Effect of ZnO/PbO and FeO_x/SiO_2 Ratio on Viscosity of Lead Smelting Slags


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Abstract

Effect of ZnO/PbO and FeO_x/SiO_2 on the viscosity of the PbO-ZnO-Fe_3O_4-SiO_2-CaO slag was measured using the rotating spindle method. The slag viscosity decreased with decreasing ZnO/PbO mass ratio because of depolymerization of the silicate structures. The viscosity decreased with increasing FeO_x/SiO_2 mass ratio as experimental temperature was above 1623 K, while the viscosity increased significantly with the increase of FeO_x/SiO_2 as the experimental temperature below 1623 K because of phase transition and change of slag melting point. According to XRD analysis of as-quenched slag, the spinel and zincite phase increased with increasing FeO_x/SiO_2. Increasing ZnO/PbO and FeO_x/SiO_2 could enhance the crystallization capacity of the slag. FTIR analysis revealed that the degree of polymerization of the as-quenched slags decreased with decreasing ZnO/PbO and increasing FeO_x/SiO_2. The temperature dependencies of the viscosity on ZnO/PbO and FeO_x/SiO_2 were investigated, and the apparent activation energies of each system were found to be between 169.5 to 227.4 KJ/mol, and 151.1 to 676.4 KJ/mol, respectively.

Keywords: Viscosity; High lead and zinc slag; Slag structure; Apparent activation energy;

1. Introduction

Lead sulfide concentrate is the main raw material for lead smelting. The traditional routes of extracting lead from lead sulfide concentrate include desulfurization process in bath smelting process, followed by reduction process to obtain metal lead. The oxygen-enriched bottom blown smelting in desulfurization process is widely used in lead and copper smelting process in China due to low energy consumption and environment friendly [1-2]. The limited resources of high grade lead concentrate has aroused interest in the use of alternative lead bearing material which is seldom used in the bottom blown furnace due to the restrictions on the composition of raw materials in the existing lead smelting process. Especially the high Zn content raw materials such as lead and zinc mixed concentrate are abundant in China. These raw materials due to the high zinc content will lead to reduction of PbO content in oxidation slag, variation in the mass transfer in desulfurization process, the increase of slag melting point, and
the change of slag viscosity. Viscosity is one of the important physiochemical properties of slag, and low viscosity could promote the efficiency of the desulfurization reaction of slag. Considering the effect of slag composition on its physical properties, the slag composition must be optimized to accommodate changes to its viscosity.

FeO_x/SiO_2 is an effective means to adjust slag physical property in desulfurization process of lead smelting [3], it is important to study the effect of FeO_x/SiO_2 on the viscosity of slag with high ZnO content. Therefore, in order to make the oxidation furnace operate effectively, it is necessary to understand the dependence of slag viscosity on temperature and composition. Gupta [4] studied the viscosity of PbO-SiO_2 melts and found that the viscosity of slag decreased with the increasing content of PbO because of depolymerization of silicate network structure. Battle and Hager [5] found that addition of SiO_2 increased the viscosity of lead smelting slag, while additions of ZnO decreased the viscosity at high temperatures, but raised the slag liquidus temperature. Lv et al [6] investigated the effect of FeO content on viscosity and structure in the SiO_2-MgO-FeO-CaO-Al_2O_3 slag system. The results showed that the viscosity decreased and the mass fraction of the suspended particles increased with the increase of FeO content.

The final desulfurization slag can usually be approximately considered a mixture of oxides, including PbO, ZnO, FeO_x, SiO_2 and CaO from oxidation of lead raw material and flux of oxygen-enriched bottom blown furnace. However, the available information with respect to the viscosity of PbO-ZnO-Fe_3O_4-SiO_2-CaO system slag is in lack. Therefore, the effect of ZnO/PbO and FeO_x/SiO_2 on viscosity in PbO-ZnO-Fe_3O_4-SiO_2-CaO system was initially investigated in this article, and the effect of ZnO/PbO and FeO_x/SiO_2 on the activation energy (E_\eta) for viscous flow was estimated as well. The phase relations of the slag were studied by XRD. Furthermore, Fourier transform infra-red (FTIR) techniques were used to obtain the structural information of the molten slag.

2. Experimental

2.1 Sample preparation

The slag used in this experiment was synthesized with analytical pure grade PbO, ZnO, Fe_3O_4, SiO_2, CaO provided by Sinopharm Chemical Reagent co., Ltd. The slag composition (FeO_x/SiO_2=1.08, CaO/SiO_2=0.5) was based on the chemical composition of oxidation slag of typical oxygen-enriched bottom blown furnace in lead smelting industry in China.

