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Recycling MgO–C refractories and dolomite fines as slag foaming conditioners: experimental and thermodynamic evaluations

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Abstract

The slag foaming process is currently applied in the steel production due to its greater benefits, i.e., reduction of energy losses, enhancement of the refractory service life and productivity, etc. Lime/dolomite, oxygen and carbon are usually added during an electric arc furnace (EAF) operation to adjust the liquid basicity and saturation and also provide suitable conditions for foam generation. Considering the high concentration of MgO and C contained in spent MgO–C refractories and dolomite fines (derived from rotary kilns), one good alternative consists of recycling those materials as slag conditioners. In this work, experimental tests and thermodynamic calculations (phase equilibrium, viscosity and isothermal solubility diagrams) were carried out in order to define the impact of some mixtures (comprised mainly of ground MgO–C spent bricks, dolomite fines and coke) on the foaming behavior of a molten synthetic slag at 1700 °C. Based on the attained results, recycling MgO–C bricks and dolomite fines led to faster slag saturation and provided suitable conditions for foam formation. Furthermore, the increase in FeO content in the liquid portion of the slag compositions presented a clear correlation with the measured foam height, indicating that this oxide plays a major role in the foaming phenomenon.

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Keywords: Slag; Foam; Recycling; MgO–C refractories

1. Introduction

Slag engineering is becoming an essential issue in many companies due to the increasing necessity faced by the end-users to cut costs and produce high quality steel [1–3]. It is well-known that molten slag plays an important role in the steel production due to its capacity to withdraw impurities and inclusions out of the molten metal, reduce energy losses and refractory wearing, as well as protecting steel from re-oxidation [2].

Slag foaming has been mainly applied in electric arc furnaces (EAF) and basic oxygen ones (BOF) in order to protect the refractory materials from the high energy intensity (radiation) generated by the electrodes, decrease the noise level, improve productivity and the energy efficiency of this equipment. Some investigations reported [3,4] that this practice can save 3–10% and 25–63% of energy and refractory consumption, respectively.

The foam generation at high temperatures can take place mainly by injecting oxygen and carbon particles into the electric furnace bath, resulting at first in FeO formation in the liquid (Eq. (1)). After that, this oxide reacts with C and gives rise to $CO_{(g)}$ (Eq. (2)), which is the main gaseous phase responsible for the foaming effect [5,6].

$$Fe_{(l)} + \frac{1}{2}O_{2(g)} \rightarrow FeO_{(l)} \quad (1)$$

$$FeO_{(s,l)} + C_{(s,l)} \rightarrow Fe_{(l)} + CO_{(g)} \quad (2)$$

$CO_{(g)}$ formation is also related to the reaction between C (from the metal) and oxygen (Eq. (3)). Furthermore, companion reactions between $FeO_{(l)}$+$CO_{(g)}$ and $CO_{2(g)}$+$C_{(s)}$ (Eqs. (4) and (5)) are also expected to occur at the slag–gas and carbon–gas interfaces, respectively [7].

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$$C_{(l)} + \frac{1}{2}O_{2(g)} \rightarrow CO_{(g)} \quad (3)$$

$$FeO_{(l)} + CO_{(g)} \rightarrow Fe_{(l)} + CO_{2(g)} \quad (4)$$

$$CO_{2(g)} + C_{(s)} \rightarrow 2CO_{(g)} \quad (5)$$

An essential requirement for a suitable slag foaming comprises attaining the right balance between the refractory (CaO and MgO) and fluxing oxides (SiO_2, Al_2O_3, FeO, MnO and CaF_2) in the liquid composition. The overall chemical composition of the slag will directly define the basicity, surface tension, viscosity and other important properties of this molten phase at high temperature. According to practical tests, the most suitable slag compositions for EAF equipment are the MgO–CaO or MgO saturated ones, containing suspended second phase particles (i.e., $MgO \cdot FeO$ and Ca_2SiO_4) [6,7]. The saturated liquids will not only positively affect the foam generation, but also result in a decrease of the refractory wearing, as most of the lining materials applied in those furnaces are MgO–C based bricks.

