The Influence of Al₂O₃/SiO₂ Ratio on the Viscosity of Mold Fluxes

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The present paper investigates how the X_{Al₂O₃}/X_{SiO₂} ratio in mold slag compositions in a range between 0.06 and 2.14 influences the viscosity of the melt. The objective was to study the variation in viscosity of mould slags if they get enriched in Al₂O₃ as a result of reactions during continuous casting with high Al containing steel melts for manufacturing TRIP steels.

The viscosity was found to increase with increasing X_{Al₂O₃}/X_{SiO₂} ratio but a change in the degree of increase was noted at a ratio of 0.57. The effect of increasing temperature was investigated and it was shown that viscosity decreased, as expected, with increasing temperature. An available model in literature for predicting the mold-slag viscosity was empirically modified to account for high Al₂O₃ contents investigated in this study.

Increasing X_{Al₂O₃}/X_{SiO₂} did not appear to influence the vaporization loss of F but increasing the ratio did influence crystallization by promoting the precipitation of CaF₂ during slow cooling of samples with a high X_{Al₂O₃}/X_{SiO₂} ratio (0.84 and 2.14).

KEY WORDS: TRIP steel; mold fluxes; X_{Al₂O₃}/X_{SiO₂} ratio; viscosity.

1. Introduction

Considerable effort is currently being invested into the development of advanced high strength steels for automotive applications. Transformation-induced plasticity assisted steels (TRIP steels) are one of the most ideal candidates for lightweight automotive applications because of their strength to weight ratio and excellent deformability.¹-³

In order to obtain these properties, TRIP steels have conventionally contained C, Mn and Si, where C and Mn are used to stabilize austenite and Si is used as a solid solution strengthener and to suppress iron carbide formation.⁴ In view of the detrimental effect of Si-based oxides that form on the surface, on the coating process, the replacement of Si by Al has been explored⁵-⁷ and the experimental results indicate that Al has similar effect to Si in influencing the iron carbide precipitation and stabilization of austenite.⁸-¹⁰

The required Al content which ranges between 0.5–3% depending on the Si content would however be expected to result in process control problems during melt-state processing due to the reactivity of the Al containing melt. Dissolved Al would be expected to react with the environment which includes the refractory containers, atmosphere and protective Si-based slags. In the case of the continuous casting process, this is of concern since conventional Si-based slags would be expected to change considerably in composition by virtue of the following reaction¹¹:

\[3[\text{SiO}_2]+4[\text{Al}]=2[\text{Al}_2\text{O}_3]+3[\text{Si}] \]  

This could result in significant changes in the thermo-physical properties that are crucial for controlling the casting process. One such property is the viscosity of the molten steel melts for manufacturing TRIP steels. In the case of the continuous casting process, this is of concern since conventional Si-based slags would be expected to change considerably in composition by virtue of the following reaction¹¹:

\[3[\text{SiO}_2]+4[\text{Al}]=2[\text{Al}_2\text{O}_3]+3[\text{Si}] \]  

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Viscosities of polymeric slags are usually Newtonian and are often expressed in an Arrhenius type of equation where the activation energy required to move one silica-unit with respect to another one. Consequently, the activation energy is expected to drop as the silica units get smaller since smaller units have less number of negative charges and therefore fewer number bonds that need to be broken. Some slag systems, such as CaO–SiO₂ containing more than 41 mol% CaO and several coal slags and mould fluxes, exhibit a variation in the activation-energy with temperature. This behavior is probably linked to phase changes in the structure but this still remains to be established. Several models have been developed for predicting viscosity from chemical compositions and temperature and several of them are based on structural parameters and some are related to thermodynamics. Several of these models are developed for the purpose of predicting mold flux viscosities but to the best of the knowledge of the present authors, no systematic study of viscosity of mold slag bearing high amount of alumina has been carried out so far. The purpose of this paper is to experimentally measure the influence of Al₂O₃/SiO₂ ratio on synthetic slags of base composition similar to those of conventional mold fluxes but the contents of Al₂O₃ and SiO₂ are varied to reflect the influence of reaction (1) on the slag composition.

