RLM-SEM CO-SITE MICROSCOPY APPLIED TO IRON ORE CHARACTERIZATION[1]

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Abstract
Despite progress in Scanning Electron Microscopy (SEM) automatic instruments, these systems are not capable of performing the identification and discrimination of major iron ore minerals (hematite and magnetite). On the other hand, Reflected Light Microscopy (RLM) can easily distinguish these iron oxides by their reflectancies, but it cannot discriminate quartz and epoxy resin, which present similar color on images. Therefore, iron ore quantitative microstructural characterization is still a challenge. In the present work, a Co-Site Microscopy methodology that combines images obtained by RLM and SEM was applied to perform a quantitative characterization of an itabiritic iron ore. The so-called RLM-SEM Co-site Microscopy can discriminate phases that are not distinguishable with either RLM (epoxy resin and quartz) or SEM (hematite and magnetite) through this multimodal approach, allowing the subsequent mineralogical quantification. The applied methodology employs an automatic routine based on Image Analysis and Pattern Recognition techniques. The mineralogical quantification computed by image analysis was consistent with independently obtained results based on the Rietveld technique.
Key words: Iron ore; Co-site microscopy; Image analysis

MICROSCOPIA CO-LOCALIZADA MO-MEV APLICADA À CARACTERIZAÇÃO DE MINÉRIO DE FERRO

Resumo
Apesar do progresso nos sistemas automatizados de microscopia eletrônica de varredura (MEV), estes instrumentos não são capazes de identificar e discriminar os principais minerais de minérios de ferro (hematita e magnetita). Por outro lado, a microscopia óptica de luz refletida (MO) pode facilmente distinguir estes óxidos de ferro por suas refletâncias, mas não consegue discriminar quartzo e resina epóxi, que apresentam cores similares. Deste modo, a caracterização microestrutural quantitativa dos minérios de ferro ainda é um desafio. No presente trabalho, uma metodologia de microscopia co-localizada que combina imagens adquiridas em MO e em MEV foi aplicada na caracterização quantitativa de um minério de ferro itabirítico. A chamada microscopia co-localizada MO-MEV é capaz de discriminar as fases que não são distinguíveis por MO (resina epóxi e quartzo) e por MEV (hematita e magnetita) através desta abordagem multimodal, possibilitando a subseqüente quantificação mineralógica. A metodologia utilizada emprega uma rotina automática baseada em técnicas de Análise de Imagens e Reconhecimento de Padrões. A quantificação mineralógica realizada por análise de imagens foi consistente com os resultados independentemente obtidos pela técnica de Rietveld.
Palavras-chave: Minério de ferro; Microscopia co-localizada; Análise de imagens

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1 INTRODUCTION

Reflected Light Microscopy (RLM) is typically applied to perform qualitative characterization of iron ores by visual examination. The most common iron ore minerals (hematite, magnetite, goethite and quartz) can be visually identified on RLM through their reflectancies. In fact, they present quite different reflectancies.[1]
Automatic image analysis systems are capable of identifying hematite, magnetite and goethite by their color on suitable RLM images. However, quartz and epoxy resin have practically the same reflectance through the visible light spectrum.[2] 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 characterization through RLM and digital image analysis.
On the other hand, a Scanning Electron Microscope (SEM) with a back-scattered 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 hematite 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 hematite and magnetite phases in back-scattered 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 Micro-analysis (EDX) can discriminate hematite and magnetite.
Therefore, iron ore quantitative microstructural characterization is still a challenge. In the present work, a Co-Site Microscopy[3] methodology that combines images obtained by RLM and SEM was applied to perform a quantitative characterization of an itabiritic iron ore that are mainly composed by hematite, magnetite, goethite and quartz. The so-called RLM-SEM Co-site Microscopy can improve the SEM analytical capacity adding color information from RLM to discriminate hematite and magnetite.
The applied methodology employs an automatic routine based on Image Analysis and Pattern Recognition techniques in order to assess quantitative mineralogy.

2 MATERIALS AND METHODS

2.1 Sample Selection and Preparation

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

2.2 Image Acquisition on RLM

A Zeiss Axioplan 2 ie motorized and computer controlled microscope was used, with an AxioCam HR digital camera (1300 x 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 and

automates many procedures like specimen x-y scanning, automatic focusing, background correction and imaging.
The following image acquisition conditions were employed:

a) 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;
b) illumination was kept constant by direct digital control of the lamp voltage;
c) camera sensitivity, exposure and white balance were optimized initially for a representative image and kept constant there on;
d) objective lens: 10X (NA 0.20), leading to resolution of 1.05 μm/pixel;
e) 81 fields regularly spaced on the sample were imaged through specimen scanning with a motorized x-y stage and automatic focusing;
f) each field position was recorded in a data base for subsequent image acquisition on SEM;
g) all images were acquired at 24 bit RGB quantization.

