Modelling the physical properties of electrofluxes through the lens of experimental data

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Hugo Lucas
RWTH Aachen University

Bernd Friedrich
RWTH Aachen University

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H. Lucas[1*], R. Schneider[1], J. Mohsseni[2] and B. Friedrich[1]

[1] IME Process Metallurgy and Metal Recycling, RWTH Aachen University, Aachen, Germany
[2] Wacker Chemie, Burghausen, Germany

[*]E-mail: hlucas@ime-aachen.de

Abstract. Electroslag remelting (ESR) and vacuum arc remelting (VAR) are widely employed techniques for producing defect-free ingots in ferroalloys, nonferrous metals, and superalloys. These technologies find applications in sectors like military, aerospace, and aeronautics, where high performance is of paramount importance. Given the critical nature of these applications, a comprehensive understanding of the smelting process is imperative. Therefore, developing precise models for predicting the physical properties of the involved slag used in the ESR process is crucial, as it enables operators to simulate and optimise process parameters effectively. This study comprehensively modelled three premelted commercial electrofluxes from Wacker and compared these models with experimental data. Key properties such as viscosity, density, electrical conductivity, specific heat (Cp), heat of fusion, and linear and volumetric thermal expansion coefficients were modelled through precise mathematical expressions, which can be subsequently integrated into process simulations. Moreover, the Roscoe-Einstein expression, traditionally used for correcting viscosity during solid-liquid phase transitions, was adapted to model electrical conductivity across the entire temperature range, from solid to liquid phase. The created models strongly correlated with the experimental data, providing operators with reliable and accurate information about Wacker Electrofluxes in their production processes.

1. Introduction

Electroslag remelting (ESR) and vacuum arc remelting (VAR) are widely employed techniques for producing defect-free ingots in ferroalloys, nonferrous metals, and superalloys [1–3]. These techniques are used in sectors where high-performance and high-reliability materials are needed, such as energy, aerospace, aeronautics or military [4–7].

In an ESR furnace, a consumable metal electrode is continuously remelted through a slag (known as electroflux) in a water-cooled copper mould. The smelting process during the stationary remelting phase is depicted in Figure 1. Electric current flows from the electrode through the liquid slag bath, then over the remelted ingot to the base plate. Since liquid slags possess significantly higher electrical resistivity than metallic conductors, most of the electric power is converted into heat within the slag bath. As the temperature of the slag bath surpasses the liquidus temperature of the electrode material, a thin layer of liquid metal forms at the tip of the electrode, from which droplets continuously detach. Due to density disparity, these metal droplets sink through the slag and gather in a pool of liquid metal before solidifying [1,8].

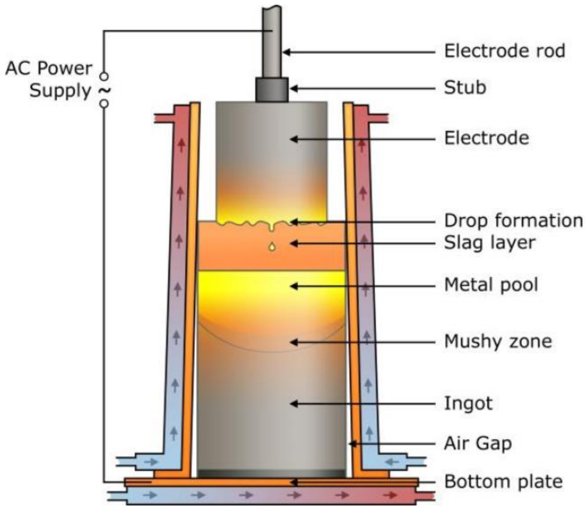

Figure 1. Schema of an electroslag remelting furnace [8].