2. Experimental

2.1. Slag Preparation

Synthetic slags were prepared, whose chemical compositions were chosen considering variations in the Al₂O₃/SiO₂ molar ratio ($X_{Al2O3}/X_{SiO2}$) which would result from reaction (1). These included eight compositions were the molar ratio ranges from 0.06 to 2.14. Constant amounts of 10.0 mass% CaO, 12.0 mass% Na₂O, 2.0 mass% Li₂O, 1.5 mass% MgO, 1.5 mass% MnO, 22.0 mass% CaF₂ were used in all the samples to simulate slag compositions that currently under trial in steel industry for casting TRIP steel. The target chemical compositions of the slags studied are listed in Table 1 and these are based on the pre-mixed powder compositions prior to melting. As will be discussed in the results section, these compositions differ from those of the fused samples due to evaporation losses.

The slags were prepared by mixing pure oxides (CaO, SiO₂, Al₂O₃, CaF₂, MgO, MnO), adding Na₂CO₃ and Li₂CO₃ as sources of Na₂O and Li₂O, respectively. The powders were dried for 4 h at 200°C and then heated to 1450°C at a rate of 5°C/min in order to obtain homogenized samples for the experiments. The liquid slag was subsequently poured into a steel mold which was cooled naturally. A slag sample of 50 g was used for the analysis of chemical composition by X-Ray fluoroscopy (XRF). In order to ensure that complete dissolution of Al₂O₃ was achieved slag of No. 8 was quenched after heating to three temperatures, 1200, 1300, 1400°C, respectively and the sample was analyzed in a SEM. This slag contained the highest amount of Al₂O₃, and thus any deviation on crystallization behavior should be apparent. While this test was carried out to ensure the liquidity of samples during the viscosity experiments, slow cooling was carried out after selected viscosity tests in order to investigate whether there was a change in crystallization tendency as a result of the $X_{Al2O3}/X_{SiO2}$ ratio.

2.2. Viscosity Measurement

The high temperature viscosity of liquid mold fluxes was measured with a rotating cylinder method which is schematically shown in Fig. 1. This instrument measures...
the torque of a spindle rotated at fixed speed in a crucible filled with the liquid. A calibration measurement was carried out at room temperature by using standard oil of known viscosity.

Graphite crucibles with inside dimension 80 mm in height and 50 mm in diameter and spindles composed of a bob and a shaft were employed in the experiments. A description of this type of apparatus and its use for high-temperature viscosity measurements has been described in literature. A MoSi2 electric furnace was used to heat and melt the samples. The temperature was controlled through a type B thermocouple inserted in the bottom of the crucible which was protected by an alumina tube. For each sample, the following procedure was followed. The crucible was heated to a temperature of 1200°C, at which point a slag sample of 250 g was added. The temperature was subsequently heated to 1400°C at a heating rate of 5°C/min and maintained isothermally for 10 min in order to ensure that a slag pool was formed. The viscosity at 1400°C was then determined by the averaged value of 20 measurements which were continuously measured. Subsequently, the temperature was decreased to 1300°C and was maintained for 10 min, after which the viscosity at 1300°C was determined in the same way as an average of 20 continuous measurements. By further lowering the temperature to 1200°C the viscosity at that temperature was measured in a similar manner. In order to determine the tendency of crystallization of mold slag, samples taken from the solidified slag pool was formed. To determine the variation of chemical composition during pre-melting of the slag, the chemical compositions of the pre-melted slags were analyzed by X-ray fluoroscopy (XRF), and the results are shown in Table 3. It can be seen, that the content of CaF2 differs from the starting powder-mixture compositions listed in Table 1. The differences in F content, resulting from evaporative loss are between 2.3 to 3.5%. The contents of alumina and silica are very close to that of the designed chemical compositions, indicating

\[ CaF_2(s) = CaF_2(l) \] (3)
\[ (CaF_2)(l) = CaF_2(g) \] (4)

Here, the bracket denotes that the corresponding component is dissolved in the melt. It is obvious that the volatilization rates are strongly depending on the partial pressures of CaF2(g), HF(g) and SiF4(g) respectively. The equilibrium partial pressures have been evaluated by using thermodynamic data on the CaO–CaF2–SiO2 and CaO–CaF2–SiO2–FeO systems, and the results are summarized in Table 2, which lists the equilibrium partial pressures. As can be seen, the partial pressures of SiF4(g) and HF(g) in reactions (6) and (7) are one order of magnitude higher than that of the other reactions, and it is thus reasonable to consider that Eqs. (6) and (7) are the most pertinent ones in the volatilization process. Furthermore, Shinmei and Machida discussed vaporization of AlF3 from slags in the CaF2–Al2O3 system using the Kushin–Knudsen method, and concluded that Eq. (8) also had to be considered.

