Color electron microprobe cathodoluminescence of Bishunpur meteorite compared with the traditional optical microscopy method

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Amanda Araujo Tosi
Mestre
Universidade Federal do Rio de Janeiro - UFRJ
Instituto de Geociências
Departamento de Geologia e Paleontologia
Rio de Janeiro – Rio de Janeiro – Brasil
amandatosi@hotmail.com

Maria Elizabeth Zucolotto
Professor Associado
Universidade Federal do Rio de Janeiro - UFRJ
Instituto de Geociências
Departamento de Geologia e Paleontologia,
Museu Nacional
Rio de Janeiro – Rio de Janeiro – Brasil
meteoritos@mn.ufrj.br

Julio Cezar Mendes
Professor-Titular
Universidade Federal do Rio de Janeiro - UFRJ
Instituto de Geociências
Departamento de Geologia
Rio de Janeiro – Rio de Janeiro – Brasil
julio@geologia.ufrj.br

Isabel Ludka
Pesquisadora
Universidade Federal do Rio de Janeiro - UFRJ
Instituto de Geociências
Departamento de Geologia e Paleontologia
Rio de Janeiro – Rio de Janeiro – Brasil
ludka@geologia.ufrj.br

Fernando de Souza Gonçalves Vasques
Geólogo contratado
Centro de Tecnologia Mineral – SCT (Centro de Caracterização Tecnológica)
Rio de Janeiro – Rio de Janeiro – Brasil
fernandovasques@gmail.com

Abstract

Cathodoluminescence (CL) imaging is an outstanding method for sub classification of Unequilibrated Ordinary Chondrites (UOC) – petrological type 3. CL can be obtained by several electron beam apparatuses. The traditional method uses an electron gun coupled to an optical microscope (OM). Although many scanning electron microscopes (SEM) and electron microprobes (EPMA) have been equipped with a cathodoluminescence, this technique was not fully explored. Images obtained by the two methods differ due to a different kind of signal acquisition. While in the CL-OM optical photography true colors are obtained, in the CL-EPMA the results are grayscale monochromatic electronic signals. L-RGB filters were used in the CL-EPMA analysis in order to obtain color data. The aim of this work is to compare cathodoluminescence data obtained from both techniques, optical microscope and electron microprobe, on the Bishunpur meteorite classified as LL 3.1 chondrite. The present study allows concluding that 20 KeV and 7 nA is the best analytical condition at EPMA in order to test the equivalence between CL-EPMA and CL-OM colour results. Moreover, the color index revealed to be a method for aiding the study of the thermal metamorphism, but it is not definitive for the meteorite classification.

Keywords: Bishunpur Meteorite, Colour Cathodoluminescence, Electron Microprobe.

1. Introduction

Cathodoluminescence (CL) is the emission of light resulted from the excitation of a target by an electron beam. The wavelength of the light emitted is related to the composition and crystal structure of the studied material. When excited, electrons of the sample move to higher energy levels, and then, promptly return to a lower energy level, resulting in emission of photons in the UV, visible, and/or IR regions of the electromagnetic spectrum (Gotze *et al.,* 2013). Cathodoluminescence is the most common luminescence phenomena used for studying minerals (Pagel *et al.,* 2000), and this technique is frequently used to classify some kinds of rocky meteorites.

The classification of meteorites is based on their chemistry, oxygen isotopes, mineralogy, and petrography. The bulk composition, mineralogy, and petrology of a meteorite are functions of the original bulk composition of its parental body and the amount of heating and melting it has experienced (Burbine *et al.,* 2002). Thus, they are set on a specific group according to these features. First, meteorites can be divided into two major categories: unmelted (unfractionated, undifferentiated) and melted (fractionated, differentiated) meteorites. The unmelted, or chondrites, are all stone meteorites with element composition close to that of the solar photosphere, minus the highly volatile elements (Weisberg *et al.,* 2006), whereas melted meteorites, or achondrites, cover a range of stone, stony-iron to iron. Bridging the both division are the primitive achondrites, which have an unfractionated composition, but showing textures that indicate they have been strongly heated, if not melted (Grady *et al.,* 2014).

