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Estimation of Spectral Emissivity and S/Cu Ratio From Emissions of Copper Concentrates at the Flash Smelting Process

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ABSTRACT The project focused on the investigation of new sensing techniques in the copper production industry, specifically in the flash smelting of copper concentrate process. In this paper, we report a direct relationship between the visible and near-infrared emission spectra in the combustion of copper concentrates by changing some operating conditions such as the sulfur–copper and the oxygen ratios provided to the reaction zone. Spectral processing techniques are applied to the measured spectra. The first one aims to separate both continuous radiations mainly associated to incandescent particles and heating walls with discontinuous emissions associated with some emitting atoms and molecules. This goal was carried out using airPLS baseline estimation algorithm. The second processing technique aims to find the continuous emission only associated with the combustion of copper concentrate particles, eliminating the background spectra associated with the smelter walls. This goal was carried out by directly measuring walls emission at operating temperature and in the absence of flame. The most relevant results show that the estimation of the total radiation associated with each measured spectra is an intrinsic parameter of the process that can provide useful information to the operator that supervises the industrial process. It allows estimate quantitatively the sulfur–copper ratio in order to online monitor the mineral characteristics of the copper concentrate that is entering the process. On the other hand, the approach of a first prototype of the emissivity model for the copper concentrate particles, which validated with measurements, becomes a promising tool that will allow increasing the development of optoelectronic applications around this industry process.

INDEX TERMS Copper, signal processing, smelting, spectral analysis, spectrophotometers, temperature.

I. INTRODUCTION

Flash smelting of copper concentrate is a process found in the primary copper production industry. Since the first furnaces manufactured by Outokumpu, the process operation and control have been mainly performed by operators based on previous empirical knowledge or by offline predictions based on mass and energy balance [1]. Therefore, there is an increasing interest in developing sensing techniques or instrumentation aimed to monitor and/or control this process.

In this context, we have been applying sensing methods for measuring the flame radiation emitted during the combustion of copper concentrates in laboratory scale setups and in industrial facilities. Such as was reported in [2], we have corroborated that spectra are composed by discontinuous spectral features over a continuous baseline. In Fig. 1 it is shown a typical flame spectrum in the visible and near infrared (VIS-NIR) bands emitted by the combustion of copper concentrates. Several discontinuous emissions can be directly identified, like Na, K and possible Cu_xO or FeO emissions at 588.9nm, 616.13nm, 606nm, 767nm, 779.1nm and 793.9nm respectively, with different intensities and spectral widths. All these narrowband features are added over a continuous broadband baseline, which is typically related to incandescent particles at high temperature [3], [4]. Both, continuous and discontinuous patterns are representative of

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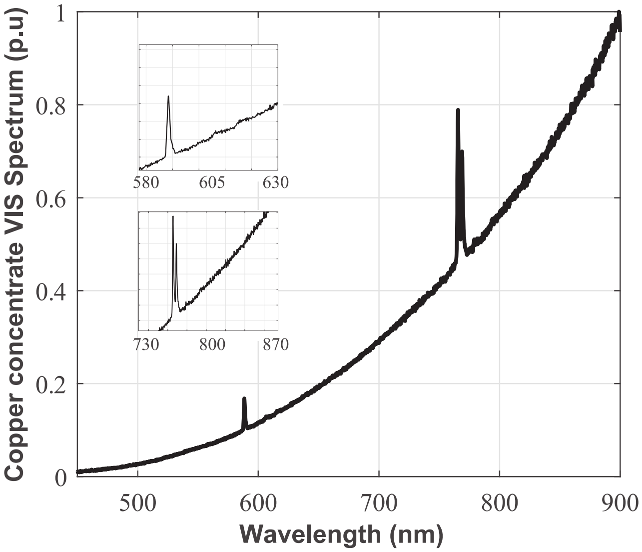

FIGURE 1. Typical flame spectra (normalized) of copper concentrate acquired during flash smelting in an industry facility.

the combustion process. Then, to carry out a proper analysis and spectral characterization, it becomes relevant to separate both, continuous baseline from the discontinuous emissions. From an exploratory analysis described in [2], a direct relationship between the S/Cu ratio, the percentage of oxygen and total radiation measure were found. Previous studies have also shown that VIS-NIR emission spectra (continuous and discontinuous features) increase when copper concentrates have greater sulfur content, producing highly exothermic reactions when sulfur reacts with oxygen to produce $SO_{2(g)}$ [5]–[7], and therefore it stimulates the radiative emission processes. In this work, we advance in the aforementioned combustion processes understanding by introducing new analytical methodologies.

