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IMPROVEMENT OF MODELING ON THE PIDGEON PROCESS FOR MAGNESIUM PRODUCTION BY INTRODUCING THE VARIABLE THERMOPHYSICAL PROPERTIES

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Abstract

The variable thermophysical properties were introduced into the coupling model of heat transfer and reduction reaction in the Pidgeon process to improve the accuracy of the numerical calculation. The distribution of temperature and magnesium reduction extent in the briquette layer, and the total magnesium reduction extent in the retort were investigated. The model results show better agreement with those of industrial production. The feature of 'layer shift' in the briquette layer during the reduction process was clearly shown. It was shown that the reduction reaction occurs only at a thin interface. The slag layer with lower thermal conductivity of $0.4 Wm^{-1}K^{-1}$ formed during reduction strongly hinders the reaction to move forward within the layers, resulting in the slow magnesium production rate in the Pidgeon process, which is important for key equipment innovation and the development of new magnesium production techniques.

Keywords: Magnesium; Pidgeon process; Numerical calculation; Heat transfer; Thermophysical property

1. Introduction

Magnesium and its alloys are being widely used in the fields of automotive, aerospace, military, electronics, and medicine, due to its excellent mechanical properties and lower density than the other engineering structural materials [1]. At present, more than 80% of the world total magnesium production mainly produced by the Pidgeon process comes from the China. The Pidgeon process extracts the magnesium from calcined dolomite (MgO×CaO) according to reaction (1), under a vacuum pressure of ~100 Pa and temperature of ~1200 °C in a horizontal steel retort charged with the walnut-shaped briquettes with a diameter of ~22 mm.

$$2(MgO \cdot CaO)_{(s)} + (Si \cdot Fe)_{(s)} \xrightarrow{-1200^{\circ}C, -100Pa} \rightarrow (2CaO \cdot SiO_2)_{(s)} + 2Mg_{(s)} + Fe_{(s)}$$
(1)

Because of operating in high temperature and high vacuum, a quick and deep understanding of magnesium production in the retort needs to recur to the simulation tool. Modeling on the Pidgeon process has been developed by researchers in recent years.

Early, Xia et al. [2, 3], and Liang et al. [4] considered the heat transfer in the briquettes layer as a twodimensional unsteady heat conduction. Since the magnesium reduction reaction was not introduced in the modeling, the simulation results can only demonstrate the temperature distribution in the retort during the heating process. Yu et al. [5] introduced the reaction heat into the two-dimensional unsteady heat conduction model as an inner heat source. The reaction heat as a function of temperature was derived from the free energy change of reaction (1). Since the reaction mechanism and kinetics were not taken into account for the modeling, the magnesium reduction extent and the model results were strongly empirical in Yu's model. Then, Yang et al. [6] used the threedimensional phase interfacial reaction model to describe the reaction mechanism of reaction (1). The magnesium reduction extent in the briquettes layer in the retort can be calculated. However, the reasonability of the mechanism and accuracy of modeling by the phase interfacial reaction were still not clear. It should be noted that the model used by Yang et al. was also strongly empirical.



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For providing a more accurate numerical modeling on the Pidgeon process, a lot of experimental and numerical modeling studies were carried out by our team in the last decade [7-12]. Zhang [7] put forward using the micro-briquette to eliminate the influence of heat transfer of briquetteself in the kinetic experiments. This method greatly improved the accuracy of kinetics on the magnesium reduction reaction, laying a foundation for exploring the kinetic of magnesium reduction process. Based on the micro-briquette method, Li et al. [8, 9] conducted pseudo-isothermal kinetic experiments to study the kinetics equation describing the reduction reaction (1). It was found that the reduction reaction was more likely controlled by the interface kinetics at lower reduction rate, while it was controlled by the diffusion of the silicon atoms or dolime molecules at higher rate. After coupling the kinetics model and the heat transfer model in the briquette layer in retort, a three-dimensional unsteady simulation was developed.

Although modeling on the Pidgeon process had made a great progress, there were still some shortcomings: a great number of assumptions in the model simplification, neglect of radiation heat transfer between briquettes and, most of all, the imprecision of the basic thermophysical properties. As far as we know, the thermophysical parameters including the density, specific heat, thermal conductivity, and surface emissivity of the briquettes in the developed models were mostly taken as constants. The constant parameters had a great difference with temperature- and briquette composition-dependent ones, which played a significant effect on the calculation results. Wang et al. [10] in our team measured experimentally the briquette density, specific heat, and the effective thermal conductivity of the Pidgeon-processed briquette. It was found that the physical parameters of briquette were largely related to temperature, forming pressure, briquette composition, and magnesium reduction extent.