Chondritic meteorites consist of metal grains (Fe, Ni alloy), iron sulfide (FeS) and silicates in the form of matrix and chondrules (Sears and Hasan, 1987), although some chondrites lack chondrules (Weisberg *et al.,* 2006), such as the meteorites classified as CI carbonaceous chondrites. A majority of chondrites also have refractory inclusions in their bulk, which include Ca,Al-rich inclusions (CAIs) and amoeboid olivine aggregates (AOAs) (Krot *et al.,* 2009). Chondrules are millimeter size aggregates of silicates that had an independent existence prior to the incorporation in the meteorite (Sears and Hasan, 1987). They are subspherical objects that formed as total or partial melts or by sintering of mineral grains (Hutchison, 2004). The achondrites are the largest class of differentiated meteorites and include meteorites from the asteroid belt, the Moon and the planet Mars (Norton and Chitwood, 2008). They are formed by igneous rock (melts, partial melts, melt residues) or breccias of igneous rock fragments from these bodies. Some meteorites have achondritic (igneous or recrystallized) textures, but retain a primitive chemical affinity to their chondritic precursors. They are referred to as primitive achondrites because they are nonchondritic, but are thought to be closer to their primitive chondritic parent than other achondrites (Weisberg *et al.,* 2006). Knowing the group in which the meteorites belong has a great significance, since it can indicate that meteorites with similar characteristics are from the same parental body as asteroids, comets, planets or moon.

Based on their textures and chemical compositions, Van Schmus and Wood (1967) proposed a petrographic classification for chondrite meteorites, ranging from 1 to 6, being type 3 the most primitives. These primitive chondrites experienced little or no metamorphic heating, showing relatively unaltered fabric. This is attested by the presence of well-defined chondrules and heterogeneous chemistry of the minerals. According to these characteristics, they are named as Unequilibrated Ordinary Chondrites (UOC). Sears *et al.* (1980) subdivided the UOC or petrologic type 3 meteorites into 10 detailed divisions ranging from 3.0 to 3.9, which encompass all variation in the metamorphic properties based on features of soft thermal metamorphism, revealed by the thermoluminescence technique. The sensitivity of the thermoluminescence is revealed with the presence of feldspar, which is formed by devitrification of the glass, present in the most primitive meteorites, during the thermal metamorphism. The cathodoluminescence technique also enables to observe these mineralogical changes. In this way, Sears *et al.* (1990) related the CL color results of a pair of meteorites with their previous petrological classification, including some of the subtypes 3 as well as types 4 and 5 of chondrites. Taking into account the diversity of chondrule composition during the studies of the CL analysis, Sears *et. al.* (1992) sorted the chondrules into bright CL group and little or no CL group, leading the CL properties to be correlated with composition and metamorphism. Thus, these works were the starting point for the petrologic scheme considering CL results, in which CL became an important response to define the subtypes of these primitive meteorites.

Meteorites classified as 3.0 are considered the most primitive and 3.9 are the most metamorphosed UOC meteorites. Most common meteoritic investigations by CL images have been done through CL device attached in an Optical Microscope (OM). Taking into account the development of the use of CL effects in minerals of meteorites, this work deals with CL results obtained on the Bishunpur chondrite meteorite by using Optical Microscopy (OM) and Electron Microprobe (EPMA) techniques. Gotze and Kempe (2008) presented a detailed comparison between CL images obtained by optical and electronic microscope, exposing pros and cons of each one, but this comparison was done only based on gray scales released by electronic signal from a SEM.

Although the CL-OM is cheaper, the greatest advantage of CL obtained from electronic microscope is the simultaneous acquisition of images such as secondary electron (SE), back-scattered electrons (BSE), as well as the quantitative analysis using wavelengths dispersive spectroscopy (WDS) detectors. Another advantage is the scanning system of electronic microscopes that allows an automatic analysis of the whole section whereas in optical microscope, it is necessary to take pictures of different regions in the same section to produce a photomosaic, considering optical microscopes without the motorized stage. All the techniques have their pros and cons, therefore this work intends to provide new alternatives to obtain the cathodoluminescence response, according to goals and available equipment.

