A reliable method for the automated distinction of quartz gangue and epoxy resin with reflected light microscopy for geometallurgical characterisation

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Abstract. The reliable distinction of gangue (typically quartz or silicates) and epoxy resin (commonly used to embed the sample, before polishing) is a pending problem for the widespread automated mineralogical characterization of ores and its application to geometallurgy. Traditional methods based on specular reflectance (**R**) measures are not able to distinguish between gangue and resin because of their similar R values. Different additives (colorants and fluorescein) have been investigated to alter the spectral signature of the resin in polished thin sections. Automated segmentation can commonly be achieved with colour dyes, only if R ratios are used, instead of R values. However, the results are most reliable with the addition of fluorescein. The acquisition of fluorescence images with the 20x objective, shows a high contrast between bright and dark areas (belonging to resin and mineral particles, respectively). This opens the way to the generalized application of automated methods of optical microscopy, such as the AMCO system, for geometallurgical characterization, with a notable increase in efficiency over traditional methods (point counter) and a significant cost reduction compared to electron microscopy.

1 Introduction

The reliable distinction of gangue (typically quartz or silicates) and epoxy resin in reflected light microscopy (through the measurement of specular reflectance in the visible and near infrared ranges, between 400 and 1000 nm), is a common problem for the automated geometallurgical characterization of ores with digital image analysis software. This is not a difficult problem in polished whole rock samples, because resin is present at the border only and can be easily isolated.

In milled ore concentrates, however, each ore grain or particle is surrounded by resin. This resin must be discriminated from gangue in order to carry out automated quantitative analysis of ore and gangue, or when the contours of the grains have to be recognized to define morphological properties. This distinction is therefore a requirement for a widespread application of automated optical microscopy, which warrants a notable increase in efficiency over traditional methods (point counter) and a significant cost reduction compared to electron microscopy.

Neumann and Stanley (2008) studied the possibility of discriminating quartz from commercial epoxy resins. They concluded: "*The close coincidence of specular reflectance for quartz and epoxy resins ensures that there is not relevant reflectance contrast between them, even when spiked with dyes. This data set is final, and precludes the application of reflectance in light optical image analysis for assessment of liberation, phase quantification, or any other signal that requires separation of resin from transparent minerals*". Delbem et al. (2015) propose for iron ore characterisation a semi-automated quartz/resin classification, defining first the particle contours, and then applying the measured R values inside each particle to identify its components, but the contour definition is not always straightforward.

The approach of this work is the direct definition of the particles based on the measured R values.

2 Background

This research benefitted from previous experiences in the GeMMe Lab (University of Liège) and in the Laboratory of Applied Microscopy (**LMA**, Universidad Politécnica de Madrid), kindly contributed by Pirard E., and Pérez-Barnuevo L., resp., which even if unsuccessful were instrumental to avoid repetition of useless attempts, including use of organic solvents to etch the polished epoxy surface, laser ablation, use of different additives to modify the surficial appearance or the refractive index (and R) of the resin, use of different types of dyes (yellow, blue and red), graphite powder, etc.

A common feature of these attempts was the use of polished blocks (**PB**), in which results are usually unsatisfactory, even with coloured resins, because the epi-illumination penetrating the surface of the block is transmitted and diffused within the resin and the gangue, and can be finally conveyed back to the surface through the transparent gangue grains, apparently tinting them with the resin's colour.

In 2017, a Grade`s Thesis funded by a GIRMI-UPM grant (Grunwald-Romera 2017) brought forward Pérez-Barnuevo's work by studying the effect of different types of resin additives (the same yellow, red and blue dyes, plus fluorescein), in Polished Thin Sections (**PTS**). PTS have a typical thickness of 30 µm, thinner than the grain size of most samples, thus minimizing the presence of dyed resin under the grains. Should the grain size be smaller, thinner PTS might be prepared. This method proved to be quite effective, and therefore PTS were chosen as the standard sample preparation method for the tests that were performed later.

From 2016 to 2018, the LMA led the development of the AMCO System, an automated mineralogical characterisation system based on optical microscopy.

