

The use of diffuse reflectance spectroscopy for the characterization of iron ores

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ABSTRACT

The aim of this work was to develop a diffuse reflectance methodology for quantifying minerals in powdered iron ores, which is a key quality control requirement for these materials. Selected samples ranging widely in their concentrations of hematite (as specularite and martite), goethite, magnetite, and quartz were collected in mines from the Iron Quadrangle, Minas Gerais State, and also in the Carajás region, Pará State, Brazil. A chemometric analysis based on the concentrations of the different minerals as determined with a combination of conventional methods (chemical analysis, X-ray diffraction, Mössbauer spectroscopy, light-reflected microscopy, and magnetic susceptibility) and the principal components derived from the diffuse reflectance spectra in the visible range was performed. Principal component regression analysis provided successful calibration for the concentrations of goethite ($r^2 = 0.94$; standard error of validation (SEv) = 4.2%) and hematite ($r^2 = 0.89$; SEv = 7.4%), in addition to good estimates for quartz ($r^2 = 0.83$; SEv = 7.4%), specularite ($r^2 = 0.80$; SEv = 11.6%), and martite ($r^2 = 0.78$; SEv = 10.6%). Our results suggest that diffuse reflectance spectroscopy is a promising tool for the simultaneous determination of minerals in iron ores within a few minutes only.

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1. Introduction

The steel industry requires accurate quantification of the main iron (Fe) oxides and oxyhydroxides present in iron ores, which entails determining goethite, magnetite and hematite present in ore concentrates, and also the distribution of hematite into martite and specularite. Quantifying quartz, which is invariably present in the ores, is also essential for various processes, particularly the flotation step (Santos and Brandão, 2003; Santos et al., 2005).

In recent years, optical microscopy has become the standard technique for quantifying the minerals in iron ores. An experienced operator can easily identify all the previous mentioned phases by reflected light with an absolute uncertainty of roughly 5–10%. Quantification is done by counting a total of at least 500 particles and then converting the volumetric percentage into weight by using the density of each phase (Donskoi et al., 2007; Ferreira, 1993). However, this technique is time-consuming and takes from 30 to 180 min depending on the particular size fractions studied. Furthermore, its results may vary with the sample preparation procedure, operator, and absolute mineral densities. Thus, densities of goethite, specularite, martite and magnetite are dependent on var-

ious factors such as porosity, and degree of oxidation and hydration (Donskoi et al., 2008). Also, there is no alternative technique to cross-check the results for martite and specularite, which precludes the suppression of systematic errors.

One other analytical technique suitable for studying of iron oxides (a term used here to designate all Fe oxides, oxyhydroxides and hydroxides) and other iron-bearing phases is Mössbauer spectroscopy (Bancroft, 1973). Identification of the iron-oxide phases relies on comparing the derived hyperfine parameters with those for well-characterized samples reported in the literature, and their quantification is based on the relative areas for each mineral. The main advantage of this technique over optical microscopy is that the densities of the involved phases need not be known. However, each spectrum takes 5–12 h of measuring time, which makes this technique unsuitable for routine analyses. Some comparative studies have exposed a relatively good consistency between both methods (de Sá et al., 1999; Toríbio et al., 2001) and also that the room-temperature Mössbauer spectra for samples containing variable proportions of martite and specularite are essentially identical.

X-ray diffraction (XRD) analysis is widely used for the identification of crystalline substances, but rarely used for quantification of the identified phases owing to the potential influence of texture, stress and crystal size, among other factors, on peak intensity (Jenkins and Snyder, 1996). The Rietveld method can be used to quantify crystalline phases, but the operational procedure is far from

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straightforward for samples exhibiting textural effects (Rietveld, 1969; Pöllmann and Angélica, 2002). In addition, the structural parameters for martite and specularite are identical, which precludes their discrimination. In spite of these shortcomings, XRD research work on some preliminarily well-characterized iron-ore samples revealed that magnetite, goethite, martite and specularite can be effectively quantified by combining the results of chemical and X-ray diffraction analyses (da Costa et al., 2002).

