SOME ASPECTS OF SLAG-METAL INTERACTION IN A TWO PLUME STIRRED LADLE

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ABSTRACT

A 1:5 scale physical model of a steelmaking ladle, simulating the protoype at V&M Brasil was built in order to assess the influence of several parameters on the metallurgical performance. The effects of gas flow rate and its distribution through 2 porous plugs on the plume eye and liquid-liquid mass transfer was investigated. Silicone oil of different viscosities and kerosene have been employed to simulate the top slag. The water properties have been adjusted by zinc chloride addition. The experimental results indicate that the eye opening is a function of the total flow from the plugs, top slag thickness but independent of slag viscosity. Plant trials on eye opening were carried out at Vallourec and Mannesmann (V&M) of Brazil, and the results of these experiments were in conformity with those carried out in the physical model. Mass transfer data obtained from the model are shown to be independent of gas distribution. Mass transfer was affected by total gas flow rate only.

INTRODUCTION

Generally the surface of the liquid steel is covered with a layer of slag in most of the refining operations. When a slag layer is present fluid-dynamic conditions in the reactor are controlled by the system of agitation, be it mechanical, pneumatic or magnetic. The intensity of agitation is chosen depending upon the specific purpose. For example moderate agitation is recommended with minimal disturbance in the slag-metal interface, to promote coalescence and flotation of inclusions. More vigorous agitation can be used to generate an "eye" (located on the opening in the slag layer) through which sampling of metal, additions of elements for alloying etc can be done. This *eye* region is unprotected and could lead to pick up of atmospheric gases like oxygen and nitrogen and is also characterized by a high state of superficial turbulence. High rates of agitation may also cause formation of emulsions, with the concomitant increase in the slag metal interfacial area thus favouring kinetics and mass transfer during the process.

There have been several investigations to study this phenomena using physical modeling to evaluate mixing times, formation of dead zones, behaviour of emulsions, plumes, and velocity and flux patterns of the liquid and during injection of inert gas as well as emptying of the liquid metal from the ladle (1-9). Cold model studies by Kim *et al.* [1] showed that critical flow of gas for starting emulsification can be determined by the Equation 1:

$$Q = 3{,}8\ 10^{-3}\ H^{1{,}81}\left[\frac{\sigma\ \Delta\rho}{\rho_s^{\ 2}}\right]^{0{,}35} \tag{1}$$

Where, Q: gas flowrate [lpm]; σ: interfacial tension between the two liquid [dynes/cm]; H: liquid level of the denser phase; Δρ difference between densities of liquid phases [g/cm^3]; ρ_s density of phase of relatively lower density [g/cm^3]. This relationship is valid for ladles with diameter/height ratios equal to 1, volumetric ratio of slag/bath equal to 0.1, and for injection of gas in the center of the ladle and results can be extended to industrial processes.

The nature of plume, velocity fields, distribution of bubbles and correlation of the eye of the plume and operational parameters in bottom blown ladles have been investigated by several researchers such as Mazumdar & Evans [2], Sahai & Guthrie [3], Brooks *et al.* [4] , Subagyo et al. [6]. It was observed that for a given height of the slag layer and level of liquid metal increasing the flow of gas increases the area of the plume eye.

The area of the spout of the plume by simple geometric analysis can be defined as:

$$A_s = (H + h)^2\, \pi \tan^2\theta_{pl} \qquad m2) \tag{2}$$

This area can be determined by the angle of the biphasic plume θ_{pl}, height of slag(h) and liquid steel(H). Krishnamurthy *et al.* [7] give the following equation to calculate the same:

$$\frac{\theta_{pl}}{180} = 0{,}915\ Fr_{mod}^{0{,}12}\left[\frac{H}{D}\right]^{-0{,}254}\left[\frac{d_o}{D}\right]^{0{,}441} \qquad F_{mod} = \frac{u_o^2}{g\ H}\left[\frac{\rho_g}{\rho_L - \rho_g}\right] \tag{3}$$

Here H = liquid steel level, m; D = diameter of the vessel, m; d_o = diameter of the orifice of gas flow, m; u_o= superficial velocity of the gas in the nozzle, m/s; g = acceleration due to gravity, m/s^2; ρ_g and ρ_L = specific mass of gas and of liquid, respectively, kg/m^3.

