doi:10.20944/preprints202109.0172.v1

Effect of basicity and Al_2O_3 on viscosity of blast furnace slag and thermodynamic analysis

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Abstract: With the increased use of laterite nickel ore, the impact of high Al_2O_3 slag on blast furnace smelting has gradually increased. In this paper, the effects of slag basicity and Al_2O_3 content on slag viscosity and enthalpy change under constant temperature conditions was investigated. The changes in slag structure were analyzed by activation energy and Fourier Transform Infrared (FT-IR) spectroscopy. The relationship between slag components and slag temperature and viscosity when slag heat is reduced was investigated. The results showed that the viscosity first slightly decreased and then significantly increased with increasing basicity at constant temperature. With the addition of Al_2O_3 content, the viscosity of the slag increases. The activation energy increases with increasing slag basicity and Al_2O_3. With increasing basicity, the $[SiO_4]^{4-}$ tetrahedral unit trough depth becomes shallow, the $[AlO_4]^{5-}$ asymmetric stretching band migrates to lower wave numbers, and the slag structure depolymerizes. With the increase of Al_2O_3 content, the trough of $[SiO_4]^{4-}$ tetrahedra deepens and the center of the symmetric stretching band moves to a higher wave number. The $[AlO_4]^{5-}$ asymmetric stretching band becomes obvious, indicating the complexity of the slag structure. When the heat decreases, the slag temperature increases as the basicity increases, and the slag thermal stability is better at the basicity of 0.95-1.05. As the Al_2O_3 content increases, the thermal stability of the slag becomes worse.

Keywords: thermodynamic; enthalpy change; viscosity; activation energy; structure

1. Introduction

As a strategic element, nickel has an important position in the development of the national economy [1]. Nickel is mainly found in nickel sulfide ores and nickel laterite ores, most of which is obtained by smelting nickel sulfide ores [2]. Laterite nickel ore accounts for about 70% of the world's total nickel resources, yet only 40% of nickel production comes from smelting laterite nickel ore. With the continuous consumption and increasing depletion of nickel sulfide ore resources, laterite nickel ore will become the main raw material for smelting nickel metal [3,4]. Laterite nickel ore is low-grade composite iron ore, mainly containing Fe, Ni, Cr and other valuable elements, of which the mass fraction of Fe ranges from 9wt% to 50wt% [5]. Since the pyro smelting technology of laterite nickel ore is more mature at this stage, laterite nickel ore is mostly used in blast furnaces to smelt nickel-bearing pig iron in China [6]. However, the complex composition of laterite nickel ore can make the slag produced in the blast furnace during the smelting process have high Al_2O_3 content [7]. The high content of Al_2O_3 in slag will cause an increase in pressure difference in the lower part of the blast furnace, deterioration in the permeability of the column, increase in the liquid phase line temperature and viscosity of the slag, difficulty in separating slag and iron, and the accumulation of slag and iron in the furnace cylinder [8,9]. Therefore, it is important to study the performance of smelting high Al_2O_3 slag to ensure the stability and improve the production efficiency when smelting laterite nickel ore.

The current research on Al_2O_3-containing blast furnace slag is more and more mature, but most of them focus on the Al_2O_3 content within 20%. Most scholars have found that the viscosity of the slag decreases with increasing binary basicity [10,11]. Chang et al. [12] found that the solid particles precipitated when the MgO content was 9.6 wt.% or higher, and the viscosity increased with increasing MgO content. Zhang et al. [13] investigated the effect of MgO/Al_2O_3 on the viscosity of high-alumina type blast furnace slag. It was found that the Al_2O_3 content in the slag should be controlled within 18% and MgO/Al_2O_3 in the range of 0.6~0.7, which can maintain good stability and fluidity of the slag. Yao et al. [14] investigated the effect of basicity and MgO on slag viscosity behavior at Al_2O_3 content of 15-20% and found that the effect of increasing Al_2O_3 content on blast furnace slag viscosity could be counteracted by a higher content of MgO. However, binary basicity has little effect on the viscosity of high alumina blast furnace slag. Shen et al. [15] found that the MgO/Al_2O_3 ratio should be controlled between 0.40 and 0.50 for the range of 15-17% Al_2O_3 in the slag, while ensuring sufficient physical heat of the iron. When Chinese iron and steel enterprises smelt ferronickel, the content of Al_2O_3 in the slag has reached more than 20%. With the increase of Al_2O_3 content, the difficulty of smelting increases, and the condition of the blast furnace is also unstable. In addition, when the blast furnace fuel and furnace conditions change, it will inevitably cause thermal fluctuations in the slag and affect the flow of slag [16]. Therefore, it is important to study the thermodynamic properties of different slag components and the relationship between the composition and its viscosity during heat fluctuations.