Diffuse reflectance spectroscopy has previously been used for the identification of Fe oxides in soils and sediments (Torrent and Barrón, 1993; and references therein). Iron oxides are important soil chromophores exhibiting absorption bands in the visible spectral range (Sherman and Waite, 1985). However, overlap and weakness in some bands requires the use of derivative transformations of the raw diffuse reflectance spectrum in order to quantify hematite and goethite (Torrent and Barrón, 2008). Chemometric methods have been successfully applied to visible and near-infrared spectra in many agricultural fields, particularly in the study of soils, where iron oxides and other components can thus be accurately identified and quantified (Ben-Dor and Banin, 1990; Reeves et al., 1999; Malley et al., 2004; Viscarra-Rossel et al., 2006). Equipment for in-line mineral quantification based on diffuse reflectance spectroscopy was recently made commercially available (de Waal, 2007). To the best of our knowledge, however, diffuse reflectance has never previously been used to quantify minerals in iron ores. This led us to investigate the use of this technique for quantifying the major phases in various iron ores. As shown below, the results were very promising.

2. Experimental

Eighty-four iron-ore samples containing variable proportions of martite, specularite, magnetite, goethite and quartz were obtained from different locations in Brazil. Most were collected in mines from the well-known Iron Quadrangle, Minas Gerais State (Rosière and Chemale, 2000). Other samples were collected in the Carajás region, Pará State (Rosière and Chemale 2000). One sample containing 92% specularite, 1% magnetite and 7% quartz, and another one containing 83% martite, 14% magnetite and 1% quartz, were mixed in different proportions in order to obtain 11 samples spanning the whole composition range at intervals of about 10% in terms of specularite–martite. Samples were sieved and the fractions below 100 mesh chosen for analysis.

Mössbauer spectra (MS) were acquired at room-temperature with a spectrometer using a constant-acceleration drive with triangular reference signal, 512 or 1024 channels and velocities over the range -11 to $+11$ mm/s. Velocity calibration was achieved by using the spectrum for a standard α -Fe foil at room temperature, and isomer shifts are quoted relative to α -Fe. Spectra were fitted either with discrete Lorentzian sextets/doublets or with one model-independent hyperfine-field distributions (Vandenberghe et al., 1994). Relative areas were calculated on the assumption that ferrous and ferric ions have the same recoil fraction. The amount of each iron-bearing phase was calculated from its relative area and the total iron content as obtained by chemical analysis.

A reflected light microscope (Leica, model MPS30) at $200\times$ magnification was used to count particles. The powdered samples were mounted in epoxy resin blocks which were then polished. At least 500 particles were identified and counted by reflected light. Volumetric percentages were converted into weight percentages by using the following average densities: specularite = 5.2 g cm^{-3} ; martite = 3.9 g cm^{-3} ; goethite = 3.0 g cm^{-3} ; magnetite = 5.0 g cm^{-3} ; quartz = 2.65 g cm^{-3} .

Powder XRD patterns were obtained on a Shimadzu XRD 6000 instrument fitted with an Fe tube and a graphite monochromator.

The main purpose of these measurements was to check for the absence of any spurious phases such as kaolinite or micas. None of the samples, however, exhibited any such phases.

Total and ferrous iron were determined by titration with potassium dichromate after dissolution in hot concentrated HCl. The residues of the attacks were weighted and assumed to be, based on the X-ray diffraction results, quartz.

Magnetic susceptibilities were measured with a Bartington MS-2B dual frequency sensor (Bartington Instruments Ltd., Oxford, UK). The magnetite concentration was calculated by using a magnetic susceptibility value of $500 \times 10^{-6} \text{ m}^3 \text{ kg}^{-1}$ (Dearing, 1999).

