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VOLATILIZATION BEHAVIOR OF TIN DURING CARBOTHERMIC REDUCTION OF TIN-BEARING MIDDLING TO RECOVER TIN

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Abstract

A new method for selecting reducing agent was proposed in this study. This study dedicated to recovery of tin from tin-bearing middling by carbothermic reduction. Volatilization behavior of tin during carbothermic reduction was systematically studied, including the tin volatilization thermodynamics and kinetics, the selecting method of reducing agent, the effects of reducing agent, reduction temperature and effect of time on the tin volatilization rate. The thermodynamic results showed that the SnO_2 was reduced to SnO by controlling CO% in the range of 9%~15%, and then volatilized as SnO. The reduction efficiency and the reaction capability with CO_2 of three reducing agent were investigated, which indicated tin volatilization was close in nature to the reductant, and it was beneficial to the volatilization by using the coke powder. The experiment results show that the volatilization ratio of tin can reach more than 81.34% when the coke powder is used as the reducing agent at 900°C for 2h. The kinetic of tin volatilization during the reduction roasting process showed that the reaction was controlled by the interface chemical reaction and the activation energy of the reaction was 44.66 kJ/mol.

Keywords: *Tin; Coke powder; Stannous oxide; Reduction roasting*

1. Introduction

Tin and its oxides are widely used in many fields, such as gas sensors, electrode conductors, liquid crystal displays, packaging containers, electronics industry, transportation, construction and so on [1]. As one of the rare metals in the world, tin content in the crust is 0.004%, and the tin reserves are about 4.8 million tons worldwide [2].Tin resources make more than 2 million tons in China which account for 41.6% of global reserves. With the development of science and technology, the usage amount of tin was also raised which resulted in reducing depletion of tin resources. At the same time, the tin-bearing middling was treated as rubbish that accumulated and caused severe problems of environment pollution. The tin-bearing middling as a second resource plays an important role in utilization of tin. Recovering tin not only solves the problem of resource depletion but also avoids the problem of environment pollution. Therefore, it has a great significance for developing an effective method of tin recovery from tin-bearing middling.

There are many volatilizing methods developed and under development to recovery tin. The volatilization of tin is mainly divided into three types, which include reduction roasting, sulfuration roasting and chlorination roasting. In the chlorination roasting process, the tin-bearing middling phases were transformed to chlorides which evaporated, and the tin volatilization rate was up to 94% [3,4]. In the process of sulfuration roasting, the tin-bearing middling was translated to SnS and the volatilization rate reached 94% [5-7]. The effect of sulfurizing reagent on the experiment is difficult to control because of the impurities in sulfurizing reagent which can also react with the tin-bearing middling. In addition, the corrosion of the facilities in the chlorination roasting process and sulfuration roasting caused the problems of production cost and polluted the environment. In the method of reduction roasting, the tin-bearing middling was translated to SnO and the tin volatilization rate reached 70% [8]. The corrosion of the facilities was successfully avoided and the removal rate of tin was desirable [9, 10].

There are many studies about the reduction process with different reduction reagents, including soild reductive agents (graphite, coke, petroleum coke, semi-coke, anthracite, charcoal, lignite, etc.) and gas reductive agents (CO, H_2, CH_4, etc.) [3,11-15]. The carbonaceous material [3] (charcoal) reduced the SnO_2 to SnO when the temperature was at 925°C ~1000°C with the tin volatilization rate of 68.4%, and the kinetics analysis showed that the reduction activation energy was 323 kJ/mol. Zhang [7] proposed that the Sn-bearing mineral can be reduced by coke

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breeze, anthracite, semi-coke and lignite, and the volatilization rates were 72.54%, 70.98%, 42.54%, 40.36%, respectively. The results of reducing SnO_2 by gas agent (H_2,CO-CO_2) [16,17] indicated that the volatilization rate reached 99% with the reduction activation energy of 62.5kJ/mol at the strict reduction atmosphere. Although the reduction of pure SnO_2 and Sn-bearing concentrate by gas agent could obtain a high tin volatilization rate, the reduction atmosphere and temperature were difficult to control. The above researches indicated that the reduction atmosphere has a significant effect on the reduction of tin-bearing middling.

