

# Effect of CaO/SiO<sub>2</sub> and Fe/SiO<sub>2</sub> Ratios on the Viscosity at 1300°C of Partly Crystallized Silicate Slags

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**Abstract**—The present study reports an experimental investigation of the effects of CaO/SiO<sub>2</sub> mass ratio (0.4 to 1.2) and Fe/SiO<sub>2</sub> ratio (1.26 and 1.7) on viscosity of the heterogeneous system of the CaO–SiO<sub>2</sub>–FeO–Fe<sub>2</sub>O<sub>3</sub>–PbO–MgO–ZnO–S slag at 1300°C. The viscosity was measured using rotating cylindrical viscometer, and structure of quenched slags was studied by X-ray diffraction and SEM-EDS analysis. It was found that the viscosity of the slurry decreased with an increase in CaO/SiO<sub>2</sub> ratio and increased with an increase in Fe/SiO<sub>2</sub> ratio. The sulphur content in the slag decreases the FeO/Fe<sub>2</sub>O<sub>3</sub> ratio in the liquid slag and consequently increases the viscosity. The FactSage thermodynamic software is utilized for the phase equilibrium and viscosity predictions. The effect of solid fractions in slag phase on the viscosity is calculated using the Einstein–Roscoe equation. Calculations show that an increase in solid fractions increases the viscosity of the slag phase.

**Keywords:** viscosity, silicate slags, partly crystallized slag

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## INTRODUCTION

The control of slag properties such as viscosity is essential in maintaining the pyrometallurgical processes efficiency. The slag viscosity is very sensitive to the changes of temperature and composition. The suitable viscous flow of molten slag can facilitate the chemical reaction to eliminate impurities and control the heat transfer, mass transfer and smelting stability related to the active multiphase reaction among molten slag, liquid metal, and gas.

Viscosities of liquid slag systems have been extensively studied and many theoretical models have been developed to predict the viscosity of a fully liquid silicate [1–4]. However, not all slag melts are fully liquid at the operating temperatures. Silicate slags may become quite viscous due to the precipitation of solid phases, leading to problems with slag tapping or appropriate separation of metallic phase. Sycheva et al. [5–7] have studied the nucleation of crystals in glass obtained by melting furnace slags with the additive of SiO<sub>2</sub>.

Several viscosity models have been established to correlate the viscosity and solid fraction. The most widely used viscosity model for heterogeneous system

is the Einstein–Roscoe (*E–R*) equation [8, 9]. Kondratiev and Jak [10] validated the applicability of the *E–R* equation with a large experimental data set in the Al<sub>2</sub>O<sub>3</sub>–CaO–FeO–SiO<sub>2</sub> slag.

Liu et al. [11] discussed a review of the viscosity of solid-bearing silicate melts where it is mentioned that several characteristics of the high-temperature melts, such as temperature, dwelling time, and shear rate, which leads to change in crystal fraction should be noticed. Wang et al. [12] showed that an increase in the CaO/SiO<sub>2</sub> ratio led to the decrease in viscosity in silicate mould fluxes with B<sub>2</sub>O<sub>3</sub>. Wright et al. [13] reported an experimental investigation of the effect of solid suspension on the viscosity of molten slags. They used up to about 20 vol % of spinel (MgAl<sub>2</sub>O<sub>4</sub>) particles of the size range from 0.10 to 0.99 mm.

FactSage thermodynamic software has been used for a better understanding of slag–metal interaction in both ferrous and non-ferrous processes. It is well known for predicting metallurgical processes, multiphase equilibria, liquidus temperature and chemical and metallurgical process modelling [14]. This software was also used by Mishra et al. [15] to study the deposition characteristics of ash particles based on the

**Table 1.** Chemical composition of the master slag

Component	mas. %
SiO <sub>2</sub>	19.35
CaO	19.88
Fe <sub>2</sub> O <sub>3</sub>	34.84
ZnO	8.4
ZnS	11.26
MgO	3.9
PbO	2.37

**Table 2.** Mass in grams of the slags used in the viscosity and the equilibrium-quenched trials. The mass of ZnO (8.4 g), ZnS (11.26 g), MgO (3.9 g) and PbO (2.37 g) were kept constant

Fe/SiO <sub>2</sub>	CaO/SiO <sub>2</sub>	CaO	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>
1.26	0.4	19.88	49.7	89.53
	0.6	19.88	33.13	59.68
	0.8	19.88	24.85	44.77
	1.027	19.88	19.35	34.84
	1.2	23.22	19.35	34.84
1.7	0.4	19.88	49.70	120.80
	0.6	19.88	33.13	80.53
	0.8	19.88	24.85	60.40
	1.027	19.88	19.36	47.05
	1.2	23.22	19.35	47.03

viscosity and phase transformation properties in boiler operation. They also studied the effect of solid or crystalline fractions in slag phase on the viscosity.

