

Viscosities of Industrial Lead Blast Furnace Slags

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ABSTRACT

Viscosities of two industrial lead blast furnace slag sets were experimentally determined as a function of temperature. A Brookfield viscometer and alumina stirrer rods and crucibles were used in this study. The experiments were carried out in argon atmosphere in the temperature range of 1273–1600 K. The experimental viscosity data is fitted to a non-linear function of both composition and temperature. The constants involved in the model equation were evaluated by regression analysis using the experimental data. The calculated viscosity values were in good agreement with experimental data of the slags.

1. INTRODUCTION

Viscosity is a very important property of slag which determines not only the flow characteristics of the slag, but also affects the mass transfer across the slag/metal interface, heat transfer in the slag, refractory attack in the blast furnace and entrainment of metal in the slag. Slag-metal reactions are important in the extraction and refining of metals by pyrometallurgical processes. New smelting processes have been developed which are cost efficient and environmentally friendly. Sintering of the lead concentrate involves roasting of concentrates in hot air that burns the sulfur content in the form of sulfur vapor and sulfur dioxide and yielding sinter which is a

porous mass consisting of lead oxide, metallic lead, and oxides of other metals. Lead oxide in sinter is then treated in a blast furnace along with coke to reduce to metallic lead. Typical lead blast furnace slag consists of iron oxide, silica, alumina, and calcium oxide and some metallic and non-metallic impurities in the form of entrainments in the slag.

Viscosity of a slag is a structure related physical property and therefore is significantly influenced by the composition and temperature of slag. Molten slags are comprised of ions. Smelting slags invariably contain silica which forms a network of silicate anions. Viscosity of molten slags is influenced by the formation or breaking of these anionic networks. Silica, alumina, and chromia are acidic oxides which form oxide network while CaO, MgO, FeO, ZnO, PbO are basic oxides that modify the oxide network. Small additions of basic oxides such as CaO, FeO, MgO, etc. can drastically decrease viscosity of the slag.

One of the authors has studied the viscosities of commercial lead smelting slags and applied the Arrhenius type of relation to describe the viscosity as functions of composition and temperature /1/. Reddy *et al.* /2, 3/ have modeled the viscosities of several silicate slag systems by considering the ionic structure of the slags. They extended the structure based model to predict the viscosities of binary and ternary borate melts /4-6/. Battle and Hager /7/ have measured viscosities and activities of certain oxides in lead smelting slags using the rotating cylinder method in the temperature

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range of 1423–1623 K. They studied the effect of addition of several oxides such as SiO_2 , Al_2O_3 , MgO , CaO , ZnO , to the master slag at various temperatures. Increase in basic oxide content of the slag resulted in a decrease in slag viscosity at all temperatures of their study. Altman *et al.* /8/ have measured the viscosities of industrial lead blast furnace slags in the temperature range of 1400–1573 K. They have fitted their experimental data to a model equation similar to that of the Arrhenius type of relation.

In this research, viscosity data for two industrial lead blast furnace slag sets were experimentally determined using a Brookfield Digital Viscometer RVT DV-II. A model equation was developed to describe the variation of viscosity as a function of both temperature and composition of slag for both the sets. The constants involved in the equation were evaluated by means of regression analysis of the experimental data. The calculated viscosities were compared with those of the experimental values. Activation energies were also determined from the Arrhenius relationship of viscosity and temperature for both the slag sets.

2. EXPERIMENTAL

A. Experimental Setup

Viscosity measurements were carried out using a Brookfield Digital Viscometer (RVT DV-II) by the rotating cylindrical spindle method. The experimental setup along with the crucible-spindle assembly is shown in Fig. 1. The viscometer rotates a spindle in a fluid, whose viscosity is to be determined, and measures the torque necessary to overcome the viscous resistance to the induced movement. This resistance is indicated by deflection of a spring and is converted to a viscosity value by means of a calibration factor. A continuous digital display of viscosity in centipoises is provided by means of an LED. The RVT model is capable of measuring viscosity of fluids in the range of 0.4–800 poise by varying the rotating speed from 100–0.5 rpm. The furnace uses a silicon carbide heating element which can be heated up to 1873 K. The furnace is placed on a vertical platform where it can be moved up and down. A controller made by Eurotherm is being used to maintain the temperature of the furnace. Heating

schedule including the heating rates and dwell times at various stages can be programmed by the controller.

