Influence of synthetic slag added during the tapping on inclusion features of a vacuum degassed LCAK steel grade

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Abstract

Synthetic slag has been added during BOF tapping in order to modify the physicochemical properties of resulting slag with the aim of assessing the influence on inclusion density, composition and size distribution. Viscosities and activities of slag oxides were calculated by FactSage 7.2, using the FToxid database and Viscosity module. All heats were treated under vacuum in an RH degasser for 15 minutes. A comparative analysis of top slags with two levels of FeO+MnO (> 10% and ≤ 10%) was carried out. The higher level of FeO+MnO led to almost thrice more inclusion than the lower level. Top slags after treatment with synthetic slag brought the best result of inclusions features thanks to their higher CaO activity, lower viscosity and lower activity of Al_2O_3. These heats presented smaller inclusions and lower initial inclusion density at ladle arrival at RH. Based on these evidences, top slags with synthetic slags added during the tapping and FeO+MnO content ≤ 10% conferred the best condition in steel cleanliness.

Keywords: slag modification; synthetic slag; vacuum degassing; inclusion removal.

1. Introduction

Steelmaking practices using synthetic slags have been developed to remove unwanted nonmetallic inclusions (NMIs), as well as, to desulfurize steel according to the customer specifications and/or final use requirements. In order to comply with these purposes, synthetic slag or final slag (resultant oxide mixture after synthetic slag addition on the carryover slag) must have its physicochemical properties, such as basicity, viscosity and activities of Al_2O_3 and CaO correctly adjusted in order to maximize inclusion removal.

Viscosity of slags plays an important role in steel cleanliness. In general, dissolution capacity increases when slag viscosity is lower, which means faster kinetics. (Valdez *et al.*, 2002; Capurro *et al.*, 2015). A suitable synthetic slag composition is a key point to guarantee physicochemical properties for inclusion removal, and a higher thermodynamic driving force. Mohanasundaram *et al.* (2019) modeled four different kinds of CaO-Al_2O_3 synthetic slags. They found that MgO-bearing and $CaO/Al_2O_3 > 3.0$ synthetic slags led to the best result, due to lower viscosity and a higher fraction of the liquid phase. Gavanescu (2011), pointed out that the CaO content should lie in a 50%-55% range for better results of desulfurization and alumina absorption.

FeO and MnO dissolved in carryover slags could be the main suppliers of oxygen to the steel bath. These oxides are easily reduced by aluminum acting as a deoxidant agent in steel. Anderson and Zimmerman (1985), Di Napoli Guzela *et al.* (2003) and Aydemir (2007) reported how the aluminum is lost by the system, reducing slag and producing alumina inclusions.

A decrease of inclusion density after vacuum degassing, as well as a size distribution displaced to smaller diameters was found by Rocha *et al* (2017). They observed a higher removal rate for larger inclusions due to their easier flotation characteristics. A similar behavior was observed in a VD route combined with a high stirring LF (Pereira *et al.*, 2018), where virtually no inclusions larger than 15µm were found.

Most studies reported in literature, deal with the dynamic behavior of inclusions during secondary treatments in Ladle Furnaces and VODs with longer treatment time (> 30 minutes); few articles reported operational findings regarding RH vessels (shorter treatment time – approx. 15 minutes) as the sole secondary refining reactor prior to casting. Therefore, the aims of this study are: to know how synthetic slag modified the physicochemical properties of resultant slag; and to understand how inclusion features (quantity, dimension, and composition) are affected by synthetic slag addition, considering RH vacuum degassed heats and two levels of oxidation (FeO+MnO ≤ 10% and > 10%) in a standard and optimized (in case of top slag mixed with a synthetic one) $Al_2O_3 - CaO - SiO_2 - MgO - FeO - MnO$ normalized slag.

2. Materials and methods

An industrial experiment involving four heats of an LCAK – HSLA grade 0.07C – 1.00Mn – Nb – Ti – Cr with 50ppm S max was carried out at Gerdau – Ouro Branco steelworks. The standard process route was: BOF → RH → CC. The nominal capacity of BOF tapping is 224t per heat. No alloy addition or blowing has been made in RH during the first 15 minutes of treatment, in order to avoid any kind of disturbance of the process. All four heats were treated according experimental conditions as stated in Table 1.

Table 1 – Experimental conditions.

