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Role of extrusion process on kinetic of carbothermal reduction of barite

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ABSTRACT

In present study, the effect of extrusion process on kinetic of carbothermal reduction of barite was investigated. In order to improve the gasification rate potassium carbonate was doped on coke as catalyst. The mixture of barite and coke powder was shaped by a laboratory extruder and the reduction process was isothermally carried out at four temperatures, ranging 850–1000 °C. The effect of extrusion process on conversion of barite was evaluated by iodometry method. Also, a modified kinetic model was used to analyze the conversion data. The experimental results revealed that the extrusion process can effectively promote the rate constant both in catalytic and non-catalytic runs. It was proved that the rate of reaction is controlled by active site density in the presence of potassium carbonate and the extrusion process reduces activation energy approximately, 20 kJ/mol. Finally the reduction time was optimized to achieve the maximum conversion in the absence and presence of catalyst.

1. Introduction

Barium compounds such as $BaCO_3$, $BaCl_2$ and $Ba(NO_3)_2$ are the most important materials in chemical, ceramic and oil industries [1]. These materials widely used in manufacturing bricks, glass and pigments [1]. The manufacture of barium chemicals was estimated to be 14% of barites consumption in 2005 [2]. Barite ore is the natural starting material for producing various barium components. Currently, there are two main methods for manufacturing barium components and the reduction process is based on two-steps [3]. Firstly, barite ore is reduced by coke or methane, leading to produce barium sulfide as intermediate material in rotary kiln or fluidized bed reactor as follows [4,5]:

$$BaSO_4 + 4C \rightarrow BaS + 4CO \quad (1)$$

$$BaSO_4 + CH_4 \rightarrow BaS + CO_2 + 2H_2O \quad (2)$$

In the second step, the barium components are subsequently produced from barium sulfide solution by the addition of acids or alkali materials [3]. However, the reduction of barite by coke which is called as black ash process, presents important problems such as high consumption of coke, impurity of barium sulfide and high residence time in the kiln. In addition, production of BaS by this procedure involves environmental drawbacks and it is energy-intensive consuming. The development of manufacturing technology in the reduction process is aimed to finding more efficient and cost effective ways to increase product content. An assessment on the role of different processing steps has shown that the compaction of sample plays effective role on reduction [6]. The later cost can be significantly reduced through the increase of reduction rate, which produces acceptable amount of product.

The second industrial method for BaS production is the carbothermal reduction in the presence of catalyst. In this case $BaSO_4$ reacts with carbon monoxide as following reaction [7,8]:

$$BaSO_4 + 4CO \rightarrow BaS + 4CO_2 \quad (3)$$

The generated CO diffuses back into coke, therefore the content of CO increases according to Boudouard reaction:

$$C + CO_2 \rightarrow 2CO \quad (4)$$

The major role of catalyst is to promote the rate of gasification reaction. The formation of CO improves the barite reduction rate, therefore the importance of this process depends on gasification rate. Concerning the catalysts used in gasification of coke, it is well known that alkali metal affects the gasification rate [9,10]. After the first report on the reduction of barite in the presence of Na_2CO_3 and $Fe(NO_3)_3$ by Jagtap et al. [11], these materials have been found to be excellent catalysts for improving reduction rate. These catalysts should be doped on active charcoal powder in order to increase the number of active sites and to achieve a maximum conversion in short time in industrial application [9,10].

Gokarn et al. [12] studied the effect of sodium vanadate on gasification of active charcoal and reduction of barite. They reported compensation behavior for first time. The calculated isokinetic temperature provided some important information relating to the observed anomalous behavior of catalytic carbothermal reduction of barite at high temperature. Salem and Tavakkoli Osgouei [13] were reported the suitable particle size distribution of barite for

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Table 1
Chemical and mineralogical analysis of barite ore.

Chemical analysis								
SiO_2	BaO	Na_2O	K_2O	MgO	CaO	Al_2O_3	Fe_2O_3	SO_3
3.58	59.00	0.01	0.01	0.01	0.29	0.62	1.03	34.12

Mineralogical analysis calculated by chemical analysis		
Barite	Quartz	Others
89.62	3.58	6.80

using in reduction process. They presented that the pre-grinding of barite (−230, +400 mesh) allows reaching the best results in the presence of sodium carbonate due to modification in active sites.