Data acquisition associated with the combustion process is currently an open research field aiming to increase the performance of their processes and to reduce the emission of pollutants [8]. The design of new sensors and the implementation of techniques allows to carry out the monitoring and control of process variables. The development of optoelectronics, and in particular of spectroscopic techniques, allows measuring the spectral emission of flames using non-invasive methods. These techniques allow delivering information such as flame stability, oxygen concentration, pollutants emission, flame geometry, released energy, elemental and molecular composition, and their relation with operational variables like temperature, pressure, or feeding rate [9].

Thus, in this paper we propose a monitoring technique to characterize the copper concentrates that enters the flash smelting process in a laboratory combustion setup and in an industrial facility, such a technique is based on empirical and physical models developed in this work. The models consider physical assumptions, e.g. particles emissivity, S/Cu ratio of copper concentrate and the spectral behavior of particles during combustion to perform predictions. Linear and non-linear least squares regression methods are applied to preprocess spectra and to fit models parameters. This information, together with flame temperature retrieved from spectral data, will help flash smelter operators to set optimal process variables during furnace operation. Models performance are evaluated with standard error metrics such as the RMSE (Root Mean Square Error) value and the GFC (Goodness of fit coefficient) [10].

VIS-NIR spectroscopy techniques are used in this work for signal acquisition as described in the methodology section. Moreover, signal preprocessing methods are applied to separate the discontinuous spectra from baseline spectral features. In this work, the airPLS (Adaptative iteratively reweighted penalized least squares) baseline correction method is implemented for such a purpose [11]. Once discontinuous spectra are separated, and background emissions from heating walls are eliminated using a linear treatment and least square methods, models are proposed to relate the spectral measurements with the operating variables of the combustion process. Emissivity model is proposed according to: particles size, assessed with mineralogical analysis; feeding rate; optical path and wavelength bands according to considerations taken from Bourger's law, and the results of Godridge and Hammont for the solution of Mie's scattering equation [12].

We begin by introducing the dataset and the signal processing performed to have data representative of each operating condition. In Section II, the airPLS baseline method is described. Then, the separation of the background from the real spectrum of the flame is carred out by using a linear model. Also, exploratory analysis is presented with the proposed model to estimate the S/Cu ratio. In Section III, experimental validation results are shown and finally, our conclusions are drawn.

II. METHODOLOGY

Preprocessing and post-processing methods are discussed in this section together with the measurement techniques and experimental design.

A. SYSTEM DESCRIPTION

A flash furnace laboratory prototype (droptube) was setup to conduct combustion experiments at the Metallurgical Engineering Department, University of Concepcion. A scheme depicting the test bench is shown in Fig. 2, and Table 1 describe its main components.

The copper concentrate fed into the furnace flows with an average rate of 5.7 [g/min]. The concentrate particles fall to the reaction zone inside the droptube, where they combust, releasing large amounts of energy and thus raising the combustion chamber temperature. The feed spear is a structure designed to refrigerate the optical probe by means of a water jacket and to facilitate the entrance of nitrogen and oxygen into the reaction zone. Gasses flows are controlled and monitored by using mass flow controllers, while reaction zone temperature is monitored by the furnace controller. A suction system pulls out the gases from

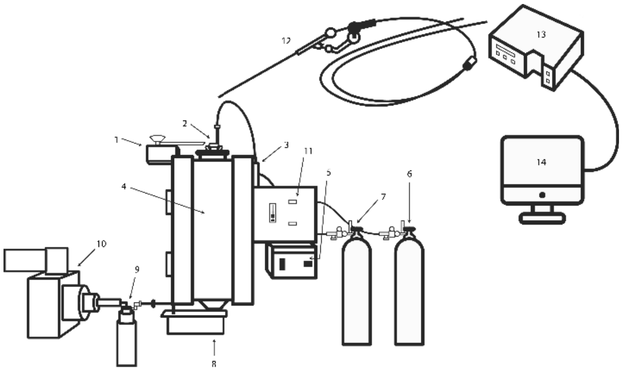

FIGURE 2. Experimental setup scheme for copper concentrate combustion experiments.

TABLE 1. Setup description.

#	Label	#	Label
1	Feeder	8	Manifold
2	Feed spear	9	Dust recovery
3	Gas Mixer	10	Gas suction
4	Furnace	11	Flowmeters
5	Electric Controller	12	High temperature probe
6	N_2	13	Spectrophotometer
7	O_2	14	Laptop

inside the furnace, then they go through a scrubber system which extracts part of the powders generated in the process. The flame spectral emission produced inside the reaction zone is guided to a calibrated VIS-NIR spectrophotometer (USB4000, Ocean Optics) through a high temperature optical fiber (Avantes Inc.), specially adapted for operations in harsh environments. Spectral data acquisition was performed with a graphical user interface developed in LabView^{TM} (National Instruments), further signal processing and analysis were performed in Matlab®.