The reagent was weighted according to the required composition given in Table 1 and mixed to become homogeneous. The loss caused by PbO volatilization [7] at high temperature was compensated by excess addition of PbO (~1.09 times the theoretical quantity).

<table>
<thead>
<tr>
<th>No.</th>
<th>PbO</th>
<th>ZnO</th>
<th>SiO_2</th>
<th>Fe_3O_4</th>
<th>CaO</th>
<th>FeO_x/SiO_2</th>
<th>CaO/SiO_2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>45</td>
<td>15</td>
<td>15.52</td>
<td>16.72</td>
<td>7.76</td>
<td>1.08</td>
<td>0.5</td>
</tr>
<tr>
<td>2</td>
<td>42.5</td>
<td>17.5</td>
<td>15.52</td>
<td>16.72</td>
<td>7.76</td>
<td>1.08</td>
<td>0.5</td>
</tr>
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<td>40</td>
<td>20</td>
<td>15.52</td>
<td>16.72</td>
<td>7.76</td>
<td>1.08</td>
<td>0.5</td>
</tr>
</tbody>
</table>
The composition of slag after the experiment was tested, which was presented in Table 2. It can be seen that there were some changes in the composition of slag after viscosity measurement. Besides, the test results showed that Al₂O₃ concentration (less than 2.5 wt. %) was dissolved in slag from corrosion of the crucible in viscosity measurement. In addition, the Al₂O₃ content in slag before and after viscosity measurement was measured. The results illustrated that the Al₂O₃ content varied in the range of 2.32 wt. % to 2.45 wt. %, and the change of Al₂O₃ content before and after viscosity measurement was less than 0.15 wt. %, which indicated the enrichment of Al₂O₃ in slag from corrosion of the crucible was mainly in the process of heating and equilibrium (1623 K). Therefore, the influence of the change of Al₂O₃ content in slag on viscosity measurement could be neglected. The same method was used to measure the viscosity of copper smelting slag [8-9], and the results demonstrated that the solubility of Al₂O₃ was negligible in the copper smelting or “Cu₂O”-SiO₂-Al₂O₃ melt slag under experimental conditions.

Table 2. The chemical composition of slag after the viscosity testing (wt. %)

<table>
<thead>
<tr>
<th>Number</th>
<th>PbO</th>
<th>ZnO</th>
<th>SiO₂</th>
<th>Fe₃O₄</th>
<th>CaO</th>
<th>Al₂O₃</th>
<th>Fe²⁺/TFe</th>
<th>FeO₃/SiO₂</th>
<th>CaO/ SiO₂</th>
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<td>32.6</td>
<td>1.07</td>
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<tr>
<td>2</td>
<td>42.9</td>
<td>17.15</td>
<td>14.53</td>
<td>15.55</td>
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<td>19.56</td>
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<td>15.72</td>
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<td>1.09</td>
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<td>32.9</td>
<td>1.07</td>
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<tr>
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<td>32.6</td>
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<td>32.4</td>
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<td>2.3</td>
<td>32.8</td>
<td>0.66</td>
<td>0.507</td>
</tr>
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<td>12.12</td>
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<td>2.41</td>
<td>32.1</td>
<td>1.65</td>
<td>0.502</td>
</tr>
</tbody>
</table>

2.2 Experimental apparatus and procedure

In this research, the viscosity of molten slag was determined by the rotating cylinder method. Fig. 1 shows the schematic diagram of the viscosity experimental device. An alumina crucible (inner diameter: 40 mm, height: 115mm) was put in a vertical tube furnace with MoSi₂ heating elements. The Pt-13 pct Rh/Pt thermocouple at the bottom of the crucible was used to monitor the slag temperature. Meanwhile, the furnace temperature was controlled by computer program. The rotating bob was made of alumina, and the viscometer was calibrated with castor oil with known viscosity before the experiment. It should be point out that the bob which used to measure the
viscosity was almost no change in the diameter before and after the experiment, which indicated the influence of diameter variation on viscosity measurement could be neglected.

Figure 1. Schematic diagram of viscosity experimental device

About 250 g of mixture was heated to 1623 K and equilibrated for 1 h to obtain the homogeneous slag. The bob was inserted into the molten slag and maintained at 10 mm above the bottom of the crucible, and then the viscometer (model: RTW-10) started measuring with the spindle at a fixed speed of 200 rpm. The value of slag viscosity was calculated and recorded by computer. The Ar was introduced into the furnace at a flow rate of 1.2 L/min to prevent the oxidation of slag. The errors of viscosity measurement were less than 0.002 Pa·s. When the temperature reached at each target temperature, the slag equilibrated at each experimental temperature for 30 minutes.