Diffuse reflectance measurements were made on powder samples sieved below 100 mesh and placed in a sample holder with a quartz window 17 mm in diameter. In order to investigate the particle-size effect on the reflectance spectra, we also obtained the spectra for micaceous hematite ranging from $5 \mu\text{m}$ to $500 \mu\text{m}$ in particle size. No sample preparation (e.g. cleaning) was required. The spectrum for a white standard (Halon, polytetrafluoroethylene powder) was periodically obtained in order to calibrate the equipment. Spectra were recorded on a Cary 5000 Vis-IR spectrophotometer (Varian Inc., Palo Alto, CA) equipped with an integrating sphere of 110 mm. Reflectance values were acquired at 1 nm intervals over the 320–800 nm range. Because four replicates were recorded, about 10 min per sample were required. The visible portion of the spectrum was used to calculate color parameters, namely: the tristimulus values (X, Y, and Z), using the equations provided by Wyszecki and Stiles (1982), which were converted into Munsell notation (Hue, Value, and Chroma) with the aid of software downloaded from <http://wallkillcolor.com>. Whole spectra were used for the chemometric analysis, where reflectance data were transformed into absorbance measurements [$\text{Log}_{10}(1/\text{Reflectance})$]. Other data transformations such as the Kubelka-Munk function [$(1-\text{Reflectance})^2/(2 \times \text{Reflectance})$], scatter and smoothing corrections, or derivative pre-treatments, were also tested, but their results were not significantly better. Principal component analysis (PCA) using the NIPALS algorithm (Martens and Næs, 1989) and partial least-squares regression (PLSR) analysis (Geladi and Kowalski, 1986) as implemented in the commercial software package Unscrambler 9.02 (CAMO technologies, Inc., Woodbridge, NJ) were used in order to relate spectra to mineral composition. The calibration accuracy was evaluated via the determination coefficient, r^2 , and the root mean square errors of calibration (SEc). The results were validated by using the one-out cross validation procedure, which involves withdrawal of each sample in turn from the whole set and development of a calibration equation from that set with prediction of the value for the withdrawn sample; this method provides a good estimate of the standard error of prediction (SEv) (Shenk and Westerhaus, 1996).

3. Results and discussion

3.1. Iron ore mineralogy

Hematite was the major constituent in the studied ore samples (Table 1), which is typical of Brazilian iron ores. The water contents

Table 1
Mineralogical composition of the samples.

	Mean	Standard deviation	Minimum	Maximum
Hematite (%)	61.5	22.8	6.1	92.0
Specularite (%)	37.3	25.9	0.0	92.0
Martite (%)	24.1	21.2	0.0	83.0
Goethite (%)	13.4	16.9	0.0	86.3
Magnetite (%)	3.6	4.1	0.0	17.0
Total Fe (%)	54.1	12.7	4.3	69.2
Quartz (%)	21.2	18.4	1.0	93.9

of Brazilian ores are low relative to Australian ores, which typically contain greater amounts of goethite (Morris, 2003). In addition, Brazilian ores have low Fe^{2+} contents (i.e. low magnetite contents) and insubstantial amounts of alumina. The other major iron-bearing phase in addition to hematite was goethite. This oxyhydroxide is normally present as fine-grained material, which is named “earthy goethite” in order to distinguish it from the high-crystallinity variety.

The hematite crystals in the ores exhibited various morphologies that were normally classified as tabular (HT) or granular (HG) as seen under the optical microscope (Fig. 1a). The main difference between tabular and granular hematite is that the former has a higher aspect ratio and more regular edges. Micro granular hematite was observed in some samples. Non-porous crystals were collectively grouped as “specularite”. The identification of martite was quite simple as the mineral exhibited a porous structure with occasional magnetite relicts. “Earthy goethite” was seen as a brown material, whereas magnetite exhibited a pink color. Quartz grains appeared as black spots. All these phases were present in the sample of Fig. 1b.

The studied samples were chosen in such a way to span the broadest possible range of compositions, even if economically unviable (e.g. ores that contain goethite as the major iron-bearing phase). These samples were included in order to check whether the method would be applicable to all kinds of compositions. As can be seen from Table 1, the mineralogy varied widely except for magnetite, which exhibited peak content of about 17%.