According to the availability of minerals and operating capacity of the droptube, the copper concentrates coming from Las Tórtolas smelter facilities (located in the Metropolitan region, Chile, 56 km from Santiago de Chile) were used for the combustion experiments. Previous mineralogical analysis of these concentrates depicted three different concentration S/Cu ratios: 1.79 (High ratio), 1.21 (Medium ratio) and 1.19 (Low ratio), with particle sizes about 50 ± 20 microns. The concentrates are composed mainly of three mineralogical phases: pyrite, chalcopyrite and bornite in different proportions, which produce changes in the total composition of sulfur and copper. Experiments were conducted under five different oxygen ratio conditions (30%, 50%, 60%, 70% and 80%).

B. SPECTRAL ACQUISITION AND PRACTICAL ISSUES

When droptube walls are heated by the furnace electrical resistances to raise the reaction zone temperature, it causes the walls to emit radiation. This effect produces a background in the spectral measurements that depends on the setting operating furnace temperature. Therefore, it is necessary to subtract this background emission on the measured spectra so only the continuous spectra related to incandescent particles on the flame can be analyzed [4]. Thus, a first step to carry out this treatment, a first step is to separate the discontinuous emissions from measured spectra, by computing airPLS algorithm. Later, through a curve fitting procedure (linear model) and by assuming the background emission from the walls as an additive process, the true radiance emitted by the flame can be retrieved.

C. DISCONTINUOS SPECTRA SEPARATION

Measured spectra have both, a continuous part that follows the Planck's radiation behavior, and discontinuous features that are related to the emission from atoms and molecules present in the concentrate, Fig. 1. In order to make feasible to separately analyze both spectral patterns, we have applied a classical and effective baseline correction algorithm.

Due to spectral patterns present throughout the combustion process, the most effective algorithm is airPLS [11], since it does not require *a-priori* information for the baseline estimation, it has a low computational cost, and it delivers small estimation errors [5].

This method generates a flexible smoothing but balanced between the fidelity of the original data and the roughness of the adjusted data. Considering E_c, like an adjusted vector, the next equation represents the balance between the fidelity and smoothness:

$$Q = F + \sigma R = ||y - E_c||^2 + \sigma\, ||DE_c||^2\,, \tag{1}$$

where Q is the result of equilibrium, F is the fidelity expressed as the sum of squares errors between y (original data) and E_c, R is the roughness calculated as the first derivative of E_c, σ is a parameter to control the smoothness of the adjusted vector and D is the derivative of the identity matrix, such that $DE_c = E_c$. When finding the derivative vector, partial and equal to 0, the solution of Eq. (1) minimization is:

$$E_c^{'} = \left(I + \sigma D^{'} D\right)^{-1} y^{'}. \tag{2}$$

The vector of weights W, is introduced as follow:

$$E_c^{'} = \left(W + \sigma D^{'} D\right)^{-1} Wy^{'}. \tag{3}$$

Then, the adaptive iterative procedure is carried out by calculating the weights and adding a penalty element to control the smoothness of the adjusted baseline. Each k-iteration of the adaptive procedure, re-weighted iteratively, implies to solve a weighted problem of least squares penalized in the following way:

$$Q^k = \sum\nolimits_{i=1}^{n} w_i^k \left(y_i - E_{c_i}^k\right)^2 + \sigma \sum\nolimits_{j=2}^{n} \left(E_{c_j}^k - E_{c_{j-1}}^k\right)^2. \tag{4}$$

A value $w_0 = 1$ is considered as an initial step. After initialization, the w of each iterative step k can be obtained using the following expressions:

$$w_i^k = \begin{cases} 0, & y_i \geq E_{c_i}^{k-1} \\ e^{\frac{t\left(y_i - E_{c_i}^{k-1}\right)}{|d^k|}}, & y_i < E_{c_i}^{k-1}, \end{cases} \tag{5}$$

where the vector d^k consists of negative elements of differences between y and E_c^{k-1}. If d^k contains elements negative in the previous iteration, the value of E_c^{k-1} in that iteration is a candidate for the baseline. If the value of the *i-th* point is greater than the candidate for baseline, it can be considered as part of a peak. Therefore, its weight is established as zero to ignore this point in the next iteration.

The iteration will stop with the defined maximum number of iterations or when the detention criterion is reached, defined by:

$$d^k < 0.001 \sum\nolimits_{i=1}^{n} |y_i|. \tag{6}$$

The separation of both spectral patterns from the measurements allows us to work with the part of the spectrum that only has a relation with the emission energy of flame generated by the heating particles [11].