The lead ore produced in Mexico has historically been different from that around the rest of the world as the Mexican lead ores contains high concentrations of silver and bismuth. The recovery of precious metals during lead production in a blast furnace makes this process more profitable. Losses of silver and lead are common in this process and can be due to both physical and chemical phenomena. Lead can be trapped as metallic particles in the slag [16], due to its composition and viscosity.

The present study has been conducted to provide experimental data of the viscosity of the heterogeneous system of the CaO–SiO<sub>2</sub>–FeO–Fe<sub>2</sub>O<sub>3</sub>–PbO–MgO–ZnO–S slag at 1300°C (1573 K) with the following mass ratios: CaO/SiO<sub>2</sub> = 0.4 to 1.2 and Fe/SiO<sub>2</sub> = 1.26 and 1.7. These composition parameters have been selected because they represent the slags typically encountered in the Mexican lead blast furnaces. The equilibrium-quenching method was also used to estimate the solid fraction and the species that are stable at high temperature experimentally.

Factsage thermodynamic software [14] has incorporated an extensive amount of results on the PbO–ZnO–CaO–SiO<sub>2</sub>–Fe<sub>2</sub>O<sub>3</sub>–FeO–Al<sub>2</sub>O<sub>3</sub> slag systems in order to obtain one set of model equations for the Gibbs energy of the liquid slag. This software is used in this work as a tool to calculate the solid fraction of the system. This computer program can also calculate the viscosities of fully liquid silicate slags. The model links the viscosities of silicate melts to their structure and thermodynamic properties.

## EXPERIMENTAL

### Materials

A typical slag sample obtained from the smelting of lead concentrate in a blast furnace was used as master slag. The as-received sample was crushed to –100 mesh size for the X-ray fluorescence analysis. Table 1 shows the chemical composition of the slag. The master slag contains the following mass ratios Fe/SiO<sub>2</sub> = 1.26 and CaO/SiO<sub>2</sub> = 1.027. It is worth to note that the chemical analysis reported 3.7 mas. % of sulphur, which is considered as ZnS since this sulphide is thermodynamically more stable than FeS and PbS. To estimate the effect of the CaO/SiO<sub>2</sub> and Fe/SiO<sub>2</sub> ratios on the viscosity and the solid fraction at high temperature, reagent-grade chemicals (above 99.5 mas. % purity) of SiO<sub>2</sub>, CaO and Fe<sub>2</sub>O<sub>3</sub> powders were added to the master slag. The slag compositions used in the viscosity and the equilibrium-quenching trials are reported in Table 2.

### Procedure to Estimate Slag Viscosity

The viscosity was measured by the rotating cylinder method. The scheme of the apparatus used is shown in Fig. 1a, where the slag sample was melted in a chromemagnesite crucible 4.4 mm in diameter and 80 mm in height and placed in a vertical tubular resistance furnace, using a molybdenum spindle. The industrial slag contains some sulphur, so argon atmosphere were used as a protective gas to prevent the corrosion at high temperature. The temperature was measured by a Pt/Pt–Rh 10 thermocouple. The system was calibrated using a standard oil with known viscosity for the range from 0.29 to 1.8 Pa·s at 298 K. The reproducibility of the measurements was approximately ±10%.

### Equilibrium-Quenching Trials

Figure 1b shows a schematic illustration of the experimental setup to estimate the mineralogical species and the fraction of solid in equilibrium with liquid slag at high temperature. It consists of a tubular resistance furnace where the tube was of alumina with an internal diameter of 65 mm. 20 g of each slag sample with the composition given in Table 2 were homogenized and equilibrated in molybdenum crucible