In the present work, the slag viscosity was determined for different slag basicity and Al_2O_3 content. Based on the thermodynamic data, the enthalpy change of slag was calculated and the relationship between the slag components and its viscosity was studied when the slag heat was reduced. In addition, the effects of different slag basicity and Al_2O_3 content

 doi:10.20944/preprints202109.0172.v1

on the slag structure were analyzed using Fourier Transform Infrared (FTIR) spectroscopy.

2. Material and Methods

2.1. Thermodynamic Calculations

The chemical composition of the slag is shown in Table 1. The total mass of slag used for the calculation is set to 100g. Theoretically, the specific heat capacity and enthalpy change of the slag can be calculated according to the following equations (1) to (4). However, the enthalpy change of crystallization transition, the enthalpy change of phase change and the enthalpy change of chemical reaction should also be considered in the slag melting process. Therefore, in order to improve the accuracy of the slag specific heat capacity and enthalpy calculations, the data calculated by FactSage software is usually used as a reference. Factsage is one of the largest computational systems in the field of chemical thermodynamics and is widely used to calculate metallurgical thermodynamic data [17,18]. The enthalpy changes of slag at 1773 K, 1823 K, and 1873 K were calculated. According to the first law of thermodynamics, the enthalpy change at a certain temperature is equal to the heat absorbed during the melting process when no work is done by the non-volume under constant pressure conditions. The average values of enthalpy change of slag with different alkalinity and Al2O3 content were calculated at 1873 K and set as Q1 and Q2. The equilibrium temperature of the slag when the heat is reduced is obtained by calculation.

Table 1. Chemical compositions of slag (wt%).

No.	Chemical compositions (mass%)				Basicity	MgO/Al_2O_3
	CaO	SiO_2	MgO	Al_2O_3	(C/S)	
1	28.03	32.97	11.00	28.00	0.85	
2	29.72	31.28	11.00	28.00	0.95	
3	31.24	29.76	11.00	28.00	1.05	0.39
4	32.63	28.37	11.00	28.00	1.15	
5	35.34	33.66	11.00	20.00	1.05	0.55
6	33.29	31.71	11.00	24.00	1.05	0.46
7	29.20	27.80	11.00	32.00	1.05	0.34

$$C_{pi} = (A+Bt+Ct^2+Dt^3+E/t^2)/M_i \quad (1)$$

$$C_p = \sum m_i C_{pi} \quad (2)$$

$$\Delta H_i = \int_{298}^{T_{tr}} C_{pi(s)} dT + \Delta_{tr} H_i + \int_{T_{tr}}^{T_M} C'_{pi(s)} dT + \Delta_s^l H_i + \int_{T_M}^{T} C'_{pi(l)} dT \quad (3)$$

$$\Delta H_T = \sum m_i H_i \quad (4)$$

C_{pi} is the specific heat capacity, J/g·K; i is the components of the slag, CaO, SiO_2, Al_2O_3,and MgO. Where A, B, C, D and E represent the corresponding thermodynamic parameters, which can be found out [19]. T is temperature(K)/1000. M_i is relative molar mass. ΔH_i is the enthalpy change at a certain temperature of the slag, J/g; ΔtrH_i and $\Delta^l_s Hi$ are the enthalpy of crystal phase

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transformation and the enthalpy of melting of blast furnace slag of unit mass during heating up, respectively, J/g. ΔH_T is the enthalpy change of a slag sample at a given target temperature, J. T_{tr} is the crystal transition temperature, K. T_M is slag melting temperature, K.