Kim and Sohn [10] used the breaking temperature to describe the property of slag. The breaking temperature is the critical temperature at which the viscosity varied greatly during the cooling cycle. And above the breaking temperature, the molten slag could be assumed to be full liquid [11].

After the viscosity measurement, the slag was reheated to the target temperature. Subsequently, parts of the slag were removed from the slag by iron rod and quenched in water for structure analysis. The slag phase and structure were analyzed by X-ray diffractometer (Bruker, D8 ADVANCE, Germany) and Fourier Transform Infrared Spectrometer (Nicolet, iS50, USA), respectively.

3. Results and Discussion

3.1 Effect of ZnO/PbO on slag viscosity

Fig. 2 shows the viscosity changes of the PbO-ZnO-Fe$_3$O$_4$-SiO$_2$-CaO system as a function of temperature with varying ZnO/PbO and a constant FeO$_3$/SiO$_2$ of 1.08 and CaO/SiO$_2$ of 0.5. The variation of ZnO/PbO in PbO-ZnO-Fe$_3$O$_4$-SiO$_2$-CaO slag system has a significant influence on slag viscosity. The viscosity increased as the ZnO/PbO increased. Further increase of ZnO/PbO above 1 has a remarkable effect in enhancing the viscosity. According to work done by Gupta [4] and Jin [12] et al, both PbO and ZnO suggested to be a weak basic oxide in a silicate melts, which could decreased the slag viscosity by depolymerizing the slag network structure. It seems that PbO is better than ZnO in depolymerizing the network structure, thus increasing PbO/ZnO decreases the slag viscosity. According to the study of Jin et al [12], the increasing ZnO content in slag would lead to the increase of crystallization capacity of the slag. In this study,
the breaking temperature increased rapidly with the increase of PbO/ZnO, which further confirmed this point.

**Figure 2. Viscosity of PbO-ZnO-Fe$_3$O$_4$-SiO$_2$-CaO system as a function of temperature with different ZnO/PbO**

Fig. 3 shows the XRD analysis of the as-quenched slags with various ZnO/PbO in the PbO-ZnO-Fe$_3$O$_4$-SiO$_2$-CaO slag system. It can be seen that the main crystalline phases for the ZnO/PbO = 0.33 (ZnO=15 wt. %, PbO=45 wt. %) was identified as spinel phase ($\text{Zn}_x\text{Fe}_{3-x}\text{O}_{4+y}$). The peaks intensity of spinel phase was gradually increased as the increase of ZnO/PbO. When the ZnO/PbO increased to 0.714 (ZnO=25 wt. %, PbO=35 wt. %), the diffraction peaks for zincite phase (ZnO) emerged. As the ZnO/PbO continue to increase, the diffraction peaks for ZnO and Fe$_3$O$_4$ became more pronounced. It is reported that the increase of spinel and zincite phase in slag is the main reason for the increase of viscosity in lead bath smelting process [13]. Thus, a subsequent decrease of ZnO/PbO ratio shows an increase effect in lowing the slag viscosity.

**Figure 3. The XRD analysis of as-quenched slag with different ZnO/PbO**

The temperature dependence of viscosity can be represented by the Arrhenius equation [8].
\[ \ln \eta = A + \frac{E_\eta}{R \times T} \]  

(1)

where \( \eta \) is the viscosity, Pa·S; \( A \) is a constant, Pa·S; \( R \) is the gas constant, 8.314 J/mol·K; \( T \) is the absolute temperature, K; \( E_\eta \) is the apparent activation energy stand for the sensibility of viscosity to temperature, and the change of \( E_\eta \) can illustrates variation in frictional resistance of viscous flow and the change of slag structure [14], J/mol;

Fig. 4 shows a linear fit between the natural logarithm of the viscosity (\( \ln \eta \)) and the reciprocal temperature (1/T). Clearly, the viscosity decreased with increasing temperature. The value of \( E_\eta \) could be obtained from the linear fitting results of \( \ln \eta \) with 1/T. The corresponding \( E_\eta \) with variation slag composition was calculated to be between 169.5 and 227.4 KJ/mol, and the results were listed in Table 3. It can be seen that the increase of ZnO/PbO led to the increase of apparent activation energy of the viscosity. Moreover, the decrease of \( E_\eta \) illustrated the formation of small structural units in slag and the decrease of resistance for viscous flow [12], which was similar to the change of measured viscosity.