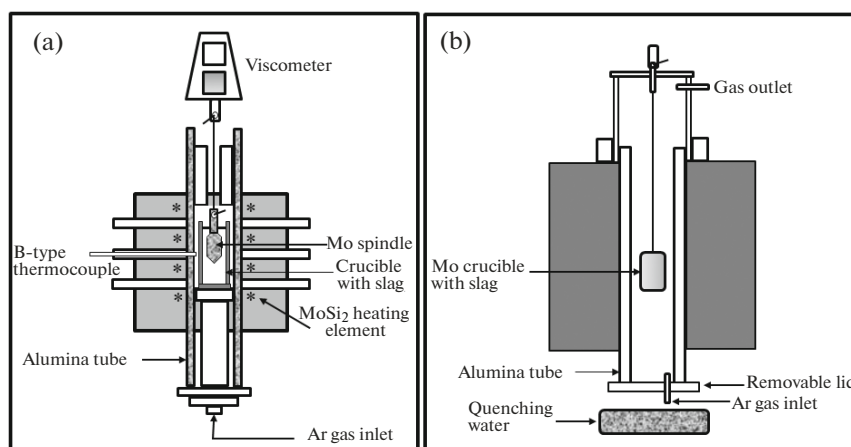


Fig. 1. Schematic representation of experimental setup for the viscosity (a) and equilibrium-quenching trials (b).

(15 mm inner diameter and 40 height) in argon, at 1300°C. After holding for 2 h at the set temperature (the time required to achieve equilibrium between the condensed phases of slag) the samples were quenched in iced water. On rapid cooling, the silica-containing liquid phase converts to glass so that the phase assemblage that exists at high temperature is frozen.

The phases present were determined by first mounting and polishing the samples, and then by examination using optical microscopy and scanning electron microscopy coupled with an energy-dispersive spectra analyzer (FEI Quanta 600, EDAX EDS). Samples of each slag were also characterized by X-ray diffraction (XRD Bruker D8 Focus) using CuK<sub>α</sub> (λ = 1.5406 Å) radiation over a 2θ of 20° to 100° at a speed of 2 min<sup>-1</sup> to determine the mineralogical species.

#### Viscosity Calculations Using FactSage and the Einstein–Roscoe Equation

The simple and most well-known model based on Einstein’s approach was due to Roscoe. The Einstein–Roscoe type equation is given as follows [8, 9, 17–21]:

$$\eta = \eta_L [1 - a\phi_s]^{-n}, \quad (1)$$

where  $\eta$  and  $\eta_L$  are the viscosity of the solid-containing and the solid-free melt, respectively,  $\phi_s$  is the volume fraction of solid particles in the melt,  $a$  and  $n$  are empirical parameters that depend on the morphology of the solids.  $a$  and  $n$  are considered equal to 1 and 2.5, respectively, for rigid spheres of diverse sizes [9]. The volume fraction of solids is expressed as:

$$\phi_s = \frac{V_s}{V_s + V_L}, \quad (2)$$

$$V_s = \sum_j v_j^s \frac{m_j^s}{M_j^s}; \quad V_L = \sum_i v_i^L \frac{m_i^L}{M_i^L}, \quad (3)$$

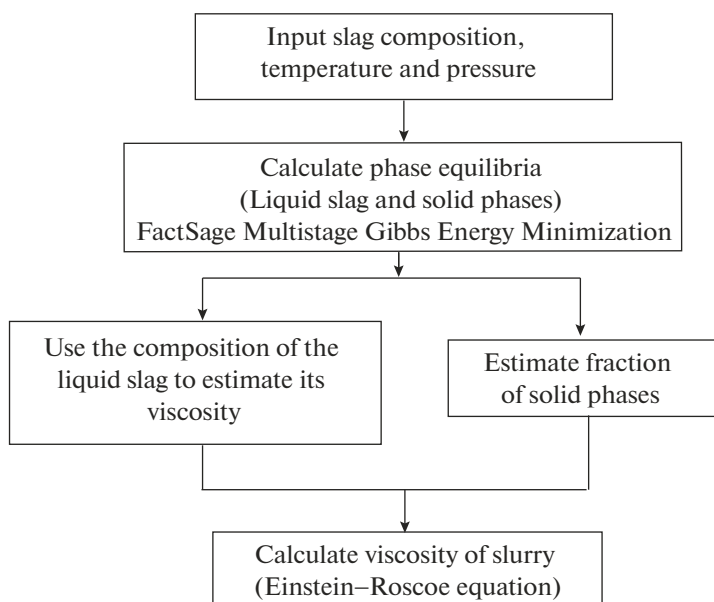
where  $V_s$  and  $V_L$  are the volumes of the crystallized solids and the residual liquid,  $v$  is the partial molar volume,  $m$  is mass and  $M$  is molar mass of the component in solid or liquid phases.