To determine the variation of chemical composition during pre-melting of the slag, the chemical compositions of the pre-melted slags were analyzed by X-ray fluoroscopy (XRF), and the results are shown in Table 3. It can be seen, that the content of CaF2 differs from the starting powder-mixture compositions listed in Table 1. The differences in F content, resulting from evaporative loss are between 2.3 to 3.5%. The contents of alumina and silica are very close to that of the designed chemical compositions, indicating

\[ CaF_2 + H_2O = CaO + 2HF \] (5)
\[ 2(CaF_2)(l) + SiO_2 = 2(CaO)(l) + SiF_4(g) \] (6)
\[ (CaF_2)(l) + H_2O = (CaO)(l) + 2HF(g) \] (7)
\[ 3CaF_2(l) + Al_2O_3 = 2AlF_3(g) + 3CaO(l) \] (8)

3. Results and Discussion

3.1. Evolution of the Slag Composition and Crystallization Prior to the Experiments

It is well known that one of the main problems with CaF2 containing slags is the volatilization of fluorides which occur during pre-melting and molten states. This is a known issue from corrosion and health hazard point of views in the continuous casting process, but in laboratory investigations it also is of concern since the slag composition might be varying during the experimental time. In order to elucidate the volatilization mechanism, thermodynamic calculations have been carried out, and the possible reactions resulting in a chemistry change of slag were those listed below,\(^{30-34}\)

\[ CaF_2(l) + H_2O = CaF_2(g) \] (3)
\[ (CaF_2)(l) = CaF_2(g) \] (4)
that the Eqs. (6) and (8) are negligible under the present conditions. The change in F content is also relatively independent of the $X_{\text{Al}_2\text{O}_3}/X_{\text{SiO}_2}$ ratio and thus there would not be an expected change in fluorine evaporation from the liquid slag as a result of $\text{Al}_2\text{O}_3$ pick up during casting of high $\text{Al}$ bearing steel melts. It should be pointed out that the quantity of calcium oxide in Table 3 includes the calcium of calcium fluoride.

The slags investigated in this study contain high $\text{Al}_2\text{O}_3$ contents for which the crystallization tendency and melting point are not known, and un-dissolved crystals would influence the measured viscosity. Sample 8 contains 40% $\text{Al}_2\text{O}_3$ and this would present the strongest influence of $\text{Al}_2\text{O}_3$ on the precipitation of crystals. In order to investigate whether crystals are appreciably present during the experimental temperatures, the slag containing 40 wt% alumina (No. 8) was quenched from 1200, 1300 and 1400°C, respectively, after holding the samples isothermally for 30 min. Figure 2 shows the SEM images of quenched samples at different temperatures. It can be seen from the images that the compositions of quenched samples are homogeneous and no crystallized particles are observable even when the sample was quenched from 1200°C.

### 3.2. Viscosity Measurements

The results of the viscosity measurements for the various slags at different temperatures are shown in Table 4. Figure 3(a) gives the viscosity as a function of $X_{\text{Al}_2\text{O}_3}/X_{\text{SiO}_2}$ ratio. It can be seen that the viscosity increases with increasing $X_{\text{Al}_2\text{O}_3}/X_{\text{SiO}_2}$ ratio. It is particularly noteworthy that viscosity suddenly increases as the ratio reaches 0.57. In order to examine the effect of temperature on the slag viscosity, the viscosities of mould slags are shown in Fig. 3(b). As can be seen, that the viscosity decreases, as expected with an increasing of temperature. It should be pointed out that the results obtained in this study are not in agreement with the common idea that an increase of alumina content results in the increase of viscosity, following by a decrease with further increasing of alumina content.35–37)

The viscosity in ternary slag systems CaO–$\text{SiO}_2$–$\text{Al}_2\text{O}_3$ (CaO/$\text{SiO}_2=1.0$ or $1.3$) were found to increase with increasing of alumina and to reach a maximum value at the amount of 10 wt%, followed by a decrease with further increase of alumina in the range of 10 to 15 wt%.35–37)

The variation of viscosity in quaternary slag systems CaO–$\text{SiO}_2$–10wt%MgO–$\text{Al}_2\text{O}_3$ (CaO/$\text{SiO}_2=1.0$ or $1.3$) showed the similar tendency with that in the ternary slag systems.35,36) The CaO/$\text{SiO}_2$ ratios (basicity) in the slags in-

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**Table 4.** The measured viscosity values of various compositions and temperatures.