According to Subagyo *et al.* [6], the area of the eye of the plume considering the presence of slag in the top, can be estimated by the following Equation:

$$A_{es} = \pi \; d_{es}^2 \approx 0{,}02 \; (H + h)^2 \left[\frac{Q^2}{g \; H^5} \right]^{0{,}375} \quad (m2) \tag{4}$$

Where H and h = height of the metal and slag layers, respectively in meters, Q: gas flow rate, m^3/s. From the above equation the diameter of the plume eye (d) can be calculated. The number of eyes is equivalent to the number of nozzles for injection of inert gas.

Lin [8] conducted experiments on emulsification to clarify the phenomena of dispersion. To simulate dispersion in the process of bottom blowing, he used a solution of 7.34 M $ZnCl_2$ in water as the lower phase, and then obtained a ratio of density of the solution $ZnCl_2$ /oil equal to 1.72. Meanwhile the ratio density of the steel/slag ranges from 2.0 to 2.2 in practice. Silicon oil of different densities and viscosities have been used to simulate the slag layer. Han *et al.* [9] used various operating conditions in order to examine the formation of the eye plume in a physical model (water/oil) along with a mathematical model for validation. The evolution of the plume eye with time was determined and a comparison was made between measured and calculated values. The size of the plume eye was influence by both air flow and thickness of the oil layer.

In this brief literature review, general principles governing the formation of emulsions and the opening of *eye* of the plume are dealt with. It should be remembered that specific results depend on geometry of the system, such as one two or 3 plume ladle. In this paper results of physical modeling of a 2 plume ladle with validation from industrial practice are presented.

METHODS AND MATERIALS

A 1:5 scale physical model was built in acrylic adopting various similarity criteria. It simulates a 75 ton capacity industrial ladle with provision for injecting gas in the 2 different bottom locations (Figure 1). The physical model experimental setup is equipped with mass flow meters for controlling gas flow and compressors. Water or a water-zinc chloride solution was used for simulating the steel. Zinc chloride allows the control of density and viscosity of the underlying liquid phase. Silicone oil of different viscosities and kerosene(in case of mass transfer studies) have been used for simulating the slag layer. Air flow rate in the model can be calculated using dynamic similarity criteria, Silva *et al.* [11]:

Froude and turbulent Reynolds $$Q_{\text{mod }el} = \lambda^{2.50} Q_{industry} \tag{5}$$

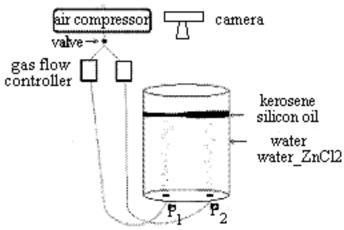


Figure 1: Schematic of the physical model of the ladle

Experimental Procedure for Determining Factors affecting Opening of Plume *Eye*

In these experiments silicone oil with viscosity of 50 and 500 centistokes (approximately 50 and 500 centipoise) was used to simulate the slag. Water was employed to simulate the liquid metal. The ladle was first filled with 85 liters of water. Three thicknesses of oil to simulate slag was tested, 2, 3 and 4 cm, with various combinations of flow rates in both the plugs, which ranged from 1 lpm to 20 lpm of air. A camcorder camera was installed above the model with the help of a support in order to film the process from top. There was also provision for filming from sides in order to monitor the formation of emulsions. During filming, the camcorder was connected to a digitalizing board for capturing in video. Accordingly the films were digitalized and then portions of them were captured as pictures. For each combination of operational parameters five randomly chosen photos from top were used for evaluating eye opening. Eye opening was evaluated as a percentage of total top cross sectional area of the ladle. A snap shot is given in Figure 2. Dark areas are of silicone oil. The light ones are the uncovered plume eye. The ratio *eye opening/ladle cross section* was then determined by weighing after photo printing. Thereafter the same procedure for determining the area of the opening formed in each photo was adopted. Data provided through the tables in this study correspond to the average of the five photos for each configuration.