In this study, based on the calculation model developed by our previous study [9], the variable thermophysical properties were introduced into the model to improve the accuracy of numerical simulation. The distribution of temperature and magnesium reduction extent simulated by the improved model was presented. The effect of introducing the variable thermophysical properties (especially the thermal conductivity) on the simulation of Pidgeon process was significantly analyzed and discussed.

2. Modeling

2.1. Physical model

In industry, the retort used in the Pidgeon process, as shown in Fig. 1, was made of heat-resistant alloy steel with 330 mm in outer diameters and 30 mm in thickness, 2.5-2.7 m in length [12, 13]. The walnut-shaped briquette was shaped with ferrosilicon 75% (FeSi) and calcined dolomite with specific molar ratio of Si/2MgO = 1.2, a small quantity of fluorite was added as the mineralizer [14-16].

In order to save the calculation cost, three simplifications were made:

(1) the \sim 22 mm walnut-shaped briquette was simplified to a regular sphere with 20 mm in diameter.

(2) the retort was charged fully with briquettes in the radial.

(3) one eighth of the briquettes layer perpendicular to the axis of the retort and one briquettes layer (20 mm in thickness) parallel to the axis direction were taken out as the calculation area.

Fig. 2 shows the calculation model where stacking state of the pre-reduction briquettes was used in the calculation. Particularly, three briquettes marked the No. 1 (near-wall), No.2 (middle) and No.3 (near-axis) in Fig. 2(a) were selected for the subsequent calculation results demonstration and discussion in convenience.

The grid independence of numerical solution was firstly conducted. Tetrahedral Cell with number

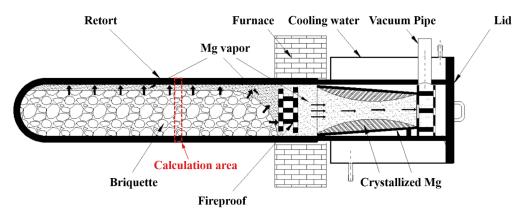


Figure 1. Magnesium production by the typical Pidgeon process



268377, 707666, 981013 and maximum cell size of 2.5 mm, 1.8 mm, 1.6 mm were tested respectively. The grid system with number 707666 and maximum size of 1.8mm was finally used in this study, as shown in Fig. 2(b).

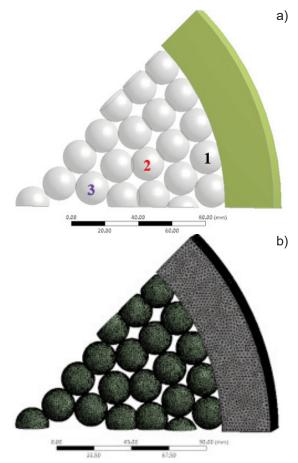


Figure 2. Calculation model used in this study. (a) Geometrical model; (b) Grid system

2.2. Governing equation

Accordingly, the magnesium reduction process in the retort was modeled as a three-dimensional, unsteady process with inner heat source, and the control equation was expressed as follows,

$$\rho_{MB}C_{pMB}\frac{\partial T}{\partial t} = \frac{\partial}{\partial x}(\lambda_{MB}\frac{\partial T}{\partial x}) + \frac{\partial}{\partial y}(\lambda_{MB}\frac{\partial T}{\partial y}) + \frac{\partial}{\partial z}(\lambda_{MB}\frac{\partial T}{\partial y}) + (2)$$

$$\frac{\partial}{\partial z}(\lambda_{MB}\frac{\partial T}{\partial z}) + S$$

where r_{MB} , C_{pMB} , l_{MB} , and T are the density (kg×m⁻³), specific heat (J·kg⁻¹·K⁻¹), effective thermal conductivity (W·m⁻¹K⁻¹), and temperature (K) of the shaped briquettes, respectively. The physical parameters used in this study are described in section 2.2.1.

The reaction heat of magnesium reduction S was

calculated by Eq. (3), which was added to Eq. (2) as a source term via the User Defined Function (UDF).

$$S = -\Delta H_T^{\Theta} \omega \tag{3}$$

$$\omega = M \frac{d\alpha}{dt} \tag{4}$$

where w is magnesium production rate of unit volume briquette (mol^{-m-3}·s⁻¹), which was introduced to multiply by the standard reaction heat ΔH_T^{Θ} to obtain the reaction heat. The magnesium production rate w was calculated by the Eq. (4) related to the change rate of reduction extent with time $\frac{d\alpha}{dt}$ and the theoretical magnesium production of unit volume briquette M. The $\frac{d\alpha}{dt}$ was determined according to the kinetic experiments described in section 2.2.2.

