Iron Ore Quantitative Characterisation Through Reflected Light-Scanning Electron Co-Site Microscopy

O F M Gomes¹ and S Paciornik²

ABSTRACT

The traditional trading of iron ores is based on chemical specifications and particle size distribution. However, recent characterisation studies, that bring additional information like mineralogical composition and microstructural (textural) aspects, have become important. They contribute to determination of the iron ores downstream beneficiation operations and subsequent steelmaking processing, allowing improvements on both new and existing processes.

Despite progress in commercial scanning electron microscope/energy dispersive spectrometer (SEM/EDS) automatic instruments in the last decade, these systems are not capable of performing the identification and discrimination of major iron ore minerals (haematite and magnetite) and the consequent description of its microstructure. On the other hand, reflected light microscopy (RLM) can easily distinguish the iron oxides by their reflectancies, but it cannot discriminate quartz and epoxy resin, which present similar colour on images.

The present work proposes an innovative method to perform a quantitative mineralogy characterisation of iron ore based on RLM-SEM co-site microscopy. This technique combines images obtained by RLM and SEM through an automatic registration procedure that accurately aligns the two kinds of images of each field. In fact, it creates multi-dimensional images, which are then analysed using image processing and pattern recognition techniques.

The case study is an itabiritic iron ore from Quadrilátero Ferrífero (Brazil) which contains mainly quartz, goethite, magnetite and haematite. The discrimination of phases that are not distinguishable with either RLM (epoxy resin and quartz) or SEM (haematite and magnetite) can be performed through this multimodal approach, allowing the subsequent mineralogical quantification.

The mineralogical quantification computed by image analysis was consistent with independently obtained results based on the Rietveld technique.

INTRODUCTION

The traditional trading of iron ores is based on chemical specifications and particle size distribution. However, recent characterisation studies, which bring additional information like mineralogical composition and microstructural (textural) aspects, have become important. They contribute to determination of the iron ores’ downstream beneficiation operations and subsequent steelmaking processing, allowing improvements on both new and existing processes (Vieira et al., 2003; Pirard and Lebichot, 2004; Santos and Brandão, 2005).

Itabiritic iron ores from Quadrilátero Ferrífero (Brazil) have generally a simple mineral assemblage mainly composed by haematite, magnetite, goethite and quartz. Despite their simple mineralogy, the quantitative microstructural characterisation of these ores is still a challenge.

Reflected light microscopy (RLM) is typically applied to perform qualitative characterisation of iron ores by visual examination. The most common iron ore minerals can be visually identified on RLM through their reflectancies. In fact, haematite, magnetite, goethite and quartz present quite different reflectancies (Criddle and Stanley, 1993).

Automatic image analysis systems are capable of identifying haematite, magnetite and goethite by their colour on suitable RLM images. However, quartz and epoxy resin have practically the same reflectance through the visible light spectrum (Neumann and Stanley, 2008). Therefore, they cannot be distinguished only by their reflectancies. Actually, this is a classical problem in ore microscopy that renders unfeasible this kind of microstructural characterisation through RLM and digital image analysis.

On the other hand, a scanning electron microscope (SEM) with a backscattered electron detector can produce grey level images where quartz and epoxy resin present different intensities, due to their distinct average atomic numbers. Goethite also exhibits a different intensity, but haematite and magnetite have similar average atomic numbers, respectively 20.59 and 21.02, and consequently show similar grey levels in such kind of images, preventing their discrimination.

The discrimination of haematite and magnetite phases in backscattered electron images requires a strong image contrast. However, this contrast condition avoids the segmentation of the other phases. The complete discrimination of these minerals is hence not possible with this kind of signal. Besides, in practice, not even SEM systems with energy dispersive X-ray microanalysis (EDX) can discriminate haematite and magnetite.

The present work proposes an innovative method to perform a quantitative mineralogy characterisation of iron ore based on RLM-SEM co-site microscopy (Gomes, 2007). The method can improve the SEM analytical capacity adding reflectivity information from RLM to discriminate haematite and magnetite.

EXPERIMENTAL

Sample selection and preparation

An itabiritic iron ore from Quadrilátero Ferrífero (Brazil) was selected as a case study. The ore was classified and segregated with a dense liquid. Thus, the samples -297+210 μm, -149+105 μm, -74+53 μm with density greater than 3.2 were employed. The samples were cold mounted with epoxy resin and subsequently ground and polished. After image acquisition on RLM, the cross-sections were covered by an evaporated carbon layer to make them conductive and suitable for SEM analysis.