Experimental Procedure for Mass Transfer Studies

In addition, mass transfer experiments were carried out using iodine distribution between kerosene and water in the cold model. Kerosene dissolves approximately 5.7 g of iodine/100 ml. Solubility of iodine in pure water is relatively very low (about 0.335 grams per liter of water at 25°C). Iodine is also easily lost by volatilization. In order to facilitate iodine transfer from kerosene to water an aqueous solution of Potassium Iodide should be preferred since solubility of iodine increases considerably after dissociation of potasss-sium iodide.

$$KI = K^+ + I^- \qquad I_2\text{ (kerosene)} + I = I_3^{--}\text{(aqueous)} \tag{6}$$

The amount of KI previously added to the water was in excess of the molar amount of iodine contained in 2.5 liters of kerosene, considering transfer complete, according to the reaction,

$$KI\text{ (water)} + I_2\text{ (kerosene)} = KI_3\text{ (water)} \tag{7}$$

Initially the air flow rate was adjusted at pre-determined levels until reaching steady conditions. At zero time kerosene is added on top, and at the same time sampling of 100 ml of the aqueous solution, for a 0.006M thiosulfate titration begins. The titration volumes are proportional to the concentration of iodine in aqueous solution and can be used as a measure of mass transfer kinetics.

RESULTS AND DISCUSSION

A photograph taken from the top for the condition: air flow rate 1 lpm in the first plug and zero in the other (1 - 0), 2 cm layer of oil of 50 cSt, is given in Figure 2 as an example of typical results generated by this technique.

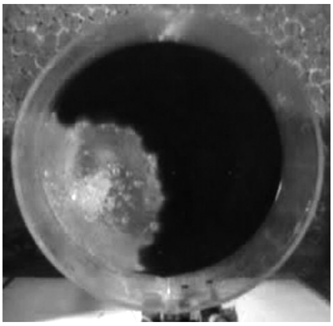


Figure 2: Photo of plume eye taken from top. Water (light area)-silicone oil (dark area) system 1 lpm in the first plug and zero in the system: 10. Other (1-0), 2 cm layer of oil of 50 cSt.

Figure 3: Photo of plume eye taken from top. Water/$ZnCl_2$ solution (light area)-silicone (dark area) air flowrate lpm in the first plug and 5 lpm in the other (10 - 5), 3 cm layer of oil of 500 cSt.

In another set of experiments, the same methodology was applied, but using a solution of zinc chloride, containing 50 kg of $ZnCl_2$ in 80 liters of water. The solution had the density of 1443 kg/m^3 and viscosity 22.24 mPa.s. A typical result is shown in Figure 3.

Analysis of Results of Water/Oil System

The specific masses of water and silicone oil are very close, unlike the system steel/slag having a ratio of densities around 2.5. Thus there is greater tendency to dispersion in the system water-oil. The methodology used does not distinguish precisely the layer of the supernatant portion from the eye opening when there exist oil dispersion under the water or zinc chloride solution level. The silicon oil layer contour is not well defined from a top view in such cases. Thus one could acquire reliable data up to the flow rate of 15 lpm in the case of oil with viscosity of 50 cSt, and up to the flowrate of 20 lpm with oil of 500 cSt. The results for the silicone oil of 50 cSt and 500 cSt / water are given in Table 1.

Table 1: Average values of % opening, oil-water system (50 cSt; 500 cSt); P1 and P2 stand for plug #1 and plug #2 respectively

Flow Rate (lpm)		% opening (water-oil)					
P1	P2	50 cSt			500 cSt		
		2 cm	3 cm	4 cm	2 cm	3 cm	4 cm
1	0	29.26	23.17	17.76	37.18	32.00	28.70
2	0	35.52	26.36	19.08	50.19	45.66	30.90
3	0	46.15	28.26	23.27	54.98	52.53	39.55
4	0	50.07	30.33	25	56.92	53.97	45.69
5	0	52.22	38.95	32.44	61.78	58.71	48.52
10	0	63.04	51.58	42.8	65.04	57.14	48.85
15	0	60.04	49.57	42	63.04	58.26	48.79
20	0				62.71	54.65	49.45

It can be observed that opening of the eye increases with the flow, tending to stabilize after a critical flow rate of about 10 lpm for oil with 50 cSt and 5 lpm for 500 cSt. Higher the thickness of the oil layer , smaller is the eye opening probably reflecting the higher amount of mass to be pushed to the ladle periphery. One should expect also a larger slag layer stability when the viscosity increases.Studies by Han *et al.* [9] suggest the same trends reported here in respect of the influence of height of the top layer and the observation that increased rate of gas injection increases the eye opening. Increasing the top layer thickness means higher mass of top layer. Thus a higher rate of injection is necessary for opening of the plume eye and also a higher liquid velocity is necessary for the formation of emulsions.