At present, the coal was the main industrial reduction agent with the advantages of low cost and high reserves which was widely used in rotary kiln reduction process. During the reduction roasting process, the tin was reduced to SnO and Sn by controlling the partial pressure of CO which was provided by combustion solid reducing agent. The previous studies [7,18] investigated that the atmosphere of the CO was the most important factor which affected the reduction of SnO_2 to SnO [9,11,13,19-22]. Further studying of the behavior of tin volatilization by solid reductants could have a great significance for the improvement and optimization of reduction and volatilization of tin-bearing middling.

There are few studies which suggest a suitable method for distinguishing the better reduction agent. In this study, a novel method for selecting reducing agent was proposed. The reaction capability of coal was used to describe the reductive capability. The reduction and volatilization thermodynamics of tin oxides were also analyzed. The volatilization behavior of tin from tin-bearing middling was investigated, focusing on the effect of reductant, reduction temperature, reduction time and the reduction kinetic of tin oxides. These findings provide the technical basis for the utilization of Sn form the Sn-bearing second resource.

2. Experimental and methods

2.1 Raw materials

The raw material used in the experiment was tin-bearing middling from Inner Mongolia. The chemical composition of tin-bearing middling, shown in Table 1, indicated that the tin-bearing middling contained only 3.87% Sn and the main oxides were CaO and SiO_2. The XRD pattern is shown in Fig. 1. It can be seen that the major tin-bearing middling is cassiterite, and other minerals are fluorite and silicate. The reducing agents used in the experiment were anthracite, coke powder, bituminous coal, and the corresponding industrial analyses (by GB/T212-2008) are shown in Table 2. It can be seen that the bituminous had the highest volatiles and the anthracite was in the second position, while the coke powder had the worst volatiles, which corresponded with the strength of reduced atmosphere.

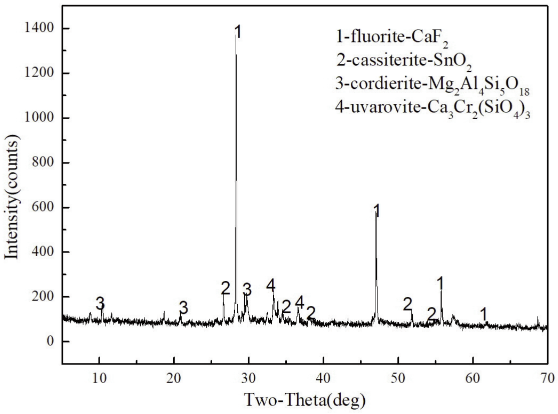

***Figure 1**. XRD pattern of raw material*

***Table 2.** Industrial analysis of reductant /wt%*

Species	F_{cad} (%)	Ad (%)	Vad (%)	Mad (%)
Anthracite	54.83	27.07	14.36	3.74
Coke powder	39.49	40.15	10.2	10.16
Bituminous coal	52.12	4.49	30.41	12.98

2.2 Equipment and methods

The reduction roasting experiments were carried out in a shaft furnace with the $MoSi_2$ heating element and an automatic temperature control system, which was shown in Fig. 2. Firstly, the samples of tin-bearing middling were dried in an oven at 120°C for 4 h. Then the dried samples were crushed to less than 0.045 μm size. Secondly, 2.5g sample was mixed with 8% water and agglomerate into pellets (Φ10× 10mm) with the pressure of 650 N/cm^2. The pellets were dried in an oven at 120°C for 4 h. the next step was to put 20 g reductant into a Φ50 × 200 mm iron crucible, and

***Table 1.** Chemical composition of the tin middling /wt%*

SnO_2	TFe	CaO	SiO_2	MgO	Al_2O_3	Cr_2O_3	S	F	P	Zn	As	Burning loss
4.91	6.87	45.92	20.6	1.54	3.94	1.3	1.84	4.89	0.18	1.33	2.26	6.24

then 8 pellets were put on the reductant covered with 30g reductant. When the temperature reached designed temperature, the iron crucible was located into the high temperature zone of the shaft furnace and then heated for the designed time. The iron crucible was taken out from the shaft furnace and cooled to room temperature as soon as it was possible. The cooled samples were weighed for the chemical analysis of Sn and the mineral phase analysis.