Lime and dolomite are usually added to provide the right content of CaO and MgO to adjust the slag basicity and to saturate it during steel production. However, considering the high concentrations of MgO and C contained in spent MgO–C refractories (from BOF, EAF and ladle furnaces) and the demands from increasingly tough environmental regulations and landfill costs, one good alternative is to recycle those materials as slag conditioners. Recently, some studies have been carried out to evaluate the effectiveness of recycling practices involving MgO–C refractories to allow faster slag saturation and provide suitable conditions to induce foaming [8,9]. Although promising results were obtained, the incorporation of such materials to molten slags must be carefully implemented in order to avoid adverse effects on the steel making processes and products. Additionally, the main aim should not be focused only on recycling spent refractories, but also to attain longer refractory working life, energy savings, less slag generation, and efficiency and productivity enhancements based on technical understanding and proper plant practices [3,4,8,10].

With the advent of thermodynamic and mass balance calculations [11–14], it is possible to predict the slag saturation level, its overall chemical composition and define the most suitable conditioner that should be used during the electric furnace operation, reducing the need of experimental tests. A well-known tool to evaluate the slag foaming phenomenon consists of analyzing isothermal solubility diagrams (ISD) [6,13,14], which can define the influence of each slag component and the combination that might result in the liquid saturation with some oxides at high temperatures. Therefore, based on thermodynamic calculations and the simulated diagrams, it is possible to analyze and predict the position of the dual saturation point and the MgO and CaO saturated lines as a function of the slag basicity, temperature, oxygen partial pressure and other parameters [6,13].

Considering these aspects, the main objective of this work was to evaluate the slag foaming behavior of a synthetic slag containing different conditioners (compositions based on mixtures of spent MgO–C bricks, coke and dolomite fines). It must be highlighted that the selected dolomite was also a waste material produced in rotary kilns, which cannot be used to manufacture refractory bricks due to its lower chemical reactivity [8]. Experimental tests were performed in a laboratory induction furnace in order to simulate an EAF operation and measure the foam height generated at 1700 °C. Thermodynamic calculations were also carried out using FactSage™ software to predict the slag composition and its viscosity at high temperatures, as well as to simulate isothermal solubility diagrams to verify whether the resultant slag compositions (after the incorporation of the prepared conditioner) were located in regions that favor the foaming process.

2. Experimental and calculation procedures

2.1. Experimental tests

Six slag conditioners were prepared based on spent MgO–C bricks (from steel ladles and electric-arc furnaces), dead-burnt dolomite fines (produced in rotary kilns), coke, iron scales and a natural binder (linseed oil). The chemical composition and some properties of the prepared mixtures are presented in Table 1. The main difference among these materials is related to the spent refractory and dolomite ratio used. Composition *A*, for instance, contains higher MgO amount (43.52 wt%, due to the greater addition of ground MgO–C refractories) and only 3.46 wt% of CaO, whereas mixture *F* presents 32.36 wt% of MgO and 14.96 wt% of CaO (derived from the dolomite fine particles).

The processing comprised of a blend of raw materials that after mixing were pressed as briquettes (Fig. 1). It is important to highlight that the incorporation of iron scales to the compositions aimed to increase the briquettes density, keeping them close to the slag–metal interface during the practical tests.

In order to evaluate the effect of those conditioners on the foaming process at high temperatures, a synthetic slag comprising various components was prepared (Table 2). Experimental tests were carried out in a laboratory induction furnace

Table 1
Chemical composition and properties of the designed slag conditioners.