2. Materials and methods

The object of the study is the meteorite Bishunpur, classified as Unequilibrated Ordinary Chondrite, petrologic type 3.1 with low free iron and low total iron, whose reference is the abbreviation LL. This meteorite has already been studied massively by many researchers, including Akridge *et al.* (2004), who carried out analysis of cathodoluminescence in 60 sections of different meteorites in order to develop a broad study about CL colors in different degree of thermal metamorphism of these asteroidal bodies. The image of the Bishunpur meteorite obtained by these authors was taken as a guide to make the comparisons between both of the techniques used to get CL images in this work.

Cathodoluminescence can be observed on a wide variety of electron beam instruments. Our CL images were produced by two different ways: a CL apparatus attached to an Optical Microscope (CL-OM) and an Electron Microprobe with a CL detector (CL-EPMA). Both techniques have a hot cathode system, although there are also instruments that operate with a cold cathode system.

The optical microscope used was a Zeiss Axio Imager 2 with CITL MK5-2 as an electron beam source (Figure 1a). Color slides of the luminescent features were taken by means of an adapted digital camera Zeiss AXIOCAM HRc. The electron beam was typically operated at 15 keV and 0.7 mA, the same analytical condition of Akridge *et al.* (2004). In this way, the digital camera records the real color emitted by the minerals as luminescence response. From the CL images obtained from each piece of the meteorite, it was possible to create a photomosaic through the Adobe-Bridge CS6-64bit and the final treatment of the image was done in Adobe-Photoshop CS6-64bit software (Figure 2a).

To proceed the analysis in the electron microprobe, it was necessary to apply a thin carbon coating on the surface of Bishunpur because it is an insulating sample, so that this coating avoids electron-charging effects. The measurements of CL-EPMA were performed in a microprobe Jeol JXA8230 and CL signals are caught by a photomultiplier R955P attached to the CL detector Jeol XM-Z09013TPCL (Figure 1b). In this system the radiation is not split in color wavelengths, as in the optical microscope, thus, in this case, the system is a panchromatic catodoluminescence with gray scales. The first analytical conditions were 15 keV acceleration voltage, 70 nA beam current and 1 µm of beam diameter. To establish a comparison between both techniques, it was necessary to obtain colours from the CL-EPMA results, so L-RGB filters were also used (Figure 1c). CL-EPMA generated 4 images separately, each one corresponding to Luminance, Red, Green and Blue filters. Afterwards, these images were merged to create just one L-RGB image in order to observe the luminescence colour emitted by the minerals. This technique applied to obtain CL response requires L-RGB filters to produce colors that are related to the energy of electromagnetic spectrum released by the minerals after the bombardment of electrons. The Maxim DL 5 software was used to merge the L-RGB images, the same applied in astrophotography field, in which it is possible to define the ratio among L, R, G and B images from these filters. The ratio found to achieve an approximate CL-OM color was L=100%, Red =10, Green = 5 and Blue =3 (Figure 2b).

A piece of Bishunpur meteorite was examined with different analytical conditions in the EPMA in order to aproximate the OM-CL colors. The new condition was a higher acceleration of electron beam at 20 KeV and lower current at 7.0 nA (Figure 3c).

3. Results

Figure 2a shows the cathodoluminescence result of the Bishunpur meteorite produced by CL-OM, featuring a variety of colors, mainly red, blue, green, yellow, and just a little purple. Many chondrules on Bishunpur exhibit luminescence with some of these colors, whereas some chondrules have no luminescence or dull color. The bulk surrounding the chondrules (matrix) does not have luminescence response, showing up dark areas on the image. There are different luminescence responses in the chondrules. Some chondrules exhibit luminescence in the minerals and mesostasis (bulk surrounding minerals into chondrules), and others show luminescence only in minerals or mesostasis.

Figure 2b is the L-RGB image got from CL-EPMA analysis. We can notice the similarity between both results with some differences though. For instance, red chondrules in the CL-EPMA are less intense than in the CL-OM image, as well as the blue ones. There are few red chondrules that were not detected by electron microprobe analysis or they exhibit purple rather than red color. Yellow color appeared on just few chondrules in the CL-OM, however it was not detected on the result by EPMA.