The influence of viscosity of the oil on eye opening is highlighted in Figures 4 and 5. The results suggest that for a higher viscosity leads to greater the eye opening . These results do not match with the literature. With increased viscosity one should normally expect a higher energy input needed for generating the eye opening. That is because higher viscosities imply on overcoming larger frictional forces. It should also be remembered that the density difference between oil and water is small and this could work as a compensating factor promoting greater instability at the interface. As had been stated before slag layer contours lose definition, whenever emulsion starts taking place. In that case, there is likelihood of eye opening being under evaluated. This effect would be more important at low viscosities levels when emulsification becomes easier.

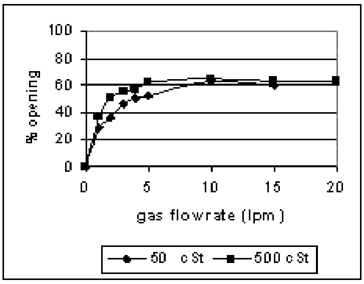


Figure 4: Relationship between % opening and gas flowrate for viscosities of 50 cSt and500 cSt, and oil layer thickness of 2 cm.

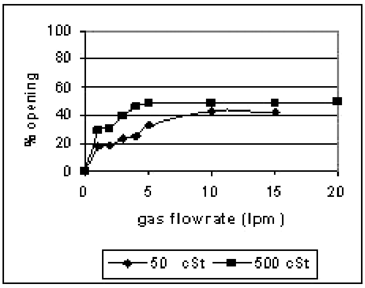


Figure 5: Relationship between % opening and gas flowrate for viscosities of 50 cSt and cSt, and oil layer thickness of 4 cm.

Analysis of Results of $ZnCl_2$/Oil System

In the case of $ZnCl_2$/oil system also similar experiments were done. In this case there exists greater density difference between the liquid phases. Thus higher flow rates could be employed. In general the experimental results are in conformity with those of Lin [8] .

In respect of these experiments, there has been greater stability of the interface $ZnCl_2$/ oil leading to a smaller number of drops of oil entering the solution of zinc chloride and a lower eye opening. Results are given in Tables 2 and 3.

Table 2: Average values of % opening, oil-$ZnCl_2$/water system (50 cSt; 500 cSt); P1 and P2 stand for plug #1 and plug #2 respectively; injection in one plug

Flow Rate (lpm)		% Opening (water/$ZnCl_2$-oil)					
P1	P2	50 cSt			500 cSt		
		2 cm	3 cm	4 cm	2 cm	3 cm	4 cm
1	0	1.84	1.1	0.33	2.05	0.86	0.63
2	0	3.7	1.77	0.67	4.58	2.39	1.07
3	0	6.15	2.77	0.8	7.0	2.64	1.99
4	0	7.97	4.48	1.26	9.4	4.06	3.59
5	0	9.77	5.33	1.98	12.2	5.26	3.90
10	0	15.13	11.38	6.63	19.74	10.16	7.14
15	0	18.61	13.27	7.96	25.49	12.73	10.12
20	0	21.75	16.38	12.63	28.25	14.75	12.69

Data for injection with two plugs are given in Table 3. There was only a slight difference in the percentage of the eye opening, when compared with data relating to the total, for 1 plug or two plugs. The biggest difference seems to be the density of bottom phase. For example analyzing the data for the water/oil (viscosity of oil-50cSt) there is greater instability in the interface between the phases, for the same injection rate of air. Thus there is greater degree of emulsification of virtually all the supernatant in the denser phase. On the contrary in the system water/$ZnCl_2$- oil, there is greater stability at the interface, and for the same flow of air, lesser amount of drops of oil enter the solution of zinc chloride.