The slag plays several crucial roles, including absorbing metal impurities, remaining inert to the alloy metal, and possessing adequate conductivity to generate sufficient heat through electric current. To maintain the electroflux remelting's focus on metal or alloy refinement, the slag's properties must be meticulously tuned through thermochemical calculations, preliminary experimental trials, and a pre-melting process, such as in an electric arc furnace. This meticulous approach enables precise adjustments of the slag properties tailored to specific applications. The large number of parameters and influencing variables results in many possible applications and advantages. The slag systems used can be used to influence metal and alloy systems via slag metallurgy and thus carry out melting treatments, e.g. the reaction of slag components with alloying elements to remove dissolved impurities, the absorption of non-metallic impurities or also to protect molten metals from the atmosphere. [6,9,10]

Given the importance of the applications utilising metals produced via this process, current research endeavours and operators prioritise their optimisation using computational models [6,10]. These models aim to forecast experimental outcomes based on various experimental parameters. One significant challenge lies in developing dependable models that accurately represent the diverse physical properties of the slag across the entire temperature spectrum of the technique, spanning from 20 °C to 1,800 °C.

Commercial electrofluxes use CaF_2, CaO and Al_2O_3, besides other minor oxides such as SiO_2, TiO_2 and MgO [8,11]. CaF_2 has the characteristic of high electrical conductivity and a low melting temperature (1,418 C) compared to other pure materials such as Al_2O_3 (2,072 °C) and CaO (2,572 °C). In addition to having properties compatible with the metals to be treated (e.g. super alloys, titanium), this element does not possess oxygen, which is an advantage in the ESR process. The use of CaO and Al_2O_3 is related not only to a lowering of the melting point but also to a high sulphur retention capacity, which is extremely important when refining a metal [11–13].

Several authors have studied the ternary system CaF_2-CaO-Al_2O_3, especially regarding liquidus temperatures and phase formation [12,14–17]. Two theories, molecular and ionic, elucidate slag behaviour. Molecular theory relies on stoichiometric oxide, sulphide, and fluoride formulas (e.g., SiO_2, CaO, FeO) to predict compound formation and elemental activity. Conversely, ionic theory views systems as cations and single or complex anions (SiO_4^{4-}, PO_4^{3-}, AlO_3^{3-}, BO_3^{3-}) to estimate slag properties like viscosity and electrical conductivity at elevated temperatures [18–20]. Although many of the theories allow for the calculation of physical properties, few studies have created equations that allow for the transition from the solid to the liquid state to be taken into account.

For example, if viscosity (η) is considered, experimental data show that the Arrhenius relation can describe it according to equation 1:

$$\eta = A_\eta \exp(E_A/RT). \quad (1)$$

Where A_η is a constant, E_A is the activation energy, R is the gas constant, and T is the temperature in Kelvins.

However, in complex slag systems, solidification occurs over a wide range of temperatures, creating a transition period where solid and liquid particles coexist, deviating the viscosity calculations according to the Roscoe-Einstein model (1952) [21]:

$$\eta_c = \eta_l.(1 - x_s)^{-n}. \quad (2)$$

Where η_c is the corrected slag viscosity, η_l is the ideal liquid viscosity, x_s is the solid fraction, and n is an empirical parameter (2.5 in this case) [22].

Like viscosity, electrical conductivity (σ) can also be described by the Arrhenius relation (equation 3), but with the difference that during cooling, it tends to zero instead of infinity like the viscosity.

$$\sigma = A_\sigma \exp(-E_A/RT). \quad (3)$$

As both properties are affected under the same parameters, nature of ions and temperature, several investigations have shown a direct quasi-linear relationship between the logarithms of viscosity and conductivity [23–27]:

$$\ln \eta = m - n \times \ln \sigma. \quad (4)$$

Where m and n are coefficients related to the ionic nature of the melt.

Concerning other physical properties, the optical basicity models of Duffy and Ingram (1971) have proven to be efficient for the prediction of several physical properties, including density [28–30].

However, it should be noted that integrating current experimental data of ESR electrofluxes such as Borowski (2023-electrical conductivity) or Akbary et al. (2007-density) with robust thermochemical models such as FactSage is perhaps the most successful method for creating continuous models [31–33].