2.2. Experimental Materials

Based on the slag design, the slag was prepared using pure chemical reagents of CaO, MgO, SiO_2 and Al_2O_3. All the reagents were heated in a resistance furnace to 1273 K and held for 120 min to get out the water of crystallization. The mixed slag is accurately weighed to 120 g and loaded into a graphite crucible with a molybdenum crucible liner. In the presence of argon as a protective atmosphere, the viscosity of the slag was measured. After the slag viscosity measurement, the samples were heated to 1873 K and held for 60 min to obtain the chemical and thermal homogeneity in the melt. The glass samples were prepared by quenching the melt and then crushing it into fine powder. The effects of basicity and Al_2O_3 content on the structural behavior of the slag were analyzed by FT-IR spectroscopy.

2.3. Viscosity Measurement

The viscosity of the slag was measured using the rotary axis method. The experimental equipment for measuring the viscosity of slag is shown in Figure 1. The $MoSi_2$ thermocouple is used to heat the chamber, and the temperature of the chamber is monitored and measured by the thermocouple (with an error of ± 2K), and then the viscosity value at the time of measurement is recorded by a computer. The pre-melted slag is loaded into a graphite crucible with an embedded molybdenum crucible and placed in the constant temperature zone of the furnace chamber. The furnace temperature is heated to 1893 K under argon protection, and the molybdenum ingot is immersed in the slag and rotated and held for 90 min to reach thermal equilibrium. During viscosity measurements, both the crucible and the spindle should be kept on the central axis of the viscometer, as a slight deviation from the axis can cause large experimental errors. Starting at 1873 K, viscosity measurements were performed every 20 K. To ensure sufficient thermal equilibrium, the melt is held for 20 minutes before each measurement. The test ends when the melt viscosity value is >5 Pa·s.

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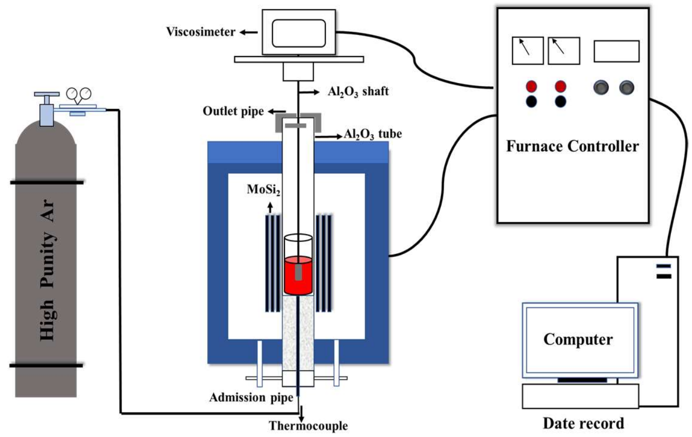

Figure 1. Experimental apparatus for the measurement of slag viscosity.

3. Results and Discussion

3.1. Effect of basicity and Al_2O_3 on slag viscosity

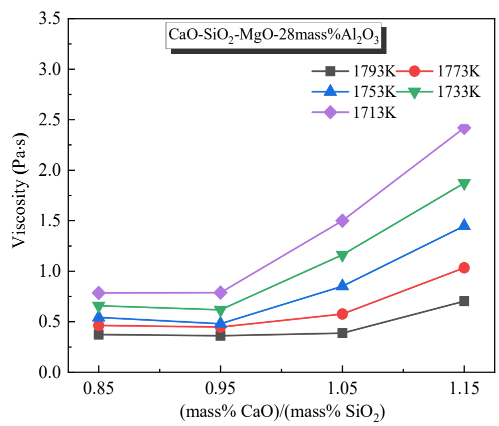

Figure 2. Effect of basicity on the viscosity of the slag.