In this model, the radiative transfer between briquettes, and briquettes and wall were accounted for by the Discrete Transfer Radiation Model (DTRM). The radiation heat flow between the radiation surfaces can be calculated as the surface emissivity was properly defined.

The retort wall temperature was set 1200 °C, 1175 °C and 1150 °C, respectively. The initial briquette temperature was 25 °C.

2.2.1. Thermophysical Properties

Table 1 lists specifically the thermophysical properties used in the previous researches and those used in our study. Different from the constant thermophysical properties in terms of density, specific heat, effective thermal conductivity, and emissivity, those with function of temperature or magnesium reduction extent were adopted in the present model.

Note: thermophysical properties used in this study measured in Si/2MgO molar ratio 1.2 and forming pressure 470 MPa.

2.2.2. Kinetic Experiments of magnesium reduction process

In this study, the fitting expression Eqs. (4)-(6) of kinetic results [11] given in segments was used for the numerical calculations.

$$T = 1050^{\circ}C \qquad \alpha = 0.95 \left(1 - e^{-0.065t}\right) \tag{5}$$

$$T = 1100^{\circ}C \qquad \alpha = 0.98 \left(1 - e^{-0.121t}\right) \tag{6}$$

$$T = 1150^{\circ}C \qquad \alpha = 1.00 \left(1 - e^{-0.234t} \right) \tag{7}$$

It should be noted that the Eqs. (5)-(7) described



	Model in the Previous Research	Model in our study [10]
Density (kg·m ⁻³)	Constant: 1.05-1.5 [4]; 1.25 [6]; 1.8 & 1.5 (Before & after reaction) [5]; 1.72 [11]	$ \rho_{MB} = 1715 \cdot (1 - 0.1938\alpha) $
Specific Heat (J·g ⁻¹ ·K ⁻¹)	Constant: 1.44 [3]; 1.2 [4, 6]; 1.2&1.0 (Before & after reaction) [5]	Heating process: $C_{pMB} = \begin{cases} 0.7805 + 4.262 \times 10^{-4} T + 2.593 \times 10^{-3} T^{-2} & (100 - 300^{\circ} \text{C}) \\ 0.9009 + 4.796 \times 10^{-5} T - 9.612 \times 10^{2} T^{-2} & (300 - 850^{\circ} \text{C}) \\ -3.106 + 2.794 \times 10^{-3} T + 1.255 \times 10^{6} T^{-2} & (900 - 1100^{\circ} \text{C}) \end{cases}$ Reduction process: $C_{pMB} = (1 - \frac{0.6867\alpha}{1 - 0.1938\alpha})(-3.106 + 2.794 \times 10^{-3} T + 1.255 \times 10^{6} \times T^{-2}) + \frac{0.6867\alpha}{1 - 0.1938\alpha}(0.7081 + 2.677 \times 10^{-4} T) & (900 - 1100^{\circ} \text{C}) \end{cases}$
Effective Thermal Conductivity (W·m ⁻¹ ·K ⁻¹)	[17];	Heating process: $\lambda_{S+ME2} = \begin{cases} 1.151 - 1.220 \times 10^{-3}T + 6.126 \times 10^{-7}T^2 & 350 MPa \\ 1.560 - 1.610 \times 10^{-3}T + 7.628 \times 10^{-7}T^2 & 470 MPa & 100 \sim 800 \degree C \\ 1.917 - 1.890 \times 10^{-3}T + 8.437 \times 10^{-7}T^2 & 590 MPa \end{cases}$
	1.3 [2, 3, 8, 9, 18]; 0.16 & 0.13 (Before & after reaction) [5]	Reduction process: $\lambda_{\alpha,T} = \frac{(-2.958 \times 10^8 T^2 + 2.203 \times 10^4 T + 0.203)(7.628 \times 10^7 T^2 - 0.00160T + 1.560)}{7.914 \times 10^7 \alpha T^2 - 0.00181 \alpha T - 2.958 \times 10^8 T^2 + 2.203 \times 10^4 T + 1.364 \alpha + 0.203}$
Emissivity	Constant: 0.8 [5, 8]	$\varepsilon = \begin{cases} -1.65 \times 10^{-4} T + 0.591 & \alpha = 0 \\ -2.81 \times 10^{-4} T + 0.737 & \alpha = 1 \end{cases}$

the relationship between magnesium reduction extent a and reaction time under different reaction temperatures. These kinetic relationships were the core link in the coupling calculation of briquettes heating process and reduction reaction. They could be used to calculate the reduction extent distribution of the single briquette or the overall briquettes layer at different temperatures and reaction times.

