomposition is very complex because of its complicated compounds. On the basis of TGA experiments and XRD analysis, the reaction process and mechanism of phosphogypsum decomposition have been studied. The intermediate production of CaS formed depends on the heating rate and the size of coal used, which was produced mainly through a reaction between  $CaSO_4$  and C, and it will decrease with an increase of the coal size. 60 mesh is a suitable size of coal for phosphogypsum decomposition to get high recovery of the main production of  $CaO$ .

The reaction kinetic model of phosphogypsum decomposition in a solid-state reaction was researched based on a chosen mechanism function. Using the Kissinger, FWO, and Coats-Redfem methods, the mechanism model of phosphogypsum decomposition was confirmed; the decomposition process belongs to the core-form and growing mechanism ( $n = 2$ ) and the kinetic model is  $d\alpha/dt = 1.6 \times 10^{16} e^{-475.99 \times 10^3 / RT} \times 2(1 - \alpha)[-ln(1 - \alpha)]^{1/2}$ .

## Appendix

### Nomenclature

A = constant

E = reaction active energy

$f(\alpha)$  = differential form of the reaction mechanism function

$G(\alpha)$  = integral form of the reaction mechanism function

R = gas constant

T = temperature of the reaction, °C

t = time, s

### Greek Letters

$\alpha$  = conversion of the reactant

$\beta$  = rate of temperature increase, °C/min

## Acknowledgment

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