Heat Id	Vacuum treatment time [min]	Circulation flow rate [t/min]	Top slag composition	FeO+MnO in top slag
1	15	130 - 135	Standard	≤ 10%
2			Optimized	
3			Standard	> 10%
4			Optimized	

Key for top slags:

- Typical Standard Composition: Al_2O_3 35 – 45%, CaO > 30%, MgO < 10%, SiO_2 < 15%;
- Optimized Composition – Top slag with synthetic slag addition: Al_2O_3 < 30%, CaO > 40% (aiming 45 – 50%), MgO 6 - 9%, SiO_2 < 13%;
- Synthetic Slag specification: CaO 55 - 65%, Al_2O_3 9 – 14%, metallic Al: 6 – 10%, MgO > 7%, SiO_2 < 5%, CaF_2 6 – 10%.

Top slag treatment is performed by addition of 1250kg of synthetic slag during tapping, using the turbulent energy at this time to modify top slag composition and inclusions. The main objective here is to have a favorable thermodynamic driving force for the highest dissolution rate of alumina inclusions. In order to investigate the evolution of inclusion features, steel and slag samples were taken each five minutes, from ladle arrival at RH (instant 0min) until vacuum treatment end (instant 15min). Oxygen activity was measured at tapping, and then, each five minutes, in the same way as steel samples. An SEM-EDS analysis was performed in steel to check the size, quantity, and composition of inclusions through a semi-quantitative analysis of elements. Then, the semi-quantitative composition was transformed into oxide contents using a set of stoichiometric rules (Table A.1 see appendix). The scanned area was

$100 mm^2$ and inclusions smaller than 2 µm were not considered.

Slag samples were ground into a very fine powder, and after that they were press-formed. Slag compositions were determined by XRF. Viscosity and oxide activities (pure solid as a standard state) were calculated considering the four heats stated in Table 1. For these calculations, the Ftoxid slag database and Viscosity module from FactSage 7.2 were used.

3. Results and discussion

3.1 Top slag sampling, composition, calculated viscosity and oxide activities

The top slag composition of four heats are given in Table 2. After the addition of synthetic slag, during tapping, all slags complied with specification ranges previously informed. Chemical composition of each slag is normalized for the six most representative oxides.

Table 2 – Composition of slags obtained by XRF – mass %.

Sample	FeO+MnO Level	Top Slag	Heat Id	T [K]	Al_2O_3	CaO	MgO	SiO_2	FeO	MnO
0	≤ 10%	Standard	1	1875	43.09	33.27	6.69	9.66	1.60	5.69
		Optimized	2	1857	27.96	48.12	7.07	12.87	1.59	2.39
15		Standard	1	1843	42.72	31.87	6.59	11.98	1.41	5.43
		Optimized	2	1839	29.02	45.02	6.98	12.68	2.35	3.95
0	> 10%	Standard	3	1879	39.69	31.85	7.05	10.08	2.73	8.60
		Optimized	4	1884	22.97	40.68	6.59	10.20	8.84	10.72
15		Standard	3	1848	41.64	30.37	7.70	9.97	2.31	8.01
		Optimized	4	1850	24.41	40.11	6.04	9.90	7.35	12.19

The carryover slag weight was estimated by dipping a steel rod across the slag/metal interface. The slag carryover ranged from 5.5 – 7.0 kg/t.

Table 3 shows the calculated results of viscosity and oxide activities performed by FactSage 7.2. This calculation considers the process temperature for each sampling point.

Table 3 – Viscosity and activities calculated for heats 1 – 4.

Sample	FeO + MnO Level	Heat Id	T [K]	µ [Poise]	Activities					
					Al_2O_3	CaO	MgO	SiO_2	FeO	MnO
0	≤ 10%	1	1875	1.37	0.0285	0.0235	0.0593	0.0011	0.0371	0.0579
		2	1857	0.84	0.0042	0.0816	0.1313	0.0002	0.0149	0.0334
15		1	1843	1.84	0.0359	0.0158	0.0509	0.0017	0.0344	0.0482
		2	1839	0.98	0.0056	0.0603	0.1155	0.0003	0.0303	0.0507
0	> 10%	3	1879	1.23	0.0241	0.0225	0.0665	0.0009	0.0606	0.0847
		4	1884	0.49	0.0030	0.0745	0.1269	0.0001	0.0963	0.1589
15		3	1848	1.52	0.0270	0.0179	0.0667	0.0011	0.0560	0.0772
		4	1850	0.59	0.0034	0.0654	0.1102	0.0001	0.0876	0.1761

According to the Equilib module in FactSage 7.2, the calculated slags are 100% liquid at their process temperatures. It is important to highlight that for all heats of this industrial trial, there has been not observed any remarkable variation in top slag composition for both sampling conditions – before RH vacuum treatment (point 0) and after 15min of treatment (point 15). Whether from simulation or experimental trials, the available literature (Anderson, 2000; Treadgold, 2003) reported no significant differences in top slag composition after vacuum treatment.