2. Experimental methodology

For this study, one commercial electrofluxe called "ESR 2015" from the company "Wacker Chemie AG" was chosen (see chemical composition in Table 1). Simulation regarding Thermodynamic parameters describing a slag system in the function of temperatures such as Enthalpy (H), Gibbs energy (G), specific heat capacity (Cp), heat of fusion (L), solid fraction (x_s) and density in solid state (ρ_s) were carried out using Equilib module with FactPS, FToxid databases in FactSage 8.2. The simulation was also used to predict the nature of ions formed in liquid state compared with the slag's commercial chemical composition based on solid-state XRF analyses. Considering calculated ion speciation in liquid state in the studied range of temperatures (20 °C-2,000 °C), viscosity was first calculated using the Viscosity module in FactSage 8.2 and then corrected using the Roscoe-Einstein correlation (equation 2) for the transition of viscosity between a fully liquid phase and a solid-state (infinite viscosity).

Table 1. Wacker commercial ESR2015 electroflux.

Type of analyse	CaF_2	CaO	Al_2O_3	TiO_2	MgO	SiO_2	AlF_3	MgF_2	SiF_4
Chemical[a]	31.8%	29.7%	33.8%	0.1%	3.0%	1.5%	0.0%	0.0%	0.0%
Model[b,]	14.4%	42.1%	27.1%	0.1%	2.6%	1.2%	10.9%	1.0%	0.5%

[a] Analised by XRF. CaF_2 and CaO speciation investigated by dissociation and titration methods [1].
[b] Modelled oxides and fluorides distribution according to FactSage 8.2 for Temperatures higher than melting point. Fluor not only combines with calcium but also distributes and react with other chemical elements present in the slag depending on their activity.

Several authors have pointed out the interrelationship between viscosity and electrical conductivity [13,20,26]. If adding a solid fraction increases the viscosity, it must act inversely to the conductivity. Here, electrical conductivity measurements have been taken based on the method described by Nikolaus Borowski [31] and the inverse of the function proposed by Roscoe to correct viscosity (equation 2). This correction allows a continuous electrical conductivity model below the melting point.

Liquid and solid fractions (x_l) calculated with FactSage were fit using the logistic regression (equation 5) and the itineration algorithm Levenberg-Marquardt:

$$x_s = \frac{A}{1+\left(\frac{T}{B}\right)^C} \ ; \ x_l = 100\% - x_s \quad (5)$$

Where A, B and C are constants calculated by the regression. Constant B is also close to the middle point between the temperature of the first liquid (T_f) and the melting point (T_m)

Regarding density, experimental raw data obtained by buoyance methods (Archimedes) at high temperatures [33], were utilised together with the distribution and nature of ions in the melt (see Table 1), melting point and solid fraction (x_s) calculated in FactSage 8.2 and fit it with equation 6 [20,34]. Using this data, molar volumes and thermal expansion coefficients were calculated, carrying out regressions based on equation 6. As a reference, the density of slags was also measured using a He pycnometer at room temperature.

$$V_{liquid} = V_m \times [1 + \beta \times (T - T_m)] \quad (6)$$

Where Vm is the molar volume (cm^3/mol) at melting point (T_m), and β is the volumetric thermal expansion coefficient.

Linear thermal expansion coefficients (α) are approximately one-third of the volumetric thermal expansion coefficient (β) [34].

Diferencial temperature analysis (DTA) was performed with a Netzsch thermobalance STA 409 in Ar atmosphere with a 10 °K/min heating rate up to 1,500 °C to compare the ESR2015 electroflux with the thermochemical model.

2. Results and discussion

2.1 Liquid fraction and melting point

In the ESR process, the slag has a lower melting point than the metal to be treated. However, although the melting point is important, a multicomponent slag can have a wide range of temperatures between its solid and liquid state. In the case of ESR 2015, the thermochemical model predicts that this slag will start its melting process between 1,116 °C and 1,379 °C (slag melting point). When comparing the model with the DTA test performed with the real sample, it can first be seen that the curve remains constant up to 1,000 °C and then reaches a transition point almost at the same initial temperature (T_f) predicted by the model until reaching a peak at 1,116 °C. After this maximum peak, the DTA values decrease rapidly, coinciding with a liquid-solid transition process. The right axis plot in Figure 2a shows that the slag should have a period between 1,234 °C and 1,320 °C where two liquid phases coexist. DTA has shown a change of slope in this same area. Finally, the lower peak of the DTA curve coincides exactly with the melting point, showing that the thermochemical model fits almost perfectly with the experimental data.