Figure 2 shows the effect of slag basicity on the viscosity of high alumina slag at different temperatures. It can be seen from the figure that the change in basicity has less effect on the viscosity at higher slag temperatures. As the slag temperature decreases, the effect of basicity on slag viscosity increases. When the Al_2O_3 content in the slag was 28%, the viscosity decreased slightly as the slag basicity increased from 0.85 to 0.95, but as the basicity continued to increase, the viscosity of the slag increased significantly. The addition of basic oxides can provide free oxygen ions (O^{2-}) to react with silicate structure to form non-bridging oxygen, which reduces the degree of polymerization of the slag and the viscosity of the slag [20]. However, as the basicity increases, the liquidus temperature of the slag increases significantly [21]. Consider that in the process of temperature reduction, solid phase mass points will be precipitated in the liquid slag, thus causing a large impact on the slag viscosity.

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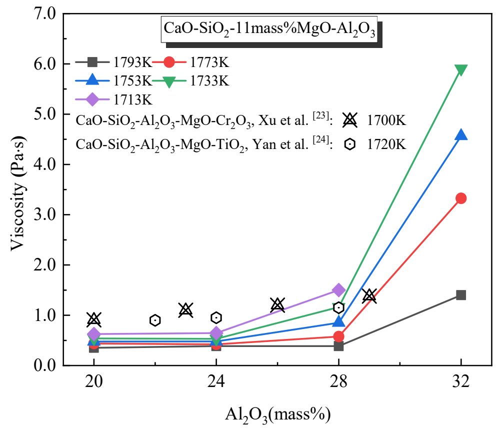

Figure 3. Effect of Al_2O_3 on the viscosity of the slag.

Figure 3 shows the variation of slag viscosity with Al_2O_3 content at different temperatures. It can be seen from the graph that the viscosity of the slag gradually increases with decreasing temperature. The slag viscosity tends to increase with the increase of Al_2O_3 content in the slag. This is similar to the results of previous studies on low-alumina slag [22,23]. This indicates that Al_2O_3 continues to be present in this slag system in the form of acidic oxidation. The two main factors affecting the slag viscosity, analyzed at the atomic level, are the strength of the chemical bonds between the atoms and the complexity of the slag structure. As the Al_2O_3 content increased from 20% to 24%, the slag viscosity did not change significantly. This is mainly considered as the increase of Al_2O_3 content and decrease of SiO_2 content under the existing chemical composition, the strength of Al-O bonding is weaker than that of Si-O bonding, which may be the reason for the insignificant viscosity change [24]. And with the further increase of Al_2O_3 content, the structure of slag becomes complex and the migration resistance of complexed anion groups in slag increases, thus increasing the viscosity of slag.

3.2. Apparent activation energy for viscous flow

The viscous flow activation energy is an index characterizing the shear resistance of the slag. To a certain extent, its change also qualitatively reflects the change in slag structure [25]. The higher the viscous flow activation energy, the higher the energy barrier overcome by the slag flow and the higher the viscosity. This can be calculated from the Arrhenius-type equation [26,27], as expressed by Equation (5). Where η is the viscosity of the melt, (Pa·s); A is the pre-exponent factor, and Ea is the apparent activation energy of the slags, (J/mol); R is the gas constant, (8.314 J/mol·K); T is the absolute temperature, (K).

$$\eta = AT\exp\left(\frac{E_a}{RT}\right) \tag{5}$$

According to the above equation, the relationship between the natural logarithm of viscosity and the inverse of temperature is shown in Figure 4. The slope of the fitted straight line ($R^2 > 0.95$)

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is the apparent activation energy Ea. The calculation results are shown in Table 2. The results showed that the viscous flow activation energy of slag gradually increased as the basicity increased from 0.85 to 1.15. As the Al_2O_3 content in the slag increases from 20% to 32%, the activation energy increases from 202.4 kJ/mol to 660.3 kJ/mol, which indicates an increase in the frictional resistance of the melt and the proportion of complex structural units.