The results for micaceous hematite in particles sizes from 60 μm to 500 μm exhibited no significant differences.

3.2. Color

Munsell color ranged from dark gray to yellowish red and relatively little in hue (from 5.6YR to 3.5Y) and value (from 3 to 5.3), but somewhat more in chroma (from 0.6 to 3.9), which is the greatest contributor to color (Fig. 2). Samples with a high content in lamellar specularite, which possesses metallic luster exhibited gray colors with low chroma (Fig. 3c). Increasing concentrations of martite and/or goethite resulted in increasing chroma by effect of the high pigmentation power of the small-sized particles of these minerals. Sub-micrometric goethite and hematite (martite) exhibit an absorption band marked with arrows in Fig. 3b and d, which has been assigned to an electron pair transition (Sherman and Waite, 1985; Scheinost et al., 1998) that is closely related to hue. Because

this band is clearly shifted to lower wavelengths for goethite, the samples with increased proportions of this mineral exhibited yellowish hues (close to 2.5Y, Fig. 2). No significant bands were observed in the visible range for quartz (Fig. 3a) or magnetite (Fig. 3e). Consequently, the effect of these minerals was to increase (quartz) or decrease (magnetite) the Munsell value (V).

3.3. Chemometric analysis of the reflectance curves

The principal component analysis of the reflectance data showed that 99.9% of spectral variance could be accounted for by the first three components—and 99.999% by the first 10 (Fig. 4). The X matrix, which contained 95 rows (samples) and 480 columns (absorbance data from 320 to 800 nm in 1-nm steps) was reduced by using the iterative NIPALS algorithm (Martens and Naes, 1989) to:

$$X = TP + E1$$

where T is the matrix of scores, P are the spectral loadings and $E1$ the residuals. T is a new reduced matrix consisting of 95 rows but only 10 columns and still retaining virtually all significant spectral information. This is so because the spectra contained much redundant information (i.e. high multi-collinearity).

Multiple correlation by least squares regression between the Y matrix containing 95 rows and 7 columns (hematite, specularite, martite, goethite, magnetite, total Fe and quartz) and T matrix (95×10) of scores of the principal component led to the following equation:

$$Y = TB + E2$$

where B are the matrix coefficients and $E2$ are the residuals. The software used the B matrix to construct a model in order to predict the mineral content of new samples based upon the diffuse reflectance spectra. Other statistical parameters derived from the least-squares regression confirmed the accuracy of this technique. Thus, the correlation coefficients for calibration (r_c^2) between predicted and measured values were close to 0.9 for all minerals except magnetite (Fig. 5a–g). The correlations for specularite and martite (Fig. 5c and e) were also relatively good ($r_c^2 > 0.8$), which indicates that the diffuse reflectance spectrum is sensitive not only to composition, but also to grain size distribution, as can be inferred from differences in diffuse reflectance spectra [see selected spectra for samples with a high content of specularite (Fig. 3c) and martite (Fig. 3d)]. The best correlation was that for goethite (Fig. 5b),