The values of the molar partial volume are not very well-known for liquid slags, so in the present work the partial molar volumes are assumed to be equivalent to the molar volumes of pure components which are shown in Table 3.

FactSage consists of several thermodynamic calculation modules such as (1) Reaction, (2) Equilib, (3) Predom, (3) Viscosity, etc. Multiphase equilibrium in the present study is calculated by using the “Equilib” module [14]. Gibbs free energy minimization and the modified non-ideal associate species models are used for the phase equilibrium calculations. The “Viscosity” module calculates the viscosity of a liquid slag solution at a given temperature and composition. The flow sheet to estimate the viscosity of a solid + liquid

Table 3. Molar volumes of pure compounds

Compound	Molar volume of pure compounds, cm <sup>3</sup> mol <sup>-1</sup>	Reference
PbO	27.5	[18]
FeO	15.8 * [1.0 + 0.0001*(T - 1773.0)]	[19]
ZnO	13.59	[21]
SiO <sub>2</sub>	27.516 * [1.0 + 0.0001*(T - 1773.0)]	[19, 20]
CaO	20.7 * [1.0 + 0.0001*(T - 1773.0)]	[19]
MgO	16.1 * [1.0 + 0.0001*(T - 1773.0)]	[19]
Fe <sub>2</sub> O <sub>3</sub>	15.8 * [1.0 + 0.0001*(T - 1773.0)]	[19]



**Fig. 2.** Flowsheet of the method to calculate de slag viscosity of a heterogeneous system using the FactSage software and the Einstein–Roscoe equation.

mixture using the Factsage software and the Einstein–Roscoe equation is shown in Fig. 2.

## RESULTS AND DISCUSSION

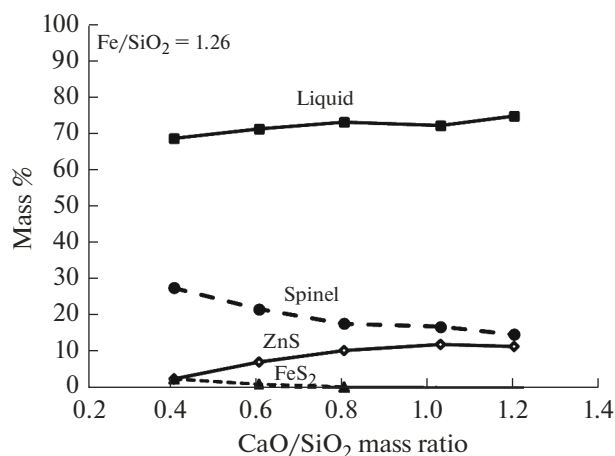
### *Thermodynamic and Experimental Viscosity Results*

The FactSage computational thermodynamic package [14] can be used to determine the equilibrium species in the slag since it contains an extensive optimized solution database. This system uses the quasi-chemical approximation [22] for the liquid oxide-based slags. The general free energy minimization method is used to estimate the equilibrium concentration once the models and the thermodynamic proper-

ties of the species in the system have been selected. For a given set of constraints (such as fixed temperature, pressure and overall concentration), the free energy minimization algorithm finds the set of mole numbers of each species as well as the compositions of all solution phases which globally minimize the free energy function. This software was used to estimate the liquid and solid phase distribution for the slags in terms of slag basicity ( $\text{CaO}/\text{SiO}_2$ ) and  $\text{Fe}/\text{SiO}_2$  mass ratios and  $1300^\circ\text{C}$  ( $1573\text{ K}$ ).

Figures 3, 4 show the amount of liquid and solid phases at equilibrium at  $1300^\circ\text{C}$  in terms of  $\text{CaO}/\text{SiO}_2$  mass ratio for slags with  $\text{Fe}/\text{SiO}_2$  ratio of 1.26 and 1.7, respectively. These figures show that increasing the slag basicity the amount of liquid phase is increased at both  $\text{Fe}/\text{SiO}_2$  ratios. It is also observed that a given basicity the liquid is slightly higher for slags with low  $\text{Fe}/\text{SiO}_2$  ratio (1.26). The main solid phases calculated by the FactSage software are wurtzite ( $\text{ZnS}$ ), pyrite ( $\text{FeS}_2$ ) and spinel, which is a solid solution mainly formed by zinc ferrite ( $\text{ZnFe}_2\text{O}_4$ ). It is observed that most of the sulphur content is associated with zinc.