Table 4 shows the evolution of the oxygen activity after de-O agent homogenization and during vacuum treatment.

Table 4 – Oxygen activity of molten steel.

Heat Id	Sampling after de-O agent homogenization		Sampling moment after ladle arrival [min]							
			0		5		10		15	
	a_O [ppm]	T [K]	a_O [ppm]	T [K]	a_O [ppm]	T [K]	a_O [ppm]	T [K]	a_O [ppm]	T [K]
1	27	1953	6.89	1875	5.93	1865	3.86	1857	3.13	1843
2	16	1957	5.12	1857	3.52	1852	3.41	1843	2.80	1839
3	67	1967	7.89	1879	6.08	1866	4.26	1860	2.77	1848
4	56	1973	8.12	1884	6.51	1870	4.38	1858	3.19	1850

Higher FeO+MnO contents, as observed in heats 3 and 4, imply higher oxygen activity in the molten steel after deoxidant agent homogenization, despite aluminum having been added according to the level of dissolved oxygen before tapping. On the other side, from point 0 on, such levels do not appear to have significantly affected the killing process.

3.2 Steel sampling – measurement of inclusion features: quantity, dimension, and composition

Quantity of Inclusions: Figure 1 indicates the influence of FeO+MnO content in inclusion content, in both situations: before and after vacuum treatment. Heats with FeO+MnO > 10% presented the highest initial and final inclusion density.

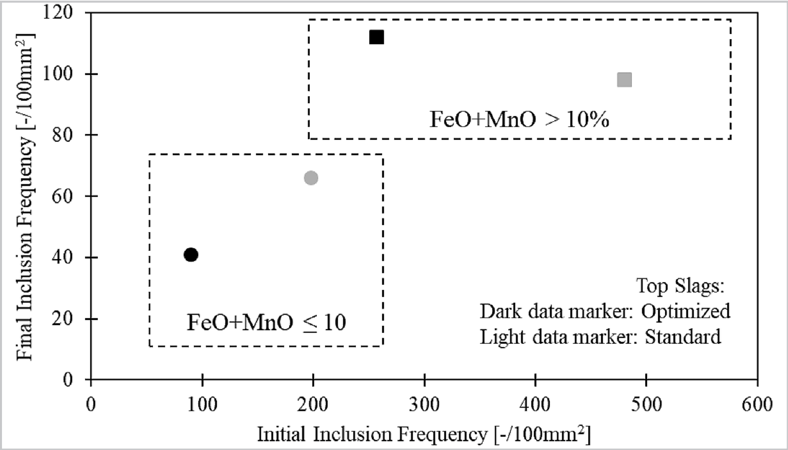

Figure 1 – Inclusion densities for both FeO+MnO conditions.

The higher amount of inclusions found in samples of point 0, for heats 3 and 4 (FeO+MnO > 10%), suggests that FeO and MnO dissolved in carryover slags are the sources of additional inclusions, due to their reduction by aluminum through favorable thermodynamics of the reactions $3(FeO) + 2[Al] = Al_2O_{3\ inclusion} + 3[Fe]$ and $3(MnO) + 2[Al] = Al_2O_{3\ inclusion} + 3[Mn]$.

A comparison of top slag features can be seen in Table 5. Heats with optimized top slag condition presented smaller inclusion content at ladle arrival in RH.

Table 5 – Comparative data of slag features and inclusions observed at ladle arrival at RH.

Heat Id	Top Slag	FeO+MnO	$\frac{CaO}{(Al_2O_3)}$	a_{Al2O3}	a_{CaO}	μ [poise]	Inclusion Quantity [-/100mm²]
1	Standard	≤ 10%	0.77	0.0285	0.0235	1.37	195
2	Optimized		1.72	0.0042	0.0816	0.84	90
3	Standard	> 10%	0.80	0.0241	0.0225	1.23	480
4	Optimized		1.77	0.0030	0.0745	0.49	257

The synthetic slag addition conferred to heats 2 and 4 smaller viscosity and activity of Al_2O_3 and higher activity of CaO, when compared with the standard top slag. The smaller amount of inclusions observed in heats 2 and 4 suggests that the modification brought by synthetic slag addition was favorable in regard to inclusion removal.