2.3. Solution

The whole simulation was completed through the ANSYS FLUENT commercial software. Particularly, the reduction extent and the thermophysical properties calculated are linked through the UDF interface.

Fig. 3 shows the UDF work flowchart. This UDF mainly implemented the following functions: (1) Calculating the magnesium reduction extent *a* with Eqs. (5)-(7) when the reduction reaction starts; (2) updating the thermophysical properties in the cell, which will be used in the next calculation period. In particular, for the cell *i*, the reduction extent *a* should be 0 if the temperature *T* is lower the reaction onset temperature. Otherwise, the *a* of the cell should be calculated through linear interpolation of the *a*-*T* curve when the cell temperature is between 1050 °C ~ 1100 °C, or between 1100 °C ~ 1150 °C. If the cell

temperature is higher than 1150 °C, the linear interpolation of a-T curve at 1150 °C and a full conversion rate was conducted.

3. Results and Discussion 3.1. Characteristic 'Layer Shift' in briquette layer in the retort

Fig. 4 demonstrates the characteristic 'Layer Shift' in terms of temperature distribution and magnesium reduction extent distribution in the briquette layer in the retort. As shown in Fig. 4(a), the temperature increased quickly at the beginning of 3 h time while it increased slowly after that. Isothermal surface in the briquettes layer was nearly parallel to the inner face of the retort, moving forward layer by layer from the wall face to the inside of the retort, demonstrating the characteristic of layer shift.

Similarly, the reaction interface (considered as single-briquette layer) also presented the characteristic 'Layer Shift', as shown in Fig. 4(b). The reaction interface inside the briquettes layer was neatly parallel to the inner face of the retort, moving forward layer by layer with time advancing. Because the temperature was the precondition of the reaction, the 'Layer Shift' characteristic in reaction interface was the same as the isothermal surface. It can be seen



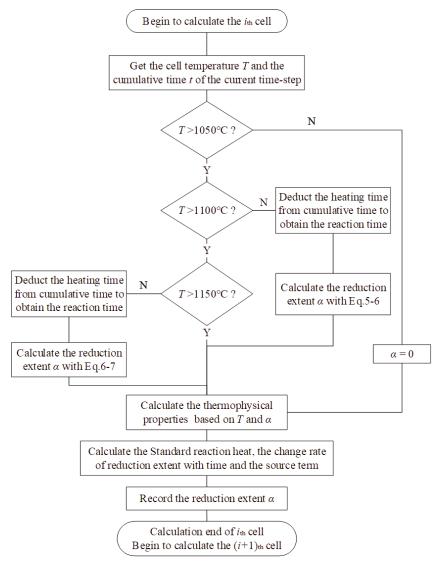


Figure 3. The work flowchart of the UDF used in the numerical calculation

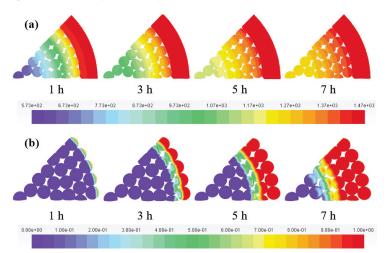


Figure 4. Characteristic of Layer Shift in briquette layer in the retort. (a) temperature distribution; (b) magnesium reduction extent distribution



that the reaction interface separates the briquette layer into two zones, that is the nearly-completed reaction zone outside the interface, and nearly un-reacted zone inside the interface. Silicothermic reduction reaction mainly occurred in the interface. From these phenomena, it can be expected that the large difference in physical parameters would play a significant impact on the actuate prediction of reduction process in the retort.