The reduction of barite by coke powder presents important environmental and safety drawbacks since it is moved by gas flow and produce large quantity of harmful dust. For the improvement of industrial application in continuous or semi-continuous kilns, barite and coke powder can be shaped in the form of granules, spheres and extrudates in order to achieve a convenient mechanical strength against hot gas flow. In general, an extruded material is manufactured from a paste, which is previously obtained by blending the powders with the following components: (a) an organic material such as coke which provides the mechanical strength, (b) a liquid phase such as water or ethanol to effectively lubricate the moving of paste during the extrusion process, (c) organic additions which increase the viscosity of the paste in order to get a suitable plasticity during extrusion [14].

In spite of some investigations that have been published about the synthesis and characteristics of barium components, almost no information can be found in literature about the effect of shaping method on reduction process. However, this process is an essential step regarding its commercial application. In this context, the first aim of the investigation has focused on development, a process for improving reduction rate. In order to achieve the most suitable catalyst composition the mixture of ferric nitrate, potassium carbonate and sodium carbonate were applied and the optimum composition was determined by mixture design method. In the second step of the study, the effect of extrusion process on catalytic and non-catalytic reduction of barite was discussed by using a modified kinetic model.

2. Experimental

2.1. Preparation of samples

The mixture design method configures a special case in response surface methodologies using mathematical and statistical techniques [15]. The basic assumption is that there is a given mixture property which depends solely on the fractions, X_i (for example catalyst fraction), of specific components in the mixture. Thus, the change in the properties, for example conversion of barite, is entirely determined by proportions of those components. In order to achieve the optimum composition, it is necessary to select firstly the appropriate mixtures from which the response surface might be calculated. A prediction of conversion can be obtained for any mixture from the change in the proportions of catalysts (K_2CO_3, Na_2CO_3 and $Fe(NO_3)_3$ in this investigation).

In a system with s components, there are $s-1$ independent composition variables, X_i, and the geometric description of the factor space containing s components consists of all points on inside or the boundaries of a regular $s-1$ dimensional simplex. The response function (conversion in this study, x) can be expressed in its canonical form as a polynomial equation typically, first or second degree as follows [15]:

$$x=\sum_{i=1}^{s}\beta X_i \qquad (5)$$

$$x=\sum_{i=1}^{s}\beta X_i+\sum_{i<j}^{s}\beta_{ij}X_iX_j \qquad (6)$$

These polynomial equations have to be evaluated over a number of points, N. These equations can represent the response surface over the entire region. Then a laboratory study consisting of N experiments ($N>s$) should be carried out and the conversion of barite on those selected points should be evaluated. A regression equation such as Eq. (5) or (6) is then fitted to those experimental values and the model is considered valid only when the differences between the experimental and calculated values are near to zero with acceptable variance.

An industrial barite powder was used as the starting raw material in present study. Chemical composition of selected barite was determined by X-ray method that is reported in Table 1. The mineralogical analysis of used barite ore indicated barite ($BaSO_4$) and quartz as main minerals, and the amounts of crystals are also reported in Table 1. The used catalysts were ferric nitrate, potassium carbonate and sodium carbonate. An industrial coke was selected to use in reduction process whose chemical analysis is reported in Table 2. In order to evaluate the carbothermal reduction fine-sized barite (−63 μm) was used. The ternary catalytic compositions prepared according to the mixture design algorithm (Fig. 1) were doped on coke based on its dry weight. The maximum amount of catalyst was considered 5 wt.% for each of them. The catalytic compositions doped on coke were added to barite powder and mixed for 2 h.

2.2. Preparation of extruded samples

The extrusion process employed in this study is based on the generation of a homogenous pastes comprised of following constituents. For the preparation of paste, the starting powders (barite + coke) were mixed with distilled water (34.3 wt.%, water/dry barite mass). Then, the pastes were left for 24 h to obtain homogenous materials. The pastes were fed into laboratory extruder (pro to type) to shape the cylindrical samples at a rate of 60 cm/min. Approximately 2 wt.% of paste humidity was removed during extrusion process. The samples were dried at 60 °C for 24 h and cut in suitable dimensions to prepare discs with the same weight.

Table 2
Specifications of used coke.

Materials	Percentage	Particle size distribution (mesh)	Percentage
Fixed carbon	70	+16	0
Ash	10	−16, +140	20
Volatile matter	16	−140	80
Moisture	4		

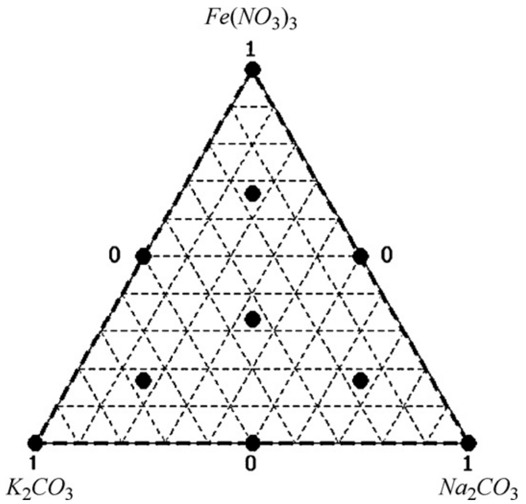

Fig. 1. Simple design plot for the ternary system of K_2CO_3–Na_2CO_3–$Fe(NO_3)_3$.