![Figure 4. The relationship between natural logarithm of the viscosity (Ln\( \eta \)) and the reciprocal temperature (I/T) at different ZnO/PbO](image)

**Table 3. The apparent activation energy of viscosity with different ZnO/PbO in PbO-ZnO-Fe_{3}O_{4}-SiO_{2}-CaO slag system**

<table>
<thead>
<tr>
<th>Slag Number of Table 1</th>
<th>Range of Temperature (K)</th>
<th>( E_\eta ) (KJ/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1448 to 1623</td>
<td>169.5</td>
</tr>
<tr>
<td>2</td>
<td>1448 to 1623</td>
<td>169.8</td>
</tr>
<tr>
<td>3</td>
<td>1448 to 1623</td>
<td>172.2</td>
</tr>
<tr>
<td>4</td>
<td>1448 to 1623</td>
<td>187.3</td>
</tr>
<tr>
<td>5</td>
<td>1548 to 1623</td>
<td>205.8</td>
</tr>
<tr>
<td>6</td>
<td>1548 to 1623</td>
<td>208.5</td>
</tr>
<tr>
<td>7</td>
<td>1548 to 1623</td>
<td>213.3</td>
</tr>
</tbody>
</table>
3.2 Effect of FeOx/SiO₂ on slag viscosity

Fig. 5 shows the viscosity variation of the PbO-ZnO-Fe₃O₄-SiO₂-CaO system as a function of temperature with varying FeOx/SiO₂ and a constant ZnO/PbO of 1.18 and CaO/SiO₂ of 0.5, it can be seen that the increase of FeOx/SiO₂ could lower the viscosity of the slag as experimental temperature was at 1623 K. The basic oxide behavior of Fe₃O₄, which, like CaO, is a modifier of the network structure of silicate [15], it was also confirmed by the FTIR results as well. The same results was reported by other investigators [6]. However, when experimental temperature was below 1598 K, the viscosity increased significantly with the increase of FeOx/SiO₂. Note that the increase of Fe₃O₄ in lead smelting slag can increase the melting point of the slag, thus causing various viscosity [13]. With the increase of FeOₓ/SiO₂, the gradient of the viscosity curve got sharper below the breaking temperature, which further confirmed that the breaking temperature of the slag increased with the increase of Fe/ SiO₂.

![Figure 5. Viscosities in the PbO-ZnO-Fe₃O₄-SiO₂-CaO slag system as a function of temperature with different FeOx/SiO₂](image)

In order to know the relationship between the viscosity and slag composition with increase of FeOx/SiO₂, the XRD analysis of the as-quenched slags with various FeOx/SiO₂ in the PbO-ZnO-Fe₃O₄-SiO₂-CaO slag system was carried out. As shown in Fig. 6, the main crystalline phases of the slag was identified as zincite when FeOₓ/SiO₂ was at0.65. With FeOₓ/SiO₂ increasing to0.86, the diffraction peaks for spinel phase became observable. Moreover, the spinel phase increased as FeOₓ/SiO₂ continued to increase. This illustrated that the addition of silica could inhibit the formation of spinel phase.

Based on the above study results, the increase of FeOₓ/SiO₂ would not only increase the slag melting point, but also led to the phase transformation of slag. These could be the reason that led to change of slag with various FeOₓ/SiO₂ ratio as experimental temperature below 1598 K.
Figure 6. The XRD analysis of as-quenched slag with different FeO$_x$/SiO$_2$

The viscosities of various FeO$_x$/SiO$_2$ slag as a function of reciprocal of the temperature were shown in Fig. 7. $E_\eta$ in this system was estimated to be between 151.1 and 676.4 KJ/mol, and the corresponding $E_\eta$ with various FeO$_x$/SiO$_2$ was listed in Table 4.