Size distribution of Inclusions: A size distribution of inclusions in RH samples along treatment (sampling point 0, 5, 10 and 15) is shown in Figure 2 for both FeO+MnO > 10% and FeO+MnO ≤ 10% conditions. As can be noticed, the optimized top slag condition brought smaller inclusion size for both conditions of FeO+MnO, after 15 minutes of vacuum treatment at RH.

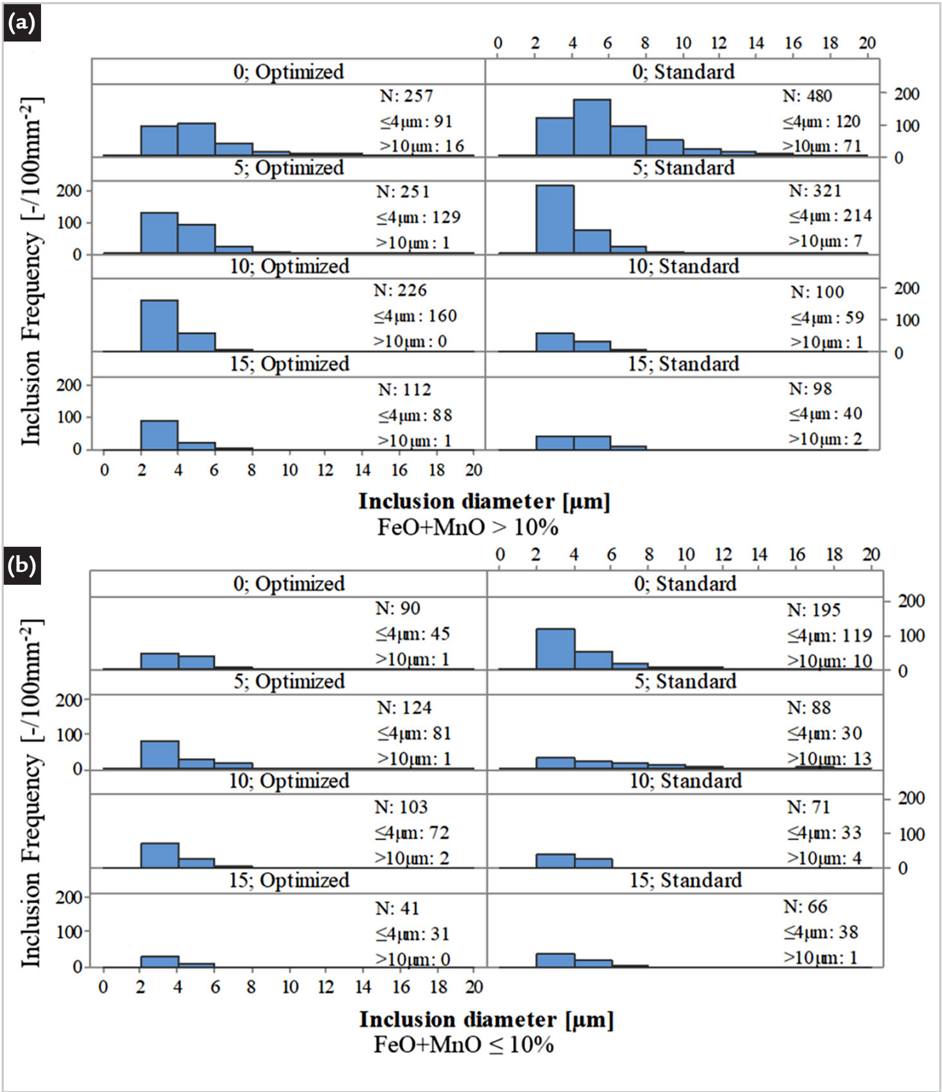

Figure 2 – Comparison of the inclusion size distribution for optimized and standard top slags.