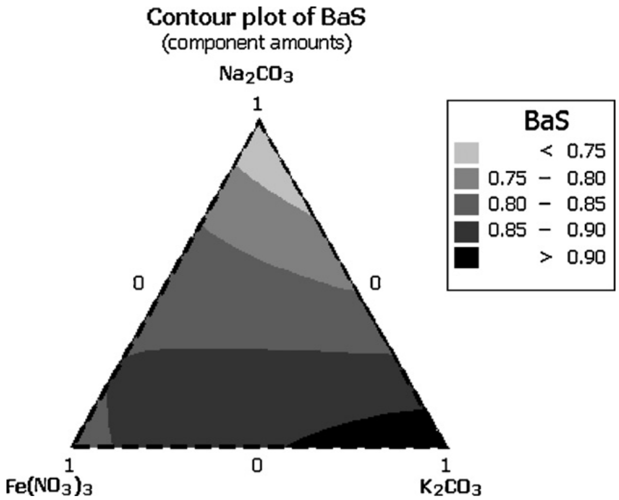

Fig. 2. The response surface plot for conversion of barite as a function of ternary catalytic composition.

2.3. Evaluation of reduction process

The carbothermal reduction of samples in the form of powder or disc was carried out in laboratory electrical kiln (Azar Furnace Company, Model F11L, 1500, Iran) under isothermal condition at 850, 900, 950 and 1000 °C in air atmosphere. The zero point of time scale was considered as starting time at which the samples enter the kiln. The cooled samples were weighed and barium sulfide percentage was measured by iodometry method. The procedure for determining BaS content is as follows [16]:

(i) The iodine and hydrochloric acid solution was prepared by mixing 50 mL of 0.5 N iodine solution and 50 mL of 1 N hydrochloric acid solution.
(ii) 2 g of black ash powder was mixed with 100 mL deionized hot water and stirred to prevent agglomeration and effective dissolution.
(iii) The black ash suspension was added into the iodine and hydrochloric acid solution and stirred for 3 min.
(iv) The solution was titrated with 0.1 N sodium thiosulfate to obtain straw color in the presence of starch indicator. The BaS percentage was calculated by the following equation:

$$C = 8.17\left(\frac{N_1V_1 - N_2V_2}{W}\right) \tag{7}$$

where C is the BaS percentage, N_1V_1 and N_2V_2 are total mole of iodine and sodium thiosulfate solution, respectively. W is the initial weight of sample. The fractional conversion of $BaSO_4$, x, was calculated according to the stoichiometric reaction presented in Eq. (1).

3. Results and discussion

3.1. The effect of catalyst on reduction process

The effect of combination ratio of catalysts on conversion of barite powder to barium sulfide at 950 °C, holding for 45 min in kiln is indicated in Fig. 2. It shows that the conversion increases significantly with increment of K_2CO_3 content. Conversion was also found to decrease with the addition of Na_2CO_3 content in catalytic composition indicating the weak effect of catalyst on gasification of coke. Maximum conversion is observed in the presence of 5 wt.% K_2CO_3. Using the measured conversion presented in Fig. 2, the following equation can be suggested for conversion as a function of catalytic composition. This model was found to be statistically most adequate with regression coefficient of 98.9%:

$$\begin{aligned} x = {} & 0.73X_1 + 0.83X_2 + 0.93X_3 + 0.24X_1X_2 - 0.12X_1X_3 - 0.04X_2X_3 \\ & + 1.11X_1X_2X_3 - 2.03X_1X_2(X_1 - X_2)^2 - 2.03X_1X_3(X_1 - X_3)^2 \\ & + 2.41X_2X_3(X_2 - X_3)^2 \end{aligned} \tag{8}$$

where X_1, X_2 and X_3 are the weight fractions of Na_2CO_3, $Fe(NO_3)_3$ and K_2CO_3, respectively. The response surface is graphical presentation of Eq. (8) and allows easy and rapid prediction over the entire composition range under investigation. Fig. 2 indicates that reduction of barite improves with increment of K_2CO_3 amount and the mixing of catalysts does not efficiently affect reduction process. Also potassium carbonate is the catalyst with effective performance in improving conversion. Therefore, the experiments which lead to the construction plots of conversion versus time were carried out in the presence and absence of K_2CO_3 to elaborate the mechanism of reduction process and to estimate the kinetic parameters.