![Figure 6: XRD analysis of as-quenched slag with different FeO$_x$/SiO$_2$](image)

Figure 7. The relationship between natural logarithm of the viscosity (Ln $\eta$) and the reciprocal temperature (1/T) at different FeO$_x$/SiO$_2$

Table 4. The apparent activation energy of viscosity with different FeO$_x$/SiO$_2$ in PbO-ZnO-SiO$_2$-Fe$_3$O$_4$-CaO slag system

<table>
<thead>
<tr>
<th>Slag Number of Table 1</th>
<th>Range of Temperature (K)</th>
<th>$E_\eta$ (KJ/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9</td>
<td>1523 to 1623</td>
<td>151.1</td>
</tr>
<tr>
<td>10</td>
<td>1573 to 1623</td>
<td>182.8</td>
</tr>
<tr>
<td>11</td>
<td>1573 to 1623</td>
<td>676.4</td>
</tr>
</tbody>
</table>

3.3 FTIR Analysis

It is generally considered that the viscosity is related to the degree of polymerization of silicate slag [16-17]. The FTIR transmittance of the as-quenched slag
as a function of wavenumbers at different ZnO/PbO and FeO\textsubscript{x}/SiO\textsubscript{2} are exhibited in Figure 10 and 11. The infrared spectra of silicate slag is mostly concentrated in the wavenumber range of 1200 to 400 cm\textsuperscript{-1} [18-20]. This wavenumber region includes the stretching vibration bands of the [SiO\textsubscript{4}]\textsuperscript{-}\textsubscript{tetrahedra} (wavenumber: 1100-850) and [AlO\textsubscript{4}]\textsuperscript{-}\textsubscript{tetrahedra} (wavenumber: 750-590), and the Si-O bending vibration bands (wavenumber: ~500), respectively [6].

In the study of silicate melt structure, the average number of non-bridge oxygen (NBO/Si) is often used to illustrate the polymerization of silicate slag structures [21], where the lower NBO/Si indicates the higher degree of polymerization of molten slag [11]. The wavenumber region between 1200 and 800 cm\textsuperscript{-1} is widely known to be the [SiO\textsubscript{4}]\textsuperscript{-}\textsubscript{tetrahedra} peaks [11] with various NBO/Si (NBO/Si=1, 2, 3, 4). According to the study of B. O. Mysen et al [22], the wavenumber region of 1100-1050 cm\textsuperscript{-1}, 980-950 cm\textsuperscript{-1}, 920-900 cm\textsuperscript{-1}, 880-850 cm\textsuperscript{-1} represent the band groups of [SiO\textsubscript{4}]\textsuperscript{-}\textsubscript{tetrahedral with NBO/Si = 1 (sheets), 2 (chains), 3 (dimers) and 4 (monomers), respectively. Clearly, as shown in Fig. 8, the [SiO\textsubscript{4}]\textsuperscript{-}\textsubscript{tetrahedra peaks at about 750 to 1150 cm\textsuperscript{-1} got weaker when the ZnO/PbO was decreased, which demonstrated that the structure of slag could be depolymerized by the decrease of ZnO/PbO. The results observed in Fig. 9 shows that the characteristic stretching vibration band for the large silicate network structures became less pronounced and the depth of the convoluted band became shallower, which illustrated increase of FeO\textsubscript{x}/SiO\textsubscript{2} were likely to depolymerize the silicate network structure into simpler polymer type units. Therefore, the depolymerization of silicate network structure could reduce the polymerization degree of complex viscous units, thus decreasing the slag viscosity.

Figure 8. FTIR results of as-quenched slag from 1623 K with different ZnO/PbO ratio
4. Conclusion

The viscosities of PbO-ZnO-Fe$_3$O$_4$-SiO$_2$-CaO lead smelting slags were measured using the rotational spindle method, and the phase structure were studied by XRD and FTIR analysis. The following conclusions could be obtained as follows:

1. The viscosity of molten slag decreased as the ZnO/PbO mass ratio decreased from 1.18 to 0.33, and the decrease trend was more obvious when ZnO/PbO was less than 0.714 and temperature was below 1523 K.

2. The effect of FeO$_X$/SiO$_2$ in PbO-ZnO-Fe$_3$O$_4$-SiO$_2$-CaO system on the viscosity were complicated due to the phase transition of the basic slag and change of slag melting point ranging from 1523 to 1573 K. XRD analysis of as-quenched molten slag samples revealed that the phase could be changed in the slags with the lower FeO$_X$/SiO$_2$ mass ratio.

3. The apparent activation energy of viscosity at different ZnO/PbO and FeO$_X$/SiO$_2$ was estimated to 169.5 to 227.4 KJ/mol, and 151.1 to 676.4 KJ/mol, respectively. The variation of the apparent activation energy was similar to the change in viscosity.

4. The PbO or FeO$_X$ is likely to be a network modifier and provided free O$^{2-}$ to depolymerize ([Si$_x$O$_y$])$^n$-tetrahedra into simpler polymertype units, which resulted in the decrease of viscosity.

Acknowledgements

This study was supported by the National Natural Science Foundation of China (51674074, 51774078) and the Fundamental Research Funds for the Central Universities (N162505002)

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