The work was carried out within a research project funded by EIT Raw Materials, with the participation of the University of Liège, the SME Thin Section Lab, and two mining companies (Cobre Las Cruces and KGHM). **AMCO** (which stands for Automated Microscopic Characterization of Ores), is the result of an upscaling of the CAMEVA system (Castroviejo et al. 2009, Catalina and Castroviejo 2017).

3 Materials and methods

A number of polished sections were prepared from two samples of quartz sand (IMOSAB and GUD), using two epoxy resins (Feroca and Struers EpoFix Kit), and four dyes from Struers: the three AcryDye pigment-based liquid colorants (yellow, red and blue), and EpoDye (fluorescein in powder). Undyed polished sections were also prepared, for comparison.

Nine PTS, two with each dye plus one without, and two PB, one with EpoDye and one undyed, were prepared from IMOSAB sand in the labs of Complutense University of Madrid (UCM) by the first author for her Grade's Thesis (2017). A set of polished sections used Feroca resin with 6 drops of AcryDye colorant for each one, while another set used EpoFix resin with 10 drops of AcryDye. For the sections with fluorescein, even if not exactly quantified, IMOSAB_F001 contained a larger amount of EpoDye than IMOSAB_F002.

Later on, five PTS and PB pairs from each sand sample (IMOSAB and GUD) were prepared at Thin Section Lab (Toul, France) using EpoFix resin and the same four dyes, plus one undyed.

Multispectral images of the polished sections were acquired at the LMA on a prototype of the AMCO system. The AMCO system prototype is based on a Leica DM6000 M reflected-light optical microscope integrating additional components, such as a fluorescence filter cube, a 12V/100W halogen light source with a custom hot mirror, a high-precision XY motorized stage, a filter wheel containing a large number of hard coated bandpass filters in the VNIR range, and a high-resolution monochrome video camera. The multispectral images are typically composed of 21 bands: 13 reflectance bands in the visible range from 400 to 700 nm with 25 nm bandpass (**BP**), 6 reflectance bands in the NIR from 750 to 1000 nm with 50 nm BP, a reflectance band in the NUV range centred at 370 nm with 36 nm BP, and an optional fluorescence band obtained through the L5 cube (excitation filter: BP 480/40 nm, dichromatic mirror: 505 nm, suppression filter: BP 527/30 nm), which is especially suited for fluorescein.

4 Multispectral measurements

Multispectral images of each of the polished sections were acquired with 5x, 10x and 20x objectives. For the images of the coloured resin sections, just the 20 reflectance bands were captured, as these resins do not exhibit significant fluorescence. However, for the images of the fluorescent resin sections, all the 21 bands (20 reflectance bands plus the fluorescence band) were captured. The fluorescence band does not measure reflectance, but the intensity of the fluorescence of the sample. Because of this, it was necessary to adjust the integration time of the camera for the fluorescence band before the acquisition, to adapt it to the brightness level of the fluorescent resin, which depended on the amount of EpoDye added (higher amounts of fluorescein made the resin shine brighter, so it needed a shorter exposure time to prevent the image from becoming saturated).

Several images of each PB and each PTS were acquired with each objective, in order to determine the effect of the different dyes on the resin. These images were examined one by one with the AMCO image analysis software (Fig. 1). In each image, a number of rectangular boxes were manually drawn with the mouse on sufficiently uniform regions of the sand grains and the resin matrix, in order to select the gangue and resin regions used to conduct the study.

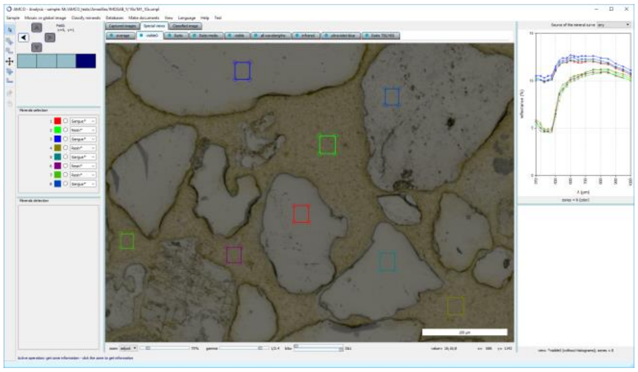


Figure 1. Window capture of AMCO image analysis software while examining an image of a polished thin section (IMOSAB-Y001)