FactSage only calculate the amount of equilibrium phases in mass (g/mol). To estimate the molar volume of the liquid phases the results in mass are combined with the molar volume given in Table 3 and equation 3. The volume of each solid is calculated considering the reported density,  $5\text{ g cm}^{-3}$  for pyrite and  $3.98\text{ g cm}^{-3}$  for wurzite. Spinel is considered mainly formed by  $\text{ZnFe}_2\text{O}_4$  with density  $5.322\text{ g cm}^{-3}$ . The volume fraction of solid is straightforward calculated with equation 2.



**Fig. 3.** Calculated phases in equilibrium at  $1300^\circ\text{C}$  in slags with  $\text{Fe}/\text{SiO}_2 = 1.26$ .

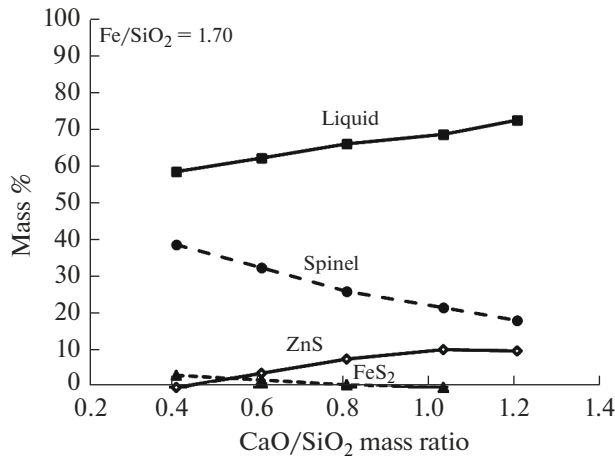


Fig. 4. Calculated phases in equilibrium at 1300°C in slags with Fe/SiO<sub>2</sub> = 1.70.

The fraction of solid phases and composition of the liquid slag phase at different CaO/SiO<sub>2</sub> and Fe/SiO<sub>2</sub> ratios were calculated by using the module “Equilib” of the FactSage software. The viscosity of the solid-free liquid ( $\eta_l$ ) was calculated through the module “Viscosity”. The mass fraction of solids was transformed to volume fraction and then the Einstein–Roscoe equation was used to estimate the viscosity of the slurry system.

Figures 5, 6 show the calculated and the experimental viscosities results of the slags with Fe/SiO<sub>2</sub> ratio of 1.26 and 1.7, respectively. From these calculations, it is evident that viscosity value increases by increasing the Fe/SiO<sub>2</sub> due to the increase of the volume fraction of solids. These figures also show that there is a qualitative agreement between the experimental and calculated results, where it is observed that the viscosity decreases as the slag basicity is increased. However, there are discrepancies between experimental and calculated viscosities which may be mainly due to the fact that the lead blast furnace slag contains many components and there is no thermodynamic database to perform the phase equilibrium calculation considering all compositions and temperatures of this silicate-based complex system. The FactSage software performs an extrapolation of thermodynamic properties to determine the phase equilibrium at compositions and temperatures that have not been obtained experimentally. Another cause of the differences between experimental and calculated results is the fact that the Viscosity module of the FactSage program does not consider the presence of metal sulphides. Finally, the Einstein–Roscoe equation employs the empirical shape parameters “*a*” and “*n*” which might vary according to the size and shape of the solid particles.

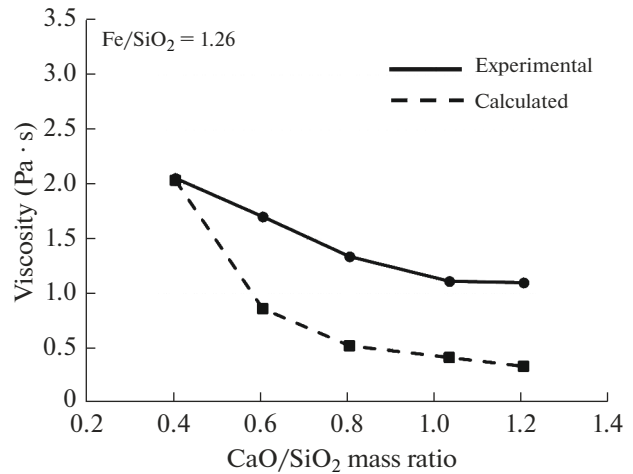


Fig. 5. Calculated and experimental viscosities at 1300°C of slags with Fe/SiO<sub>2</sub> = 1.26.