Image acquisition on a reflected light microscope

A Zeiss Axioplan 2, ie motorised and computer controlled microscope, was used with an AxioCam HR digital camera (1300 × 1030 pixels). A function implemented as a macro routine in the KS400 software (Carl Zeiss Vision) was used for microscope and camera control and for image acquisition. This function integrates automated procedures like specimen x-y scanning, automatic focusing, background correction and imaging.

The following image acquisition conditions were employed:

1. before image acquisition, a SiC reflectivity standard was used to generate a background image, which was automatically employed for background correction of every acquired image;

1. Centre for Mineral Technology (CETEM), Avenida Pedro Calmon, 900, Cidade Universitária, Rio de Janeiro RJ 21941-590, Brazil.
   Email: ogomes@gmail.com

2. Department of Materials Science and Metallurgy, Catholic University of Rio de Janeiro (PUC-Rio), Rua Marquês de S. Vicente, 225, sala 501L, Gávea, Rio de Janeiro RJ 22453-900, Brazil.
   Email: sdcini@puc-rio.br
2. illumination was kept constant by direct digital control of the lamp voltage;
3. camera sensitivity, exposure and white balance were optimised initially for a representative image and kept constant thereafter;
4. objective lenses: 5× (NA 0.13); 10× (NA 0.20); 20× (NA 0.40), leading to resolutions of 2.11, 1.05 and 0.53 μm/pixel, respectively;
5. the three samples (-297+210 μm, -149+105 μm, -74+53 μm) were imaged respectively with the 5×, 10× and 20× objective lens;
6. single fields regularly spaced on the sample were imaged through specimen scanning with a motorised x-y stage and automatic focusing;
7. each field position was recorded in a database for subsequent image acquisition on SEM; and
8. all images were acquired at 24 bit RGB quantisation.

Image acquisition on a scanning electron microscope

A LEO S440 scanning electron microscope was used to acquire a backscattered electron image of each field imaged on RLM. In this procedure, the sample must be placed in the SEM stage at a similar arrangement as positioned in the RLM stage. It is unnecessary and impractical to place the sample in the exact same way, but a similar arrangement can make image registration easier and faster.

The magnification of the SEM was set to keep the same optical resolution and other SEM operational parameters were manually tuned. Then, the field positions database was loaded with a function developed in the LEO control software. It converts RLM stage coordinates to SEM stage coordinates and subsequently performs automatic specimen scanning and image acquisition. Thus, for each sample, 81 fields were imaged with the RLM and the SEM.

Image registration

Image registration is the process of overlaying two or more images of the same scene taken at different conditions or by different sensors. It geometrically aligns two digital images pixel by pixel. Image registration is a crucial step in all image analysis tasks in which the final information is gained from the combination of various data sources. Typically, registration is required in remote sensing and medicine to combine and compare images (Zitova and Flusser, 2003).

In the present work, an automatic registration procedure for RLM and SEM images was developed in Matlab system (MathWorks) based on the maximisation of the normalised cross-correlation (Lewis, 1995) and the local weighted mean method (Goshtasby, 1988). It automatically aligns each pair of images from RLM and SEM. At the end, the aligned images are cropped to represent exactly the same field. Figure 1 shows a pair of images of a field from the -149+105 μm sample obtained by RLM and SEM, after registration.

The developed registration procedure performs an accurate alignment of the two kinds of images obtained from each field. Thus, it indeed builds an image with four layers, which can then be analysed using image processing and pattern recognition techniques to recognise different phases.

Mineral recognition

The recognition of mineral phases was performed by routines implemented in the Matlab environment. The registered images of each field went through a delineation operation (edge enhancement) to reduce the well-known halo effect (Sutherland and Gottlieb, 1991), making them more suitable for the subsequent segmentation procedures.

The composed images with four layers were segmented through the classification of their pixels in a typical supervised classification procedure (Gonzalez and Woods, 2002). Thus, each pixel was classified in one of the predefined classes that actually represent the present phases. The four components (R, G, B and SEM intensity) of these images were used as features. A Bayes classifier (Duda, Hart and Stork, 2001) was employed to recognise each pixel phase.