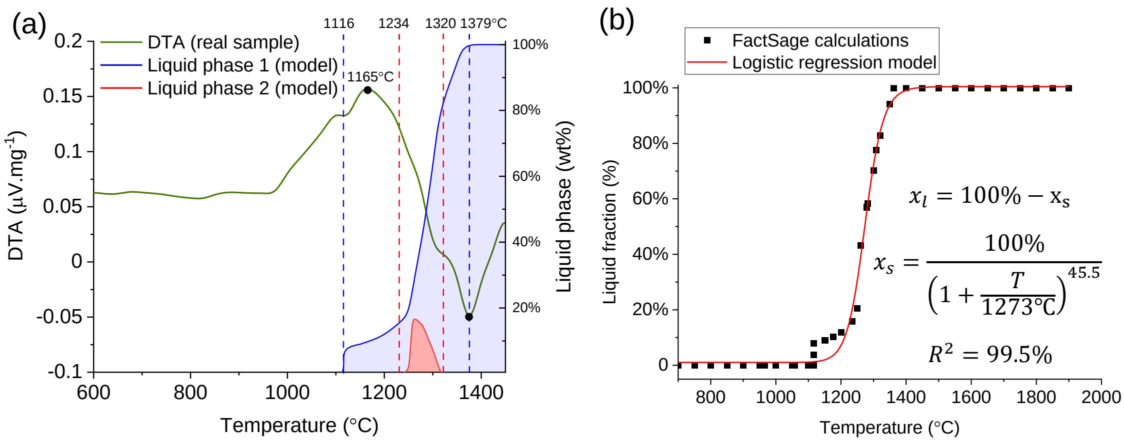

Figure 2. Modelling liquid fraction and melting point: (a) DTA on real ESR 2015 samples vs liquid phases, and (b) Calculated ESR 2015 liquid fraction versus Logistic regression algorithm.

Several regression algorithms were analysed to create simple and accurate models for liquid and solid fraction over a full temperature range. It was found that the logistic regression model, proposed by Pierre-François Verhulst for modelling population growth [35], can predict the liquid-solid transition phenomena with a confidence threshold of 99.5 % (Figure 2b). This algorithm can be used to simplify the calculations of many of the physical properties necessary for the computational modelling of the furnace process.

2.2 Thermodynamic variables

Most of the slag properties are affected by the structure of the slag. Creating simple algorithms describing a complex system that needs complex calculations is a real challenge. For example, the heat capacities (Cp) of simple components are described by equation 7, but a slag system's Cp will require a combination of each elementary Cp using molar fractions and some constants; thus, no simple algorithms can describe such a system [20].

$$C_p = A + B \times T + C \times T^{-1} + D \times T^2 + E \times T^{-2} \ldots \quad (7)$$

Where T is the temperature in Kelvins and A, B, C, D, and E are coefficients.

Suppose the calculations of the main thermodynamic variables (P), such as Enthalpy (H), Entropy (S), Gibbs energy (G), and specific Heat Capacity (Cp), are taken into account. In that case, they can be modelled almost perfectly in the solid or liquid phase using simple quadratic equations (equation 8). Combining these equations with the liquid fraction equation through the logistic algorithm (equation 5) allows the variables to be modelled over the entire temperature range using equation 9 (see Enthalpy in Figure 3a).

$$P_{s\ or\ l} = A + B \times T + C \times T^2 \quad (8)$$

$$P_{global} = P_s + (P_l - P_s).x_l \quad (9)$$

Where P_s and P_l are the thermodynamic variables of interest in solid and liquid states. A, B, and C are fitting constants, x_l is the liquid fraction, and T is the temperature in Celsius.