3.2. Thermophysical properties variable and its effect on the Pidgeon Process

Since the heat transfer in the briquette layer plays an important role in the magnesium reduction, the properties of the thermal conductivity were firstly studied. Fig. 5 shows the distribution of thermal conductivity with time in the briquette layer. Only within three hours at the beginning of reaction, the effective thermal conductivity of briquettes layer inside the retort was higher than 0.8 W·m⁻¹·K⁻¹ due to the relatively low temperature. However, for the whole reduction period of 8 h, the briquette thermal conductivity appeared to be less than 0.8 W·m⁻¹·K⁻¹ in most of the time. Hence, the constant thermal conductivity of 1.3 W·m⁻¹·K⁻¹ used in most numerical studies [2, 3, 8, 9, 19] could give an overestimation in magnesium reduction extent.

Notably, the thermal conductivity of slag after reduction reaction was about $0.4 \text{ W}\text{m}^{-1}\text{K}^{-1}$, while that of the pre-reduction briquette was about $0.7 \text{ W}\text{m}^{-1}\text{K}^{-1}$. The boundary or area between the slag and pre-reduction briquettes should be considered as the reaction interface or area, on which the thermal conductivity transited from $0.7 \text{ W}\text{m}^{-1}\text{K}^{-1}$ to $0.4 \text{ W}\text{m}^{-1}\text{K}^{-1}$.

For the whole briquettes layer, both the heating and the reaction endotherm were sourced from the retort inner face. However, there was a formed slag layer with low thermal conductivity laying between the heat source and pre-reduction briquettes, and the thickness of slag layer became larger and larger with reaction advancing. Consequently, at the later stage of reduction, the heat transfer resistance would become larger, making it hard to transfer heat to the pre-reduction briquettes inside the retort. This was the main reason for the slower increase in reduction extent at the later stage of Pidgeon process.

Fig. 6 shows the change in the reduction extent and the thermal conductivity at the center of No.1-3 briquettes (marked in Fig. 1) over the reaction time. It can be seen that the magnesium reduction reaction for the monitored briquettes started at the time of 1.6h, 4.9h, and 7.2h, respectively. The reduction reaction at each monitored briquette completed after 2~3 hours. For the thermal conductivity, which is related to the temperature and reduction extent, it presented three stages: (a) before reaction starting, the conductivity decreases rapidly from 1.5 W m^{-1.} K^{-1} to 0.7 W m^{-1.} K^{-1} with the increase of briquette temperature; (b) during reduction reaction the conductivity decreases from 0.7 W⁻¹·K⁻¹ to 0.4 W·m⁻¹·K⁻¹ with the increase of reduction extent and temperature; (c) after reaction the briquette material turn into slag and the conductivity becomes the smallest around 0.4 W·m⁻¹·K⁻¹ and slightly increases with the increase of temperature.

Particularly considering that the temperature rise during reduction reaction was less than 100 °C [10], the decrease of thermal conductivity at this stage mainly resulted from the slag producing, which was quantitatively measured to be in the range from 0.38 $W \cdot m^{-1} \cdot K^{-1}$ to 0.40 $W \cdot m^{-1} \cdot K^{-1}$. Therefore, the slag producing in the reaction with lower thermal conductivity is the key factor to extending the magnesium reduction time of the briquettes in the retort.

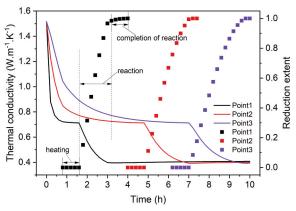


Figure 6. The variation in magnesium reduction extent and thermal conductivity with time

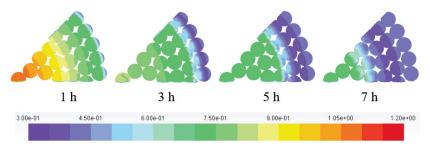


Figure 5. Distribution of thermal conductivity with time



3.3. Pidgeon process modeling improvement

Fig. 7 shows the overall magnesium reduction extent of briquettes layer in the retort at the wall temperatures of 1150 °C, 1175 °C and 1200 °C, respectively. According to the long-term industrial production statistics results of the Pidgeon process [14], the magnesium reduction extent could reach around 85% at the reaction temperature ranging from 1150 °C to 1180 °C. In comparison, the results by our present study showed that the overall reduction extent could reach 83.2% and 90.2% within 10 h in the case of wall temperature of 1150 °C and 1175 °C, respectively. There was a good agreement with the statistical results. This indicated that the accuracy of the present model was high enough to satisfy the quantitatively calculation of coupling simulation of heat transfer and reduction reaction in the Pidgeon process.