### Microstructure

There is no experimental evidence that at the temperature tested (1300°C) there exists in equilibrium a mixture of liquid slag and solid phases in the lead blast furnace process, due to the many components present in this industrial system, although thermodynamically the presence of these phases in equilibrium was determined using the FactSage software. For this reason, equilibrium and quenching tests were carried out in this work, considering the same compositions of the slags used in the viscosity tests, reported in Table 2.

The microstructures of the quenched slags with Fe/SiO<sub>2</sub> = 1.26 and CaO/SiO<sub>2</sub> = 0.4 and 1.2 are shown in Fig. 7. It is observed that at 1300°C the slag is partly melted and the rapid cooling to room temperature produces a dendritic structure. The SEM-EDS results show that in Fig. 7a the solid phases in equilibrium

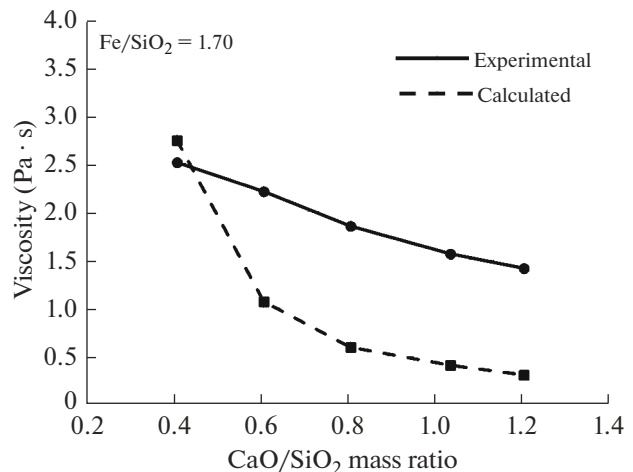
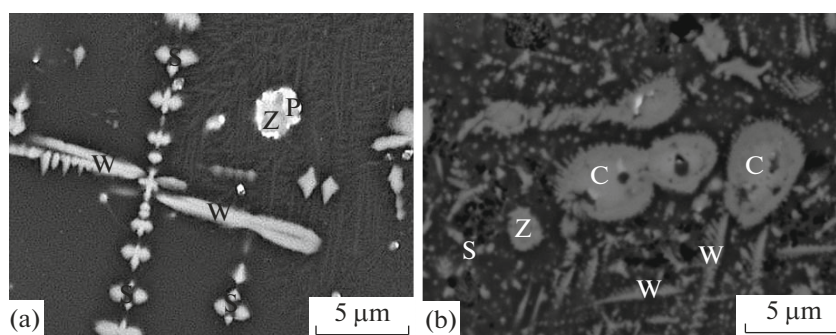
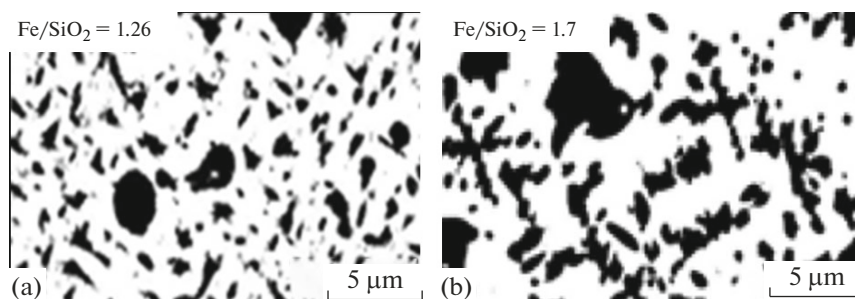


Fig. 6. Calculated and experimental viscosities at 1300°C of slags with Fe/SiO<sub>2</sub> = 1.70.



**Fig. 7.** Micrographs of the slags equilibrated and quenched from 1300°C with  $\text{Fe}/\text{SiO}_2 = 1.26$  and  $\text{CaO}/\text{SiO}_2 = 0.4$  (a),  $\text{CaO}/\text{SiO}_2 = 1.2$  (b). S:  $\text{ZnFe}_2\text{O}_4$ , W: Wustite, C:  $\text{CaFeSi}_2\text{O}_6$ , Z:  $\text{ZnS}$ , P:  $\text{FeS}_2$ .