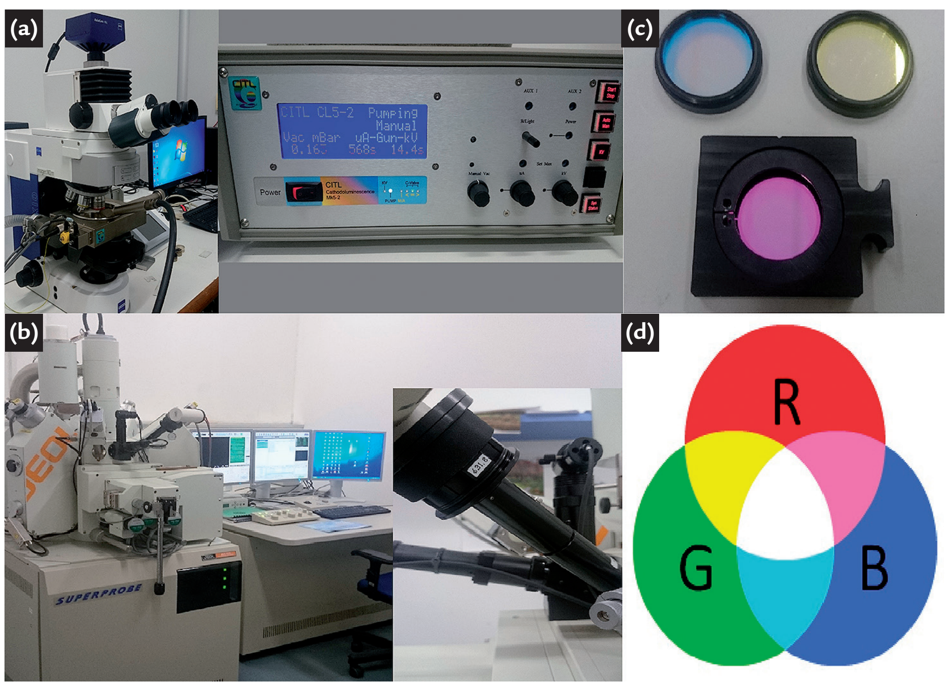


Figure 1
a) Optical Microscope Zeiss at left and the source of electron beam CITL at right, from Mineral Technology Center – CETEM; b) Electron Microprobe Jeol at left and CL detector Jeol at right, from Federal University of Rio de Janeiro – UFRJ; c) RGB filters Astronomik Type 2c used in the CL-EPMA analysis; d) Additive Color System in which the CL-EPMA result is based on, where R is Red, G is Green and B is Blue color.

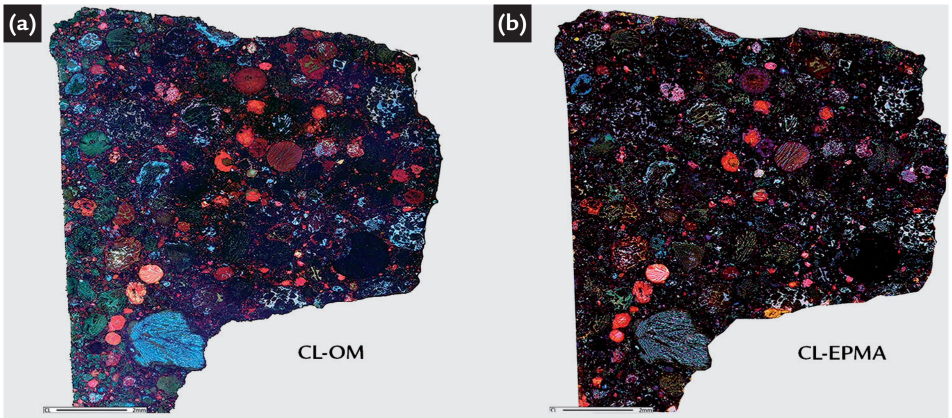


Figure 2
a) Photomosaic of cathodoluminescence of Bishunpur meteorite acquired by Optical Microscope from CETEM. The true color in the result is the strongest feature of this way of acquisition. In this image, it is possible see many colours as red, green, blue and yellow, most of them are important for classifying the meteorites by cathodoluminescence analysis. b) L-RGB image of cathodoluminescence of Bishunpur meteorite acquired by Electron Microprobe from UFRJ.