Furthermore, a comparison of overall reduction extent in 10 hours calculated by a constant thermophysical properties model and our variable thermophysical properties model were conducted in Fig. 7. It can be seen that the overall reduction extent by constant properties was generally larger than the present study in the first 8.5 hours, while over time there was little difference. It was expected that the constant properties model ignored the influence of briquette temperature and briquette composition change during the reduction process. Actually, the thermophysical properties vary with the temperature and the change of briquette composition in a large range, like the thermal conductivity of briquette being in the range of 0.4 W^{·m-1}·K⁻¹~1.5 W^{·m-1}·K⁻¹. Thus, the variable thermophysical properties should be seriously introduced to improve the model accuracy.

In recent years, new silicothermic processes like vertical Pidgeon process [5], and vertical compound retort technique [7] were being developed. Limited by

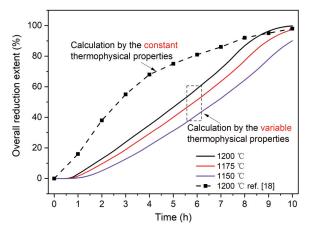


Figure 7. Comparison of overall reduction extent calculated by the constant thermophysical properties and by variable ones

the lack of knowledge of magnesium reduction process in retort and model inaccuracy, the development of new reactor including retort and crystallizer experienced lots of 'trial and error', consuming lots of manpower and material resources. The improved mode in this study can be used to provide a better guidance for the new design of reactor in silicothermic process.

4. Conclusion

The coupling numerical model of heat transfer and reduction reaction for the magnesium production by the Pidgeon process was improved by introducing the variable thermophysical parameters obtained from the experimental measurements. The effects of introducing the variable thermophysical parameters on the modeling results in terms of the distribution of temperature and magnesium reduction extent in the briquette layer, and the overall magnesium reduction extent were analyzed and discussed. The main conclusions are addressed in the following statement:

(1) Distribution of temperature and magnesium reduction extent in the briquette layer in the retort presents the characteristic 'Layer Shift' with time. A reaction interface is dynamically formed during the magnesium production. Besides, the formed slag layer between the heat source and reaction interface has a lower thermal conductivity, which makes it hard for the reaction interface to move forward, resulting into the slow magnesium production rate in Pidgeon process.

(2) The thermal conductivity plays an important role in modeling the magnesium production by the Pidgeon process. The variable thermal conductivity in the range of $0.4 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1} \sim 1.5 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ related to the briquette temperature and composition should be seriously considered in the high-accurate model.

(3) The calculated results of magnesium reduction extent by the improved model were closer to the reality than previous studies. Introducing the variable thermophysical properties can intrinsically improve the accuracy of numerical simulation for Pidgeon process, and be benefit for the innovation of key equipment and the development of the new magnesium production techniques.

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Author's contribution

R.-B. Li: conceptualization, writing original draft, response to the reviewer, manuscript revision; C. Wang: Acquisition and analysis of data, modeling, methodology; Z. Wang, P.-X. Yang, F. Xue: test, validation, discussions; F.-Q. Liu, S.-J. Zhang: discussions, supervision, resources

Data availability

All data, models, or code generated or used during the study are available from the first author or from the corresponding author by request.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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POBOLJŠANJE MODELOVANJA PIDGEON PROCESA ZA PROIZVODNJU MAGNEZIJUMA UVOĐENJEM VARIJABLE TERMO-FIZIČKIH SVOJSTAVA

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Apstrakt

Varijabla termo-fizičkih svojstava uvedena je u model spregnutog prenosa toplote i reakcije redukcije u Pidgeon procesu da bi se poboljšala tačnost numeričkog proračuna. Ispitivana je distribucija temperature i stepena redukcije magnezijuma u sloju briketa i ukupne redukcije magnezijuma u retorti. Rezultati modela pokazuju bolje slaganje sa rezultatima industrijske proizvodnje. Jasno je prikazana karakteristika 'pomeranja sloja' u sloju briketa tokom procesa redukcije. Pokazalo se da se reakcija redukcije dešava samo na tankom interfejsu. Sloj šljake sa nižom toplotnom provodljivošću od 0,4 Wm^{-1.K-1} formiran tokom redukcije snažno ometa reakciju kretanja unapred unutar slojeva, što rezultira sporom stopom proizvodnje magnezijuma u Pidgeon procesu. Poboljšani model može pružiti preciznije kvantitativno predviđanje redukcije magnezijuma u procesu Pidgeon, što je važno za inovaciju ključne opreme.

Ključne reči: Magnezijum; Pidgeon proces; Numerički proračun; Prenos toplote; Termo-fizička svojstva