Table 3: Average values of % opening, oil-water/$ZnCl_2$ system (50 cSt; 500 cSt); P1 and P2 stand for plug #1 and plug #2 respectively

Flow Rate		**%Opening (water/$ZnCl_2$ – oil)**					
P2(lpm)	**P1+P2**	**50 cSt**			**500 cSt**		
		2 cm	3 cm	4 cm	2 cm	3 cm	4 cm
5	10	17.21	9.4	3.35	17.10	6.12	4.10
5	15	18.06	11.48	6.35	22.79	11.18	7.28
5	20	21.68	13.85	9.19	23.82	13.07	9.83
5	25	23.19	15.17	11.75	27.16	13.41	11.91
10	20	26.2	14.81	10.49	29.03	12.47	8.82
10	25	27.69	18.38	14.55	31.08	14.73	11.83
10	30	27.37	19.29	17.14	31.94	19.54	13.63
15	30	29	21.82	17.31	37.15	18.04	12.79
15	35	34.38	20.13	16.19	34.88	16.37	15.34
20	40	34.01	19.62	19.08	37.40	21.69	19.84

Analyzing the size of the plume, there is a sizable variation between the results for different systems. There was relatively more eye opening of the plume for the water-oil system. This can be primarily the influence of the small difference in density between the phases causing instability in the interface. In the case of $ZnCl_2$, the opening observed is relatively less partly due to increased viscosity of the solution causing a slower rise of the bubble, circulatory flow in the solution with less intensity and relatively greater difference between the densities of liquids. The results can be seen in Figure 6.

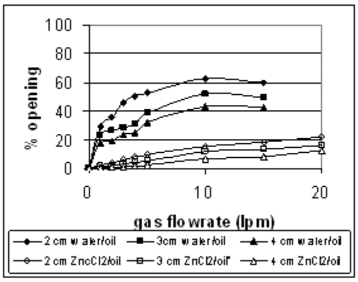


Figure 6: Comparison between eye opening in the systems water_$ZnCl_2$ and water / 50 cSt oil as function of gas flowrate and slag tickness

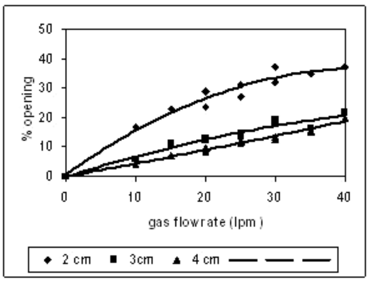


Figure 7: % opening vs flowrate for water_$ZnCl_2$ / 500 cSt oil system

The behaviour pattern of the 2 liquids with different viscosities in (500 cSt and 50 cSt) does not differ much (Tables 3 and 4 and Figure 7). As in previous cases there has been greater stability of the interface with the system $ZnCl_2$/water-oil, leading to a smaller number of drops of oil entering the solution of zinc chloride and a lower opening of the eye plume. The data were analyzed by the procedure of stepwise regression (MINITAB). The best correlation found is as follows :

$$A = 5{,}72 \frac{(P1 + P2)^{0{,}86}}{H^{1{,}52}} \quad (8)$$

P1 and P2 are the flowrates measured through the plugs and H the thickness of the oil layer. It is observed that the total opening(injecting through two plugs or injecting through one plug) depends, in these experimental conditions, only on the total flow and the thickness of the oil and the influence of oil viscosity is insignificant.

Analysis of Industrial Results in Relation to the Results of Physical Modeling

Experiments were also performed, for measuring the eye opening in slag coverage of a ladle unit of V&M of Brasil industrial plant and theses results were compared with those obtained with physical model, simulating this system, although industrial data obtained were limited to some extent. Essentially the same technique as in the physical model was used for measuring the eye opening. To facilitate the contrast , a thin layer of powder was poured over the layer of slag so to have it darkened as compared to the surface of of the steel.melt. The thickness of slag was measured through immersion of wires at different points. The values of % opening found on the industrial system as a function of flow rate (STP lpm) are shown in Table 5, along with the flow rate of air that would be needed, according to the formulation obtained in physical modeling for the same % opening (water/$ZnCl_2$ system - oil). This relationship can be mathematically expressed as:

$$Q_{model} = 0.0363\ Q_{ind} \qquad R2 = 0.94 \quad (9)$$

This expression can be compared with that used to define the conditions for the operation of the model,

$$Q_{model} = \alpha\ \lambda^{2.5}\ Q_{ind} \quad (10)$$

So considering λ=0.2, α=2.03 is obtained. This factor of correction indicates that the industrial flow reported in STP lpm needs to be corrected to reflect conditions closer to the environment. An estimate of the necessary correction can be found primarily assuming the thermal expansion of the gas injected to the temperature of liquid steel. For example T_{steel} / T_{inj} = 1873/298 = ~ 6.28 times if T_{steel} stands for metal temperature and T_{inj} for injection temperature. The column of steel has the density of 7000 kg/m^3, and exerts a high pressure on the gas bubbles compressing them. This effect could be estimated as $(P_a + \rho\ g\ L)/P_a$ = 2.66, where Pa stands for ambient pressure, ρ density of steel, g gravity acceleration and L the metal column level (in this case 2.5 m). The net effect of the two trends would lead to a *theoretical* correction factor of around 2.35. For the model, if the water column is not considerable, and for isothermal conditions no correction of this kind should be necessary. So there seems to be good agreement between the results of physical modeling and industrial practice.

Table 5: % opening in the model and industrial plant (10 cm of slag), as a function of gas flowrate

% opening	8	10	12	14	16	18	22	26	36
$Q_{industrial}$ (STP lpm)	56	112	168	224	280	336	448	559	839
Q_{model} (STP lpm)	5.02	6.51	8.05	9.62	11.24	12.89	16.27	19.76	28.84

Results of Mass Transfer Experiments

Experiments on mass transfer were made for various combinations of flow rates of air in the plugs. The experimental conditions involved full ladle (85 liters), which would be comparable to 75 ton. of steel and a volume of 2.5 liters of kerosene saturated in iodine simulating the slag.

Figure 9 give the volumes of solution of thiosulfate 0.006 mol/L needed for titrating 100 ml of aqueous solution, for two different combinations of flow rates. As already stated the volumes are proportional to the concentration of iodine in aqueous solution.

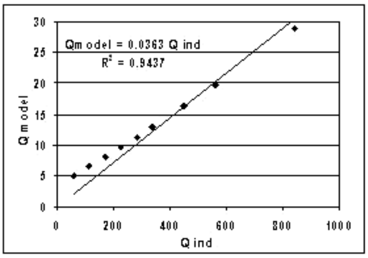


Figure 8: Flow rates (STP lpm)in the model and industrial machine, capable of producing the same % opening

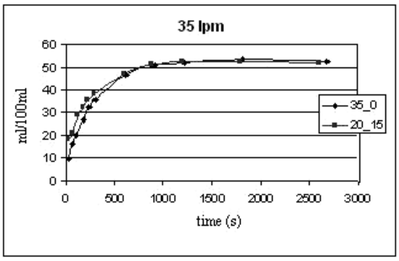


Figure 9: Kinetics of transfer for the same flow rate and different gas plug distribution

A macroscopic model (11) for the transfer of mass between the kerosene and aqueous solution gives:

$$\frac{C_{eq} - C}{C_{eq}} = e^{-\frac{AK}{V}t} \tag{11}$$

Where V is the volume of an aqueous solution; C is the concentration of iodine in aqueous solution, A is the area of interface kerosene/aqueous solution, K the apparent mass transfer coefficient, C_{eq} the equilibrium concentration of iodine, t elapsed time.

This model assumes perfect mixing of water and kerosene, except in a region corresponding to the boundary layer. The titration curves can be adjusted to the above equation yielding a value of AK/V. In most cases the exact value of A is not known as a function of eye opening in the slag layer or its emulsification. However kinetics of the process can be characterized by this factor. Higher this parameter the higher is the processing speed.