Components/Properties	**(wt%)**					
	A	*B*	*C*	*D*	*E*	*F*
SiO_2	1.71	1.62	1.64	1.70	1.73	1.50
Al_2O_3	0.94	0.77	0.80	0.82	0.74	0.77
Cr_2O_3	0.10	0.08	0.06	0.06	0.05	0.04
Fe_2O_3	10.92	9.78	9.64	9.08	9.03	8.35
MnO	0.05	0.08	0.06	0.06	0.07	0.06
CaO	3.46	5.94	7.99	10.71	12.74	14.96
MgO	43.52	41.08	38.70	36.28	35.02	32.36
Weight loss (400 °C)	8.76	8.05	7.17	6.57	6.14	6.33
Weight loss (1000 °C)	39.28	40.64	41.10	41.29	40.62	41.96
Volatiles	9.51	9.85	9.73	10.26	10.35	11.29
Ashes	51.33	50.37	52.20	52.54	53.43	51.73
R/*D*[*] ratio	10.00	4.50	2.67	1.75	1.20	0.83

[*] *R* = spent MgO–C refractories, *D* = dolomite fines.

Fig. 1. Prepared briquettes containing a mixture of spent MgO–C bricks, dolomite fines, iron scales, coke and a natural binder (linseed oil). The briquette length is 25.4 mm (~1 in.).

Table 2
Components used to prepare the synthetic slag.

Components	**(wt%)**
Reactive alumina	2.0
Lime	14.0
Hematite (Fe_2O_3)	5.0
Wollastonite ($CaSiO_3$)	25.0
Dolomite [$CaMg(CO_3)_2$]	13.0
Manganese dioxide (MnO_2)	6.0
Iron scale	35.0

simulating an EAF actual condition. The furnace was lined with refractory sections and loaded with 10 kg of SAE 1020 steel ingots (229 mm × 70 mm × 30 mm) and 200 g of the prepared synthetic slag. The temperature was kept close to 1700 °C and, after the complete melting of the steel and slag, 50 g of the conditioners (briquettes) were added into the furnace. The slag foam height was measured after 2 min based on the liquid adhesion on a steel rod that was inserted into the crucible. Samples of the resultant slag were also collected for further chemical analysis.

2.2. Thermodynamic calculations

Firstly, the thermodynamic simulations of the phase equilibrium and viscosity of the synthetic slag and slag+conditioners (*A–F*) mixtures at 1700 °C were carried out using the FactSage™ software (version 6.3.1, Thermfact/CRCT, Montreal, and GTT-Technologies, Aachen), in order to attain the chemical composition, properties of the molten phase and whether solid particles could be found in such conditions.

Phase diagrams known as isothermal solubility (ISD) in the system $MgO–Fe_2O_3–CaO–Al_2O_3–SiO_2$ were also calculated and presented, based on MgO and Fe_2O_3 contents (wt%) plotted as *Y* and *X* axes, respectively, and considering a ternary basicity ratio (B_3, Eq. (6)) in the range between 0.7 and 1.6 (according to the chemical composition of the resultant slags), temperature of 1700 °C and oxygen partial pressure (pO_2) of 0.21 atm.

$$B_3 = \frac{CaO}{Al_2O_3 + SiO_2} \tag{6}$$

FactPS and FToxid databases and Equilib, Phase Diagram and Viscosity modules were selected for this evaluation, considering as possible resulting phases: gas, liquid, solids and various solid solutions. Based on the calculated isothermal solubility diagrams and the slag compositions attained after the foaming tests, some parameters were evaluated (i.e., position of the liquids in the diagrams in order to define whether they are located in regions that favor the foaming process) with the aim of pointing out the effectiveness of the thermodynamic simulations and the possible correlations that could better explain the foaming behavior observed during the experimental tests at high temperatures.

3. Results and discussion

3.1. Major components and viscosity of the attained slags

After the foaming experiments, the chemical composition of the resultant slags (Table 3) was attained by chemical analysis. According to these results, the main differences detected, when comparing the initial synthetic slag and the ones containing the prepared briquettes, were related to the higher MgO and lower Fe_2O_3 contents of the conditioned molten liquids. The increase of the SiO_2 and Al_2O_3 amounts also led to a drop of their basicity levels (changing from 1.6 to values in the 0.7–1.4 range, Table 3), which surely reflected in some changes in the liquid properties and the phase equilibrium at high temperature.