D. BACKGROUND SEPARATION AND FEATURES EXTRACTION

As described above, the droptube furnace used for the combustion of concentrates has electric resistances that heat up the walls and reaction zone between 800°C up to 1000°C, causing them to emit background radiation that manifests itself behind the radiation of the combustion of concentrates taking place inside the furnace. After the baseline extraction procedure is applied, let's consider the measured calibrated continuum component (E_c) that can be represented as the linear combination of the flame spectra (E_f) and the background spectra (E_b), each one weighted by coefficients α_1 and α_2, respectively [13]:

$$E_c(\lambda) = \alpha_1 E_f(\lambda, T) + \alpha_2 E_b(\lambda). \tag{7}$$

Assuming that the background is known and invariant, it is possible to obtain an estimation of the real flame spectrum [10]. As a first step, a simulation of E_f is performed by using Planck's radiation law and a temperature value estimated with the two color pyrometry, TCP, method [2], then constants α_1 and α_2 involved in the linear combination are estimated with a least squares linear regression procedure implemented in Matlab® with the Levenverg-Marquardt algorithm. Finally, E_f is estimated using the retrieved constants and the residuals. After this process, a representative data set from the process is obtained without the influence of furnace walls.

Then, the total radiation, TR, is obtained by integrating the corrected spectra over all the analyzed spectral band, from 400 nm to 900 nm in this work. This feature together with the estimated temperature are used to fit the parameters of functional and physical proposed models.

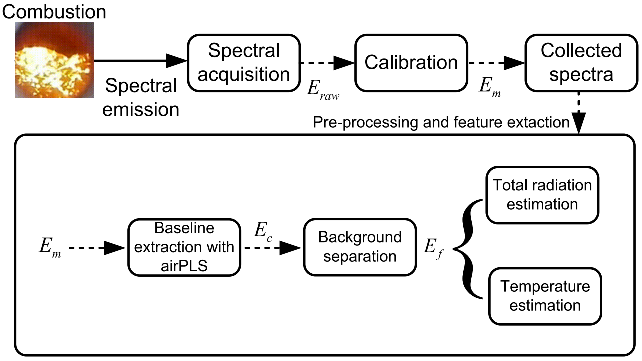

FIGURE 3. Spectral signals acquisition, pre-processing and feature extraction steps.

Finally, Fig. 3 summarizes the proposed pipelines to perform signals acquisition, pre-processing and feature extraction steps.

E. EMISSIVITY MODEL

The spectral radiation intensity $E_f(\lambda)$ is attenuated exponentially when travels through an absorbing medium. Bourger's law [14] define this physical phenomenon according to the Eq. (8).

$$E_f(\lambda) = E_0(\lambda) \exp(-K_\lambda L), \tag{8}$$

where $E_f(\lambda)$ is the flame spectrum, $E_0(\lambda)$ the spectral radiation in the optical path origin, K_λ the extinction coefficient of particles cloud and L the optical path length.

The extinction coefficient K_λ is equal to the dispersed fraction σ_λ plus the absorbed fraction A_λ. Mie's theory is one of the most used in the evaluation of the total extinction of carbon particles [15], [16] and provides a complete analytical solution to the Maxwell equations for the dispersion of electromagnetic radiation by spherical particles. The solution of Mie's equation is given in terms of the ratio between the average particle perimeter D and the measurement wavelength ($x = D\pi/\lambda$), and a complex refractive index of the particle. According to the results of Godridge and Hammont [12], scattering can be neglected for small particles of soot found in combustion. However, large soot agglomerates coupled with low wavelength ranges lead to scattering levels of up to 50% of the total extinction of soot [14]. In the Rayleigh limit of the Mie's theory ($x \ll 1$) scattering is neglected, so the absorption coefficient K_λ is given as the Eq. (9).

$$A_\lambda = \frac{36 n_\lambda k_\lambda \left(\frac{\pi}{\lambda}\right) f_v}{\left(n_\lambda^2 - k_\lambda^2 + 2\right) + 4 n_\lambda^2 k_\lambda^2} = F(\lambda) f_v, \tag{9}$$

where n_λ and k_λ are the real and imaginary components of the complex refractive index for the VIS and NIR wavelength range. f_v is the particle volume fraction and is calculated as the Eq. (10).

$$f_v = \int_0^\infty \frac{4}{3}\pi r^3 N(r)\, dr, \tag{10}$$

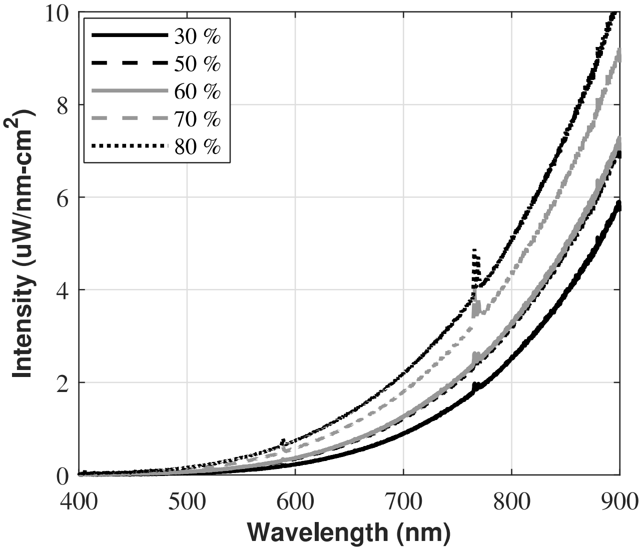

FIGURE 4. Spectra set measured in droptube furnace at 1000°C, high S/Cu and different oxygen enrichment conditions.

