Evaluation of Synthetic High Alumina Blast Furnace Slag Vis-a-Vis its Flow Characteristics

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Abstract— An attempt is made to determine the flow characteristics of synthetic high alumina blast furnace slag prepared in the laboratory in agreement with the compositional variations encountered in the industry using hot stage microscopy (German standard 51730). It is observed that additions of CaO, SiO_2, MgO, the major constituents and TiO_2, a minor constituent, influence the characteristic temperatures of the slag considerably. From the operational point of view, the combination of high C/S ratio, high MgO and high TiO_2 contents in the slag, within the range of compositions investigated, seem to be more beneficial. Here the slag formed is a short slag with a small difference between the melting temperature and flow temperature while the softening temperature is relatively high. Such a slag would soften lower-down the furnace without much interference with the permeability of the furnace bed, yet trickle down soon after it is rendered molten without needing any considerable additional heat input, thus exposing fresh interfaces resulting in enhanced rates of slag-metal reactions/exchanges. Regression analysis equations developed in terms of C/S ratio, MgO contents and TiO_2 contents at fixed high alumina content in the slag, using factorial design technique for predicting the characteristic temperatures, are validated through further experimentation with additional ten numbers of synthetic slags.

Key words: Flow Characteristics, Slag-Metal Interface, Slag-Metal Reaction Rates, Factorial Design, Short/Long Slag

I. INTRODUCTION

A slag generated in the metallurgical smelting units must possess sufficient affinity for the gangue and must have the ability to retain the gangue chemically till its final separation from the metal of choice1). The blast furnace (BF) process is a counter-current process. The efficiency of the process depends on the permeability of the burden that controls the flow of the up-coming gases which greatly influence the slag-metal reactions/exchanges, heat transfer in the furnace etc1). In light of the above, it is only pertinent that the permeability of the bed in the blast furnace under the prevailing conditions of temperature and pressure must be conducive to accelerated rates of slag-metal reactions and heat/mass transfer. The softened and subsequently melted slag as influenced by its chemical composition must acquire adequate flow ability as soon as it melts and the difference between its melting and flow temperatures should be as low as possible so that it trickles down the furnace leaving behind paths for upward movement of the ascending gases resulting in an efficient heat transfer from the hot gases to the burden material. Also the trickling slag meets with freshly-exposed, partly-reacted burden surfaces along which it trickles down, accelerating the possibilities of enhanced chemical reactions between the slag and the metal. Thus, the flow characteristics of the blast furnace slag constitute an important parameter for the evaluation of the blast furnace process of iron making. This is truer for the high alumina Indian BF slag where the amphoteric alumina (Al_2O_3) exhibits a big-brotherly attitude for deciding the characteristic properties of the slag.

Besides Al_2O_3, the components like CaO, MgO, SiO_2 etc. form the major constituents of the BF slag that control the characteristic properties of the slag[2-11)]. Both CaO and MgO provide metal cations to the slag melt, which are randomly distributed throughout the lattice of the silicate network introducing weak points to the network. The presence of these cations weaken the Si-O bonds in general, causing a general weakening effect in the lattice which renders the slag more fluid[3,4)]. Therefore, the general effect of the presence of these alkali metal oxides in the slag is to bring down its flow temperature. CaO, in particular, is a stronger base compared to MgO and its presence in the slag renders it sufficiently basic to react with and retain sulphur. However, an increase in the basicity beyond certain levels in the presence of the strongly basic CaO may result in an increase in the viscosity of the slag by the increase in the chemical potential of some primary solid phases that may precipitate out. Therefore, it may be advisable to replace some CaO by MgO, an alkali metal oxide with relatively lower basicity such that the optimum basicity is maintained and the enhanced degree of depolymerisation caused by the basic constituents, responsible for bringing down the viscosity, is not affected[3,4)].

It is also important to consider a factor, NBO/T, the non-bridging oxygen per tetragonally bonded oxygen which is a measure of the degree of depolymerisation responsible for modifying the slag structure, thus influencing its viscosity and hence its flow characteristics. It is reported that [2,5)] besides other oxides, TiO_2 contributes considerably towards NBO/T. In the present investigation, therefore, besides CaO/SiO_2 (C/S) ratio and MgO content, TiO_2 contents are also considered as a significant variable while preparing the high alumina synthetic slag in the laboratory to estimate the effect of their simultaneous variations on its flow characteristics. Al_2O_3 is kept constant at 20 wt.% for all the slags investigated while C/S ratio, MgO and TiO_2 contents are suitably varied in line with the chemical compositions of BF slags as encountered in the related industry.