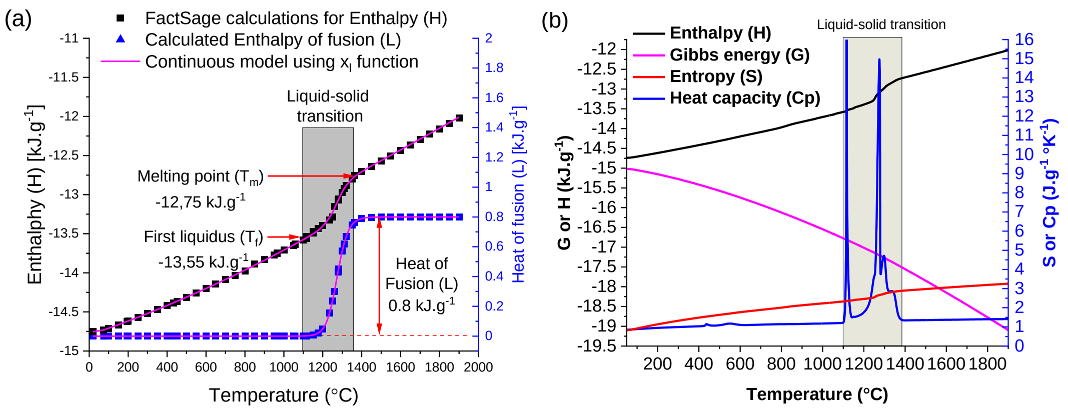

Figure 3. Modelling ESR 2015 thermodynamic variables: (a) Factsage vs equation 9 model for Enthalpy (H) and Heat of Fusion (L); and (b) full set of thermodynamic variables.

Table 2. Calculated coefficient to model main thermodynamic variables of commertial ESR 2015.

Coefficient	x_s[a]	H_s	H_l	S_s	S_l	G	Cp_s	Cp_l	L[b]
Unit	wt%	kJ/g	kJ/g	J/g°K	J/g°K	kJ/g	J/g°K	J/g°K	kJ/g
A	100	-14.8	-14.4	0.7	1.5	-14.9	0.8	0.9	0.8
B	1274	8.5E-4	1.2E-3	2.1E-3	1.2E-3	-9.8E-4	8.2E-4	3.9E-4	
C	45.5	2.3E-7	7.0E-8	-5.7E-7	-1.5E-7	-6.5E-7	-4.6E-7	-7.6E-8	

[a] To be used in equation 5

[b]Heat of fusion: $L = A \times (1 - x_s)$

Considering the performed calculations, the thermodynamic variables can be calculated with the three coefficients (A, B and C) in Table 2. From the calculations, it can be said that to melt ESR 2015, 0.56 kWh/kg is needed, while a process temperature of 1,500 °C will require at least 0.61 kWh/kg.

2.3 Densities, molar volumes and thermal expansion coefficients. Linear regression

Data from three different sources were used for the density calculations. The first is original data from Abkari et Friedrich, 2009 [4], where averages are in Table 3. The second calculation was performed by FactSage, considering the molar volumes of the formed compound at equilibrium during cooling. Finally, the density at room temperature was used as the reference value. The following equations show the linear regressions of the data obtained at high temperatures and those calculated in FactSage.

$$\rho_s = 2.99 - 2.41E^{-5} \times T \;\; \vdots \;\; \rho_l = 3.29 - 5.1E^{-4} \times T \tag{10}$$

Where T is the temperature, ρ_s is the electroflux density in solid state density in g/cm3, and ρ_l is the density in liquid state in g/cm3.

Table 3. Experimental values of electrical conductivity and density at high temperature for Wacker commercial ESR 2015 electroflux [4], [31], [36].

Temperature °C	Conductivity S/cm	Std. dev. %	Temperature °C	Density g/cm3	Molar Volume[a] cm3/mol	Std. dev. %
1,487	0.90	1.8	1,500	2.53	27.47	0.3
1,586	1.11	3.8	1,548	2.50	27.82	0.5
1,686	1.38	3.8	1,600	2.47	28.08	0.5

Density at 20°C measured with He pycnometer= 2.89 g/cm

[a]100 gr of ESR 2015 in molten state is equivalent to 1.44 mol of ionic species (see Table 1) and thus Molar mass (M_m) for the system is 69.50 g/mol. Molar Volume: $M_{vol} = M_m/\rho$

Figure 4a shows these data with an integrated model from equation 9, using the linear regressions of solid and liquid density (equation 10) and liquid fraction (equation 5 using the coefficients from Table 2).