The case study samples were composed mainly by haematite, magnetite, goethite and quartz, which were then taken as individual classes. Besides mineral phases, epoxy resin was taken as a class too. Thus, the training stage of the mineral recognition procedure involved sampling of pixels from the five classes. In practice, 6000 pixels of each phase were selected with the mouse from several images.

Furthermore, the SEM images were also segmented by the traditional thresholding method. The careful image acquisition conditions guarantee that the grey levels of each phase are stable. Thus, it was possible to use fixed intensity thresholds, effectively automating this segmentation procedure. Subsequently, the segmented images by thresholding were post-processed to eliminate small spurious objects that occurred mainly in borders between phases.

In order to compare these segmentation methods, the area fraction of the phases were measured in each segmented image.

![Image 1](https://example.com/image1.png) 200 μm

![Image 2](https://example.com/image2.png) 200 μm

**Fig 1** - Images of a field from the -149+105 μm sample obtained by RLM and SEM, after registration.
Besides, from these results, the volumetric fractions were obtained, and the mass fractions of the mineral phases were computed based on their theoretical densities.

**X-ray diffraction**

The mineralogical composition of the samples was also quantified by X-ray powder diffraction (XRD) using the Rietveld technique (Rietveld, 1967). The XRD data were collected on a Bruker-AXS D4 Endeavour equipment, with Co X-ray tube at 40 kV and 40 mA, and with a position sensitive LynxEye detector. The mineralogical quantification was performed by Bruker TOPAS R software.

**RESULTS AND DISCUSSION**

Figure 2 presents the results for area fractions of resin and quartz measured in the segmented images by supervised classification (C) and thresholding (T). In order to facilitate visualisation of this kind of figure, the fields were ordered regarding the area fraction computed from images segmented by thresholding. As one can see, the results for resin and quartz were quite similar.

On the other hand, as shown in Figure 3, it seems that the classification method led to an overestimation of the goethite phase fraction. This error occurred mainly due to undesirable relief and pull-outs that cause a considerable misclassification of haematite phase in these sites, as one can see in Figure 4. It presents the image segmented by the classification method applied to the images shown in Figure 1, followed by a look-up table that makes its visualisation easier.

This problem is less significant in the SEM segmented images because SEM is less sensitive to sample preparation defects than RLM. Therefore, the evaluated segmentation methods were combined in a hybrid method to prevent this problem. The segmented image obtained by the SEM image thresholding was applied to recognising resin, quartz and goethite, and also to determining a haematite-magnetite composed phase. Then, the classified image was used only to discriminate haematite and magnetite from the composed phase. Figure 5 presents the image segmented by the hybrid method applied to the images shown in Figure 1.

Table 1 shows phase fractions (wt per cent) measured by image analysis (classification and hybrid method) and Rietveld technique. The results from the hybrid method and the Rietveld ones were quite similar.

The results shown in this section refer to the 149+105 μm sample. The other samples presented similar results.

**CONCLUSIONS**

A co-site microscopy methodology that combines images obtained by reflected light microscopy (RLM) and scanning electron microscopy (SEM) was developed. The so-called
RLM-SEM co-site microscopy was tested in the characterisation of itabiritic iron ore samples in order to show its analytical capacity with excellent results.

The hybrid image analysis method was capable of recognising all phases, distinguishing simultaneously quartz from epoxy resin, and haematite from magnetite. The mineralogical quantification computed by image analysis (hybrid method) was consistent with independently obtained results by the Rietveld technique. Therefore, RLM-SEM co-site microscopy is an effective technique for iron ore quantitative characterisation.

REFERENCES


**TABLE 1**

Phase fractions (weight per cent) measured by image analysis and by the Rietveld technique.

<table>
<thead>
<tr>
<th>Mineral phase</th>
<th>Classification (wt %)</th>
<th>Hybrid method (wt %)</th>
<th>Rietveld (wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>7.5</td>
<td>8.9</td>
<td>10.1</td>
</tr>
<tr>
<td>Goethite</td>
<td>27.8</td>
<td>1.4</td>
<td>1.5</td>
</tr>
<tr>
<td>Haematite</td>
<td>63.7</td>
<td>88.8</td>
<td>87.2</td>
</tr>
<tr>
<td>Magnetite</td>
<td>1.0</td>
<td>1.0</td>
<td>1.2</td>
</tr>
</tbody>
</table>

**FIG 5** - The image segmented by the hybrid method applied to the images shown in Figure 1, followed by the used look-up table.