The top-right graph of Figure 1 shows spectral curves measured on an image of a PTS prepared with yellow dye. Each curve is computed as the average (or the mode, at user's choice) of the multispectral R values of all the pixels contained within a region. The measured R values correspond in fact to apparent reflectance, which is higher than the nominal specular reflectance due to the well-known contribution of light diffused within the polished section (Criddle 1990). This effect is produced in transparent minerals by internal reflections, which are seen through the surface.

Measurements on PB show that the R values are very similar for gangue and resin, making automated distinction unreliable, while for PTS there are noteworthy differences. Therefore, for the rest of the discussion we will focus on PTS only.

5 Results and discussion

Figure 2 shows average spectral curves for gangue and resin from PTS prepared with resin dyed with the three colorants. Studying the curves, it can be realised that the R values of dyed resin regions are somewhat lower than the R values of gangue regions at some bands. These bands are typically the bands near the complementary colour of the dominant hue of the dye.

For instance, with the yellow dye, resin appears significantly darker than gangue in the bands closer to the blue range (between 400 and 500 nm).

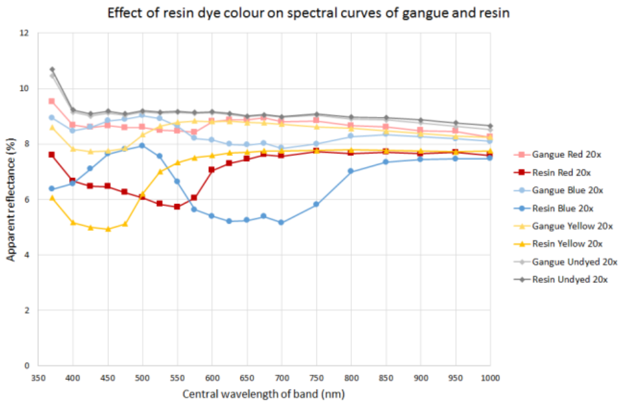


Figure 2. Effect of resin dye colour on measured spectral curves of gangue and resin

This spectrally uneven darkening of the resin can be exploited by computing ratio images of selected bands. Figure 3 shows a ratio image, in which each pixel is the quotient of the R value of the 425 nm band divided by the R value of the 700 nm band, for a PTS made with yellow-dyed resin. The difference in grey level between the gangue and the resin in this image is remarkable, as the bimodal histogram in Figure 4 clearly shows.

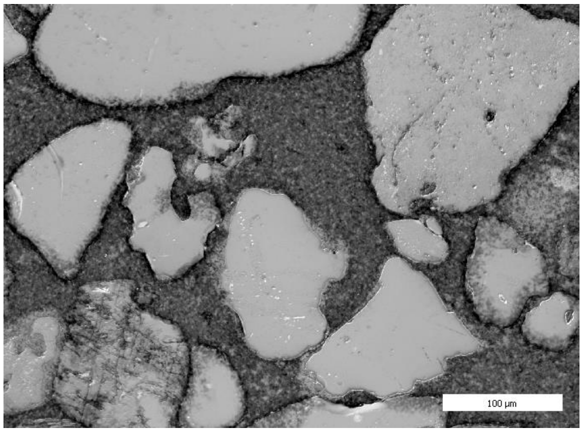
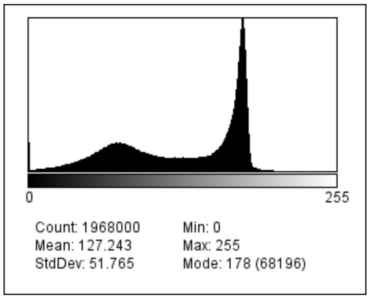


Figure 3. Ratio of 425 / 700 nm bands (yellow-dyed resin, 10x)



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Count: 1968000      Min: 0
Mean: 127.243       Max: 255
StdDev: 51.765      Mode: 178 (68196)
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Figure 4. Histogram of the 425 / 700 nm ratio image of Figure 3

The behaviour of the three AcryDye colorants is similar, although the effect is less intense for the red and blue dyes than for the yellow. Depending on the amount of colorant added, the aspect of the resin can change considerably. If the amount of dye is small, its pigments absorb part of the light that impinges on the resin, decreasing its apparent reflectance. As the amount of pigment increases, its particles make the resin behave like a higher-reflectance surface.