2.3 Image Acquisition on SEM

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 data base 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, 81 fields were imaged with the RLM and the SEM.

2.4 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.[4]
In the present work, an automatic registration procedure for RLM and SEM images,[5,6] which was developed in Matlab system (MathWorks), was employed. 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 obtained by RLM and SEM, after registration.

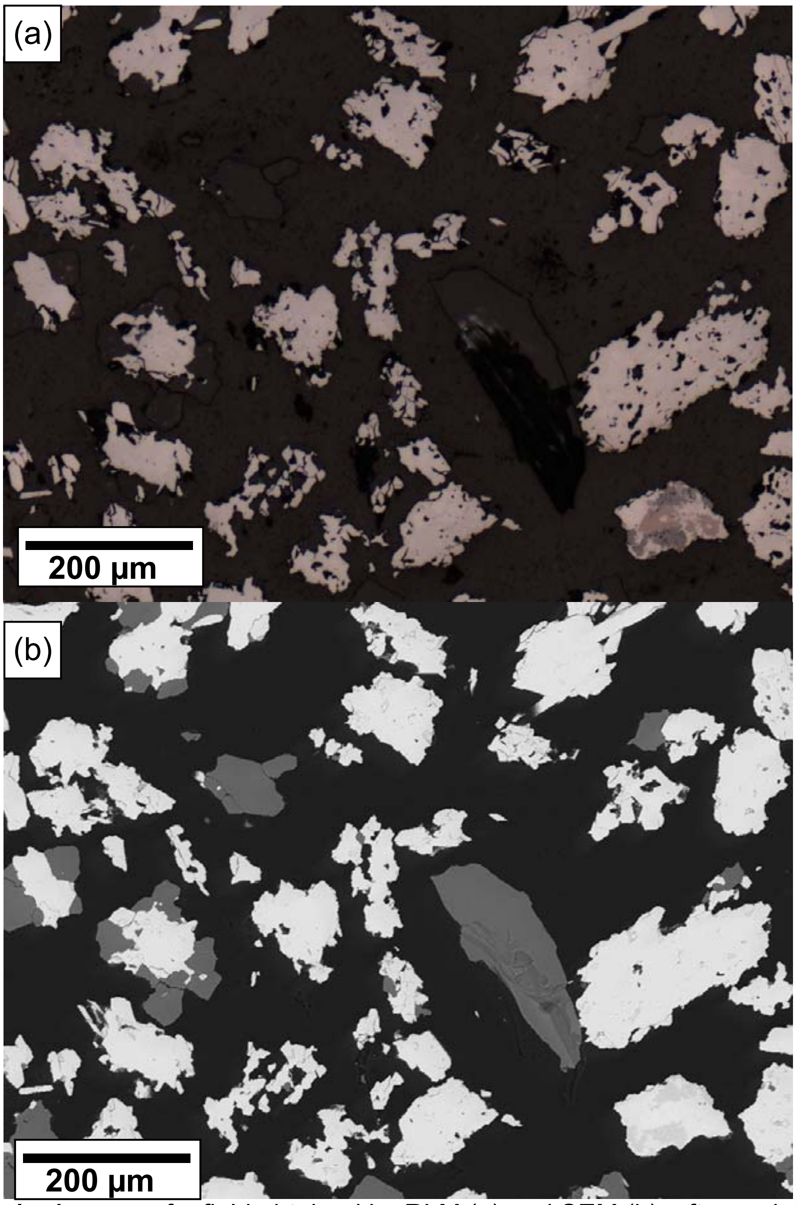

Figure 1 – Images of a field obtained by RLM (a) and SEM (b), after registration.

The registration procedure performs an accurate alignment of the two kinds of images obtained from each field. Thus, it indeed builds a RLM-SEM composed image with four components (R, G, B and SEM intensity), which can then be analyzed using Image Analysis and Pattern Recognition techniques to recognize different phases.

2.5 Image Analysis

The image analysis procedure was performed by an automatic routine implemented in the Matlab environment. This routine executes the following sequence of processing and analysis steps:

a) delineation operation of the RLM and the SEM images to reduce the well-known halo effect,[7] making them more suitable for the subsequent segmentation procedures;

b) automatic segmentation of resin, quartz, goethite, and a hematite-magnetite composed phase from the SEM images by supervised classification[8] of their pixels;
c) automatic segmentation of hematite and magnetite through the segmentation of the RLM-SEM composed images by supervised classification of their pixels;
d) logical and morphological post-processing procedure to eliminate small spurious objects that occurs mainly in borders between phases;
e) measurement of the area fraction of each present phase.