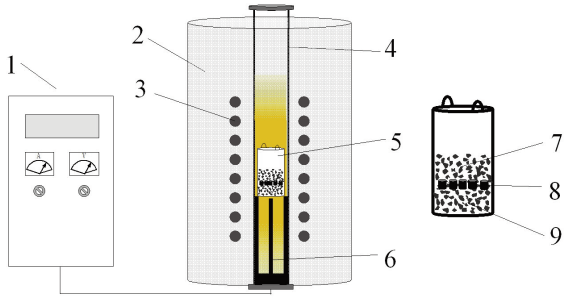

Figure 2*. Schematic diagram of the experimental apparatus for reduction roasting. 1-Constant temperature automatic control box; 2- Resistance furnace outer wall; 3- Silicon molybdenum rod heating element; 4- Corundum tube; 5- Reaction cans; 6- Thermocouple; 7- Upper pulverized coal; 8- Clumps; 9- Lower coal*

2.3 Definition of parameters

The volatilization rate of tin during roasting is calculated using Equation (1).

$$x = (\beta_1 m_1 - \beta_2 m_2) / \beta_1 m_1 \times 100 \tag{1}$$

where: m_1 is the mass of the sample before it is reduced, g; m_2 is the mass of the sample after it is reduced, g; β_1 is the content of tin in the ore, %; β_2 is the percent of Sn in the sample after calcination ; x is the tin volatilization rate (by GB/T15924-2010), wt%.

2.4 Sample analysis

The mineral composition of samples were characterized by X-ray diffraction (Rigaku D/Max 2500; Rigaku Corporation, Tokyo, Japan under the conditions of radiation: Cu Ka, tube current and voltage: 250 mA, 40 kV, scanning range: 10 to 80 (2h), step size: 0.02 (2h), and scanning speed: 8/min.)

The reaction capacity of coals with CO_2 were analyzed by Coal activity detector (SH80-X-1)(shortest maximum furnace temperature of 1500 degrees, the use of furnace temperature of 1200 degrees -1300 degrees; Constant temperature zone: 100 mm Δt<± 10 degrees; Heating rate: 20-25 degrees /minute within 1 hour, the furnace temperature of 1200 degrees -1300 degrees can be reached; Power consumption: 4-5kW; Power supply voltage: 220V ± 20V; Pyrometer: Platinum and rhodium - platinum thermocouple, range 0-1600 degrees accuracy 1.0 level.). The reaction capacity of coals with CO_2 was judged by CO production amount. In the reduction process, the SnO needed a weak reduction atmosphere. According to the CO production amount, a stable reduction agent was chosen for the experiment.

The thermodynamic used in this paper is calculated by the software Factsage7.1 with PS database.

3. Results and discussion

3.1 The Thermodynamic of SnO_2 Reduction Volatile

According to the result of XRD, the major tin phase in the tin-bearing middling is SnO_2. The volatility of tin at redox system is related to the saturated vapor pressure. There are many studies on the reduction of SnO_2 [11,23,24], which showed that the relationship between saturated vapor pressure and temperature is as follows:

$$\lg P_{Sn} = -\frac{15500}{T} - \lg T + 11.5 \tag{2}$$

$$\lg P_{SnO} = -\frac{13160}{T} + 10.775 \tag{3}$$

$$\lg P_{SnO_2} = -\frac{28800}{T} + 16.5 \tag{4}$$

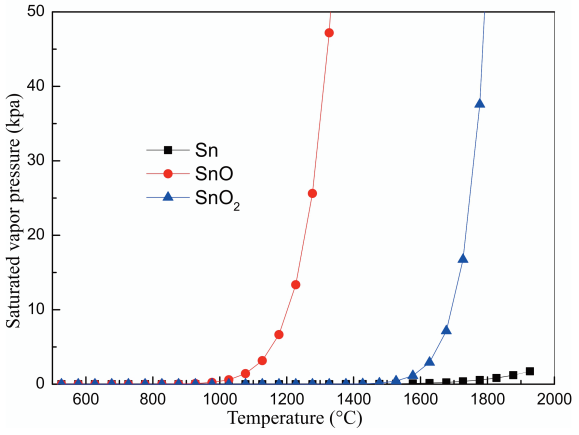

Figure 3*. Saturated vapor pressure versus temperature*

The relationships between the corresponding saturated vapor pressure and temperature are shown in Fig. 3. It can be seen that when the temperature is above 900°C, the vapor pressure of stannous oxide increased rapidly with the increase of the reduction temperature. In order to recover Sn, the tin-bearing middling phase should be controlled in the form of SnO in the reduction system.