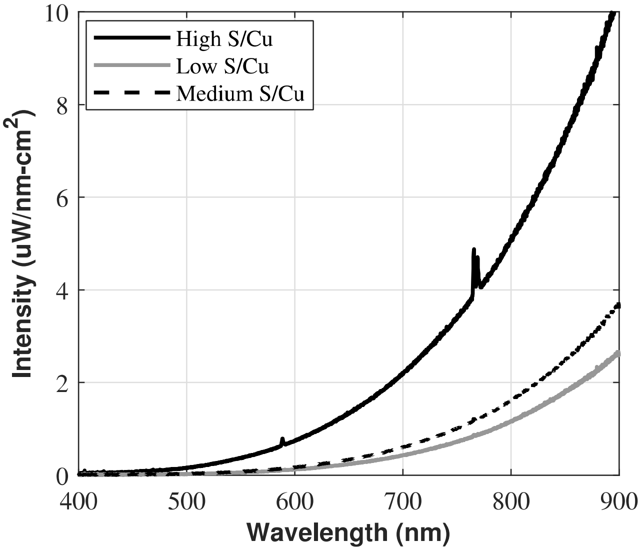

FIGURE 5. Spectra set measured in droptube furnace at 1000°C, 80% of oxygen enrichment and different S/Cu conditions.

$N(r)$ is the particle size distribution for particles of mean ratio r. To determine the emissivity in a luminous flame, one assumes the Kirchoff's law, as follows:

$$\epsilon_\lambda = A_\lambda = 1 - \exp(-F(\lambda) f_v L). \tag{11}$$

The Eq. (11) can be rewritten according to the calculation of the complex refractive index considering $n_\lambda = 1.564$ and $k_\lambda = 0.46i$ [17], resulting:

$$\epsilon_\lambda = 1 - \exp(-\frac{6.2 f_v L}{\lambda}). \tag{12}$$

A usual size distribution [15] allows that Eq. (10) can be rewritten as Eq. (13):

$$f_v = \frac{5}{27}\pi D_m^3 N_t, \tag{13}$$

where D_m is the most probable diameter for the particles, and N_t the total number of particles in an experiment. In this way, Eq. 12 is proposed as a prototype that allows modeling the emissivity of an incandescent cloud of copper concentrate particles, considering the dimensions of individual particles, the feeding rate and the distance between the particles and the optical fiber tip.

III. RESULT AND ANALYSIS

The hypothesis about the direct relationship between the oxygen ratio present in the reaction zone and measured spectra can be observed in Fig. 4 for a high S/Cu condition and different oxygen enrichment. A direct relation is depicted for the different S/Cu ratios in Fig 5 for spectra acquired at and 80% of oxygen condition. Also, note that depicted spectra are the average for each combustion experiment. Over these spectra at different operating conditions explained in the system description subsection, the signal preprocessing methods are computed.

TABLE 2. GFC values for airpls algorithm.

Oxygen %	GFC MEAN	GFC VARIANCE
30	0.9963	0.0021
50	0.9839	0.0013
60	0.9994	0.0003
70	0.9996	0.0002
80	0.9997	0.0002

To assess the performance of airPLS algorithm over copper concentrates spectra, the GFC metric was calculated over the continuous part between the calibrated measurement, E_m, and baseline estimated by airPLS, E_c. GFC is based on the Schwartz's inequality and defined as:

$$GFC = \frac{|\sum_{i=1}^{N} E_m(\lambda_i) \cdot E_c(\lambda_i)|}{(\sum_{i=1}^{N} [E_m(\lambda_i)]^2 \cdot \sum_{i=1}^{N} [E_c(\lambda_i)]^2)^{1/2}}, \tag{14}$$

where an accurate estimation requires a GFC $>$ 0.995 and a GFC $>$ 0.999 means that quite good spectral matches are achieved. In order to apply airPLS, the following parameters were set: $\sigma = 10^7$; proportional weights (at both, the start and end of the baseline) equal to 0.5; and a maximum number of iterations of 50. For instance, when applied to spectra measured from combustion experiments for different oxygen condition, GFC values are obtained and shown in Table 2, it can be seen that the baseline extraction is highly accurate with an average GFC of 0.9958 and low variance for all cases. The airPLS performance for the average spectra at each oxygen condition is shown in Fig. 6.