In order to evaluate whether the conditioned slags were saturated by MgO, thermodynamic calculations were performed aiming to predict the phase equilibrium and viscosity of the molten liquid at 1700 °C. As shown in Fig. 2a, the compositions were comprised by liquid and solid particles of $MgO \cdot Fe_xO_y$ (which corresponds to a solid solution of $MgO \cdot FeO$ and $MgO \cdot Fe_2O_3$). The presence of such solid phase attests the saturation of the attained slags, as after the

Table 3
Major components and basicity of the resulting slags attained by chemical analysis after the foaming experiments.

Oxides (wt%)	**Synthetic slag**	**Slag+Conditioner**					
		A	***B***	***C***	***D***	***E***	***F***
SiO_2	15.08	24.65	22.16	21.03	22.80	21.01	16.54
Al_2O_3	2.51	5.08	4.55	4.22	4.51	6.76	4.36
Fe_2O_3	43.15	8.25	7.00	8.29	8.26	14.38	15.27
MnO	4.95	9.21	5.74	6.31	8.00	9.11	7.82
CaO	28.89	20.45	28.76	30.47	33.36	28.25	29.55
MgO	5.08	31.78	30.73	28.97	22.31	19.73	25.49
Basicity (B_3)	1.6	0.7	1.1	1.2	1.2	1.0	1.4

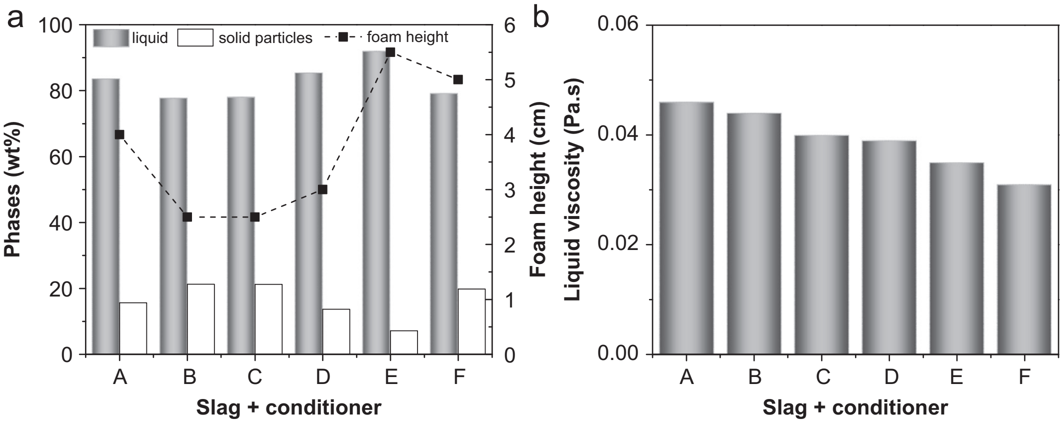

Fig. 2. Thermodynamic predictions of (a) phase equilibrium [the liquid and solid particles ($MgO \cdot Fe_xO_y$) contents] and (b) the liquid viscosity of the conditioned slags at 1700 °C. The foam height (cm) attained in the experimental tests are also highlighted.

liquid enrichment in MgO (due to the addition of the prepared briquettes), the excess of such oxide in solution most likely reacted with $FeO_{(l)}$ and $Fe_2O_{3(l)}$, leading to a further precipitation of solid particles at 1700 °C.

Some studies reported that solid particles usually act as gas nucleation sites, leading to a high amount of small gas bubbles in the molten slag and favoring the foaming phenomenon [3,6,15]. Additionally, the suspended particles can change the effective liquid viscosity, according to the following equation [6]:

$$\mu_e = \mu(1 - 1.35\Theta)^{-5/2} \quad (7)$$

where, μ_e is the effective viscosity of the slag (Pa.s) and Θ is the fraction of precipitated solid phases ($0 < \Theta < 0.74$). According to the literature [2–6], there is a close relationship between the foaming behavior and the effective viscosity (Fig. 3). The higher μ_e, the longer the residence time of the gas bubbles in the slag will be, extending the stability of the foam. However, there is a maximum amount of second phase particles that is beneficial for the foam stability.