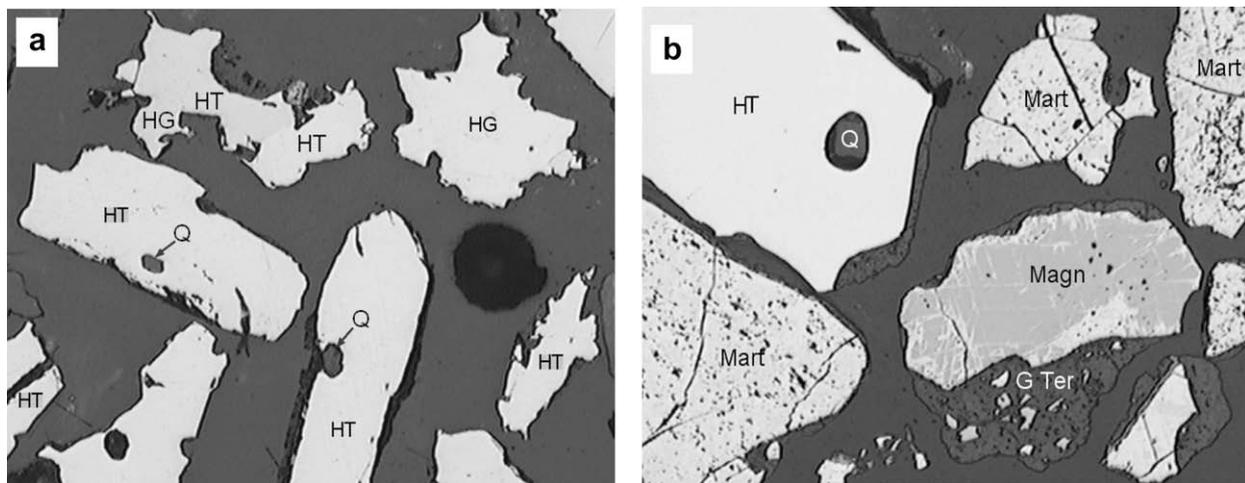


Fig. 1. Optical image of an iron-ore sample containing hematite and quartz (a), and of one containing hematite, goethite, martite, magnetite and quartz (b): tabular hematite = HT; granular hematite = GT; earthy goethite = Gter; martite = Mart; magnetite = Magn; quartz = Q.

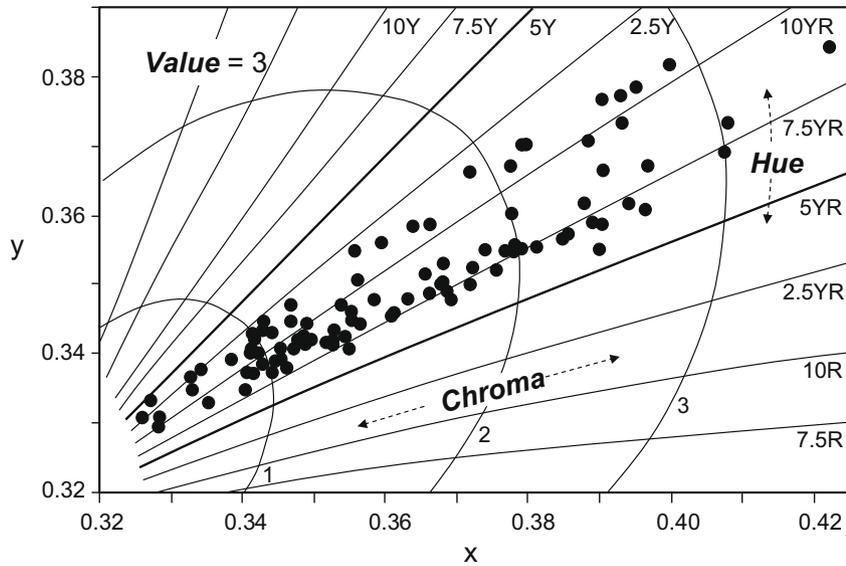


Fig. 2. Plot on the plane of Munsell value = 3 of the x and y chromaticity coordinates for the studied iron-ores samples.

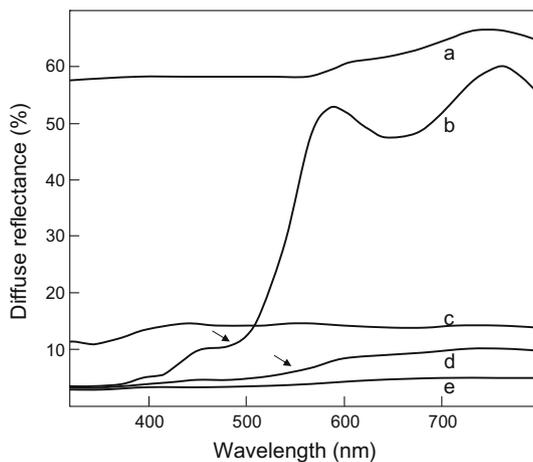


Fig. 3. Selected spectra for: (a) quartz obtained from the complete deferrification of a mixture of iron-ores samples; (b) commercial goethite powder (Bayferrox® 920, Bayer); (c) a sample with a high content (92%) of specularite; (d) a sample with a high content (83%) of martite and (e) commercial magnetite powder (Bayferrox® 318 M, Bayer).