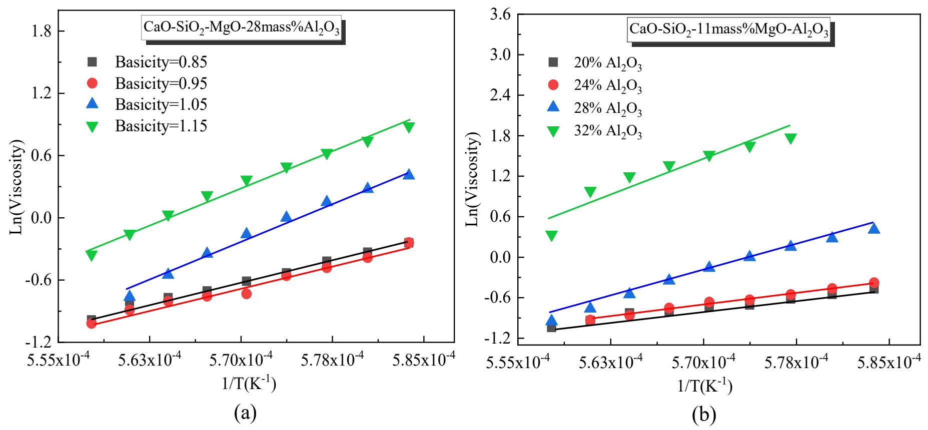

Figure 4. Natural logarithm of viscosity against reciprocal of temperature (a) different basicity (b) different Al_2O_3 content.

Table 2. Viscosity activation energy of slags.

No.	Factors	Al_2O_3	Basicity	Ea(kJ/mol)
1	Basicity	28	0.85	270.1
2		28	0.95	280.4
3		28	1.05	526.3
4		28	1.15	465.1
5	Al_2O_3	20	1.05	202.4
6		24	1.05	227.0
3		28	1.05	526.3
7		32	1.05	660.3

3.3. Effect of basicity and Al_2O_3 on slag structure and precipitated phases

To further investigate the mechanism of basicity and Al_2O_3 content on liquid slag. The Fourier transform infrared (FTIR) spectra of quenched slag were measured to obtain structural information of the quenched slag. The FTIR spectra for different basicity and different Al_2O_3 content are shown in Figure 5. According to previous studies, the vibration band of silicates ranges from 1200-400 cm^{-1} [28,29]. The strong broadband of 1200-750 cm^{-1} is generally associated with the symmetric stretching vibration of the $[SiO_4]^{4-}$ tetrahedral unit [30], and the weak broadband of 750-630 cm^{-1} is generally associated with the asymmetric stretching vibration of the $[AlO_4]^{5-}$ tetrahedral unit [31]. The band near 500 cm^{-1} is considered to be the band of [Si-O-Al] bending vibration [32,33].

Figure 5(a) shows the effect of slag basicity on the slag structure. With the increase of slag basicity, the depth of $[SiO_4]^{4-}$ tetrahedral unit trough becomes shallower and the center of $[AlO_4]^{5-}$ tetrahedral stretching band moves toward the lower

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wave number. It indicates that the composite silicate structure is simplified, while the depth of the Si-O bending groove changes less. From the FT-IR results, it appears that as the slag basicity increases, it causes the silicate structure of the slag to depolymerize, but there are differences with the measured values of slag viscosity. Therefore, we carried out water quenching of the slag at 1773 K and analyzed the precipitated phases of the slag by X-ray diffraction. The X-ray diffraction patterns of different basicity and Al_2O_3 are shown in Figure 6. As shown in Figure 6(a), there is high-temperature spinel ($MgAl_2O_4$) precipitated from the slag [34]. With the increase of basicity, the peak strength of high-temperature spinel in slag first decreases slightly, and then the peak strength increases significantly. Therefore, the increase in viscosity with increasing basicity is due to the precipitation of solid phase in the liquid slag.

Figure 5(b) shows that the depth of the $[SiO_4]^{4-}$ tetrahedral trough deepens with increasing Al_2O_3 content and the center of the symmetric stretching band moves toward the high wave number. The $[AlO_4]^{5-}$ asymmetric stretching band becomes more pronounced in the range of 750-630 cm^{-1}, indicating a change in slag structure from silicate to aluminate. The trough of the [Si-O-Al] bending vibration band becomes insignificant. The silicate structure of the slag undergoes polymerization and agrees well with the measured values of the viscosity.