**Fig. 8.** Micrographs of the image analyzer for slags with  $\text{CaO}/\text{SiO}_2 = 1.027$ .

with liquid are wustite ( $\text{FeO}$ ) and zinc ferrite ( $\text{ZnFe}_2\text{O}_4$ ) for the system with low slag basicity ( $\text{CaO}/\text{SiO}_2 = 0.4$ ). There were also observed in this sample rounded particles with combined metallic sulphide phases, pyrite ( $\text{FeS}_2$ ) and wurtzite ( $\text{ZnS}$ ). The slag with high slag basicity ( $\text{CaO}/\text{SiO}_2 = 1.2$ ) also shows solid particles of wustite and zinc ferrite together with pyroxene hedenbergite  $\text{CaFeSi}_2\text{O}_6$ .

The micrographs of the quenched slags were used to estimate the volume fraction of solids through an image analyzer, such as is shown in Fig. 8. These images correspond to slags with  $\text{CaO}/\text{SiO}_2 = 1.027$ . The experimental volume fractions of solids are 26.1 and 31.9% for slags with  $\text{Fe}/\text{SiO}_2$  with 1.26 and 1.7, respectively. These results are close to the calculated volume fractions of solids calculated by the software FactSage, which are 25.7 and 28.1%, respectively. The average difference between the experimental and the calculated volume fraction of solids were less than 10%.

The X-ray diffraction (XRD) analysis was used to confirm phase identification carried out by SEM-EDS. Figures 9, 10 show the XRD patterns for slags with different  $\text{Fe}/\text{SiO}_2$  ratio and  $\text{CaO}/\text{SiO}_2$  being 0.4 and 1.2, respectively, equilibrated at 1300°C and quenched to room temperature. Zinc ferrite  $\text{ZnFe}_2\text{O}_4$  (JCPD file 22-1012), wustite  $\text{Fe}_{0.932}\text{O}$  (JCPD file 06-

0615) and  $\text{CaFeSi}_2\text{O}_6$  (JCPD file 70-1876) were observed at most of the experiment conditions as the main solid phases obtained in these slags. Small peaks of wurtzite  $\text{ZnS}$  (JCPD file 39-1363) and  $\text{CaO}$  (JCPD file 37-1497) were also observed. It is worth noting that the presence of crystals of  $\text{CaO}$  and  $\text{CaFeSi}_2\text{O}_6$  were not clearly observed in the SEM-EDS microanalysis. These figures also show that increasing the  $\text{Fe}/\text{SiO}_2$  basicity, the amount of wustite and zinc ferrite was increased.

The thermodynamically determined solid species were spinel, which consists mainly of zinc ferrite, and two sulphides ( $\text{FeS}_2$  and  $\text{ZnS}$ ). It should be mentioned that wustite ( $\text{Fe}_{0.932}\text{O}$ ) was not determined as a solid phase but it was reported as a component of the liquid slag in the equilibrium calculations; however, this phase was detected in the experimental tests by XRD and SEM-EDS.

Park et al. [23] studied the viscous behavior of  $\text{FeO}_t\text{-Al}_2\text{O}_3\text{-SiO}_2$  copper smelting slags and found that the slag viscosity as a function of  $\text{Fe}/\text{SiO}_2$  ratio showed a common pattern of decreasing viscosity with increasing  $\text{Fe}/\text{SiO}_2$ . Osugi et al. [24] also found that the viscosity of silicate melts decreased with increasing the amount of  $\text{FeO}$ , since it behaves as network modifier and depolymerizes the silicate melts. Unlike Park's and Osugi's works, we observed that the slag