It is important to highlight two groups of inclusions: smaller, with diameter ≤ 4µm, and larger, with diameter > 10 µm. Top slags with synthetic slag addition (optimized condition) present a well-defined behavior for both groups, as can be seen in Figure 3. The smaller inclusions have their share increasing steadily, independently of the FeO+MnO level. Regarding the larger inclusions, an almost constant level could be seen for FeO+MnO ≤ 10% class, while for FeO+MnO > 10% a decrease is perceived for the first 5 minutes of vacuum treatment, and after that, an almost constant level is also seen until the final sampling. Also, it is important to note that for both levels of FeO+MnO, the difference between the fraction of smaller and larger is quite the same, while for optimized top slags the spread is higher than standard ones.

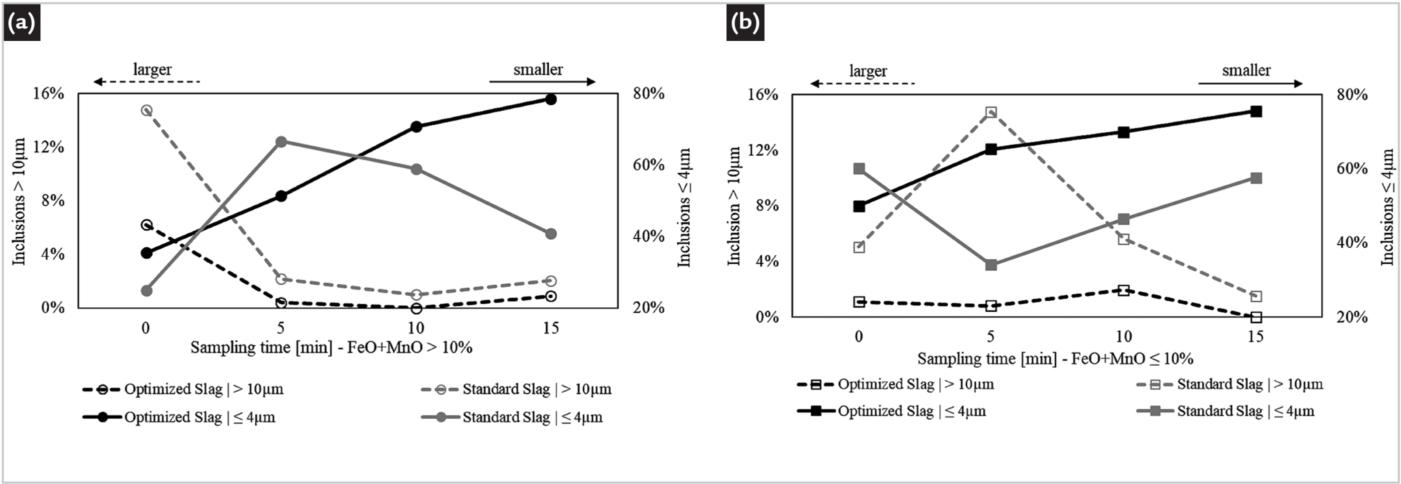

Figure 3 – Sharing of smaller and larger inclusions along RH treatment for both top slag conditions: a) FeO+MnO > 10%, b) FeO+MnO ≤ 10%.

Composition of Inclusions: Alumina was the observed major inclusion type, according to a morphological and chemical analysis in SEM-EDS. However, sulfides and oxysulfides were found as major ones only at ladle arrival (sample 0min – just before vacuum starts) for slags with FeO+MnO > 10%. Very few amounts of other oxides, like calcium aluminates and spinel ($MgO.Al_2O_3$) were observed. Figure 4 shows a qualitative chemical analysis. In case of FeO+MnO > 10%, (Figure 4a), after the first 5 minutes of vacuum treatment, it is believed that most of the oxysulfides have floated up. Yan *et al*., (2014) reported that oxysulfides tends to nucleate and grow quickly as temperature decreases. Therefore, it is believed that turbulent flow in RH catalyzes the flotation of this inclusion. According to Li *et al*., (2018) MnS is unlikely to precipitate in Alumina-rich regions of an Alumina-Silica based inclusion. This fact could be explained by a high lattice disregistry between MnS and Alumina. This finding may explain the reason why oxysulfides are not observed from 5 minutes on.