The conversion–time plots shown in Figs. 3 and 4 refer the carbothermal reduction of barite powder at four temperatures in the absence and presence of K_2CO_3. All curves are identical in nature and are characterized by exponential behavior. This behavior of

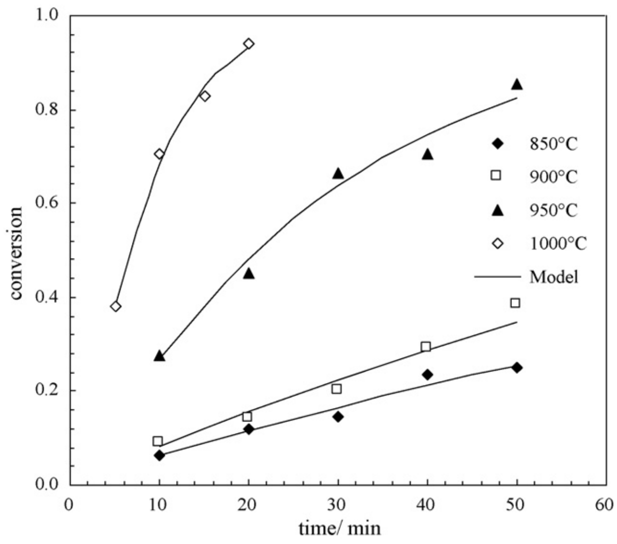

Fig. 3. Conversion–time plots for the reduction of barite powder in the absence of catalyst.

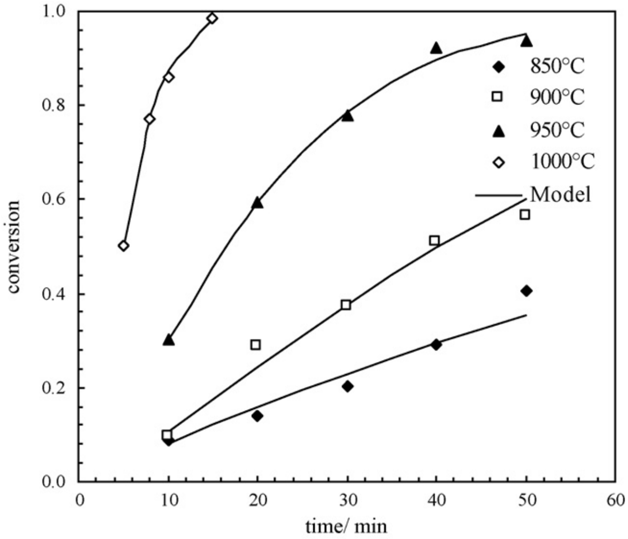

Fig. 4. Conversion–time plots for the reduction of barite powder in the presence of K_2CO_3.

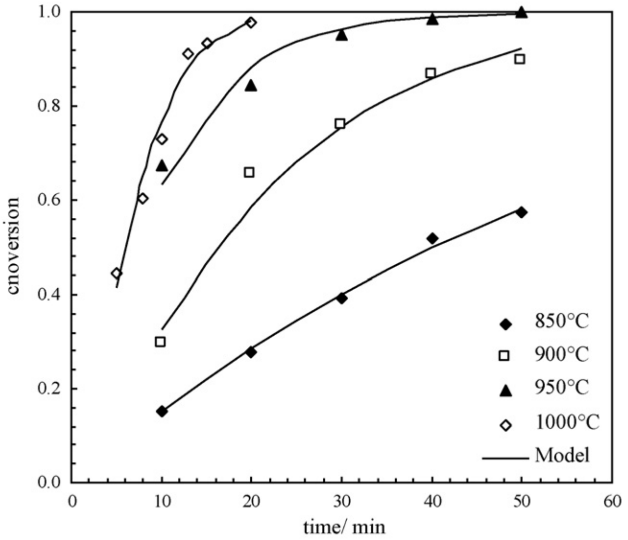

Fig. 5. Conversion–time plots for the reduction of extruded samples in the absence of catalyst.

plots is generally observed in isothermal reduction of minerals [11]. In the present study, the observed exponential behavior can be attributed to gasification of coke at high temperatures. The obtained results show that conversion increases as reduction time rises. The increase in conversion is the result of gasification of coke. As the reduction progresses, the BaS amount grows and barite completely was converted to barium sulfide. There is a clear increment in reduction rate with increasing temperature and a remarkable improvement in reduction rate is observed at above 950 °C.