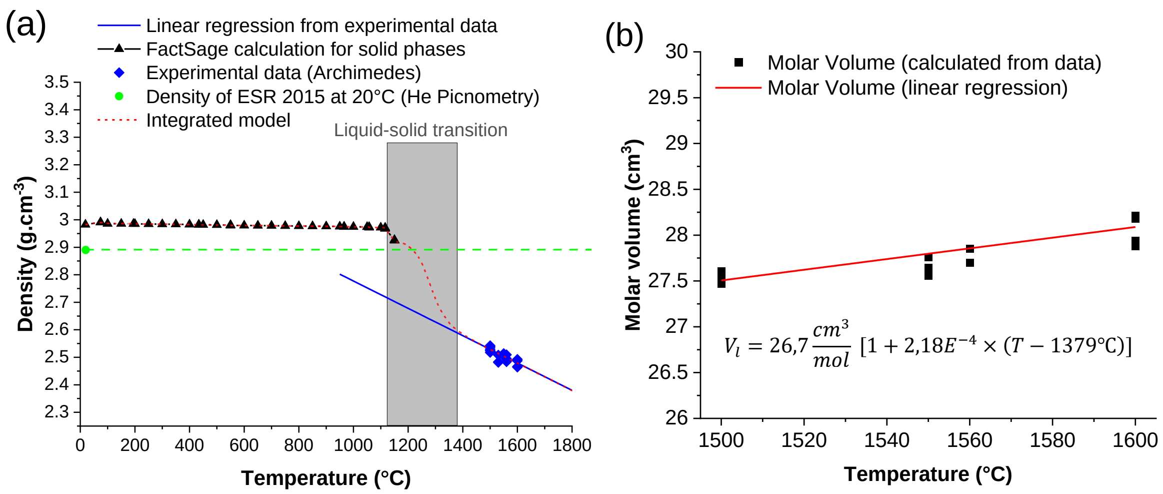

Figure 4. Modelling ESR 2015 thermodynamic variables: (a) Factsage vs equation 9 model for Enthalpy (H) and Heat of Fusion (L); and (b) full set of thermodynamic variables.

The density at room temperature shows a difference of 3.5 % compared to the calculated thermochemically. In real slags, the material rarely cools down too slowly to reach equilibrium, which closes its physical properties to the temperature of its liquid state [35]. Figure 2a shows that the DTA curve increases at temperatures above 1,000 °C, which may indicate that the diffusion mechanisms have been activated within the material to reach its equilibrium state. As with the thermodynamic variables, the use of equation 9 for the calculation of density is an interesting tool for modelling this property over the whole temperature range.

Using the density information and the ion distribution of the slag, volumetric and linear thermal expansion coefficients could be determined by performing a non-linear regression from equation 6 (Figure 4b). These coefficients are 2.18E^{-4} cm^3.cm^{-3}.ºK^{-1} (volumetric) and 7.28E-5 cm.cm^{-1}.ºK^{-1} (linear).

2.4 Electrical conductivity and viscosity

The black line in Figure 5a shows the behaviour of the viscosity calculated in FactSage, taking into account the distribution and nature of the ions. As stated above, this can be described as an exponential function (equation 1), and therefore, its logarithm should give a quasi-linear function (see coefficients in Table 4). If one wants to model the viscosity in the full temperature range between the solid and liquid state, the viscosity calculations show illogical values with the material in the solid state (infinite viscosity=solid). The use of the correction proposed by Roscoe (equation 2), using the solid fraction during cooling (equation 5 and coefficients in Table 2), allows to obtain viscosities more consistent with what should be the real behaviour (red curve in Figure 5a).