Once discontinuous part of the spectra is separated, it was applied the background separation method (Eq. (7)) over entire spectra to separate the background radiation from the flame radiation emitted by the cloud of incandescent particles during combustion. To carry out this goal, the E_b was directly

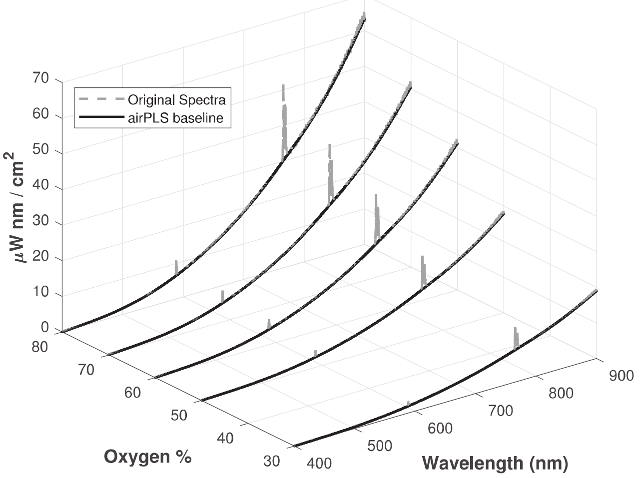

FIGURE 6. Set of spectra measured at different % oxygen for 1000°C for the droptube controller temperature applying airPLS.

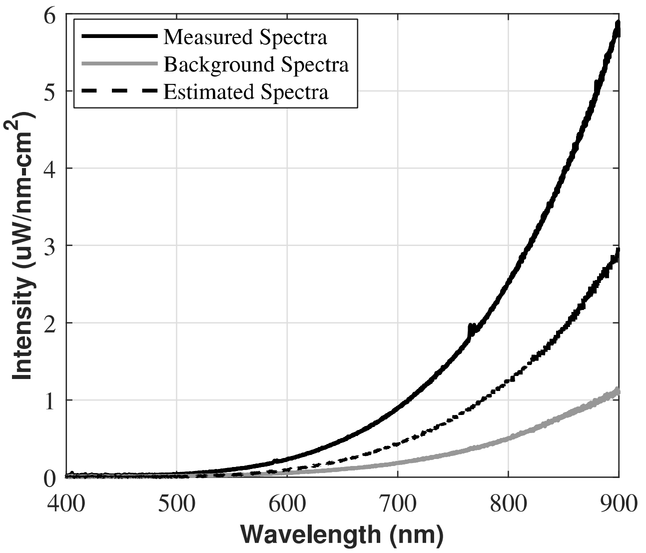

FIGURE 7. Example of background separation using the Eq. (7) for measured spectra at 50% oxygen and medium S/Cu ratio.

measured from the furnace walls set at an operating value of 1000° C, in absence of flame. In Fig. 7 it is shown the retrieved flame spectra for an enrichment atmosphere of O_2 at 50% and medium S/Cu ratio.

By having the spectra attributed to the process under different combustion operating conditions, the total radiation, TR, is estimated and presented in Fig. 8. From this analysis, it is possible to infer that there is a relationship between the total radiation and the oxygen present in the combustion process at different copper concentrates S/Cu ratios. A no linear model is proposed to fit a representative curve to the data in Fig. 8.

From an exploratory analysis, the function that best follows the trends in our data has an exponential structure of the form $TR = a \cdot \exp(b \cdot \mathrm{S/Cu})$, a and b are estimated as a function of oxygen percentage present in the combustion zone, according to the following models:

$$\sum\nolimits_{i=0}^{2} k_i x^i = a, \quad k_i = [-27.18 \;\; 1.982 \quad -0.01736] \tag{15}$$

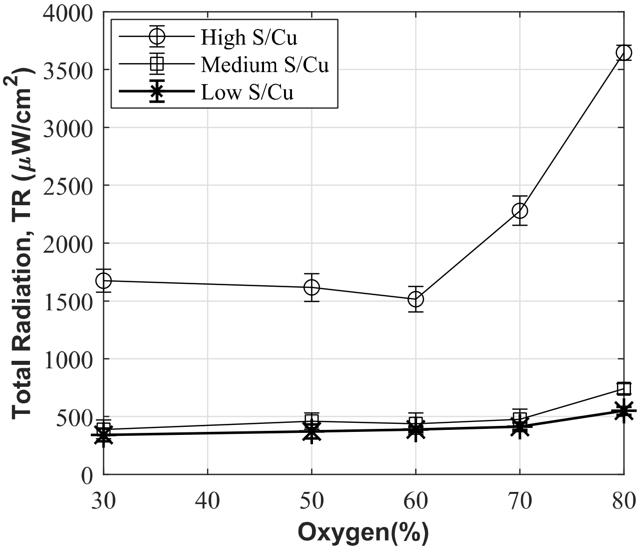

FIGURE 8. Total radiation vs Oxygen % for different S/Cu ratios.