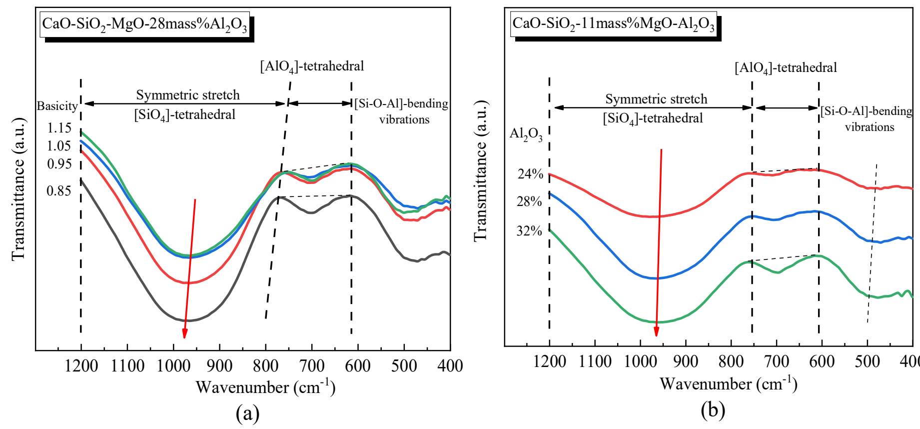

Figure 5. FT-IR results of the as-quenched samples with (a) different basicity (b) different Al_2O_3 content at 1873K.

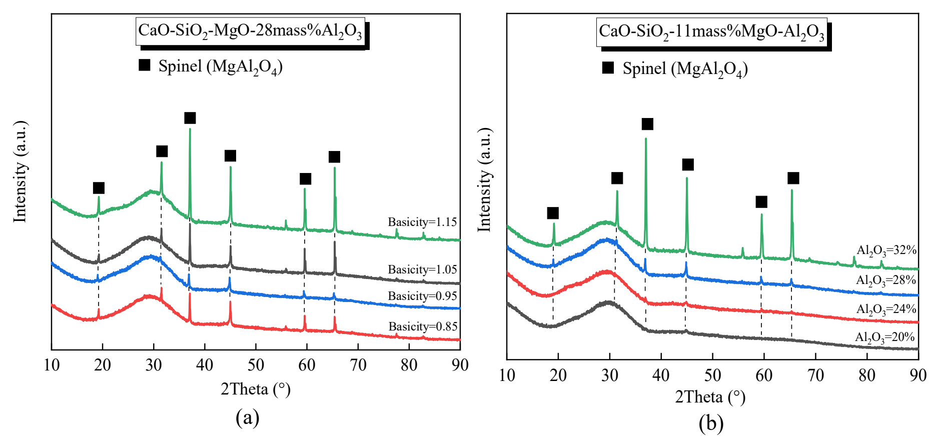

Figure 6. XRD patterns of the rapidly quenched slag samples with (a) different basicity (b) different Al_2O_3 content at 1773K.

3.4. Effect of basicity and Al_2O_3 on slag equilibrium temperature and viscosity at heat reduction

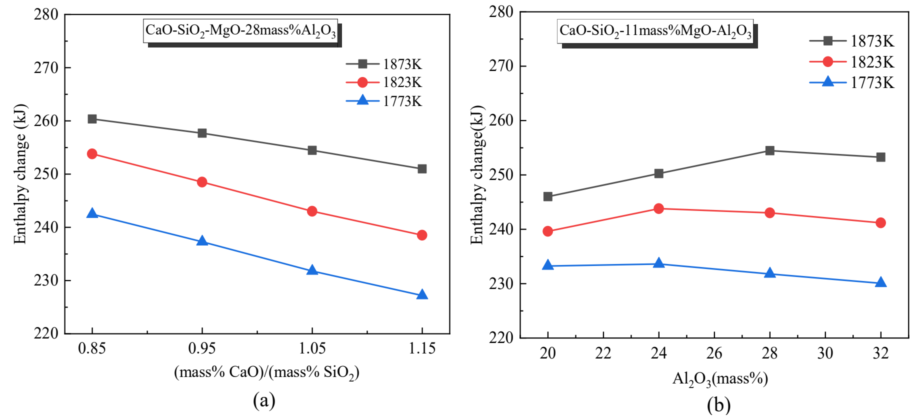

Figure 7. Effect of basicity and Al_2O_3 on the enthalpy change of slag.