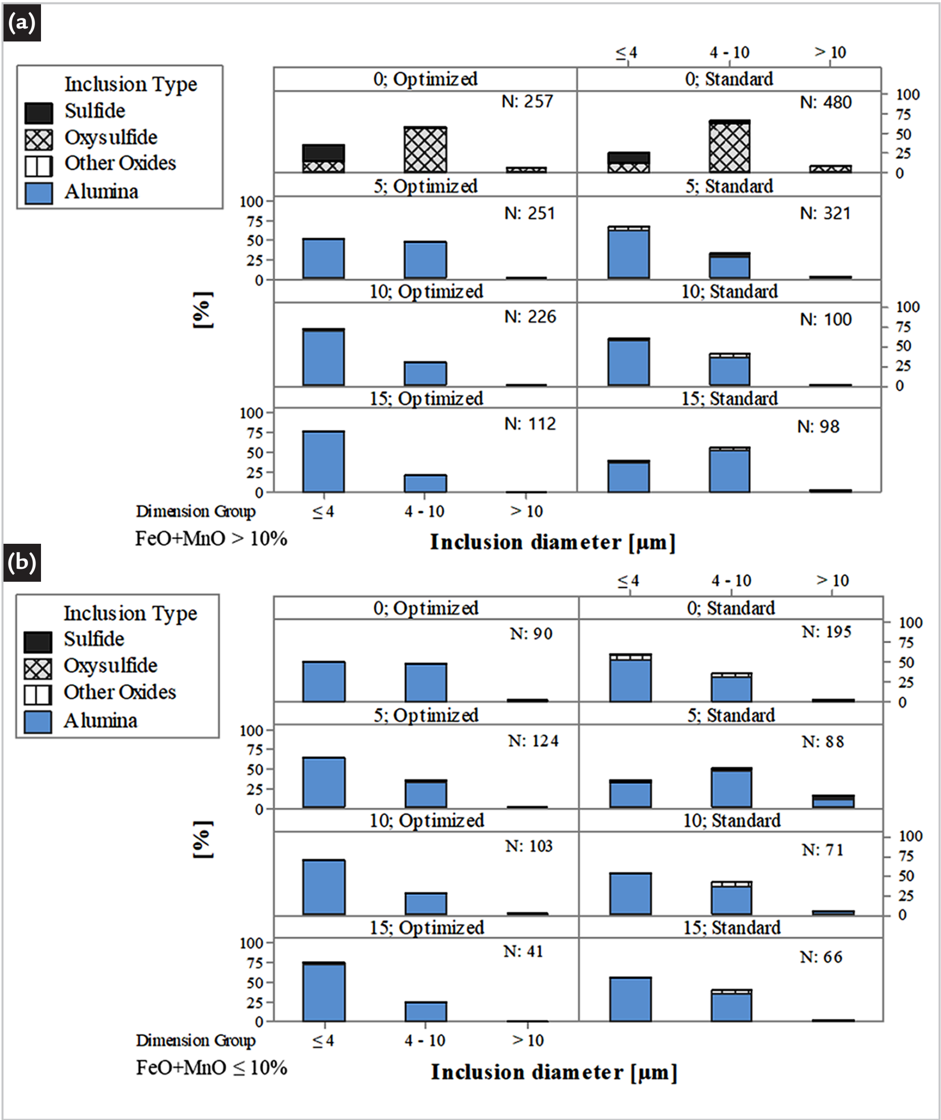

Figure 4 – Qualitative chemical analysis of inclusions following size distribution: top slag conditions a) FeO+MnO > 10%, b) FeO+MnO ≤ 10%.

An important point to pay attention to in Figures 4a and 4b is the shape of size distribution. As far as optimized slags are concerned, for both low and high FeO+MnO contents, after 15 minutes almost 75% of inclusions are smaller than 4µm. Optimized top slags reach smaller inclusions faster than with the standard practice and also keep increasing ≤ 4µm fraction until reaching 15 minutes of vacuum treatment.

Figure 5 shows SEM-EDS x-ray maps of a typical oxysulfide observed in this study. This inclusion presents itself as a double oxide – Alumina + Silica – surrounded by an Mn shell with embedded MnS precipitated on SiO_2-rich regions of the inclusion. Yan *et al*., (2014) reported the formation of oxysulphide inclusions with higher [O] and [S] as a consequence of the diffusion of Mn, O, and S towards an inclusion acting as a sink.