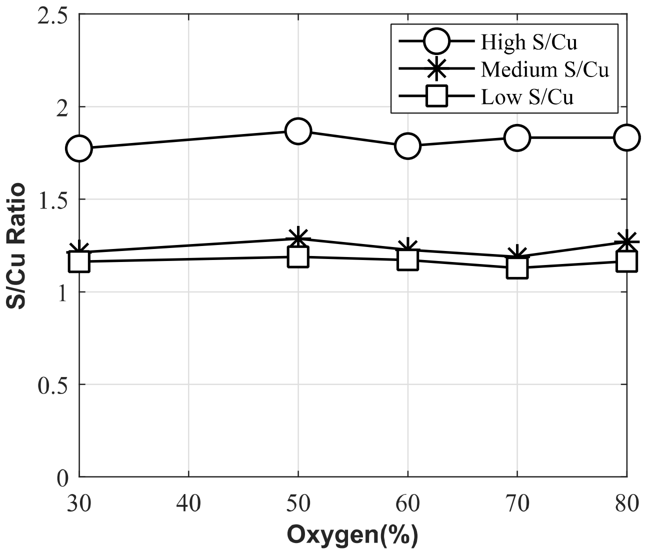

FIGURE 9. S/Cu ratio estimation under different furnace operating conditions.

$$\sum\nolimits_{i=0}^{2} l_i x^i = b, \quad l_i = [4.581 \quad 0.09261 \quad 0.0008846], \tag{16}$$

where x is the oxygen percentage of the furnace atmosphere and k_i and l_i are constants estimated from the spectral data with a non-linear curve fitting procedure. Then, it is possible to make an estimation of S/Cu ratio as follows:

$$S/Cu = \frac{\ln(\frac{TR}{a})}{b}. \tag{17}$$

Estimation results for different copper concentrates are depicted in Fig. 9.

The estimations of S/Cu ratios depicted in Fig. 9 give RMSE values of 0.0441, 0.0198 and 0.0379 for the reference S/Cu ratio values of 1.79, 1.21 and 1.19 respectively, which makes the model a good alternative for future predictions.

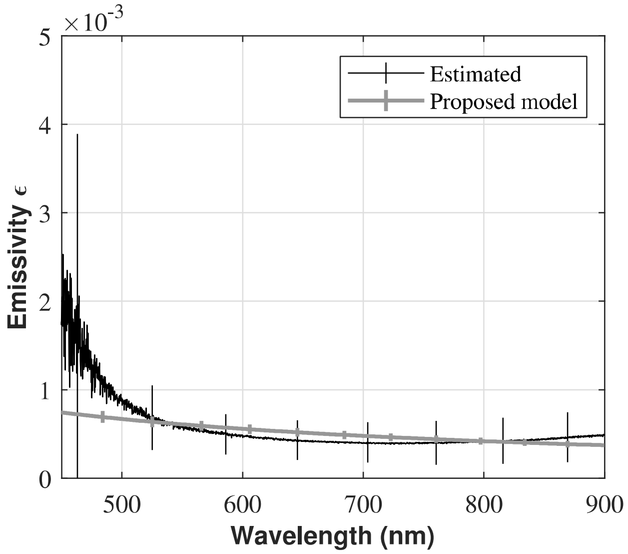

FIGURE 10. Estimated emissivity from data vs proposed method.

On other hand, from the pre-processed spectra, the lame temperature is estimated using the TCP method, using 650 nm and 750 nm as the sample wavelengths [2], [18], [19] and a simulated spectrum is generated by Planck's law at the estimated temperature of 1430°C. In this way, by dividing a real measured flame spectrum with respect to the blackbody spectrum associated with that temperature, an estimate of the emissivity is obtained. The estimated emissivity curve from a spectral sample can be observed in Fig. 10.

The emissivity was also estimated with the model in Eq. (12), f_v was estimated using Eq. (13), a D_m of 50 ± 10 microns according to the previous mineralogical analysis, and a number of particles of $N_t = 1800 \pm 300$ for each integration time, estimated by assuming a constant feeding rate for an experiment. An optical path of $L = 15\ cm$ is considered in Eq. (12), in order to the distance between the optical fiber and the center of the particles cloud. The estimated emissivity using the proposed model can be also seen in Fig. 10. Both graphs are displayed with their variance according to the existing changes in the time at which the measurement is made. Thus, Eq. (12) proposed for the emissivity of copper concentrate particles combustion could be used to improve temperature estimation models implemented with optical techniques and in numerical simulations in the field of material and metallurgical sciences.