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Table 3. *ΔG-T Equations of the chemical reactions [7,20]*

Equation	Chemical Reactions	ΔG°=A+BT(J/mol)		$T_{Reaction}$ [K(°C)](ΔG=0)
		A	B	
(5)	$SnO_2(s) + 2CO(g) = Sn(s) + 2CO_2(g)$	10492	-34.051	581.2(308.1)
(6)	$SnO_2(s) + CO(g) = SnO(s) + CO_2(g)$	3909.2	-15.05	532.8(259.74)
(7)	$SnO(g) + CO = [Sn] + CO_2(g)$	6582.8	-19	619.6(346.4)
(8)	$[Sn] + SnO_2(s) \rightleftarrows 2SnO(g)$	-17729	9.05	1959(1685)

Due to the lower reaction rate between the pellets and coals, the reaction between SnO_2 and CO produced by the Budol reaction of carbon instead of coal was chosen [3,25]. Finally, all reactions of coal and SnO_2 performed in the study were considered to be the reactions of CO with SnO_2. The reactions and the temperatures when ΔG equals 0 are shown in Table 3.

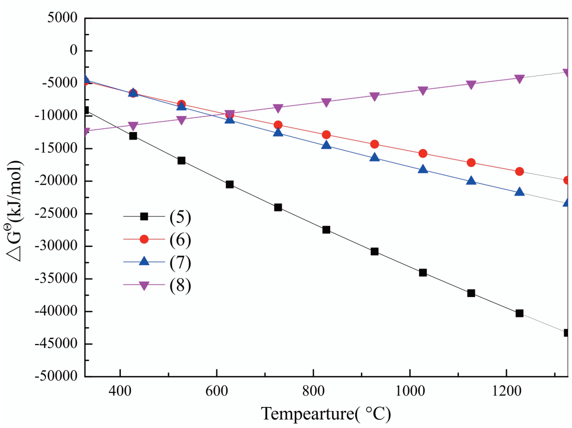

Figure 4. *ΔG-T Relations of reductive roasting*

The ΔG-T relations during the reduction roasting are shown in Fig. 4, which indicated the reactions (5)~(8) occurred in the range of 327°C -1327°C. In general, the reduction of tin oxides by the CO can be seen as the following gradual reduction principle: $SnO_2 \rightarrow SnO \rightarrow Sn$. The reactions (5) and (6) were related by ΔG-T, the sum of the volume fractions of CO and CO_2 in the gas phase was 100% ($nCO+ nCO_2 = 100\%$) when the reduction reaction reached equilibrium. The relationship between the vapor phase composition and the temperature can be obtained by the reactions which are shown in Fig. 5. The result indicated that the reduction equilibrium curves of SnO_2 and SnO were very close and the two curves intersected at 570°C. When the temperature was above 570°C, the stable region of SnO was between the two curves, as shown in the shadow region of Fig. 5.

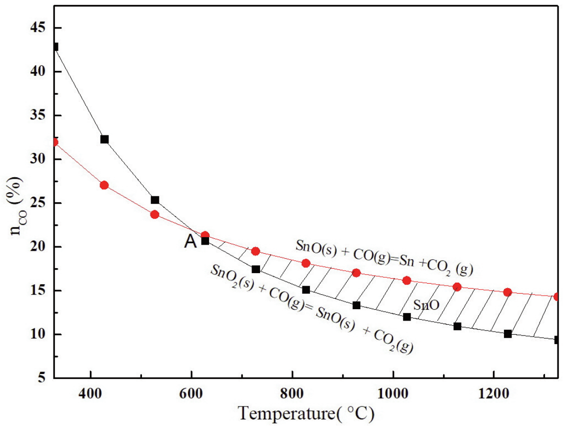

Figure 5. The relationship between contents of CO and the temperature

3.2 Effect of reducing Agent

The reaction capacity of coal can be taken for the reductive capability at high temperature. The reaction capacity of coal with CO_2 is expressed by CO production. Fig.6 shows that the results of reaction capacity of coal with CO_2. The result which is presented in Fig. 6 shows that the anthracite had the best reactivity, which indicates that anthracite has a strong reaction with CO_2 and provides an intensely