Nevertheless, many chondrules and matrix of Bishunpur meteorite obtained the same luminescent responses when excited by an electron beam. In both results, we can notice similarities such as: mostly green chondrules and luminescent green traces into the same chondrules; a great number of red chondrules appeared with a strong intensity color, although a higher intensity was detected in the CL-OM, as mentioned above; and finally the lack of luminescence of matrix confirmed by EPMA imaging.

Trying to solve a lack of yellow in the CL-EPMA result, a different analytical condition (20 KeV and 7.0 nA) was applied to measure the luminescence of meteorite. Figure 3 shows the comparison between the luminescence of the slice of Bishunpur meteorite got at CL-OM and the same slice measured in the EPMA at 20 KeV and 7.0 nA. This part of the meteorite was chosen because it presents the main colors used on the Sears system. Under the new conditions, all colors showed up, including the yellow into the chondrule. The red and blue colors appeared in the same chondrules in both results; however, the green one was less brilliant in the CL-EPMA than in the CL-OM.

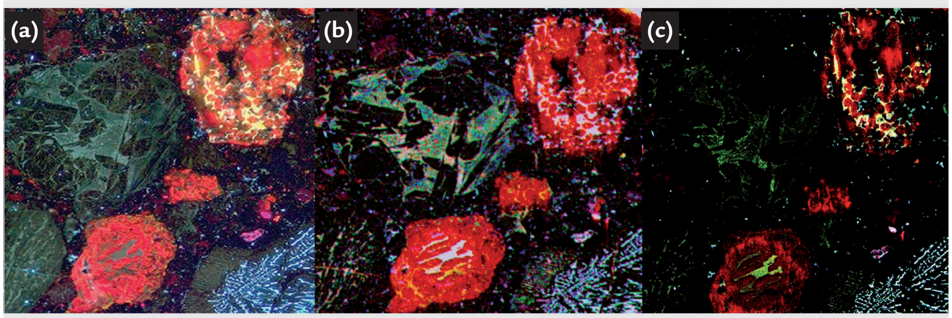


Figure 3
a) CL-OM image: a slice of Bishunpur meteorite took from the whole CL analysis showed in Figure 2a. b) CL-EPMA image: a slice of the Bishunpur took from the whole CL analysis acquired with the first analytical condition at EPMA. c) CL-EPMA image: Analysis of the same slice using the new analytical condition to get the yellow CL signals. It is possible to notice that the first condition applied at EPMA was not suitable to reveal the yellow signal. In contrast, with the new analytical condition, one can see the colors red, blue, green and yellow into the chondrules such as displayed in the CL-OM image.

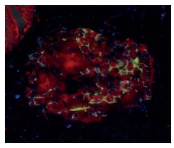
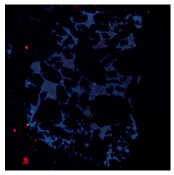
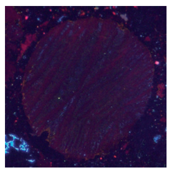
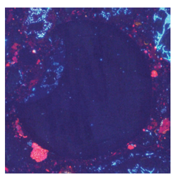
4. Discussion

It is outside the scope of this work to discuss the reasons of color in minerals because its origin has been investigated for a long time. Despite the fact that color in minerals is caused by either crystal-structure defects or trace elements (Reed, 2005), there are several mechanisms that can contribute to the coloring of gems (Fritsch and Rossman, 1987); thus, in most of the cases, it is very difficult to set a reason for the color emitted by these materials.

Regardless of the lack of certainty about the origin of the color, we can point some causes on the CL results of meteorites. One of them is the presence of Fe^{2+} in mineral lattices, such as olivines and pyroxenes. The luminescence is dependent on the amount of this ion because it acts as a quencher (Meier et al., 2003), in other words, it can reduce or eliminate the luminescence. Minerals that exhibit luminescence either is Fe^{2+} poor or do not have this ion in the chemical composition. On the other hand, some ions act as an activator of luminescence when they are included as trace elements on the lattice. For example, the transition metal Mn^{2+} is a very prominent activator element in silicates which compose the chondrules, such as olivines, pyroxenes and feldspars. These minerals contain cations of the alkaline earth metals (Mg^{2+}, Ca^{2+}) in which Mn^{2+} easily substitutes any of these ions (Ramseyer and Mullis, 2000). As example, the blue-green region of the spectrum has been commonly associated with Mn^{2+} impurity in the plagioclase, however Ca-rich feldspars exhibit yellow CL, suggesting a different crystallographic site for Mn^{2+} (Sears *et al.*, 2013), such as exemplified by CL colours in some chondrules of the Bishunpur shown in Table 1. According to the same work, synthetic forsterites doped with Mn have red CL colour.