Comparing the foam height and $MgO \cdot Fe_xO_y$ particles content shown in Fig. 2a, one can notice that the conditioned slag *E* presented higher foam generation and a lower amount of precipitated particles at 1700 °C. Conversely, the excess of the solid phase in compositions *B* and *C* resulted in the over-saturation of those liquids and led to a reduced foam level. Nevertheless, a direct correlation between the foam height and $MgO \cdot Fe_xO_y$ content was not observed, as slag+conditioner *F*, for instance, showed high foamability and approximately 20 wt % of precipitated solid particles (which is similar to the ones presented by compositions *B* and *C*, 21 wt%) might be formed.

If the synthetic slag:conditioner mass ratio was adjusted in order to attain only compositions presenting solid particles content close to 7 wt% (as the *E* composition presented in Fig. 2a)., it is expected that a more favorable condition for the slag foaming might be attained. Nevertheless, other parameters should also be taken into account, such as the viscosity of the liquid itself and the amount of FeO available in this phase.

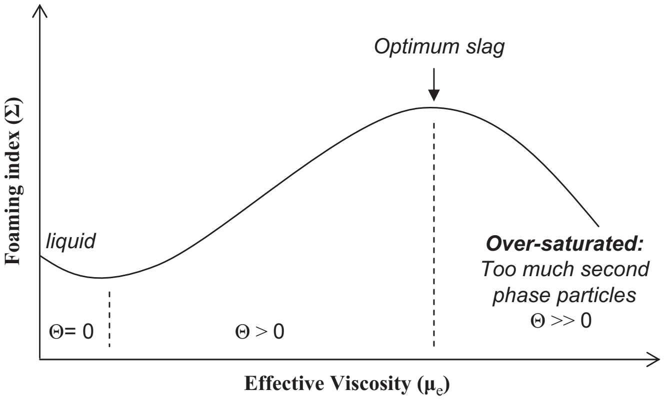

Fig. 3. The relationship between foaming index and effective viscosity [15].

Although the thermodynamic calculations could not predict the effective viscosity of the slags+$MgO \cdot Fe_xO_y$ particles, some simulations were carried out considering only the liquid portion attained (Fig. 2b). The higher the basicity, the lower the viscosity of the liquid phase was. However, again it was not possible to obtain a correlation between the viscosity values and foam height.

It must be pointed out that for a liquid with low viscosity, the gas bubbles will be easily eliminated and the foam may be suppressed. Conversely, the bubbles cannot be even generated in a highly viscous slag [3,16]. Hence, an optimized chemical composition (for a chosen temperature) should be attained to assure the formation and stability of the gas bubbles. Usually, this condition is only achieved when the slag is properly conditioned.

3.2. Isothermal solubility diagrams and ΔMgO

Isothermal solubility diagrams (ISD) were also calculated for the $MgO–CaO–Al_2O_3–SiO_2–Fe_2O_3$ system considering specific ternary basicity values (B_3=0.7–1.6), oxygen partial pressure (pO_2) of 0.21 atm and 1700 °C. The resultant composition of the synthetic slag and synthetic slag+conditioners and their respective ΔMgO (which represents the