The variation curve of slag enthalpy change with slag basicity is shown in Figure 7 (a). It can be seen that the enthalpy change of slag shows a decreasing trend with the increase of slag basicity. As the temperature increases, the enthalpy change of the slag increases significantly. As mentioned earlier, the heat absorbed by the slag is approximately equal to the enthalpy change of the slag. Therefore, an appropriate increase in basicity can reduce blast furnace energy consumption and coke ratio to some extent [35]. Figure 7 (b) shows the relationship between the enthalpy change of slag and Al_2O_3 content at different temperatures. As an important physicochemical property of slag, the enthalpy change is mainly related to the heat capacity and temperature of the slag system. As the slag temperature increases, the enthalpy change of the slag increases. And with the increase of Al_2O_3 content, the effect of temperature on the enthalpy change of slag is more significant. It can be seen that with the increase of Al_2O_3 content, the heat storage capacity of slag is increased. Therefore, to guarantee the stability of slag temperature, the heat supply to the blast furnace should be increased with the increase of Al_2O_3 content.

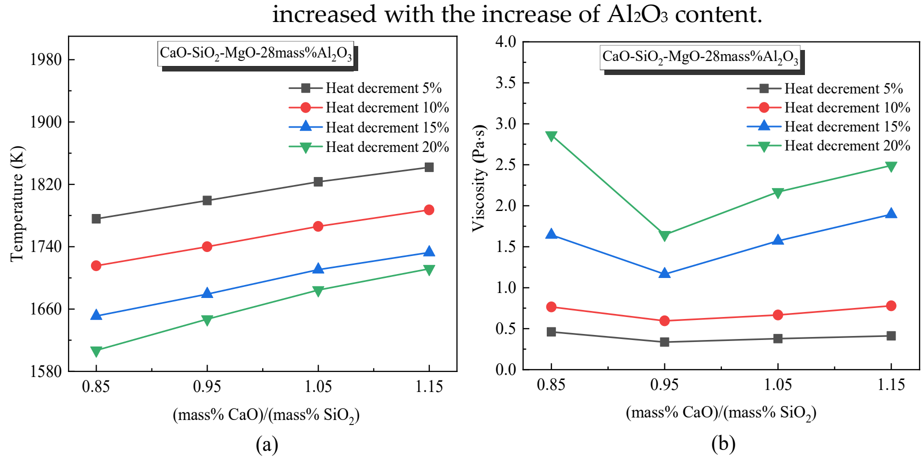

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Figure 8. Effect of basicity on (a) equilibrium temperature and (b) viscosity of the slag.

Figure 8 shows the effect of basicity on slag temperature and viscosity at 1873 K when the slag heat is reduced by 20%, 15%, 10% and 5%, respectively. As shown in Figure 8(a), the slag temperature increases significantly with the increase of basicity under certain heat conditions. And as the slag basicity increases, the temperature fluctuation due to the reduction of slag heat tends to be insignificant. Therefore, an appropriate increase in slag basicity will make the slag more resistant to temperature. Figure 8(b) depicts the variation of slag viscosity with basicity for different heat reductions. When the heat reduction is certain, with the increase of basicity, the viscosity of slag first decreases significantly and then increases slowly. The viscosity of the slag is lowest at a slag basicity of 0.95. Therefore, for this slag system, the slag basicity is maintained at 0.95-1.05 to improve the thermal stability of the slag.