The magnification of the objective also has a clear effect in the separation between the spectral curves of gangue and resin: it is wider for the 5x and 10x objectives than for the 20x objective (Fig. 5). Nevertheless, 20x is the preferred magnification for ore characterisation, so the selected solution should be the one working best with 20x objectives.

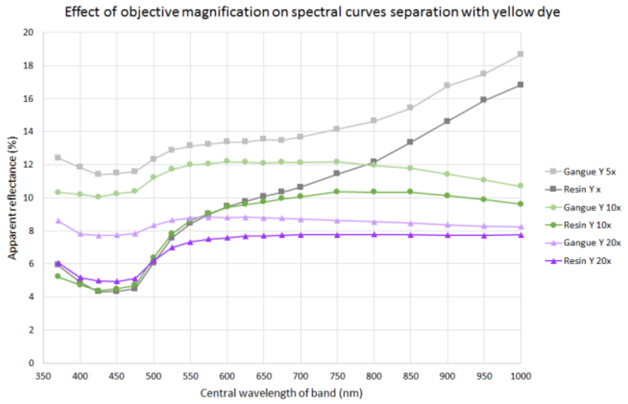

Figure 5. Effect of objective magnification on spectral curves separation

EpoDye behaves as a colorant very similarly to the yellow dye, and produces very good ratio images as well. However, its effect is even more outstanding when the image is acquired in fluorescence mode, as the resin appears bright while the gangue looks almost black, as Figure 6 shows. The histogram of fluorescence images is clearly bimodal (Fig. 7), implying a straightforward segmentation.

Therefore, the best solution is to use fluorescein as dye (EpoDye), and acquire a band in fluorescence mode, because of its higher effectivity with the 20x objective. While in bright-field mode differences between gangue and resin are small, in fluorescence mode resin becomes very bright and is clearly distinguished from mineral grains. Fluorescent minerals that might exist in the sample could be the only disadvantage of this method, but they are not common components of milled ore concentrates, and their fluorescence would also help to detect them.

6 Conclusion and applications

The results of this work for Polished Blocks (PB) agree with the conclusions reported by Neumann and Stanley (2008).

However, our work has shown that an effective method to distinguish between resin and gangue minerals is possible for Polished Thin Sections (PTS), without altering the properties of the ores or the gangue present in the sample.

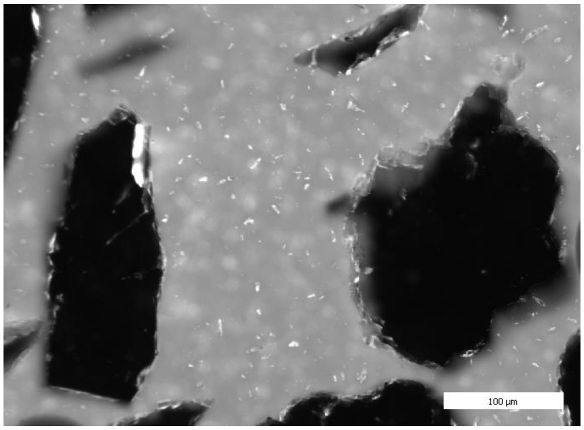

Figure 6. Image of fluorescence (fluorescein-dyed resin, 20x)

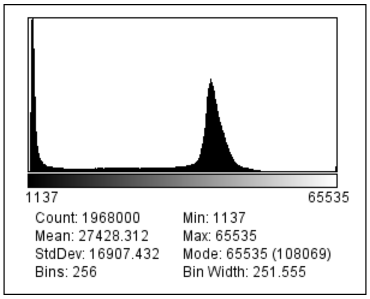

Figure 7. Histogram of the fluorescence image of Figure 6

Although all the studied colorant dyes can provide ratio images presenting a good discrimination between resin and gangue with 5x or 10x objectives, the best solution appears to be the use of the fluorescent dye, because of its high effectivity with the 20x objective, both for ratio and fluorescence images. This enables the application of Digital Image Analysis based on Reflected Light Microscopy to milled ore samples or concentrates.