The plot of Fig. 4 illustrates the reduction behavior of barite powder in the presence of 5 wt.% K_2CO_3. The tests were conducted at isothermal condition to compare the results with those obtained in the absence of K_2CO_3. The increase in reduction rate at low temperatures is due to promoting gasification rate. The district changes in the curves are evident at 1000 °C, where the conversion reaches maximum value of 0.99. These observations are also supported by the results of previous investigation [11]. The rapid increment in conversion continued at initial reduction times in the absence and presence of K_2CO_3, beyond which a rapid gasification is observed due to diffusing CO_2 into the coke and generating more CO. As the temperature rises, the reduction rate increases considerably compared to that in the absence of catalyst, in which finally conversion reaches maximum value during 15 min at 1000 °C.

The effect of extrusion process on carbothermal reduction of barite in the absence and presence of K_2CO_3 was demonstrated in Figs. 5 and 6. The conversion–time plots indicate the rate of reduction at four temperatures. For both cases a remarkable increase in reduction rate is observed at the same conditions. Hence potassium carbonate can reduce the reduction temperature, which is 50 °C lower than the temperature required for the composition prepared without catalyst.

The reduction rate of extruded samples increased significantly in the absence and presence of K_2CO_3 at different temperatures compared to those for barite powder. It is well known that contact area between barite and coke particles would be improved after extrusion process. Therefore, the reduction rate is sensitive to contact surface area and it increases with the increment of contact surface area between the particles due to pressure acting in extrusion process. In this case, the controlling step of solid–gas reaction is related to diffusing CO through the porous media. The diffusion path of reducing agent, CO, becomes longer and more complex compared to the case that powder of barite and coke is used. The improvement of contact surface area between barite and coke results the decrease in back diffusion rate of CO. After extrusion process the gas–solid reaction area is usually divided into the following regions [7]:

(i) diffusion of CO to the surface of barite;
(ii) adsorption of CO on the surface of barite particles;
(iii) reduction of barite;
(iv) desorption of CO from the surface of barite particles;
(v) diffusion of CO away from the barite surface.

Though, the diffusion rate of CO to the barite surface and the diffusion rate of CO away from solid surface seem to be indirectly rate-determining factor but the increase in gasification rate of coke leads to improve the overall reduction rate. It seems that the addition of K_2CO_3 significantly affects reduction rate of extruded samples. It is worth to note that the reduction rate is much affected by K_2CO_3 at 950 °C and is more rapid than that for the reaction between barite and coke in the absence of catalyst.

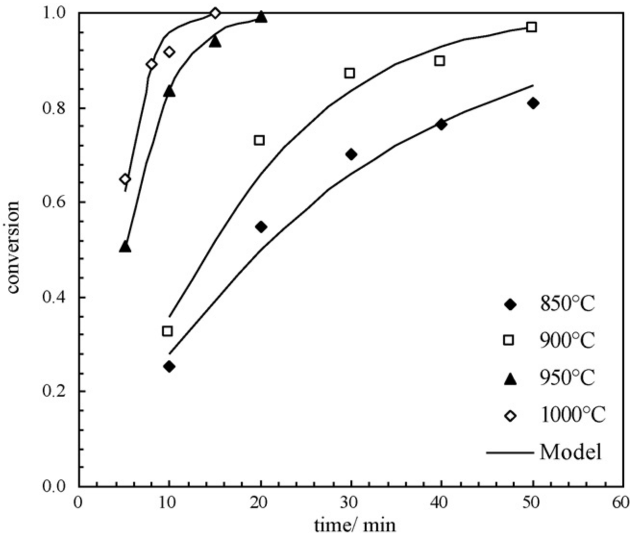

Fig. 6. Conversion–time plots for the reduction of extruded samples in the presence of K_2CO_3.