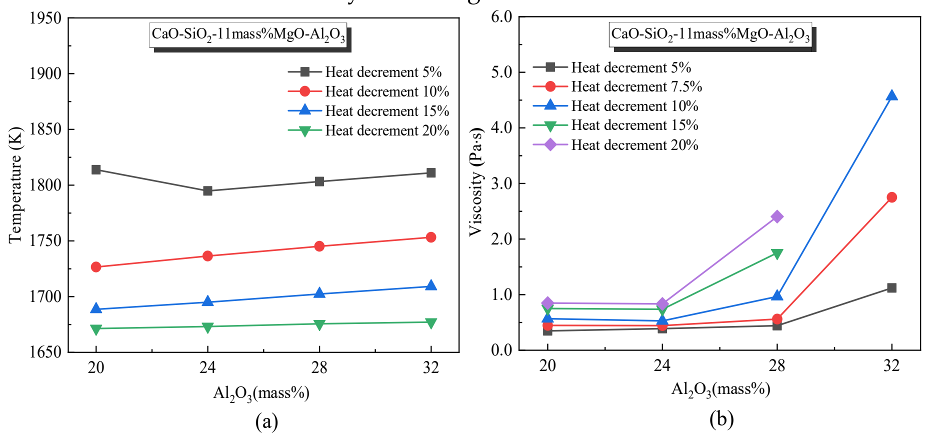

Figure 9. Effect of Al_2O_3 on (a) equilibrium temperature and (b) viscosity of the slag.

The variation of slag temperature with Al_2O_3 content for different heat reductions is given in Figure 9(a). As the heat of the slag decreases, the temperature of the slag decreases. At a 5% reduction in heat, the slag temperature first decreases and then increases slightly as the Al_2O_3 content increases. This is because as the Al_2O_3 content increases, the liquid phase line temperature of the slag increases, and when the heat of the slag decreases, there will be solid-phase precipitation exotherm in the slag, resulting in a slight increase in slag temperature. Figure 9(b) shows the effect of Al_2O_3 content on the slag viscosity when the heat is reduced. With the addition of Al_2O_3, the viscosity of slag firstly changed insignificantly and then showed a significant increasing trend under the condition of a certain heat reduction. Especially when the Al_2O_3 content in the slag exceeds 24%, the effect of heat fluctuations on the slag viscosity is obvious. Therefore, for slags with high Al_2O_3 content, the fluctuation of heat in the blast furnace should be strictly reduced.

4. Conclusions

The effects of different slag basicity and Al_2O_3 content on the viscosity and enthalpy change of high alumina slag at constant

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temperature were investigated. The effect of components on slag temperature and viscosity when slag heat is reduced was investigated. The slag structure with different slag basicity and Al_2O_3 content was studied by using FTIR spectroscopy. The following conclusions could be drawn.

(1) Under constant temperature conditions, the viscosity of the slag first slightly decreases and then significantly increases with increasing basicity. With the increase of Al_2O_3 content, the viscosity of slag shows a significant trend of increasing. With the increase of basicity and Al_2O_3 content, the activation energy of the slag system of this design both tends to increase.

(2) With the addition of slag basicity, the trough depth of $[SiO_4]^{4-}$ tetrahedral unit becomes shallow and the $[AlO_4]^{5-}$ asymmetric stretching band migrates toward the lower wave number. With increasing Al_2O_3 content, the center of the $[SiO_4]^{4-}$ tetrahedral symmetric stretching band shifts to higher wave numbers and the $[AlO_4]^{5-}$ asymmetric stretching band becomes pronounced, indicating the complexity of the slag structure.

(3) When the slag heat is reduced, the slag temperature increases and the viscosity first decreases and then increases as the basicity increases. The thermal stability of slag is better when basicity is controlled at 0.95-1.05. As the Al_2O_3 content increases, the temperature of the slag gradually increases and the viscosity of the slag increases significantly. Considering Al_2O_3 below 24%, the thermal stability of slag is better.

Acknowledgments: The present work was financed by National Natural Science Foundation of China (Grant No. 51874025) and the Fundamental Research Funds for the Central Universities (No. FRF-TP-20-048A2). The authors gratefully acknowledge the supports.

Conflicts of Interes: The authors declare no conflict of interest.

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