The significance of the characteristic temperatures [softening temperature (ST), hemispherical temperature (HT) and flow temperature (FT)] which explain the flow characteristics of the slag and the procedure adopted for their measurements using the high temperature microscope as provided in German Industrial Standards 51730, have been reported earlier elsewhere[12-14)].

In the present case, regression equations have been developed to predict the combined effect of the significant variables on the flow characteristics of the slag. The equations are validated in the laboratory with further ten numbers of synthetically prepared BF slags. A careful analysis of the results establishes the interdependence of all the three significant variables, namely C/S ratio, MgO and TiO_2 contents, in deciding the flow characteristics of the BF slag, with a major impact on the process of iron making in the BF route.

II. EXPERIMENTAL WORK

A. Chemical Composition and Sample Preparation

Synthetic BF slags are prepared by using analytical grade oxides available in the commercial market. The purity of such oxides is presented in table 1. As mentioned earlier three significant variables namely CaO/SiO_2 ratio (R), MgO content (M) and TiO_2 content (T) are considered for the regression analysis. The amount of other oxides like Al_2O_3, Na_2O, K_2O, Fe_2O_3 and MnO are kept constant in all the slags at 20 wt.%, 1.1 wt.%, 0.5 wt.%, 1.0 wt.% and 0.5 wt.% respectively. The base levels for the three significant variables are set at 1.15, 9.0 wt.% and 0.55 wt.% respectively. Table 2 shows the chemical composition (in wt.%) of the base slag. The chemical composition (in wt.%) of slags for factorial design calculation are presented in table 3. Table 4 represents the composition of the ten numbers of salgs exclusively prepared within the prescribed ranges of the variation of significant variables for validation of the developed equations.

Specimens of 3 mm cube size are prepared by a die assembly and a hand press for the measurement of the flow characteristics of BF slag as presented elsewhere[12-14].

B. Hot Stage Microscopy

A Leitz heating microscope, with a Leica camera and 1750°C furnace as a supplementary attachment is used to determine the flow characteristics of the slag samples. The characteristic temperatures, softening temperature (ST), hemispherical temperature (HT) and fusion temperature (FT), are determined for different slags in accordance with German Industrial Standards 51730. ST is the temperature at which the outline of the shape of the specimen starts changing and is reported as the temperature at which the specimen shrinks by one division or the temperature at which the distortion of the sample starts. HT is the temperature at which the specimen attains a hemispherical shape and is measured as the temperature at which the height of the specimen is equal to half of its base length. This temperature represents the liquidus temperature of the slag at which the slag is rendered a sluggishly flowing one. FT is the temperature at which the specimen liquefies and is reported as the temperature at which the height of the specimen is equal to one-third of the height that it had at the hemispherical temperature. At this temperature the specimen is rendered free-flowing.

C. Factorial Design

A two-level factorial design technique[14-15] is used in the present study. As per the design each factor i.e. the slag composition in the present case, is assigned two levels w.r.to the base level slag: ±0.25 for R, ±3.0 for M and ±0.45 for T. Initially, characteristics temperatures of different level slags are performed to develop the relationship and then 10 tests are conducted to verify the derived equations[1].

III. RESULTS

The characteristics temperatures, ST, HT and FT, of the salgs used for factorial design technique are shown in table 5. It may be noted that the average characteristics temperatures are represented here. However, the measurements are carried out four times for base slag and twice for other slags respectively. Variance analyses for ST, HT and FT are shown in table 6. Using Snedecor's table (for 95% confidence) and the calculated values of Fischer's ratio (shown in table 6), it can be seen that the significant factors are for ST: R and T; for HT: R and R-M; and for FT: R and T. The relative significances of these factors are shown in table 7. Depending on the relative significances of the factors, the individual responses, namely ST, HT and FT, are found out as follows:

$$ST = 1502.375 + 20.125R + 9.375M - 13.625T \quad (1)$$

$$HT = 1649.625 + 28.625R + 4.625M - 10.875T - 12.375M\text{-}R \quad (2)$$

$$FT = 1677.375 + 29.625R - 5.625M - 14.875T \quad (3)$$

Subsequently, the observed and calculated values of the response for ST, HT, and FT can be used to verify the fit of above equations as per χ^2 test[16]. According to this test at 95% confidence, $\sum \frac{(\rho_{obs} - \rho_{cal})_2}{\rho_{cal}}$ should be less than 2.17 for seven degrees of freedom. ST, HT, and FT are calculated according to the Eq. 1 through Eq. 3, and the results are represented in Table 8. In the above mentioned equations, signs (not values) of R, M, T, and R-M are taken to calculate ST, HT, and FT. Table 8 shows that the above equations fit into the result pattern because the $\sum \frac{(\rho_{obs} - \rho_{cal})^2}{\rho_{cal}}$ for all the three responses, namely ST, HT, and FT, lies much below the value of 2.17. Finally characteristics temperatures of ten slags of composition shown in table 4 are compared to verify the accuracy and usefulness of these equations. The observed and calculated values of all the three responses i.e. ST, HT, and FT are presented in table 9. The percentage variation for ST, HT, and FT is found out to be within 1.99%, 1.69%, and 1.74% respectively. This may, therefore, be concluded that the equations developed for the prediction of ST, HT, and FT of the blast furnace slags in the range of CaO/SiO_2 ratio from 0.9 to 1.4, MgO percentage from 6 to 12 wt.%, and TiO_2 percentage from 0.1 to 1.0 wt.% give good fit for all the characteristic temperatures.

Oxide	Purity (minimum percent of the concerned compound)
Al_2O_3	99.90%
$CaCO_3$	98.50%
MgO	98.53%
Na_2CO_3	99.50%
MnO	99.00%
SiO_2	99.50%
Fe_2O_3	98.00%
TiO_2	99.50%
K_2O	99.40%

Table 1: Percentage purity of oxides used for preparation of different slags

Al_2O_3	CaO	MnO	Na_2O	K_2O	Fe_2O_3	SiO_2	MgO(M)	TiO_2(T)	CaO/SiO_2(R)
20.0	36.29	0.01	1.1	0.5	1.0	31.56	9.0	0.55	1.15

Table 2: Chemical composition (in wt.%) of base slag used for statistical calculation

Sl. No.	Al_2O_3	CaO	MnO	Na_2O	K_2O	Fe_2O_3	SiO_2	MgO(M)	TiO_2(T)	CaO/SiO_2(R)
1	20.0	37.57	0.01	1.1	0.5	1.0	26.83	12.0	1.0	1.4
2	20.0	41.59	0.01	1.1	0.5	1.0	29.71	6.0	0.1	1.4
3	20.0	30.93	0.01	1.1	0.5	1.0	34.37	12.0	0.1	0.9
4	20.0	33.35	0.01	1.1	0.5	1.0	37.05	6.0	1.0	0.9
5	20.0	41.07	0.01	1.1	0.5	1.0	29.33	6.0	1.0	1.4
6	20.0	30.50	0.01	1.1	0.5	1.0	33.89	12.0	1.0	0.9
7	20.0	33.77	0.01	1.1	0.5	1.0	37.53	6.0	0.1	0.9
8	20.0	38.09	0.01	1.1	0.5	1.0	27.21	12.0	0.1	1.4

Table 3: Chemical composition (in wt.%) of slags used for statistical calculation

Sl. No	Al_2O_3	CaO	MnO	Na_2O	K_2O	Fe_2O_3	SiO_2	MgO(M)	TiO_2(T)	CaO/SiO_2(R)
1	20.0	34.44	0.01	1.1	0.5	1.0	36.25	6.5	0.20	0.95
2	20.0	35.05	0.01	1.1	0.5	1.0	35.05	7.0	0.30	1.00
3	20.0	35.09	0.01	1.1	0.5	1.0	34.40	7.5	0.40	1.02
4	20.0	35.29	0.01	1.1	0.5	1.0	33.60	8.0	0.50	1.05
5	20.0	35.46	0.01	1.1	0.5	1.0	32.83	8.5	0.60	1.08
6	20.0	35.22	0.01	1.1	0.5	1.0	32.02	9.5	0.65	1.10
7	20.0	35.38	0.01	1.1	0.5	1.0	31.31	10.0	0.70	1.13
8	20.0	36.41	0.01	1.1	0.5	1.0	29.13	11.0	0.85	1.25
9	20.0	36.73	0.01	1.1	0.5	1.0	28.26	11.5	0.90	1.30
10	20.0	37.16	0.01	1.1	0.5	1.0	27.53	11.75	0.95	1.35

Table 4: Chemical composition (in wt.%) of slags used for testing accuracy statistical calculation