With this solution, using PTS, the way to the generalized application of automated methods of optical microscopy for geometallurgical characterization is wide open. Nevertheless, for whole-rock studies automated identification of gangue minerals is possible even in PB, except for porosity measurement or for very porous samples, because the presence of resin is limited to the borders and can be easily isolated.

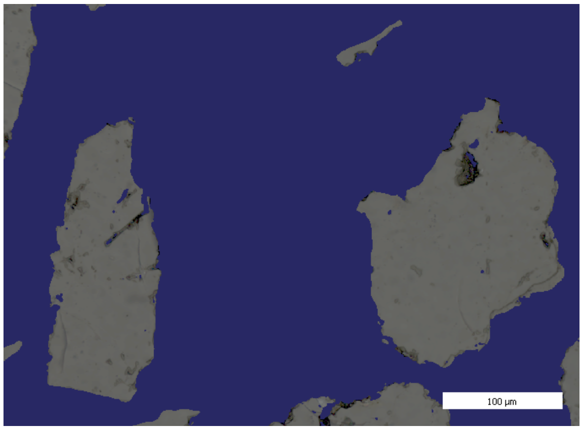

Figure 8. Mask for resin computed from image of Figure 6

The AMCO image analysis software incorporates a routine to implement the automated distinction between gangue and resin that creates a mask for the resin area, isolating the particles present in the microscopic field (Fig. 8). AMCO is thus able to offer an accurate modal analysis of milled ore concentrates, as well as whole-rock samples, automatically discounting the regions of resin from the sample.

Acknowledgements

This study has been undertaken as a UPM Grade Thesis and as a contribution to one of the scheduled tasks of AMCO (EIT Project no. 15039. 2016-2018), R+D+i Project funded by EIT Raw Materials, EU-H2020 Program. The colleagues of the AMCO Consortium (particularly TSL and E. Pirard and his ULiège team) and of the Sample Preparation Lab of the Department of Geochemistry and Petrology (Faculty of Geological Sciences, Madrid Complutense University) are thankfully acknowledged.

EIT RawMaterials is supported by the EIT,
a body of the European Union

References

Castroviejo R, Brea C, Pérez-Barnuevo L, Catalina JC, Segundo F, Bernhardt HJ, Pirard E (2009) Using computer vision for microscopic identification of ores with reflected light: preliminary results. Pp. 682-684, in: Williams et al., eds. Smart Science for exploration and Mining (Proc.10th biennial SGA Meet. Vol. 2. Townsville, Australia), ISSN: 9780980558685.

Catalina JC and Castroviejo R (2017) Microscopía de Reflectancia Multiespectral: Aplicación al Reconocimiento Automatizado de Menas Metálicas. Rev. Metal. 53 (4):1-20. ISSN 0034-8570. doi: http://dx.doi.org/10.3989/revmetalm.107.

Criddle A (1990) Microscope photometry, reflectance measurement, and quantitative colour. Chapter 6, pp. 135-169, in Jambor JL and Vaughan DJ, editors: Advanced Microscopic Studies of Ore Minerals. MAC Short Course 17, Ottawa.

Delbem ID, Galéry R, Brandao PRG, Peres AED (2015) Semi-automated iron ore characterisation based on optical microscope analysis: quartz/resin classification. Miner. Eng. 82:2-13.

Grunwald-Romera, U (2017) Detección de ganga en concentrados de planta por microscopia óptica de reflexión mediante métodos automatizados. Proyecto Fin de Carrera / Trabajo Fin de Grado, E.T.S.I. de Minas y Energía (UPM). http://oa.upm.es/48414/

Neuman, R and Stanley, C J (2008) Specular reflectance data for quartz and some epoxy resins - Implications for digital analysis based on reflected light optical microscopy. Ninth International Congress for Applied Mineralogy, Brisbane, pp. 703-705.