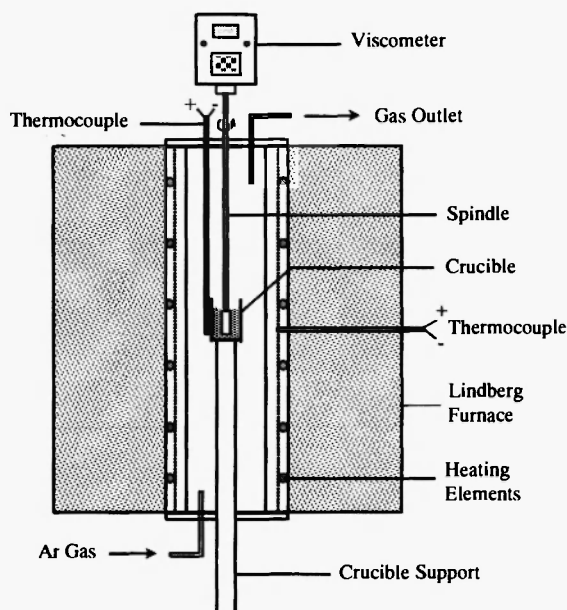


Fig. 1. Schematic Diagram of the Experimental Setup

The instrument was calibrated using the standard lead silicate glass and the obtained results were in good agreement with the reference experimental data suggested by the Theta Industries /9/.

B. Sample Preparation

Two industrial lead blast furnace slag sets were obtained from various lead primary processing industries and are labeled as S1 and S2 in this paper. Table 1 lists the compositions of the five slags in set S1 labeled as S11, S12, S13, S14, and S15. Table 2 lists the compositions of the four slags in set S2 labeled as S21, S22, S23, and S24. The two sets of slags were grouped as S1 and S2 based on the similarity of their sources. The composition of slags in each slag set (S1 and S2) result from a mixture of slags obtained from several furnaces involved in the extraction of lead and copper. For a given slag set, say S1, the concentration of individual components varied within a narrow range. The slag mixture was melted in a resistance furnace to obtain a homogenized composition. Following the melting, the solidified composites were crushed to a particle size of 2–3 mm. Viscosities of the slags in both

sets were measured at different temperatures in the temperature range of 1273–1600 K. Alumina stirrer rods and alumina crucibles were used in the experiments.

Table 1
Compositions of the S1 slag set

Component	S11	S12	S13	S14	S15
Pb	3.93	1.48	1.33	2.88	2.45
Cu	0.41	0.48	0.57	0.39	0.53
Ni	0.035	0.013	0.014	0.022	0.027
Co	0.135	0.079	0.066	0.098	0.112
As	0.118	0.027	0.025	0.097	0.105
Sb	0.113	0.036	0.026	0.092	0.08
Sn	1.37	0.5	0.36	0.86	0.93
ZnO	8.82	10.11	9.81	10.31	9.93
MgO	1.48	1.5	1.74	1.48	1.49
CaO	16.72	17.94	18.67	17	17.59
SiO ₂	26.68	27.36	29.3	27.02	26.8
FeO	32.30	32.97	32.19	31.30	31.58
BaO	0.37	0.43	0.46	0.43	0.45
Al ₂ O ₃	3.22	3.32	3.46	3.3	3.15
Cr ₂ O ₃	0.11	0.12	0.12	0.12	0.12
BI	1.98	2.03	1.90	1.97	2.01

Table 2
Compositions of the S2 slag set

Component	S21	S22	S23	S24
Pb	1.68	2.54	2.86	1.67
Cu	0.45	0.37	0.71	0.64
Ni	0.017	0.019	0.027	0.019
Co	0.055	0.05	0.036	0.041
As	0.042	0.081	0.098	0.049
Sb	0.067	0.131	0.144	0.082
Sn	0.33	0.53	0.43	0.32
ZnO	8.23	8.21	8.55	9.00
MgO	1.54	1.57	1.66	1.63
CaO	17.13	17.38	17.36	17.96
SiO ₂	29.96	27.16	28.1	27.34
FeO	31.61	31.69	30.81	31.45
BaO	0.53	0.72	0.71	0.78
Al ₂ O ₃	4.26	4.23	4.11	3.76
Cr ₂ O ₃	0.23	0.14	0.21	0.17
BI	1.70	1.87	1.80	1.92

C. Experimental Procedure

Major oxides present in the lead/copper blast furnace slags are CaO, FeO, and SiO₂, although other oxides such as PbO, MgO, ZnO, Al₂O₃, ZnO, along with some metal values such as Co, Sb, Sn, Ni, are present in minute quantities. The silicon carbide furnace was heated to the desired temperature which is higher than that required to melt a given slag. After attaining the desired temperature, an alumina crucible filled with the slag is kept in the furnace. The furnace is maintained at this temperature for several minutes to allow thermal equilibrium. Once the melting of the slag begins, the alumina spindle is lowered and centered from the bottom of the crucible. Viscosity measurements were carried out in the temperature range of 1273–1600 K. Temperature was measured with the help of two Pt/Pt-10% Ph thermocouples with an accuracy of ± 0.5 K. All the experiments were carried out in argon atmosphere. Measurements were also repeated during cooling in order to check the reproducibility of the data.