Test No.	Factors	Levels		Response (ρ)	
			ST, K	HT, K	FT, K
1	$R_0M_0T_0$	------	1474	1660	1673
2	$R_1M_1T_1$	+ + +	1518	1668	1681
3	R1M2T2	+ - -	1534	1691	1726
4	$^{R}2^{M}1^{T}2$	- + -	1503	1653	1675
5	$R_2M_2T_1$	- - +	1451	1583	1623
6	$R_1M_2T_1$	+ - +	1499	1681	1718
7	$R_2M_1T_1$	- + +	1487	1623	1628
8	R2M2T2	- - -	1488	1625	1665
9	$^{R}1^{M}1^{T}2$	+ + -	1539	1673	1703

Table 5: Softening temperature (ST), hemispherical temperature (HT) and fusion temperature (FT) at different levels of R, M and C, measured by heating microscope. R_0 = 1.15, M_0 = 9.0, T_0 = 0.55, R_1 = 1.4, R_2 = 0.9, M_1 = 12.0, M_2 = 6.0, T_1 = 1.0, and T_2 = 0.1

Source of variance	Degree of freedom	Sum of Squares			Fischer Ratio		
		ST	HT	FT	ST	HT	FT
R	1	3240	6555	7021	529	1070	693
M	1	703	171	253	115	28	25
T	1	1485	946	1770	242	154	175
R-M	1	91	1225	703	15	200	69
M-T	1	135	36	45	25	6	4
R-T	1	1	406	435	0.184	66	43
Residual	1	6	6	10	1	1	1
	Total = 7						

Table 6: Variance analysis for softening temperature (ST), hemispherical temperature (HT) and fusion temperature (FT)

Specification	R	M	T
ST [AI]	+20.125	+9.375	-13.625
HT [AI]	+28.625	+4.625	-10.875
FT [AI]	+29.625	-5.625	-14.875

Table 7: Relative significance of R, M and C on softening temperature (ST), hemispherical temperature (HT) and fusion temperature (FT)

Test No.	Levels	Response Observed ρOBS			Response Calculated ρCAL			$(\rho_{OBS}-\rho_{CAL})^2/\rho CAL$		
1		ST	HT	FT	ST	HT	FT	ST	HT	FT
2	+ + +	1518	1668	1681	1518	1660	1687	0.0000	0.0423	0.0179
3	+ - -	1534	1691	1726	1527	1697	1728	0.0344	0.0203	0.0013

4	- + -	1503	1653	1675	1505	1649	1657	0.0034	0.0106	0.1955
5	- - +	1451	1583	1623	1459	1593	1639	0.0466	0.0643	0.1466
6	+ - +	1499	1681	1718	1500	1675	1698	0.0001	0.0206	0.2415
7	- + +	1487	1623	1628	1478	1627	1628	0.0548	0.0105	0.0000
8	- - -	1488	1625	1665	1487	1615	1668	0.0015	0.0635	0.0063
9	+ + -	1539	1673	1703	1546	1681	1716	0.0273	0.0417	0.1023
						$\sum(\rho_{OBS} - \rho_{CAL})^2/\rho CAL$		0.1681	0.2737	0.7114

Table 8: Results of χ^2 test

Test No.	*R*	*M*	*T*	Response Observed			Response Calculated			Percentage Variation		
				ST	*HT*	*FT*	*ST*	*HT*	*FT*	*ST*	*HT*	*FT*
1	0.95	6.5	0.2	1465	1625	1649	1487	1615	1668	+1.50	-0.62	+1.15
2	1.00	7.0	0.3	1476	1628	1641	1487	1615	1668	+0.75	-0.80	+1.65
3	1.02	7.5	0.4	1458	1629	1648	1487	1615	1668	+1.99	-0.86	+1.21
4	1.05	8.0	0.5	1465	1641	1657	1487	1615	1668	+1.50	-1.58	+0.66
5	1.08	8.5	0.6	1462	1618	1668	1459	1593	1639	-0.21	-1.55	-1.74
6	1.10	9.5	0.65	1470	1608	1650	1478	1627	1627	+0.54	+1.18	-1.39
7	1.13	10.0	0.7	1467	1600	1645	1478	1627	1627	+0.75	+1.69	-1.09
8	1.25	11.0	0.85	1516	1643	1682	1518	1660	1687	+0.13	+1.03	+0.30
9	1.30	11.5	0.90	1516	1658	1685	1518	1660	1687	+0.13	+0.12	+0.12
10	1.35	11.75	0.95	1518	1663	1688	1518	1660	1687	0.00	-0.18	-0.06