The training stage of the classification procedures involved sampling of pixels from the five classes (epoxy resin, quartz, goethite, magnetite, and hematite). In practice, 1000 pixels of each phase were selected with the mouse from a RLM-SEM composed image.

The SEM images were segmented through the classification of their pixels in epoxy resin, quartz, goethite, or a hematite-magnetite composed phase. The intensity of these images was used as feature and a Bayes classifier[9] was employed to recognize each pixel phase.

The RLM-SEM composed images were segmented through the classification of their pixels in one of the five pre-defined classes. The four components (R, G, B and SEM intensity) of these images were used as features and a Bayes classifier was employed. These segmented images were used only to discriminate hematite and magnetite in the SEM segmented images. Subsequently, logical and morphological operations were employed in a post-processing procedure to eliminate small spurious objects.

The area fraction of the phases was measured in each final resulting image. Besides, from these results, the volumetric fractions were obtained, and the mass fractions of the mineral phases were computed based on their theoretical densities.

2.6 X-Ray Diffraction

The mineralogical composition of the samples was also quantified by X-ray powder diffraction (XRD) using the Rietveld technique.[10] 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.

3 RESULTS AND DISCUSSION

The result of the image analysis routine was a grey level image per field where each phase was represented by a grey level. Thus, pixels recognized as epoxy resin have intensity 1, pixels recognized as quartz have intensity 2, and so on. Figure 2 presents the resulting image from the analysis of the images shown in Figure 1. In order to facilitate visualization, a look-up table was applied.

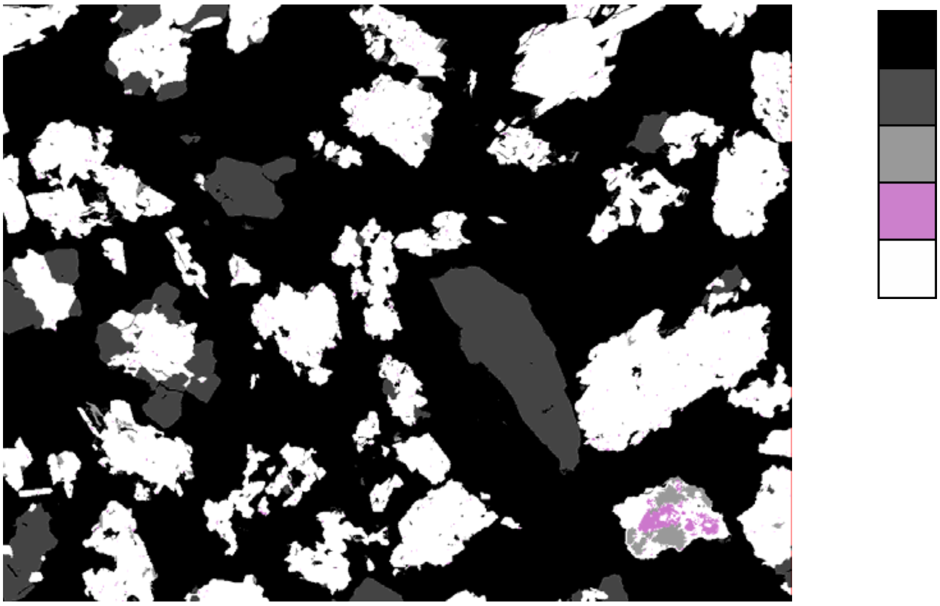

Figure 2 – The resulting image from the analysis of the images shown in Figure 1, followed by the used look-up table.

The present case study sample was already used in a Gomes and Paciornik[6] previous paper. In that work, a semi-automatic hybrid method was employed to analyze the images.

Table 1 shows phase fractions (wt. %) measured by the automatic image analysis routine, the semi-automatic hybrid method[6] and the Rietveld technique. The results from the image analysis (automatic and semi-automatic) and the Rietveld ones were quite similar.

Table 1 – Phase fractions (wt. %) measured by image analysis and Rietveld technique.

Mineral phase	Image analysis		Rietveld (wt. %)
	Automatic (wt. %)	Semi-automatic (wt. %)	
Quartz	8.7	8.9	10.1
Goethite	1.6	1.4	1.5
Magnetite	1.0	1.0	1.2
Hematite	88.7	88.8	87.2

4 CONCLUSION

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 characterization of an itabiritic iron ore sample in order to show its analytical capacity with excellent results.

The automatic image analysis routine was capable of recognizing all phases, distinguishing simultaneously quartz from epoxy resin, and hematite from magnetite. The mineralogical quantification results were very similar to the previously reported for the same sample. In fact, the present work consists of an improvement of the previous work since it automated the image analysis procedure.

The mineralogical quantification computed by image analysis was consistent with independently obtained results by the Rietveld technique. Therefore, RLM-SEM Co-site Microscopy consists in an effective technique for iron ore quantitative characterization.

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