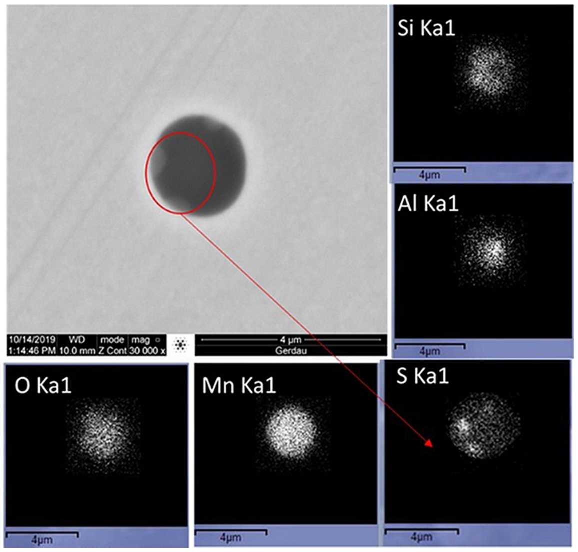

Figure 5 – SEM-EDS X-ray map of a typical Oxysulfide found in both conditions of top slag and FeO+MnO > 10%, prior to vacuum.

Regarding alumina formation, Zhang *et al.*, (2002) concluded that the growth of inclusions > 2µm is mainly controlled by turbulent collisions. Inclusions in this range tend to form clusters which retain minimum size in the 1 – 2µm range. A typical morphology for alumina inclusion, Figure 6a, as well as its composition, Figure 6b are in agreement with Rastogi and Cramb, (2001); Doo *et al.*, (2002) who predicted alumina agglomeration in clusters after earlier stages of nucleation, precipitation, and growth in different stages of the steelmaking process. Riyahimalayeri and Olund, (2013) reported a size distribution during vacuum degassing with skewness concentrated in smaller diameters, which is the same behavior seen in Figure 4a. Xuan *et al.*, (2016) also have shown that for Al deoxidation, morphologies are similar to clusters and shape of the size distribution curve every 5 minutes, very similar to the synthetic slag addition condition in Figures 2a and 2b.

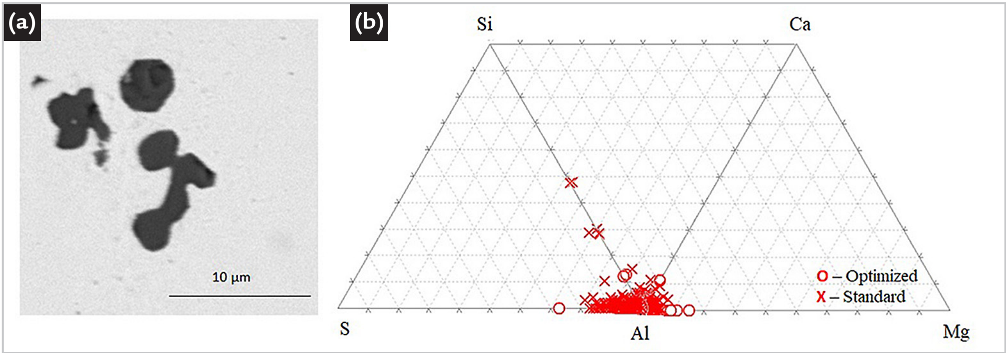

Figure 6 – a) Typical Alumina cluster found after synthetic slag addition conditions of top slag at 5 minutes after vacuum. b) Semi-quantitative analysis normalized to 5 main elements of the inclusion population at the same time.

3.4 Influence of slag parameters in inclusion features

As could be seen previously, the levels of FeO+MnO in top slag have a strong influence on the inclusion content in both situations, before and after during vacuum treatment. Higher content of dissolved CaO in slags helps to increase the basicity and decrease the Al_2O_3 activity in slags. Both are important driving forces for alumina removal. Another important feature in top slags for dissolution capacity is slag viscosity. A change in slag composition would result in a change of viscosity which affects the absorption ability of inclusions. Choi *et al.*, (2002) reported an increase of mass transfer coefficient following viscosity decrease, intensifying the dissolution rate of inclusions by slag.

Table 6 shows the mentioned slag parameters and how they affect the inclusion features. CaO/Al_2O_3 ratio is given by final slag measurement (sampling point 15 – Table 2), oxide activities and slag viscosity were calculated by FactSage 7.2, using the composition of the same sampling point. Based on this evidence, top slags with synthetic slags added during the tapping and FeO+MnO content ≤ 10% conferred the best condition in steel cleanliness. Also, the synthetic slag addition was crucial to bring the finest inclusion size distribution. These findings are similar to those reported by Valdez *et al.*, (2002) which concluded that dissolution was faster for small inclusions or slags with lower viscosities.