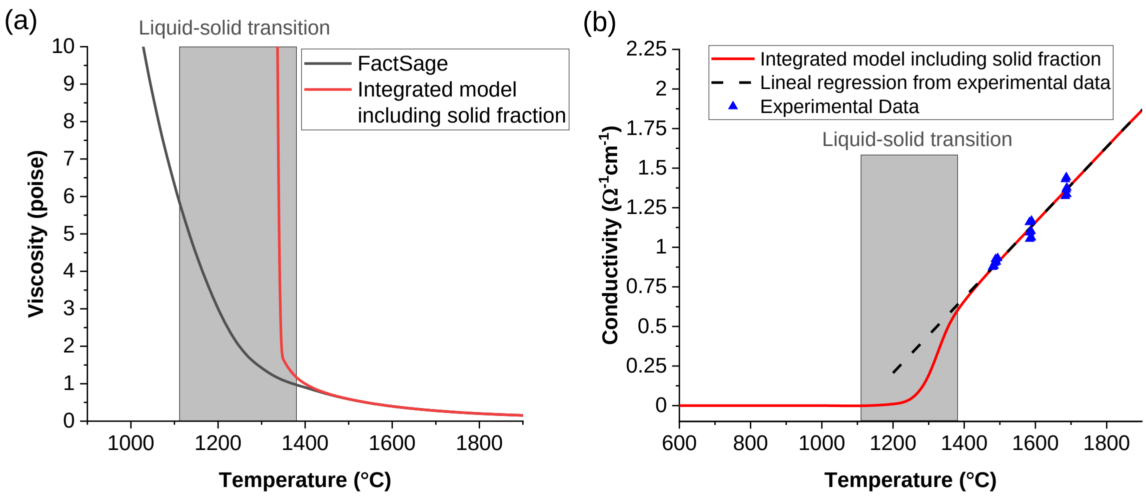

Figure 5. Modelling ESR 2015 thermodynamic variables: (a) Factsage vs equation 9 model for Enthalpy (H) and Heat of Fusion (L); and (b) full set of thermodynamic variables.

Table 4. Regresion coeficient to calculate electrical conductivity and viscosity at temperatures higher than melting point.

Ph. property:	Electrical conductivity [Ω-1.cm-1]			Viscosity [ln(poise)]			
Coeficients	n	m	R^2	A	B	C	R^2
ESR 2015	-2.65	2.38E-3	95.9%	14.7	-1.74E-2	4.33E-6	99.7%

[a]100 gr of ESR 2015 in molten state is equivalent to 1.44 mol of ionic species (see Table 1) and thus Molar mass (M_m) for the system is 69.50 g/mol. Molar Volume: $M_{vol} = M_m/\rho$

Regarding electrical conductivity (σ), there is a wide diversity of calculations made by different scientists over several decades [24,36–38]. Such calculations in high-temperature fluids are complicated and are not a direct method, as they interact with the electrodes, generating a polarisation of the electrodes (capacitance). Modern methods of viscosity calculation, such as those developed by Borowski (2023) [31] and Drude et al. (2024) [37], have shown that at high temperatures, ESR 2015 has an almost linear electrical conductivity (coefficients in Table 4).

There is currently no method to calculate the conductivity during the solid-liquid transition. What is known is that conductivity and viscosity depend on the nature of the ions and the temperature. Therefore, both physical properties are interrelated, and while viscosity tends to infinity, conductivity tends to zero during cooling. If the correction proposed by Roscoe applies to viscosity, the same should hold for electrical conductivity in reverse. Equation 11 describes this new methodology. Figure 5b shows the model integrating the solid fraction (red line) compared to the linear regression of the experimental results (black dashed line). This model shows a behaviour coherent with ESR slags real behaviour.

$$\sigma_c = \frac{\sigma_l}{(1-x_s)^{-n}} \tag{11}$$

Where σ_c is the corrected electroflux electrical conductivity, σ_l is the ideal electrical conductivity, x_s is the solid fraction, and n is an empirical parameter (2.5 in this case) [22].

4. Conclusions

Thermochemical calculations were shown to correlate well with experimental results. Modelling with simple quadratic or linear equations for the physical properties and the basic thermodynamic descriptors of the slag was shown to be effective when combining both quadratic equations (one for solid and one for liquid) through the logistic algorithm describing the solid-liquid transition (equation 5).

Using the solid fraction information through the Roscoe-Einstein equation (equations 2 and 11) allows the transformation of the viscosity and conductivity values into simple and efficient continuum models to be implemented in computational models describing the behaviour of ESR furnaces.

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