Finally, a set of measured spectra from Chagres smelter (Chile) was analyzed, from which it is unknown the S/Cu ratio and mineralogy. Since the intensity of flame spectra was much greater than the laboratory tests (Fig. 11) mainly due to the great number of combusted particles of copper concentrates, it is assumed that walls emission of the furnace have no major influence on the radiation captured by spectrophotometer.

According to data provided by the plant operators, it is assumed that the furnace maintains a constant oxygen enrichment, set to 40 % in the reaction zone. In Fig. 12 is depicted the S/Cu ratio estimated from the industry spectra data measured in Chagres smelter, where the average ratio is 1.63, very close to values found in the industry in concentrates with high pyrite content [20].

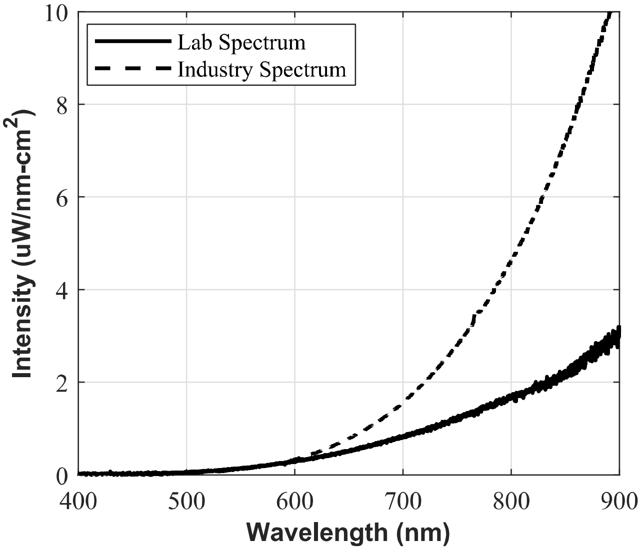

FIGURE 11. Emission spectra measured in laboratory vs industry.

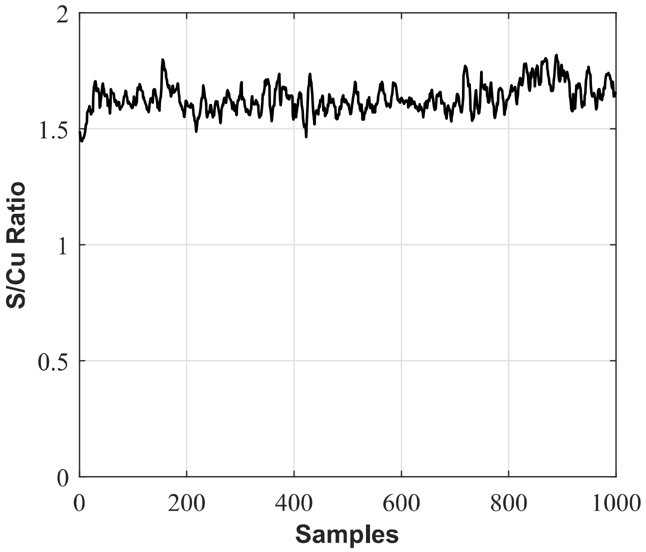

FIGURE 12. S/Cu ratio estimation from Chagres smelter spectral data set.

In future work, deeply considerations will be included in order to generalize the proposed models and procedures, such as performing combustion experiments over a greater number of copper concentrates samples and minerals species present in these processes.

IV. CONCLUSIONS

In this work, calibrated spectral measurements from laboratory scale setup at different operating conditions as well as from an industrial copper flash smelting furnace are reported. The measurements showed that spectral emission from combustion of copper concentrates are composed of continuous and discontinuous features, all related to the type of concentrates, mineralogical compositions, and operating conditions. Due to continuous spectra exhibit a direct relation with sulfur-copper and oxygen ratios, the airPLS algorithm was applied over spectra to separate both patterns. The airPLS algorithm provided a good performance aimed to separate both, continuous and discontinuous patterns, exhibiting an excellent average of GFC metric. Then, and to avoid background spectral effects from heating walls of the laboratory facilities, a simple but effective linear model was applied over measured spectra. Thus, it was possible to relate the total radiation calculated from continuous emission spectra, with the operating conditions of the process, enabling to online estimate the sulfur-copper ratio of the copper concentrate that is entering the reaction zone. On the other hand, the proposed model of emissivity depending on the physical characteristics of the copper concentrate particles and the feed rate towards the furnace faithfully describes the real emission compared with an estimated emissivity using flame spectra and blackbody radiation at the same temperature. All these results represent a great advance in the generation of knowledge enabling to continually developing new techniques of spectral measurement and process analysis.

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