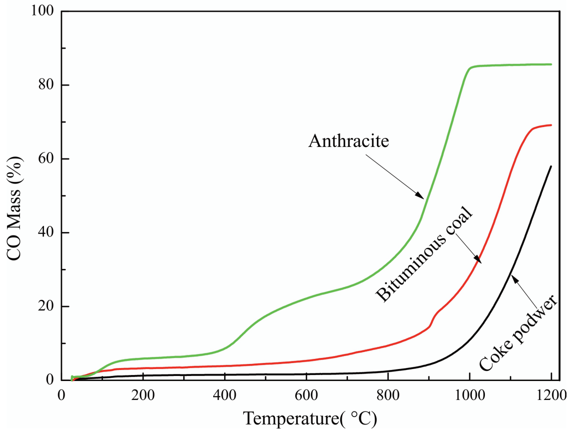

Figure 6. The coal capacity of reaction with CO_2

reduction atmosphere. The reaction capacity of coke powder is worse than that of others. The coke powder provides a weak reduction atmosphere during the reduction process. As it can be seen from Fig. 6, the tin was reduced from SnO_2 to SnO, and the process needed the CO content from 9%~15% and that was a weak reduction process. Therefore, the coke powder was the best reduction agent. Fig. 7 shows the effect of coal type on the tin volatilization rate at 900°C for 2 h. As shown in Fig. 7, the tin volatilization rate reduced by coke powder was higher than that of the other coals. The coke powder is high volatile with low ash content, which caused the coke powder to have the worst reaction capacity between carbon and CO_2. The poor reaction capacity would provide a weak reductive atmosphere during reduction process. Furthermore, it can be seen that the coke powder in the reaction process provided a weak reduction atmosphere which is reasonable to be used in the process of SnO volatilization. Therefore, coke powder was chosen for the following investigations.

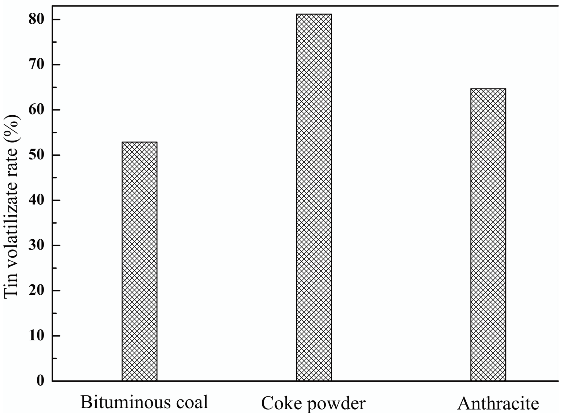

Figure 7. Effect of different coal types on SnO volatilization

3.3 Effect of roasting temperature

The effect of roasting temperature on the volatilization behavior of tin oxide reduced by coke powder was carried out for 2 h, as shown in Fig. 8. It can be seen that the volatilization rate of tin firstly increased and then decreased with the increase of the temperature. The highest volatilization rate of tin was 81.34% at 900°C. The tin volatilization rate increased from 800°C to 900°C due to the higher temperature show by the Reaction (6). The tin volatilization rate decreased when the temperature was higher than 900°C, because the saturated vaporization of stannous oxide increased rapidly after 900°C which made the reverse reaction of Reaction (8) which proceeded to produce metallic tin and tin dioxide.

The XRD patterns of reduced samples under different temperature are shown in Fig. 9. It can be seen that the Sn-bearing mineral phase was SnO_2 at 800°C~900°C and the intensity of SnO_2 peaks decreased with the increase of reduction temperature. The peaks of metallic tin were detected when the reduction temperature exceeded 900°C. The intensity of Sn peaks increased with the increase of reduction temperature, which was consistent with the result of tin volatilization rate at different temperatures. Thus, the optimal temperature was 900 °C.