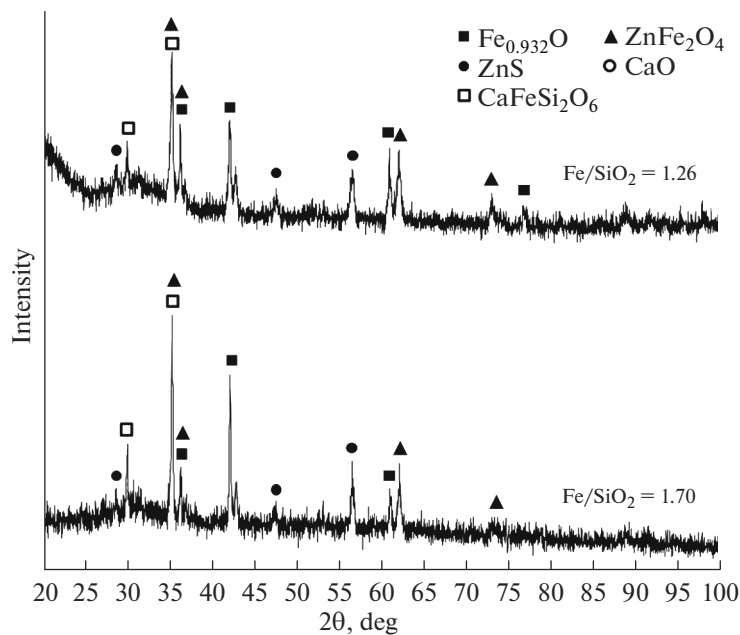


Fig. 9. X-ray diffraction patterns for slags with CaO/SiO<sub>2</sub> = 0.4.

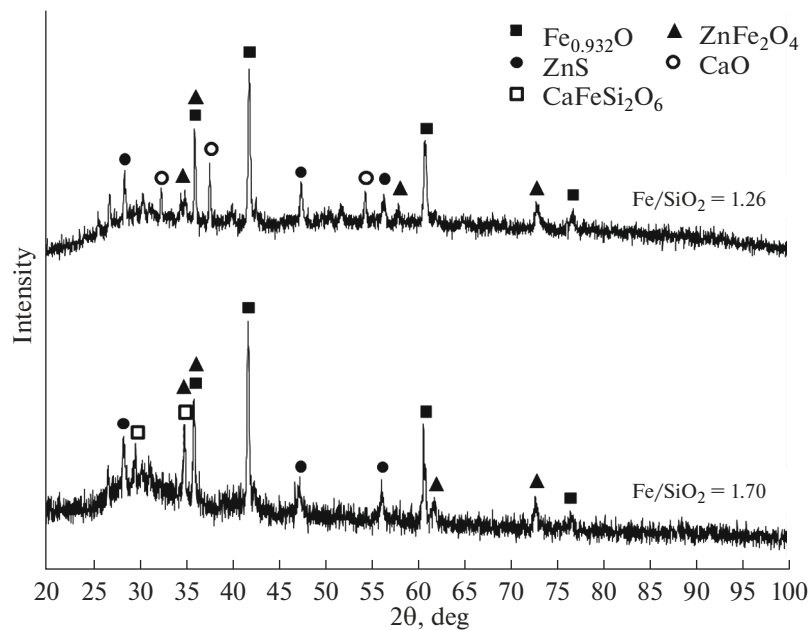


Fig. 10. X-ray diffraction patterns for slags with CaO/SiO<sub>2</sub> = 1.2.

viscosity is increased by increasing the Fe/SiO<sub>2</sub> ratio. The reason is that in the slags from the lead blast furnace at 1300°C the fraction of solid phases, as well as the viscosity of the slag, increase as Fe/SiO<sub>2</sub> increases.

Despite the simplifications made in this work to calculate the viscosity of a heterogeneous slag using the FactSage thermodynamic software and the Einstein–Roscoe, this method provides an insight of the rheological behavior of complex silicate-based slags.

## CONCLUSIONS

An experimental study of the viscosity and the equilibrium and quenching of slags from the lead blast furnace with the mass ratios: CaO/SiO<sub>2</sub> = 0.4 to 1.2 and Fe/SiO<sub>2</sub> = 1.26 and 1.7 at 1300°C was carried out. The major results are summarized as follows:

Change in the CaO/SiO<sub>2</sub> ratio from 0.4 to 1.2 lowers the solid fractions as well as the slag viscosity.

Whereas a change in the Fe/SiO<sub>2</sub> ratio from 1.26 to 1.7 increases the fraction of solid phases and the slag viscosity.

The calculated slag viscosity combining the Factsage thermodynamic software and the Einstein–Roscoe equation provides a method to have an insight of the rheological behavior of heterogeneous slags systems at high temperature. The experimental viscosity behavior compares qualitatively with the calculated results.

The equilibrium–quenching experiments and thermodynamic modelling show similar solid fractions in slags at 1300°C. These results also agree in the formation of spinel, mainly formed by ZnFe<sub>2</sub>O<sub>4</sub>, wurtzite and pyrite as solid phases. Wustite was also obtained experimentally, even though it was not obtained in the thermodynamic calculations.

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#### CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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