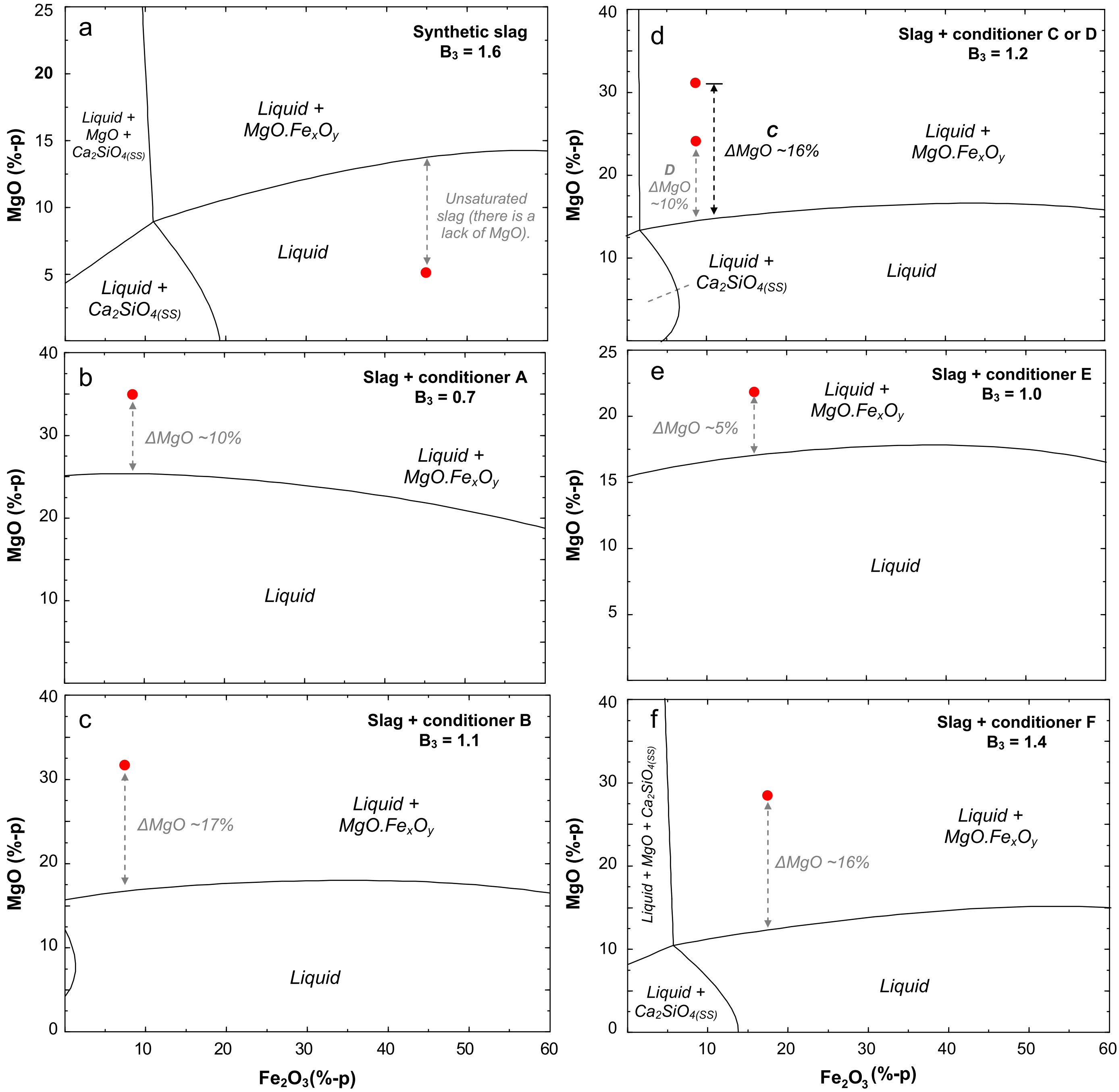

Fig. 4. Isothermal solubility diagrams (ISD) calculated for $MgO–Fe_2O_3–Al_2O_3–CaO–SiO_2$ system, considering distinct basicity values ($B_3=0.7–1.6$), temperature = 1700 °C and $pO_2=0.21$ atm. The resultant composition of the (a) synthetic slag and (b–f) synthetic slag+conditioners and their respective ΔMgO (related to the liquid saturation) were highlighted.

difference between the MgO wt% of the slags and the liquidus line, keeping constant the Fe_2O_3 content), were highlighted in Fig. 4.