3.4 Effect of roasting time

In this work, the reduction time of 15min, 30min, 45min, 60min, 75min, 90min, 105min and 120min were chosen for studying the effect of reduction time

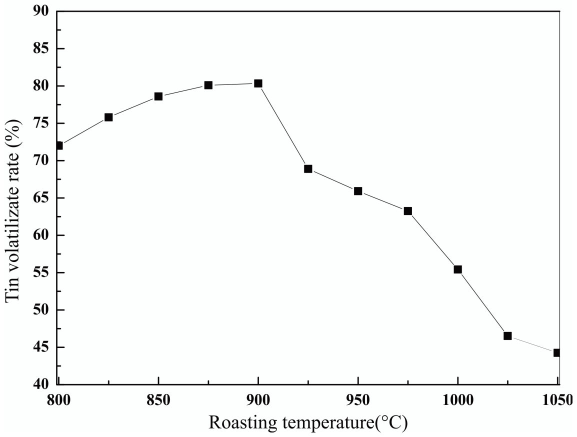

Figure 8. Effect of reduced calcination temperature on tin volatilization results

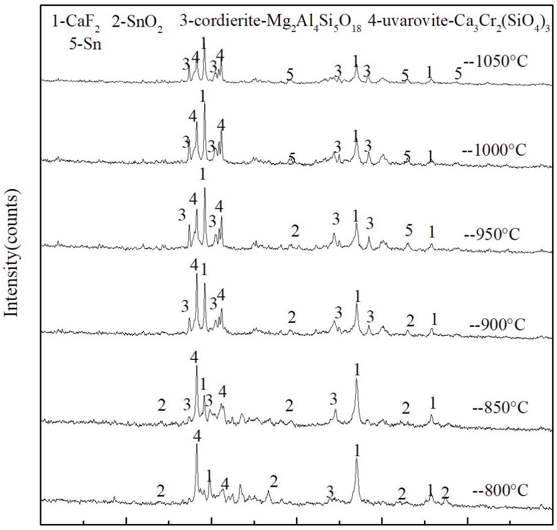

Figure 9. XRD patterns of the reduced samples at different conditions

on the volatilization behavior of tin oxide at 900°C. Coke powder was used as the reductant, and the result was shown in Fig. 10. It can be seen that the volatilization rate of tin gradually increased with the increase of reduction time. The volatilization rate of tin increased sharply below 60 min. The volatilization rate of tin reached 75.16% when the reduction time was 60 min. The volatilization rate of tin increased slowly as the reduction time was over 60 min. Therefore, the suitable reduction time of tin volatilization was 60 min.

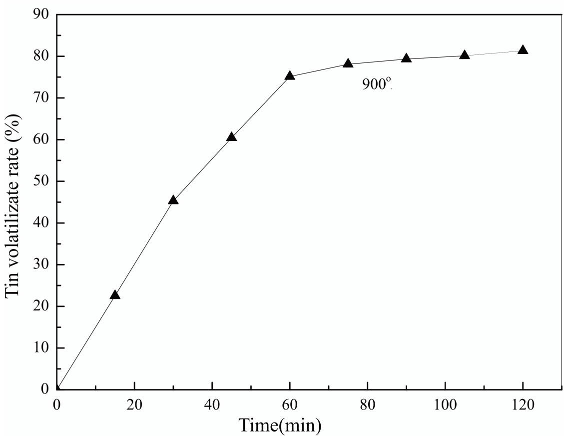

Figure 10. Effect of reduced burn time on tin volatilization

3.5 The kinetic of reduction roasting

3.5.1 Kinetic models

In this paper, the sample was pelleted by hydraulic machine which lead to the sample having a tight structure. According to the previous studies [3,20], unreacted core shrinking model was chosen to describe the reduction kinetic of SnO_2 briquetting. Therefore, the reduction reaction of SnO_2 can be established as Eq(6), which was contended with unreacted core shrinking and it can be divided into three steps: in the first step the CO diffused through the gas boundary layer to the surface of Sn-bearing middling; the next step was to diffuse CO through the product layer to the reaction interface; finally, the CO reacted with Sn-bearing mineral on the interface. The rate-control steps of unreacted core shrinking model included interfacial chemical reaction, gas internal diffusion or the combination of them. It is obvious that different speed limits correspond to different kinetic equations [9]. The rate equations of different rate-control steps are shown in Table 4.