In spite of several precautionary measures, the possibility of some errors in the results cannot be ruled out. The main sources of errors are as follows: The fuming of melt during the measurement leads to a change in its composition which, in turn, results in a variation in its viscosity. Viscosity measurements were carried out during heating as well as cooling. On the basis of these observations, it can be estimated that the fuming of melts may lead to $\pm 2\%$ error in viscosity values. Another source of possible error is the presence of micro-crystalline solid particles such as Fe₂O₃, zinc ferrite in the melt. Viscosity of lead blast furnace slags is subjected to error due to the presence of some amount of solid particles such as spinels, ferrites etc. Their presence causes a variation in the actual viscosity of the slag melt. The only way to eliminate any errors during viscosity measurement is to allow sufficient settling time for solids particles, if any, before making the measurements and also to repeat each experiment for any spurious data.

3. RESULTS AND DISCUSSION

Viscosities of two industrial lead blast furnace slag sets S1 and S2 were measured in the temperature range

of 1273–1600 K. Unlike synthetic slags, industrial slags usually contain more than 7 or 8 elements. The minor components include PbO, CuO, Al₂O₃, MgO, FeO, CaO, SiO₂, ZnO, and other impurities such as As, Sb, Sn, Ni, Co. Experimental viscosities determined for the five slags in slag set S1 at various temperatures are shown in Fig. 2. The viscosity data, in mPa.s, are plotted as function of temperature for different slags whose identity is given by the basicity index (BI). Basicity Index (BI) of a slag is defined as the ratio of sum of the amounts of basic oxides to those of acidic oxides and is given by:

$$BI = \frac{\%CaO + \%FeO + \%MgO + \%ZnO}{\%SiO_2 + \%Al_2O_3 + \%Cr_2O_3}$$

where the symbol '%' refers to 'mass %' of the oxide. The BI values for the five slags in the slag set S1 are also shown in Table 1.

The variation in the measured viscosities to those of the reported values is usually < 5%, which is less than the experimental uncertainty range of 6 to 10%. The solid line in the plot is a trend line indicating the variation of viscosity with temperature for the slags in set S1. The wide band of viscosity data of the five slags with respect to the trend line is due to the variation in the CaO and SiO₂ contents of the slags, which strongly influence the viscosity. Significant variation in the MgO and ZnO contents of the slags can also be seen from Table 1. Figure 3 shows the plot of experimental viscosities of the four slags (S21, S22, S23, and S24) of the slag set S2 at various temperatures. The slags are identified by their corresponding BI values which are also listed in Table 2. The experimental data fall in a narrow band and comply with the trend line since the variation of the contents of major oxides such as CaO, SiO₂, FeO, MgO and ZnO among the four slags is narrow.

Both the slag sets (S1 and S2) displayed Newtonian behavior, i.e., the viscosity is independent of the shear rate at constant temperature and composition of the slag. To minimize the erroneous data, every measurement should have the same Reynolds number (*R_e*) and the same geometric conditions. Viscosities of slags in slag set S2 were higher than those measured for slag set S1 due to the higher average contents of SiO₂, Al₂O₃, and

lower average content of FeO of slags in the S2 slag set than that in the S1 slag set.

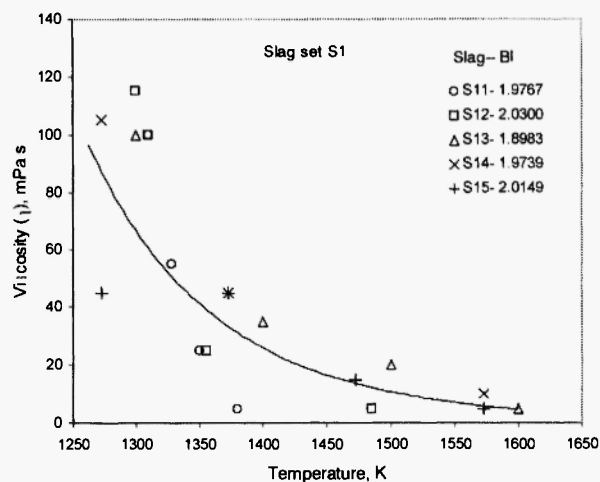


Fig. 2: Viscosities of slags in the Slag set S1 as a function of temperature. The various slags are indicated by their basicity index (BI) values. Solid line indicates the trend.