3.2. Kinetic model of reduction process

In this part of study a mathematical model is described for carbothermal reduction of barite. According to the previous investigation carried out by Kasaoka and Sakata [17], the following modified kinetic model was presented for conversion of barite:

$$x = 1 - \exp(-at^b) \tag{9}$$

where t is reduction time, a and b are the constants of equation. The reduction process of barite is usually carried out at a constant temperature in industrial scale to obtain maximum conversion in short time. In order to obtain the parameters of Eq. (9) and thereby to calculate the conversion in each condition, the kinetic model has been used to correlate conversion–time data. This equation is complex model and depicts structural changes take place in the mixture of samples during the reduction process. Eq. (9) also is used to predict exponential behavior of conversion–time data. Because the reduction of barite occurs by gasification of coke; therefore, the application of this model should be substantiated. Moreover, the model parameter, a, is related to kinetic rate constant and depends on temperature. Parameter b is related to changes that occur in matrix of samples. This parameter is very important since the value of b determines the matrix characteristics. If $b = 1$, the reduction of barite follows first order kinetic. If $b > 1$ the conversion will present the exponential behavior. The model given by Eq. (9) is a general form and it is possible to apply for different shapes of samples (powder or disc) in all temperatures. A linear plot of $\ln[-\ln(1-x)]$ versus $\ln t$ provides the calculation of a and b. Fig. 7 shows the fitting of kinetic model for the reduction of barite powder in the absence of catalyst. It is interesting to note that conversion–time data fit the modified kinetic model with high accuracy. Fig. 7 is a typical example for application of modified kinetic model and it also correlates the other studied cases that were not presented graphically. The constants of a and b were easily calculated and it is possible to calculate the rate constant by using constant parameters. The values of a, b and linear correlation coefficients, r, are reported in Table 3. It is observed that the value of b does not remain constant for each composition.

Table 3
The constant parameters of modified kinetic model for the isothermal reduction of barite.

Temperature (°C)	Non-catalytic reduction			Catalytic reduction		
	a	*B*	*r*[a]	*a*	*b*	*r*
Powders						
850	0.007	0.948	0.987	0.008	1.032	0.980
900	0.008	1.013	0.986	0.006	1.294	0.989
950	0.026	1.075	0.989	0.017	1.321	0.996
1000	0.065	1.248	0.997	0.052	1.603	0.997
Extruded discs						
850	0.015	1.042	0.996	0.027	1.081	0.984
900	0.027	1.157	0.985	0.023	1.282	0.984
950	0.084	1.077	0.984	0.076	1.372	0.996
1000	0.053	1.444	0.988	0.062	1.7112	0.978

[a] Linear regression coefficient.

Because reduction process of barite takes place by gasification of coke, the reduction rate defined in terms of rate constant, $k(x)$, changes continuously with reduction time. The reduction rate can be expressed by the following equation:

$$\frac{dx}{dt} = k(x)(1-x) \tag{10}$$

The rate constant can be calculated by the following expression:

$$k(x) = ba^{1/b}[-\ln(1-x)]^{(b-1)/b} \tag{11}$$

$k(x)$ can be computed at each conversion for example in maximum conversion, $x = 0.98$. Arrhenius plots for studied cases were illustrated in Fig. 8 as follows:

$$k(x) = k_0 \exp\left(-\frac{E_a}{RT}\right) \tag{12}$$

where E_a is the activation energy, k_0 is the frequency factor and R is the constant of ideal gas. The parameters of Arrhenius equation are reported in Table 4. The value of activation energy approximately remains constant and is not affected by the addition of catalyst. Also, this parameter was significantly reduced for samples in the disc shape both in the absence and presence of K_2CO_3. This phenomenon indicates that the mechanism of reduction or structural changes in coke matrix is different for powder and extruded samples but it does not change by the addition of catalyst. The effect of catalyst on activation energy of reduction process was also reported by previous investigations [11].

The value of frequency factor is significantly different for the reduction of barite in the absence and presence of catalyst. It is

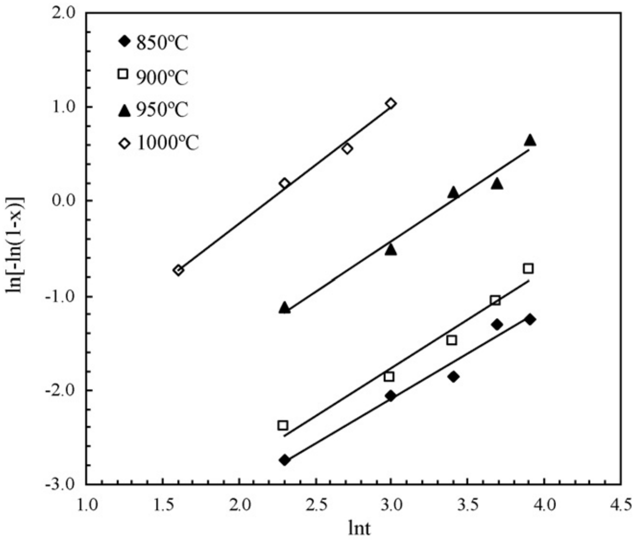

Fig. 7. Application of modified kinetic model for the reduction of barite powder in the absence of catalyst.