Table 4. Kinetic equations for different controlling steps [20,26]

Rate-control step	Rate equation
Interfacial chemical reaction	$t=\alpha(1-(1-X)^{\frac{1}{3}})$
Internal diffusion	$t=b(1-\frac{2}{3}X-(1-X)^{\frac{2}{3}})$
Combination of interfacial chemical reaction and gas internal diffusion	$t=\alpha_1(1-(1-X)^{\frac{1}{3}})+b_1(1-\frac{2}{3}X-(1-X)^{\frac{2}{3}})$
Where a is a constant $\alpha=r_0\rho/Kc_0$; r_0 is reactor particle radius; ρ is the density of the solid reactant; k is reaction rate constant; c_0 is reactant surface gas concentration. Where, $b=r_0^2\rho/2D_Ec_0$; D_E is diffusion rate constant.	

3.5.2 Kinetics of SnO_2 volatilization

According to the effect of time on the tin volatilization rate, 60 min was considered as the suitable time for reaction. The kinetic of SnO_2 reduction was investigated within 60 min at 800 °C, 850 °C, and 900 °C, respectively. All the experimental data were analyzed by linear regression using the equations of interfacial chemical reaction and internal diffusion in Table 4, and the results are shown in Fig. 11 and Fig. 12, respectively. It can be seen from the Fig. 11 and Fig. 12 that the plots of $1-(1-X)^{1/3}$ obtained a very good linear relationship with time. Therefore, the reduction of Sn-bearing mineral was controlled by chemical reaction. The specific equations which were deduced by the Arrhenius equation are shown in (9) ~ (12).

$$t=\alpha(1-(1-x)^{\frac{1}{3}}) \tag{9}$$

$$1-(1-x)^{\frac{1}{3}}=\frac{1}{\alpha}t \tag{10}$$

$$\frac{1}{\alpha}=K_{app}=K_0e^{-\frac{E_a}{RT}} \tag{11}$$

$$Ln(K_{app})=Ln(K_0)-\frac{E_a}{RT} \tag{12}$$

where, K_{app}= f (d) f(c) f (T) t; d is the particle size of pellets; c is the contents of CO%; T is the temperature of reactions. In the reactions, the particle size and the density were constant which can be consider as K (K is a constant).

In order to further calculate the activation energy of the reaction, the relationship of Ln (Kapp) and 1/T is shown in Equation (13).

$$Ln(K_{app})=\frac{-E_a}{RT}+k \tag{13}$$

The relationship between ln(k) and 1/T are shown in the Fig.13, and the result shows that the plots of ln(k) obtained a very good linear relationship with 1/T. The activation energy of the reaction was 44.66 kJ/mol.

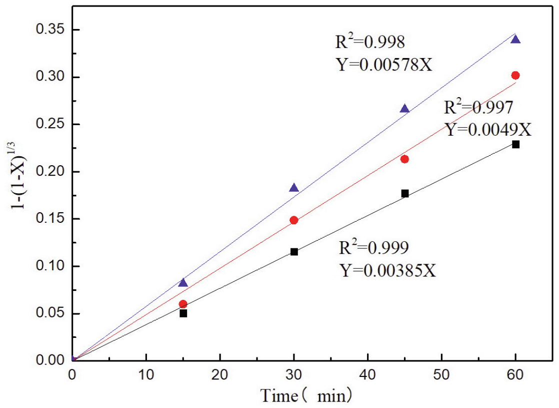

Figure 11. Relationship between $[1-(1-X)^{1/3}]$ and temperature at 800°C ~ 900°C