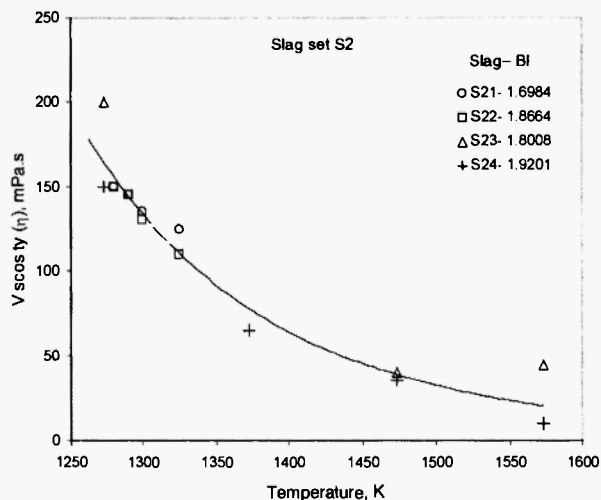


Fig. 3: Viscosities of slags in the Slag set S2 as a function of temperature. The various slags are indicated by their basicity index (BI) values. Solid line indicates the trend.

To study the viscosity of the slag, it is necessary to review the models proposed to describe viscous behavior of liquids by several researchers. Sridhar /10/ reviewed several estimation models that are available to predict viscosities of molten alloys and slags.

In this study, the following viscosity-temperature-composition relation was used to model the experimentally determined viscosities of the two industrial blast furnace slag sets (S1 and S2).

$$\log (\eta) = C_1 \log (BI) + \frac{C_2}{RT} + C_3 \quad (1)$$

where C_1 , C_2 , C_3 are constants to be evaluated by regression analysis using the experimental viscosity data as function of composition and temperature, BI is the basicity index, R is the gas constant, and T is the absolute temperature. The regression constants C_1 , C_2 , and C_3 estimated for both the slag sets are listed in Table 3.

Table 3

Regression constants (C_1 , C_2 , and C_3) of equation (1) estimated for the two industrial slag sets

Slag set	Regression Constants		
	C_1	C_2	C_3
S1	-7.046318	68398.221599	-2.392868
S2	-2.507960	47852.147305	-1.659107

The calculated viscosity values are also plotted along with the experimental data in Figs. 4 and 5 for both the slag sets. An excellent agreement between the predicted viscosity values and experimental data can be seen for both the slag sets. A simple expression given by equation (1) was sufficient to describe the viscosity behavior of the slag sets as function of both composition and temperature.

From equation (1), a plot of $\log (\eta)$ vs. $1/T$ should yield a straight line whose slope refers to the activation energy of viscosity. Figures 6 and 7 show the plots of $\log (\eta)$ vs. $1/T$ for both the slag sets (S1 and S2). Activation energies of 157 kJ and 110 kJ were determined for the slag sets S1 and S2 respectively. The activation energies determined in this study are comparable to 130 kJ, which can be derived from the model equation as described by Altman *et al.* [8].

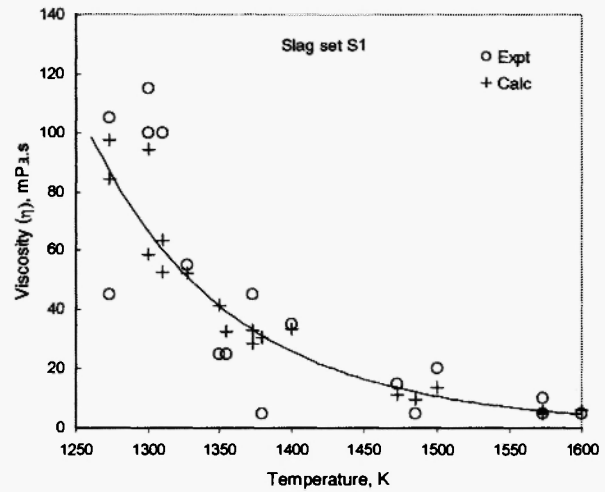


Fig. 4: Comparison between calculated and experimental viscosities of slags in Slag set S1. Solid line refers to the trend line for the calculated viscosities.