<table>
<thead>
<tr>
<th>Slag Type</th>
<th>Temperature (Pa s)</th>
<th>1200°C</th>
<th>1300°C</th>
<th>1400°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>No1</td>
<td>0.502</td>
<td>0.294</td>
<td>0.207</td>
<td></td>
</tr>
<tr>
<td>No2</td>
<td>0.627</td>
<td>0.317</td>
<td>0.223</td>
<td></td>
</tr>
<tr>
<td>No3</td>
<td>0.731</td>
<td>0.42</td>
<td>0.238</td>
<td></td>
</tr>
<tr>
<td>No4</td>
<td>0.844</td>
<td>0.441</td>
<td>0.244</td>
<td></td>
</tr>
<tr>
<td>No5</td>
<td>1.197</td>
<td>0.557</td>
<td>0.293</td>
<td></td>
</tr>
<tr>
<td>No6</td>
<td>1.451</td>
<td>0.988</td>
<td>0.486</td>
<td></td>
</tr>
<tr>
<td>No7</td>
<td>1.939</td>
<td>1.367</td>
<td>0.561</td>
<td></td>
</tr>
<tr>
<td>No8</td>
<td>2.543</td>
<td>1.437</td>
<td>0.707</td>
<td></td>
</tr>
</tbody>
</table>

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**Fig. 3.** Viscosity change as a function of (a) $X_{\text{Al}_2\text{O}_3}/X_{\text{SiO}_2}$ and, (b) temperature. The labeled numbers in the figure correspond to Table 1.
vestigated in this study are significantly lower than those in the aforementioned studies. Thus, the degree and type of polymerization of Si is expected to be different and furthermore the slag structure is affected by other constituents, such as CaF$_2$. Finally, the variation range of alumina content is much higher than that reported in earlier papers, and the total amount of alumina and silica is maintained constant in the present experiment, i.e., the amount of alumina increases corresponding to the decrease of the amount of silica, which also differs with reported by earlier papers. For these reasons, it is difficult to compare the current results to the earlier studies but it appears that for the mold-slag compositions investigated here, Al$_2$O$_3$ does not exhibit amphoteric behavior and instead continuously increases the viscosity.

Based on the experimental data, the activation energies for viscous flow of mold fluxes bearing high alumina content were evaluated according to Weymann–Frenkel’s equation,

$$\eta = AT \exp \left( \frac{E}{RT} \right)$$ \hspace{1cm} (9)

The results are presented in Fig. 4 and it can be seen, that the activation energy increases with increasing of $X_{Al_2O_3}/X_{SiO_2}$, and the degree of activation energy increase was found to decrease at the ratios above 0.5. This is consistent with viscosity change with increasing of $X_{Al_2O_3}/X_{SiO_2}$. Figure 4 also includes the activation energies calculated by the viscosity of three CaO–CaF$_2$–Al$_2$O$_3$ slags and one CaO–CaF$_2$–SiO$_2$ slag. Although the chemical compositions of these four slags are different with that of present slags, and the CaF$_2$ content does vary, there is an indication that aluminate complexes, may have a stronger influence on the viscosity than silicate complexes when alumina content is higher than 20 wt%, which could be related to complexes formed by Al ions.