Table 9: Comparison of observed and calculated values of the response

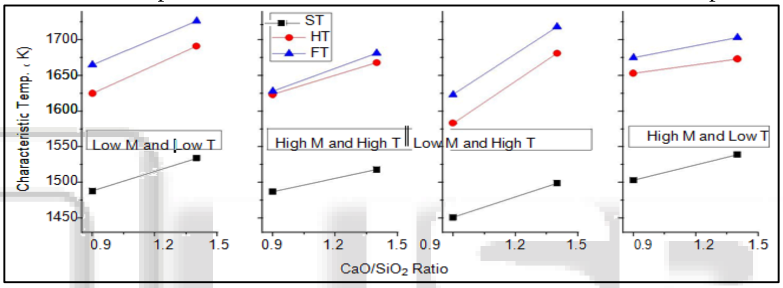

Fig. 1: Effect of CaO/SiO$_2$ ratio on flow characteristics of BF slag. Low and high values for M and T are 6 and 12% & 0.1 and 1.0% respectively.

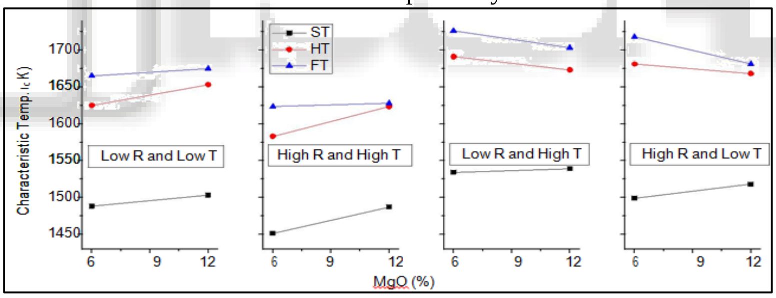

Fig. 2: Effect of MgO content on flow characteristics of BF slag. Low and high values for R and T are 0.9 and 1.4 & 0.1 and 1.0% respectively.

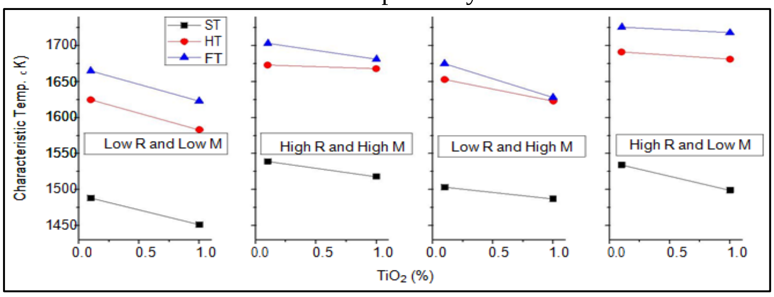

Fig. 3: Effect of TiO$_2$ content on flow characteristics of BF slag. Low and high values for R and M are 0.9 and 1.4 & 6 and 12% respectively.

IV. DISCUSSION

Slags listed in table 3 with a systematic variation of the significant factors (C/S ratio of 0.9 and 1.4, MgO content 6 and 12 wt.%, and TiO$_2$ content 0.1 and 1.0 wt.%) and in table 4 with simultaneous ascending variation of these factors, are analyzed at length to read into the impact of these variables on the characteristic temperatures. The results are tabulated in figure 1 through figure 3.

A. Effect of C/S ratio on the Characteristic Temperatures

The flow temperature of the BF slag which represents the temperature, at which the slag is rendered free-flowing, is indicative of the temperature as a stimulant for lowering the activation energy of the slag to make it free-flowing. In the present investigation, for all the cases, the characteristic temperatures including the flow temperature tend to increase with the increase of the C/S ratio. This trend may be attributed to the presence of high quantities of alumina (Al_2O_3 in all the cases is kept at 20 wt.%) as indicated below.

In slag systems where Al_2O_3/CaO ratio is high (> unity), Al_2O_3 works as a network breaker[2,5]. However, in the present case Al_2O_3/CaO ratio for all the slags investigated is below unity (especially, as the C/S ratio increases at the fixed Al_2O_3 content the Al_2O_3/CaO ratio further decreases). Therefore, in these slags both Al and Si are expected to occupy similar sites in the lattice[2] and the total network forming ions are presented by (Al+Si), Al_2O_3 contributing towards an increase in the degree of polymerization hindering the free-flowing nature of the slag. This explains the increase in the characteristic temperatures with an increase of the C/S ratio.