According to the ISD calculated for the synthetic slag ($B_3=1.6$, Fig. 4a), this composition would be located inside the liquid phase field and far from the liquidus line (which separates the liquid and liquid+$MgO \cdot Fe_xO_y$ fields), indicating that MgO addition is required to obtain a saturated slag.

As observed in the other diagrams in Fig. 4, adding 50 g of the prepared briquettes (containing spent MgO–C refractories and dolomite fines) to 200 g of synthetic slag should induce the saturation or over-saturation ($\Delta MgO > 5\%$, based on the liquidus line) of the liquid and, consequently, the precipitation of solid $MgO \cdot Fe_xO_y$ particles.

Some authors [4,6,8,15] stated that the maximum foaming should only take place when the slag composition is located close to the liquidus region (about 6% out of liquid phase field). In this work, this condition was only achieved by conditioned slag *E* which was the one that presented the highest foam height (5.5 cm), corroborating the results presented in the literature.

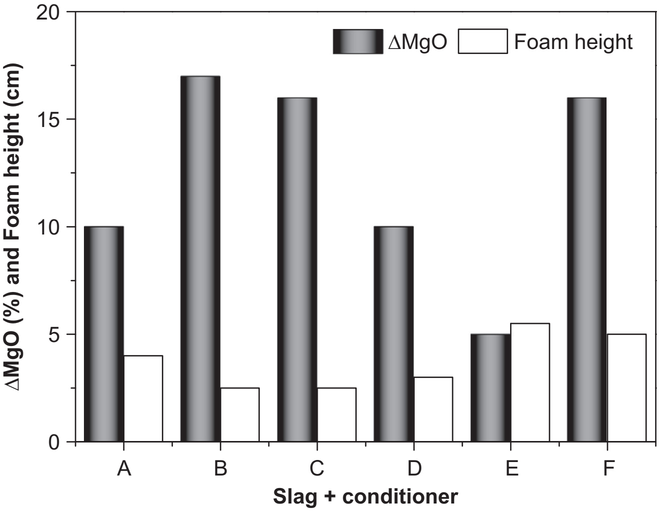

Fig. 5. MgO difference (related to the slag saturation) and foaming behavior of the slags containing the conditioner additives (briquettes).

For the other slag+conditioner compositions, the attained ΔMgO values varied from 10% to 17%.

Fig. 5 presents a summary of the ΔMgO values (based on thermodynamic calculations) and foam height (cm) generated during foaming experiments carried out in an induction furnace at 1700 °C. A direct correlation between those parameters was not observed, as for a ΔMgO = 16%, compositions *C* and *F* presented distinct foam generation (2.5 and 5.0 cm, respectively).

3.3. Correlation between slag components and foam height

In order to figure out the role of the liquid components on the foaming behavior, some additional thermodynamic and mathematical fitting calculations were carried out considering only the predicted liquid portion of the slag compositions. As presented in Table 4, a mixture of SiO_2, Al_2O_3, CaO, MgO, FeO, Fe_2O_3, MnO and Mn_2O_3 should comprise the liquid phase of the conditioned slags. The amount of each oxide was plotted versus the attained foam height in an attempt to identify which phase should directly affect the foam generation at 1700 °C.

Table 4
Chemical composition (predicted by the thermodynamic calculations) of the liquid portion of the slag+conditioner mixtures at 1700 °C.