The nature of Al$_2$O$_3$ in slag melts has been discussed in literature quite extensively, and Al-ion complexes have been reported to be present as [AlO$_4$]$^5$-$, [AlO]$^7$-, [AlO$_2$]$^9-$, [AlO$_3$]$^{11-}$ and [AlO$_4$]$^{13-}$ in molten slags. In an alumino-silicate melt for the case were molar ratio between Al$_2$O$_3$ and the sum of basic oxides is less than 1, Al$_2$O$_3$ behaves as acid oxide and Al$^{3+}$ can replace Si$^{4+}$ to form [AlO$_4$]$^{13-}$ tetrahedron. These [AlO$_4$]$^{13-}$ tetrahedron complexes acting as a network former join in the continuous [SiO$_4$]$^{4-}$ networks due to a similar ion radius (2.5 Å). This process would generate additional cations which are located in holes to preserve electrical neutrality, and the additional cations (e.g. Ca$^{2+}$ or Na$^{+}$) are thus needed to provide electrical charge balance by forming [1/2Ca(AlO$_4$)]$^{1+}$ or [Na(AlO$_4$)]$^{2-}$. IR-spectroscopy studies showed that when the alumina content increases, the [AlO$_4$]$^{13-}$ tetrahedral band decomposes to form free aluminate ions (Si–O–Al bending) and a new [AlO$_3$]$^{11-}$ octahedral, and this new formed octahedral complex is expected to act as the network modifier. It should be noted that these IR-spectroscopy were made in ternary Al$_2$O$_3$–CaO–SiO$_2$ melts that contained no CaF$_2$ or Na$_2$O and thus were significantly more viscous than those in this study however it shows that network modifying [AlO$_4$]$^{13-}$ units are created as Al$_2$O$_3$ increases. The results in Fig. 4 suggest that increasing Al$_2$O$_3$ increases the activation energy but at a decreasing rate at $X_{Al_2O_3}/X_{SiO_2}$ ratios higher than 0.57. To the best of the authors knowledge there has not been a study made for the high ratio slags investigated in this study. Therefore the sudden change in activation energy that is apparent at $X_{Al_2O_3}/X_{SiO_2}$=0.57 in Fig. 4 can not be quantitatively linked to the creation or destruction of specific Al-complexes. It is possible that there is a change from a silicate-based network structure to an aluminate-based one at this ratio. Increasing the alumina content beyond such a ration could be linked to the creation of the aforementioned [AlO$_4$]$^{13-}$ units that have been reported in literature.

It is well known that the tendency of crystallization of slag is the primary factor to affect the radiation heat transfer rate in continuous casting. Thus, the post-experimental slag samples, that were furnace cooled after the viscosity measurements, were examined by SEM-EDS to determine tendency of crystallization of slag. The results are shown in Fig. 5. When investigating sample through SEM no crystals were found in the slags with low $X_{Al_2O_3}/X_{SiO_2}$ (<=0.38) and the images appeared as what is shown in Figs. 5(a) and 5(b). However in the slags with the $X_{Al_2O_3}/X_{SiO_2}$ of 0.84 and 2.14, small particles were observed as shown in Figs. 5(c) and 5(d), i.e., the tendency of crystallization increased with increasing $X_{Al_2O_3}/X_{SiO_2}$ ratio in the present study. The EDS results indicate that the precipitated particles observed in the above SEM photographs appear to correspond to calcium fluoride based on the elemental ratio found from EDS analysis of point 3 shown in Table 5. The amorphous parts of slags have also been analyzed, and the analysis results of points 1 and 2 have a close of alumina content, demonstrating that the chemical compositions of alumina is homogenous which is consistent with that measured by XRF analysis shown in Table 3. The basicity of present slag increases with an increasing of $X_{Al_2O_3}/X_{SiO_2}$ ratio, which correspondingly enhances the tendency of crystallization of slag. However, to the best knowledge of present authors the slag systems containing high fluorine content (>10 wt%) such as SiO$_2$–AlO$_2$–CaF$_2$ have not been well assessed so far. For this reason, it is difficult to evaluate how the effect of Al$_2$O$_3$ increase on the variation of thermodynamic properties of CaF$_2$, and further evaluations are therefore needed.
3.3. Estimation of Viscosity

A number of silicate-aluminate slag viscosity models have been developed over the years. The theoretical models are generally classified into four types, statistical mechanical theory, hard sphere theory, theory of corresponding states and semi-empirical and empirical models. Among these models, the semi-empirical and empirical models have been widely developed over the past decades, which are generally expressed in the form of the Arrhenius equation as shown below or the Weymann–Frenkel equation which was shown in Eq. (9),

\[ \eta = A \exp \left( \frac{E}{RT} \right) \]  