The higher inclusion removal observed for standard top slags are related to favorable collision-agglomeration mechanisms during vacuum treatment at RH and higher initial inclusion density. This study highlights this favorable combination. RH degassers are not deemed as reactors depicting a strong top slag/metal interaction as compared with LF furnaces. Thus, such remarkable influence of top slag characteristics on inclusion absorption should be also ascribed to those mechanisms.

Table 6 – Comparative data of final slag parameters and inclusion features.

Heat Id	Top Slag Type	FeO+MnO Level	$\frac{CaO}{(Al_2O_3)}$	a_{Al2O3}	a_{CaO}	μ [poise]	≤ 4μm Share [%]	Inclusion Density Change [-/mm^2]
1	Standard	≤ 10%	0.75	0.0359	0.0158	1.84	58	1.95⟶0.66
2	Optimized		1.55	0.0056	0.0603	0.98	76	0.90⟶0.41
3	Standard	> 10%	0.73	0.0270	0.0179	1.52	41	4.80⟶0.98
4	Optimized		1.64	0.0034	0.0654	0.59	78	2.57⟶1.12

4. Summary and conclusions

A comprehensive study of inclusion distribution considering different conditions of top slag in an RH vacuum degasser treatment was made. Top slag composition was determinant for inclusion chemistry, quantity and size distribution.

The addition of synthetic slags modified the physicochemical properties of top slags, such as basicity, viscosity and activities of Al_2O_3 and CaO. Activity of Al_2O_3 decreased dramatically and aCaO increased. Viscosity decreased by half in heats with FeO+MnO level ≤ 10% and by 2/3 in heats with FeO+MnO level > 10%. This modification in such key parameters meant initial inclusion density decreased around 50%, if compared to standard heats. Moreover, inclusion distribution for heats with optimized top slags became narrower than standard ones.

FeO+MnO content in slags was decisive for inclusion content. Top slags with FeO+MnO contents greater than 10% resulted in a higher inclusion content than top slags with FeO+MnO ≤ 10%. This behavior was observed in both situations, before and after, during vacuum treatment.

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Appendix

Table A.1 – Set of stoichiometric rules used in this work.

Type	Inclusion	Class	Rule
Oxide	Alumina	Alumina	Al > 65%
	C3A	Other Oxides	(Ca+Al) ≥ 50% and 0.2 ≤ Al/Ca < 0.4
	C12A7		(Ca+Al) ≥ 50% and 0.4 ≤ Al/Ca < 0.8
	CA		(Ca+Al) ≥ 45% and 0.8 ≤ Al/Ca < 1.5
	CA2		(Ca+Al) ≥ 45% and 1.5 ≤ Al/Ca < 2.5
	CA6		(Ca+Al) ≥ 45% and 2.5 ≤ Al/Ca ≤ 3.0
	Spinel		Al ≥ 35% and Mg/(Al+Mg+Ca) ≥ 0.15 and (Al +Mg) ≥ 70% Mg ≥ 2%
	AlSi		(Al+Si) ≥ 70% and Al ≥ 10% and Si ≥ 10% and Ca < 10%
	MnSi		Mn ≥ 10% and Al ≥ 10% and Si ≥ 10%
	TiAl		Ti ≥ 10% and Al ≥ 10% and (Ti+Al) ≥ 65%
	CaSiAl		Ca ≥ 10% and Si ≥ 5% and Al > 10%
Oxysulfide	AlSi-MnS	Oxysulfide	Mn ≥ 20% and Al ≥ 10% and Si ≥ 10% and S ≥ 10%
	Si-MnS		Mn ≥ 20% and Si ≥ 10% and S ≥ 10% and Al < 5%
	CA-(Mn,Ca)S		(Ca+Al) > 40% and Ca ≥ 20% and Mn ≥ 10% and S ≥ 20%
Sulfides	CaS	Sulfides	Ca ≥ 20% and S ≥ 20% and Si < 5% and (Mn+Al+Mg) < 15%
	MnS		Mn ≥ 10% and S ≥ 10% and (Mn+S) ≥ 50%
	(Ca,Mn)S		Ca ≥ 20% and S ≥ 20% and Mn ≥ 20% and Al < 20%

Key for abbreviations: CxAy is the Calcium Aluminate x CaO .y Al_2O_3.

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