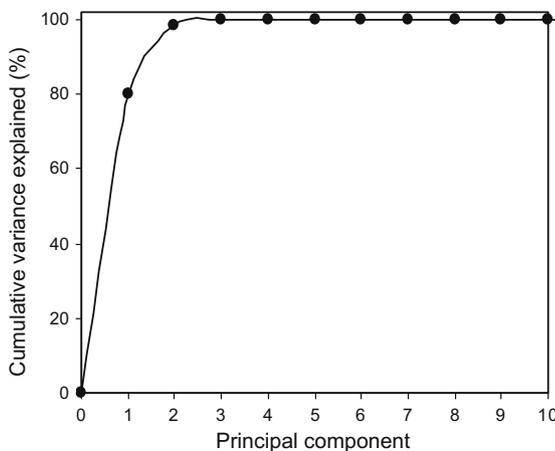


Fig. 4. Cumulative variance explained by the principal components for the spectra in the 320–800 nm range.

probably because its reflectance curve exhibited the best defined bands (Fig. 3b). This can be also the reason for its low standard error of calibration ($SE_C = 3.7\%$) relative to hematite and quartz.

The cross-validation test was more restrictive with a view to evaluating the accuracy of the proposed method. As expected, the values of the two cross-validation statistics (r_c^2 and SE_v) were invariably slightly lower than their calibration counterparts (r_c^2 and SE_C) (Fig. 5). One other statistical criterion which is commonly used in chemometric analysis to compare different constituents is the ratio of performance deviation (RPD), which was calculated by dividing the standard deviation by the standard error of validation (Table 1). Use of the guidelines of Malley et al. (2004) for evaluating calibration for environmental samples such as soils or sediments resulted in excellent calibration ($r^2 > 0.95$, $RPD > 4$) for goethite, successful calibration ($r^2 = 0.90$ – 0.95 , $RPD = 3$ – 4) for hematite, moderately successful calibration ($r^2 = 0.8$ – 0.9 , $RPD = 2.25$ – 3) for quartz, and moderately useful ones ($r^2 = 0.7$ – 0.8 , $RPD = 1.75$ – 2.25) for specularite and martite. Except for magnetite, all calibrations fell in these four categories; even for this mineral, however, the procedure was useful for screening purposes. In any case, magnetite abundance can be estimated from magnetic susceptibility.

In studies on soil samples and other materials analysed by applying chemometric procedures to near infrared reflectance data (Malley et al., 2004; Viscarra-Rossel et al., 2006, and references therein), partial least-squares regression (PLSR) typically provided more robust calibration than did PCA regression. For our samples, however, the results were comparable, i.e. r_c^2 , SE_C , r_v^2 , SE_v and RPD were not significantly different.

In conclusion, diffuse reflectance spectroscopy is a fast—only about 10 min/sample—inexpensive, accurate, environmentally safe—no chemical extractants—tool for the simultaneous quantification of the minerals commonly found in iron ores powders. Although we used a limited number of samples and composition range, the proposed methodology is quite dynamic and allows the introduction of other samples simply by remodeling the calibration curves. The influence of contaminants such as kaolinite and carbonates on the regression curves was not evaluated since Brazilian iron-ores generally contain only small amounts of these phases. However, their influence on reflectance spectra in the visible range can be anticipated to be quite weak. The particle-size effect on reflectance, which was evaluated through the spectra for