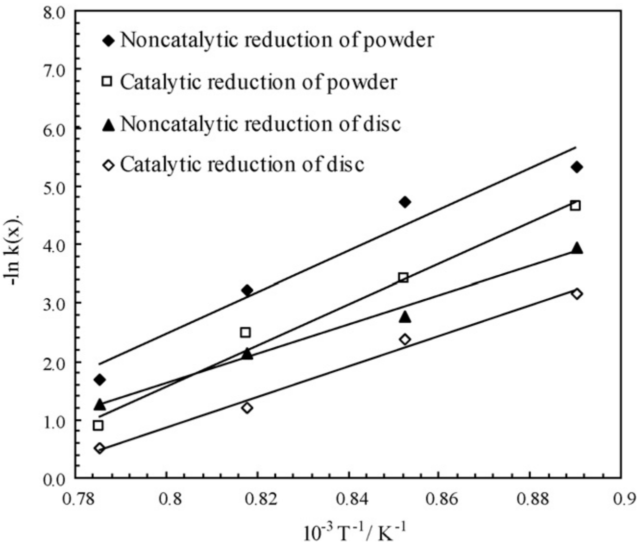

Fig. 8. Arrhenius plots for catalytic and non-catalytic reduction of barite powder and extruded samples.

Table 4
The frequency factor and activation energy of reduction process.

Sample shape	Non-catalytic reduction			Catalytic reduction		
	k_0 (min^{-1})	E_a (kcal/mol)	r	k_0 (min^{-1})	E_a (kcal/mol)	r
Powder	1.5×10^{11}	69.7	0.996	3.0×10^{11}	69.2	0.994
Extruded disc	9.8×10^{7}	49.5	0.978	4.5×10^{8}	51.5	0.992

observed that the value of frequency factor increases if K_2CO_3 is doped on coke. This parameter increases approximately 2 times when catalyst is used in the reduction of barite powder. According to the results reported by Jagtap et al. [11], the pores development in coke matrix involves typically with micropores development during non-catalytic gasification. In the catalytic gasification the pore development is due to micropores development with an enhanced rate due to the presence of catalyst. The values of frequency factors show an overall positive effect of catalyst on number of active sites. The different rates of reaction in the absence and presence of catalyst can be tentatively explained by two factors that control the reduction of barite. One of them is the effect of micropores development that increases the surface area to improve the gasification rate. The second factor acting on reduction rate is the number of active sites in the presence of K_2CO_3. As a matter of fact, the value of frequency factor which is a measure of active site increases when catalyst is added to starting composition.

In the first part of the study it was inferred that the carbothermal reduction of barite is related to the gasification of coke through the Boudouard reaction and intermediate gaseous phase diffusion, CO, is the limiting step in overall reduction. The conversion of barite involves with concentration of CO that is controlled by gasification of coke. Therefore, the micropores development and the increment in number of active sites can affect the reduction rate consequently.

Furthermore, it is obvious that the value of rate constant increases when extrusion process is used in the preparation of samples. To verify the influence of extrusion process on reduction rate, it should be noted that the rate of reduction is proportioned to contact surface area of barite and coke particles. It is evident that the contact surface area increases after the extrusion process. Thus the extrusion process plays a beneficial role in decreasing activation energy accompanied by a corresponding decrease in frequency factor. This is evident from the fact that the value of frequency factor approximately decreases compared to the case in which barite and coke powders were used directly. The trends of kinetic parameters indicate an overall positive effect on enhancement reduction rate.

The conversions of barite calculated by Eq. (9) have been plotted versus those determined experimentally in Fig. 9. In order to compare the experimental data with model prediction, the deviations, D_i, were calculated between experimental points and theoretical values by following equation:

$$D_i = \left[\frac{x_{cal} - x_{exp}}{x_{exp}}\right] \times 100 \qquad (13)$$

where x_{cal} and x_{exp} are the theoretical and experimental values of conversion, respectively. Overall root mean square, rms, was then calculated from deviations:

$$\text{rms} = \left[\sum_{i=1}^{M} \frac{D_i^2}{M}\right]^{1/2} \qquad (14)$$

where M is the total number of data in each studied cases. Very good agreement can be observed between the experimental data and calculated values with acceptable rms reported in Table 5, which substantiates the validity of modified kinetic model.