In addition, an increase in the C/S ratio indicating a decrease in the SiO_2 wt.% results in an increase in the Al/Si ratio in the slag at the fixed Al_2O_3 content of 20 wt.%. This increase in the Al/Si ratio is responsible for the increase in viscosity of the slag[17,18]. Hence, the flow characteristic of the slag is adversely affected, presented by a general upward trend with the increase of the C/S ratio.

In a silicate melt the activation energy of viscous flow is increased resulting in an increase in its viscosity, when Al_2O_3/SiO_2 ratio and hence the Al/(Al+Si) ratio is increased. This is because in Al-Si melts Al^{3+}- O^- type interactions are also present in addition to Ca^{2+}-O^- and O^--O^- type interactions. Higher extents of Al^{3+}- O^- type interactions are responsible for increasing the activation energy of viscous flow and is favoured at a high Al_2O_3/SiO_2 ratio i.e. at high Al/(Al+Si) ratio[2]. This explains the increase of viscosity and hence, the increasing trend in the characteristic temperatures at high C/S ratio. This is because, with a high C/S ratio the SiO_2 wt.% assumes a relatively lower value thereby increasing the Al_2O_3/SiO_2 ratio i.e. Al/(Al+Si) ratio.

The driving force for increase in viscosity and worsening of the flow characteristics can also be attributed to the precipitation of a solid phase, namely dicalcium silicate[19] ($2CaO.SiO_2$) when C/S ratio is greater than 1.3.

The above explains how amphoteric Al_2O_3 plays spoilsport in all the cases investigated shadowing the singular effect of C/S ratio, thus justifying the concerns at all levels in relation to a high alumina BF slag.

B. Effect of MgO on the Characteristic Temperatures

Increasing MgO contents always increased the softening temperatures of the slags investigated irrespective of the high/low values of C/S ratio and the TiO_2 contents in agreement with the findings of Dash et al.[11]. This trend is beneficial in the process of iron making because a slag with high softening temperature softens lower-down the furnace with lesser possibilities of interfering with the permeability of the bed. In this case also the distance travelled by the SiO bearing ascending gases lessens before encountering the descending metal droplets, resulting in low Si pick-up by the hot metal.

As indicated in the related plot (figure 2), a combination of high C/S ratio and high MgO contents, results in the formation of a short slag irrespective of the high/low TiO_2 contents. This is in line with the literature[11,17,18]. Such a trend is beneficial to the process of iron making in the BF since it refers to a situation when the slag is rendered free-flowing soon after its melting without the requirement of higher extents of additional heat inputs. Under this condition as soon as the slag melts it trickles down exposing fresh, partially reacted burden interface that accelerates the slag-metal reactions/exchanges.

C. Effect of TiO_2 on the Characteristic Temperatures

Irrespective of the variations in C/S ratio and MgO additions, an increase in TiO_2 addition always brought down the characteristic temperatures. This may be attributed to the fact that TiO_2 in CaO-SiO_2-Al_2O_3-MgO slags within the range of composition investigated, behaves as a basic oxide increasing the NBO/T value and therefore depolymerise (NBO/T, the non-bridging oxygen per tetrahedrally bonded oxygen, is a measure of depolymerisation) the silicate network structure making the slag more fluid thus bringing down the characteristic temperatures, especially the flow temperature[2,9].

From the process point of view, the combination of high C/S ratio, high MgO content and the highest TiO_2 content (1.0 wt.%) seem to generate the best slag from its composition point of view. This slag has the highest ST coupled with the least difference between the HT and the FT.

V. CONCLUSIVE REMARKS

- Al_2O_3 being amphoteric in nature plays spoilsport in predicting the impact of compositional variations on the characteristic temperatures of high alumina BF slag.
- Under the range of compositions investigated, the statistically developed regression equations using factorial design technique can be used fairly accurately to assess the flow characteristics of high alumina BF slag.
- TiO_2 additions increase the NBO/T of a slag system, thereby increasing the degree of depolymerisation of the silicate network.
- The characteristic temperatures increase with increase of CaO/SiO_2 ratio, for all the slags investigated with 20 wt.% Al_2O_3.
- MgO contributes substantially in selection of the slag composition vis-a-vis the flow characteristic for a smooth operation of the BF.

ACKNOWLEDGEMENT

The authors express their gratitude to the Department of Science and Technology (DST), Govt. of India, for providing the financial support.

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