Where, \( A \) is constant, \( E \) is the activation energy and \( R \) is the gas constant. Estimated viscosities of mould fluxes containing CaF\(_2\) have been evaluated using these various models, which indicate that the models, such as NPL, Riboud, Koyama and Kim models are suitable for prediction of viscosity of mould fluxes containing CaF\(_2\). It should be pointed out that Koyama, Kim and NPL models are based on Arrhenius relationship, and Riboud and Iida models are based on Weymann–Frenkel equation. Riboud, Iida, Koyama and Kim models are based on the effects on the structure of slag melts of the acidity and basicity of the components, and NPL model is related with the viscosity of slags to the structure through the optical basicity corrected for the cations used for charge balancing. Thus efforts in the present study were focused predominantly on the above five models. Figure 6 shows the comparison of the experimental data with the viscosities calculated by Riboud, NPL, Koyama, and Iida models. As indicated, the agreements between estimated viscosities by aforementioned models and measured values are reasonable at lower \( \frac{X_{\text{Al}_2\text{O}_3}}{X_{\text{SiO}_2}} \) ratios and higher temperatures. With an increase in the amount of alumina, there is a large discrepancy between the measured viscosities and calculated values especially when the alumina content exceeds 25 mass%. The prediction model proposed by Riboud was found predict a consistent trend for the effect of \( \frac{X_{\text{Al}_2\text{O}_3}}{X_{\text{SiO}_2}} \) ratio and it also widely used for mold-flux viscosity predictions in industry.

In Ribouds model, Eq. (9) is used and the parameters \( A \) and \( B \) (where \( B=E/R \)) are fitted to experimental data. In Ribouds original model, \( A \) and \( B \) were given as:

\[ A = \exp(-17.51 + 1.73X_{\text{CaO}} + 5.82X_{\text{CaF}_2}) \]
Based on the experimental data in the present study, the two parameters in Riboud's model were re-evaluated and denoted as $A'$ and $B'$ respectively. Figure 7 shows the variation of re-evaluated $A'$ and $B'$ as a function of $X_{Al_2O_3}/X_{SiO_2}$. As can be seen, both $A'$ and $B'$ increase with increasing $X_{Al_2O_3}/X_{SiO_2}$. The values for $A'$ and $B'$, re-evaluated based on the experimental data in this study are shown in Eqs. (13) and (14) below,

$$
A' = \exp(129.371 + 213.983X_{CaO} - 258.324X_{CaF_2} + 248.768X_{Na_2O} + 106.978X_{Al_2O_3}) \quad \cdots \cdots \cdots (13)
$$

$$
B' = 31 140 - 23 896X_{CaO} - 46 356X_{CaF_2} - 39 159X_{Na_2O} + 68 833X_{Al_2O_3} \quad \cdots \cdots \cdots (14)
$$

A comparison of the experimental data with the viscosity calculated by Eqs. (9), (13) and (14) is shown in Fig. 8. It can be seen here that agreement is quite reasonable under
of those proposed by Riboud (Eqs. (11) and (12)), it can be noted that the coefficients for $X_{\text{CaO}}/X_{\text{CaF}_2}$, $X_{\text{Al}_2\text{O}_3}$ in parameter $A$ increased whereas the constant term and coefficient for $X_{\text{Al}_2\text{O}_3}$ increased in sign. The coefficients for $X_{\text{CaO}}/X_{\text{CaF}_2}$, $X_{\text{Na}_2\text{O}}$ in parameter $B$ increased whereas the coefficient for $X_{\text{Al}_2\text{O}_3}$ decreased.

4. Conclusion

The viscosity of mould slags containing high alumina content has been investigated in the temperature range between 1200–1400°C, for $X_{\text{Al}_2\text{O}_3}/X_{\text{SiO}_2}$ ratios between 0.06 and 2.14 by using a cylinder rotation method. It was found that:

1. Increasing the $X_{\text{Al}_2\text{O}_3}/X_{\text{SiO}_2}$ ratio increased the viscosity and activation energy and there was no indication of amphoteric behavior of $\text{Al}_2\text{O}_3$.

2. Analysis of post-experiment samples showed that for the highest $X_{\text{Al}_2\text{O}_3}/X_{\text{SiO}_2}$ ratios (0.84 and 2.14), there was a tendency to precipitate $\text{CaF}_2$.

3. Increased $\text{Al}_2\text{O}_3$, did not appear to increase $F$ evaporation as a result of $\text{AlF}_3$.

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