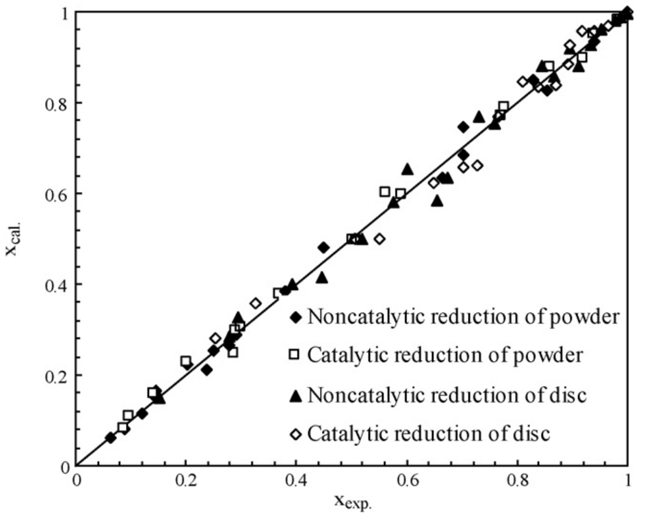

Fig. 9. Statistical evaluation of conversion for the reduction of barite powder and extruded disc in the absence and presence of catalyst.

The following equation was related to the optimum reduction time to temperature:

$$\ln t_{opt} = A + \frac{B}{T} \qquad (15)$$

where t_{opt} is the optimum reduction time to achieve maximum conversion, A and B are the model constants that were reported in Table 5. In order to well understand the validity of model, the reduction time to achieve the maximum conversion of barite, 0.98, was calculated at 850, 900, 950 and 1000 °C.

Fig. 10 indicates the variations of $\ln t_{opt}$ versus inverse temperature in semi-logarithmic scale. The validity of Eq. (15) to obtain optimum reduction time was summarized in Table 6. The

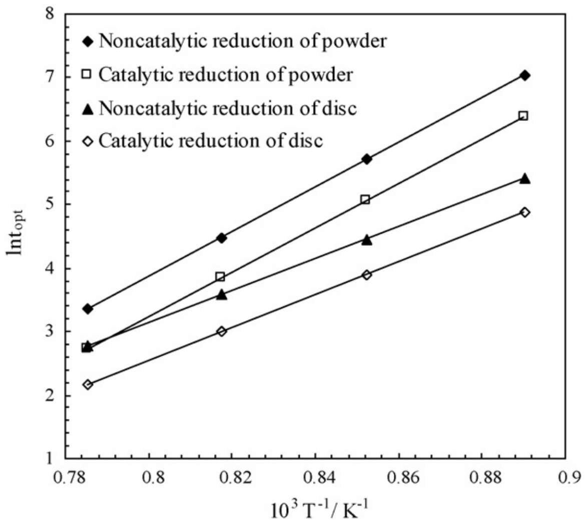

Fig. 10. The variation of optimum reduction time with temperature for catalytic and non-catalytic reduction of barite powder and disc samples.

Table 5
The rms values of barite powder and disc samples used in catalytic and non-catalytic reduction process.

Sample shape	Non-catalytic reduction			Catalytic reduction		
	rms	*A*	*B*	rms	*A*	*B*
Powder	6.6	−24.30	35204	7.1	−24.76	34968
Extruded disc	4.8	−16.87	25013	5.3	−18.26	25996

Table 6
Optimum reduction time corresponding to $x_{opt} = 0.98$ for catalytic and non-catalytic reduction of barite powder and extruded disc.

Temperature (°C)	Non-catalytic reduction			Catalytic reduction		
	t_{opt} (min)	x_{exp}	*D* (%)	t_{opt} (min)	x_{exp}	*D* (%)
Powders						
850	1149			591		
900	302			157		
950	89	0.98	0.0	46	0.94	−4.5
1000	29	0.94	−4.3	15	0.98	0.0
Extruded discs						
850	222			133		
900	86			49	0.97	−1.0
950	36	0.97	−1.0	20	0.99	1.0
1000	16	0.94	−4.3	9	0.92	6.5

agreement between experimental data and model prediction was excellent with deviation less than 6.5%.

4. Conclusions

The use of extrusion process in carbothermal reduction of barite has been successfully developed for application in industrial scale. The improvement in reduction rate was achieved in the presence of potassium carbonate as catalyst. It was found that the reduction rate of extruded samples significantly increases due to increment in contact surface of barite and coke particles during extrusion process. The optimum reduction time to achieve the maximum conversion, 0.98, was calculated by using a mathematical model and the validity of model was substantiated by experimental data.

Acknowledgement

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