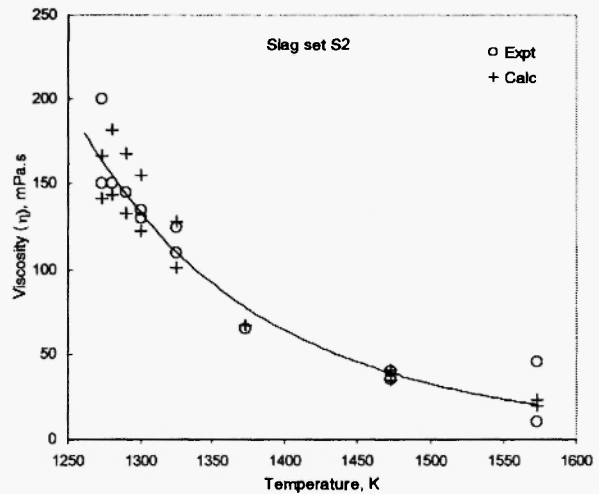


Fig. 5: Comparison between calculated and experimental viscosities of slags in the Slag set S2. Solid line refers to the trend line for the calculated viscosities.

4. CONCLUSIONS

Viscosities of two industrial lead blast furnace slag sets (S1 and S2) were measured in an argon atmosphere in the temperature range of 1273–1600 K. A Brookfield viscometer (RVT DV-II) and alumina stirrer and crucibles were used in this study. From the experimental data the following can be deduced:

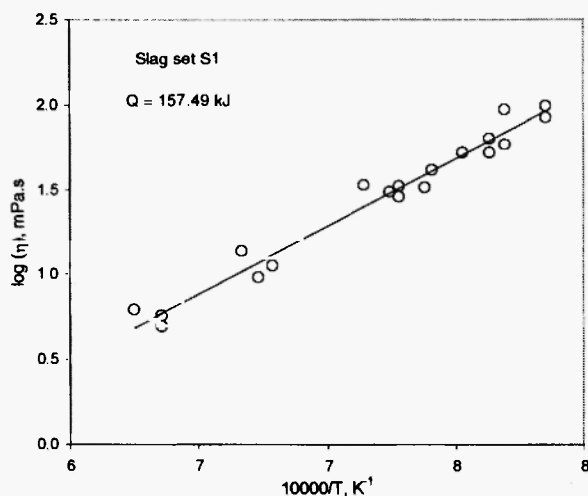


Fig. 6. Plot of $\log(\eta)$ vs. $1/T$ for the Slag set S1 and the determined activation energy (Q).

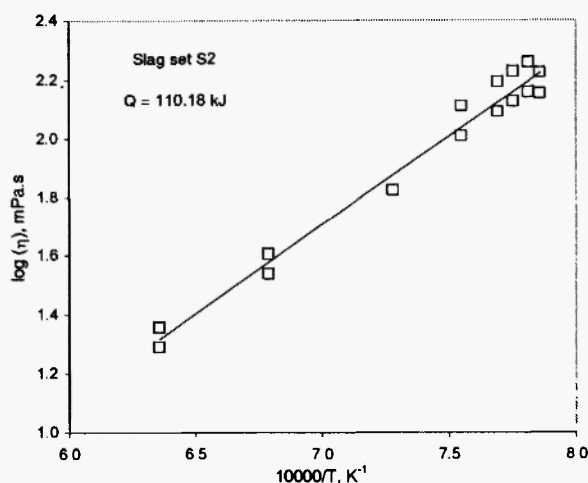


Fig. 7. Plot of $\log(\eta)$ vs. $1/T$ for the Slag set S2 and the determined activation energy (Q).

1. Viscosities of slags in slag set S2 were higher than those of slag set S1 due to the higher average contents of SiO_2 and Al_2O_3 and lower average content of FeO of slags in the S2 slag set than that of S1 slag set.
2. The viscosities of both the slag sets (S1 and S2) decreased with increase in temperature of the melt following an Arrhenius behavior.
3. A viscosity model for the slag samples has been developed to describe the viscosity behavior as a function of both temperature and composition of the

slag. An excellent agreement between the experimental data and calculated viscosity values is observed.

4. Activation energies of 157 kJ and 110 kJ were determined for the slag sets S1 and S2 respectively.

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