The data suggests an exponential relationship (Equation 12) with the total flow in the plugs, although dispersion seems substantial in the flow region of 35 lpm.

$$\frac{AK}{1000V} = 0.066\ q^{1.278} \qquad R2=0.921 \tag{12}$$

Riboud *et al.* [10] determined through comparison of a kinetic model and data from the industrial desulfurization process an expression for the calculation of mass transfer coefficient between a gas stirred steel melt and slag:

$$k = 500.\left(\frac{D_S.Q}{Area}\right)^{1/2} \tag{13}$$

Here D_S= 2.8 x 10^{-8} x EXP (-7500 / (1987 x T)),m^2/s, is the diffusivity of sulphur in steel; Q, gas flowrate at the temperature and pressure of the interface, m^3/s; Area, cross section area of the ladle, m^2. The discrepancy between the exponents obtained in this modeling experiments and those cited by Riboud *et al.* [10] may reflect the influence of flowrate upon slag emulsification.

In respect of the value of the exponent , Figure 11 shows that the apparent mass transfer coefficients in desulfurization as a function of gas flow. For low flowrates the exponent is around 0.25 and for high flow rates this is nearly 2.1. This change in behavior is attributed to the emulsification metal/slag, for larger flowrates.

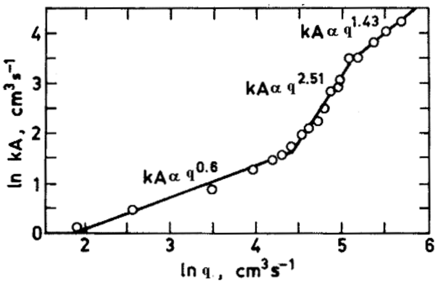
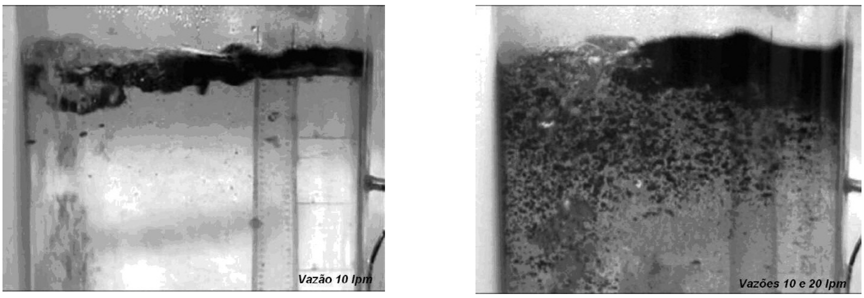


Figure 10: Apparent mass transfer coefficient for ladle desulfurization as a function of gas flowrate [1]

Figures 11 illustrates, for these experiments, the state of emulsification in the system. In view of the fact, the low viscosity kerosene and the specific mass being close to the water, emulsification is expressive, especially at higher flow rates.



(a) (b)

Figure 12: (a) Emulsification of kerosene for flow rate of 10 lpm, one plug ;
(b) Emulsification of kerosene for flow rates of 10 lpm and 20 lpm, 2 plugs.

CONCLUSIONS

Physical modeling of a steelmaking ladle of V&M of Brasil industrial plant allowed the investigation of some aspects of the interaction metal-slag. The particular feature of this ladle is the injection of inert gas through two porous plugs, in various combinations of flow rates.

It was observed that the total eye opening, measured as a fraction of ladle cross sectional area, was amplified when using the system water-oil when compared with the water/$ZnCl_2$-oil system. This is to be expected because of the small difference in densities of water and oil.

The addition of $ZnCl_2$ allowed changes to the underlying liquid phase density and viscosity. Density was adjusted to almost the same liquid/liquid ratio as in the actual steel/slag system. In this case the experiments indicated that the total % opening would be function of the total flow by the two plugs and the height of the layer of supernatant, regardless of the viscosity of the oil.

Industrial experiments conducted at V&M of Brasil industrial plant allowed a comparison with the data obtained from modeling. It has been shown that the expression developed in physical modeling can be used in industrial practice if proper correction is made for gas flowrate in respect of gas expansion at liquid metal temperatures and compressibility due to liquid metal column.

Mass transfer studies suggest that the kinetics (two liquids, water and iodine saturated kerosene) depends, too, upon the total flow of gas, not of gas distribution between the plugs.

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