Oxides (wt%)	**Slag+Conditioner**					
	A	***B***	***C***	***D***	***E***	***F***
SiO_2	29.43	28.49	26.95	26.66	22.81	20.88
Al_2O_3	5.99	5.74	5.30	5.12	7.29	5.40
FeO	1.79	1.05	1.21	1.44	2.54	2.14
Fe_2O_3	6.50	6.67	8.09	7.35	12.15	15.34
CaO	24.40	36.94	38.98	38.96	30.66	37.23
MgO	22.43	15.57	13.53	12.78	15.15	11.33
MnO	6.89	3.84	4.05	5.62	6.30	4.65
Mn_2O_3	2.54	1.65	1.85	1.95	3.06	3.00

Among the various phases evaluated, only FeO content showed a good linear correlation (regression coefficient equal to 0.989) with the foam height values (Fig. 6). The increase in the content of this oxide in the liquid induced greater foam formation.

FeO is one of the main components involved in the chemical reactions responsible for $CO_{(g)}$ and $CO_{2(g)}$ formation (Eqs. (2) and (4)) and it strongly influences the viscosity of slags (fluxing component) [2]. Consequently, it is reasonable to consider that the control of the FeO content of the liquid is a key factor to predict the foaming behavior. During the EAF operation, the actual amount of this oxide at high temperature depends essentially of its formation rate due to oxygen injection (Eq. (1)) and the rate that it is reduced by the carbon sources added (Eq. (2)). If the injected oxygen and carbon is optimized, and the slag is foaming well, the FeO content in the slag should fairly stay constant [6]. Furthermore, it must be considered a distinction between the total iron oxide of the slag (comprising solid particles+liquid) and the one that it is available in the liquid. As attested in this work, only the amount of $FeO_{(l)}$ showed a suitable correlation with the foamability of the evaluated compositions.

Although some studies [3,7,15] pointed out that the increase of FeO in the slag results in a reduction of the overall viscosity, the presence of precipitated particles also affects the effective viscosity of the system, which might still allow a suitable foam generation as detected for the conditioned slag *E* presented in this work.

4. Conclusions

Foaming is a fundamental phenomenon to improve heat transfer, refractory lining working life and to attain faster rates of melting in the electric furnace operation. According to the experimental and thermodynamic calculation results presented in this work, the recycling of spent MgO–C refractories and dolomite seems to be a good option to achieve faster slag

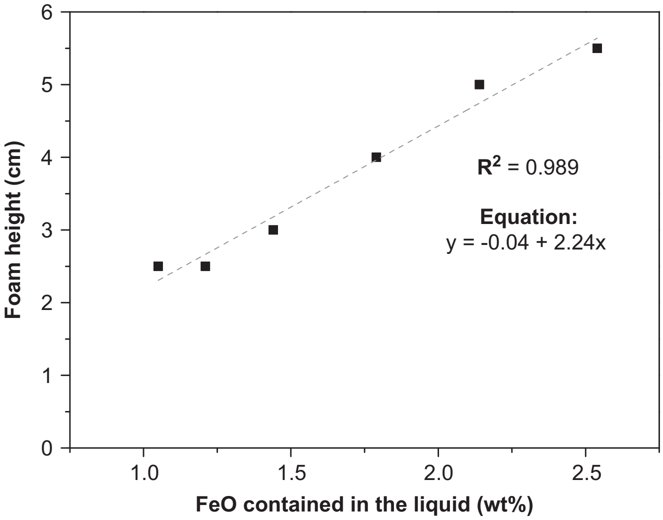

Fig. 6. Correlation between foam height (cm) and FeO (wt%) contained in the liquid slag formed at 1700 °C (liquid compositions were calculated using FactSage^{TM} software).

saturation and provide suitable conditions to induce foaming at high temperature. Nevertheless, special attention is still required to work out the proper amount of slag conditioner to be added to the slags in order to inhibit the over-saturation of the system. The increase of FeO content in the liquid portion of the slag compositions presented a clear correlation with the measured foam height, indicating that this oxide plays a major role in the foaming phenomenon. Thus, it would be important not only to adjust the MgO content, but also to control the carbon amount of the conditioners in order to attain suitable FeO levels in the resultant slag that should favor the foam generation. Additionally, the use of thermodynamic tools (such as the FactSage^{TM} software) and the isothermal solubility diagrams may allow an engineered and optimized recycling of spent materials.

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