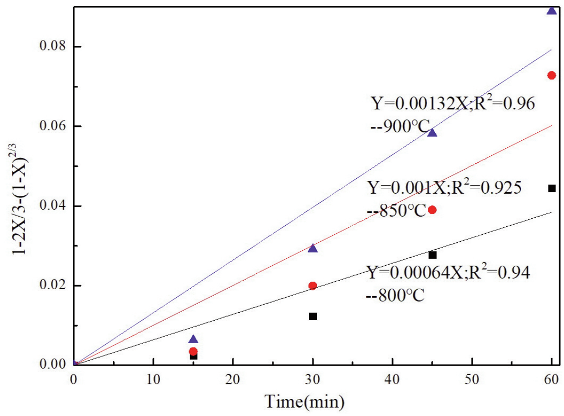

Figure 12. Relationship between $[1-2X/3(1-X)^{2/3}]$ and temperature at 800°C ~ 900°C

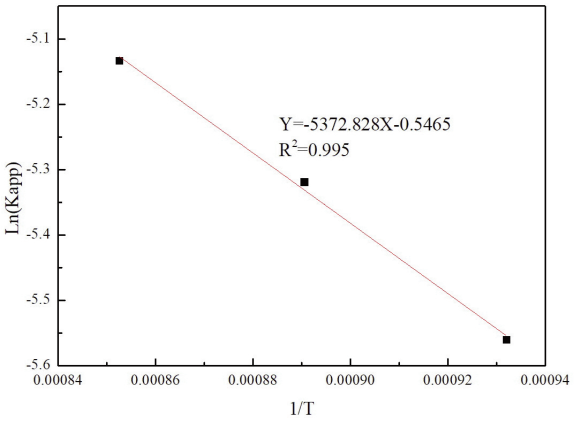

Figure 13. The relationship between Ln(Kapp) and 1/T

4. Conclusions

The results of the thermodynamic indicated that the SnO_2 can be selectively reduced to SnO by controlling the atmosphere of CO content to 9%~15% at the studied temperature. The reaction capability of coal was proposed which was a verified tool for selecting the suitable reduction agent. In the reduction process of tin-bearing middling, the coke powder was used as reductant which can control the CO content at a lower level due to its weak reactivity with CO_2. The volatilization of tin reduced by coke powder was better than that of anthracite and bituminous coal. The volatilization rate of tin reached 81.34% with coke powder as the reducing agent at temperature of 900°C for 2h. The result of kinetic analysis indicated that the reduction reaction of tin-bearing middling was controlled by the interface chemical reaction, and the activation energy of the reaction was 44.66 kJ/mol.

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ISPARAVANJE KALAJA TOKOM KARBOTERMIČKE REDUKCIJE SIROVINE KOJA SADRŽI KALAJ U CILJU NJEGOVOG POVRAĆAJA

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Apstrakt

U ovom radu je predložen novi metod za odabir redukcijskog sredstva. Ovo istraživanje se bavilo dobijanjem kalaja iz sirovine koja sadrži drugorazredni kalaj putem karbotermičke redukcije. Ponašanje kalaja prilikom isparavanja tokom karbotermičke redukcije je sistematično ispitivano, uključujući termodinamiku i kinetiku postupka, metod odabira redukcijskog sredstva, uticaj redukcijskog sredstva, redukcijske temperature, kao i uticaj perioda trajanja na brzinu isparavanja kalaja. Rezultati termodinamičke analize su pokazali da je SnO_2 *redukovan do SnO održavanjem procenta CO između 9% i 15%, a zatim je ispario u obliku SnO. Ispitivana je efikasnost redukcije i mogućnost reagovanja sa* CO_2 *u prisustvu tri redukcijska sredstva, što je ukazalo da kalaj dobijen isparavanjem ima osobine redukcijskog sredstva, i da je koristan prilikom lakog isparavanja u prisustvu koksa u prahu. Rezultati eksperimenta su pokazali da udeo isparavanja kalaja dostiže više od 81,34% kada se kao redukcijsko sredstvo koristi koks u prahu na 900°C tokom perioda od 2h. Kinetika postupka isparavanja kalaja tokom redukcionog prženja je pokazala da reakciju kontroliše hemijska reakcija na graničnoj površini, a da aktivaciona energija reakcije iznosi 44,66 kJ/mol.*

Ključne reči: *Kalaj; Koks u prahu; Kalajni oksid; Redukciono prženje*