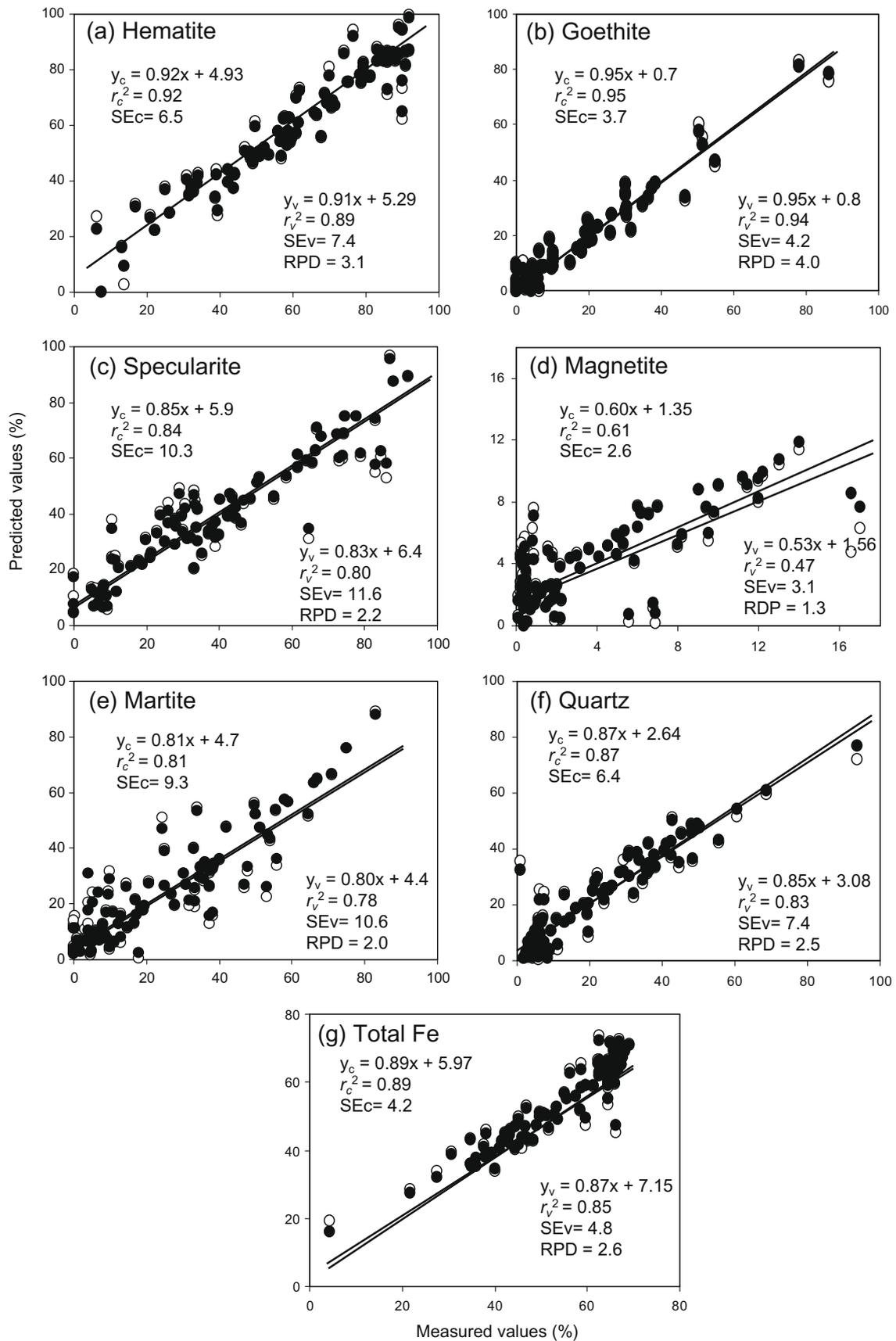


Fig. 5. Calibration (●) and cross-validation (○) regression between the predicted values obtained from the 10 principal components for the spectra and the measured values of the mineral contents as obtained with conventional procedures.

micaceous hematite, was found to be insignificant for particles ranging from 60 μm to 500 μm in size. Our results, along with others recently reported, show that diffuse reflectance is a very promising technique for the mineralogical analysis of various types of ores. Quantitative or semi-quantitative analyses can be easily performed once the reflectance spectra for previously well-characterized samples are obtained to construct the calibration model.

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