In general, olivines and pyroxenes do not exhibit luminescence trends due to the presence of Fe^{2+} in their crystal structure. However, forsterite and enstatite, commonly found on UOC, produce an outstanding red cathodoluminescence signal, whereas fayalite and ferrosilite are below 1 mol% in the minerals composition (Sears *et al.*, 2013).

Table 1
Chondrules of Bishunpur meteorite with different CL colours response. The colour is associated with the mineralogy and impurities, such as activators and quenchers ions in the crystal structure. Quantitative analyses were obtained by WDS spectrometer from the Jeol EPMA at 15 KeV and 20 nA. Values in wt.%.

Chondrules	Mineralogy/Petrografy	Activator	Quencher
	**Granular Chondrule of Fe-poor olivine in red CL signal and brilliant yellow CL in feldspar glass mesostasis** Olivine: Al_2O_3 0.04/SiO_2 43.07/MgO 56.18/FeO 0.78 CaO 0.15/CrO_3 0.28/MnO 0.03/Total 100.53% Mesostasis: Al_2O_3 22.14/SiO_2 51.80MgO 4.35/FeO 2.15/CaO 15.03 Na_2O 2.55/Mn 0.10 K_2O 0.20/Cr_2O_3 0.24/TiO_2 1.50 Total: 100.7%	Cr^{3+} Oliv. = 0.28% Mes. = 0.24% Mn^{2+} Oliv. = 0.03% Mes. = 0.10%	Fe^{2+} Oliv. = 0.78% Mes. = 2.15%
	**Porphiritic Chondrule of FE-rich olivine in dark grains or no CL signal and blue CL in fedspar glass mesostasis** Olivine: Al_2O_3 0.01/SiO_2 40.37/MgO 46.26/Fe 13.14 CaO 0.08/Cr_2O_3 0.21/MnO 0.33/Total 100.41% Mesostasis: Al_2O_3 16.91/SiO_2 61.90/MgO 0.74/FeO 9.96/CaO 3.88 Na_2O 9.14/MnO 0.41/Cr_2O_3 0.03/TiO_2 0.65/SO_3 0.03 Total: 103.72%	Cr^{3+} Oliv. = 0.21% Mes. = 0.03% Mn^{2+} Oliv. = 0.33% Mes. = 0.41%	Fe^{2+} Oliv. = 13.14% Mes. = 9.96%
	**Radial Pyroxene Chondrule of Fe-poor pyroxene in dark red CL signal and dull CL in the feldspar glass mesostasis** Pyroxene: Al_2O_3 3.75/SiO_2 61.87/MgO 31.20/FeO 2.32 CaO 0.57/Cr_2O_3 0.54/MnO 0.44/Total 100.67% Mesostasis: Al_2O_3 10.67/SiO_2 64,48/MgO 11.14/FeO 2.15/CaO 3.66 Na_2O 1.82/MnO 0.40/Cr_2O_3 0.49/TiO_2 0.36/SO_3 1.11 Total: 96.45%	Cr^{3+} Oliv. = 0.54% Mes. = 0.49% Mn^{2+} Oliv. = 0.44% Mes. = 0.40%	Fe^{2+} Oliv. = 2.32% Mes. = 2.15%
	**Chondrule of Fe-rich pyroxene in dark area or no CL signal** Pyroxene: Al_2O_3 0.54/SiO_2 54.62/MgO 24.37/FeO 19.31 CaO 0.66/Cr_2O_3 0.60/MnO 0.49/Total 100.10%	Cr^{3+} 0.60% Mn^{2+} 0.49%	Fe^{2+} 19.31%

Regarding the color as a response of minerals luminescence under an electron beam, the CL-OM provides true colors, in which the color emitted by the mineral is correspondent to the amount of energy of the electromagnetic spectrum. These colors either can be seen through objective lenses or they can be recorded by a CCD camera attached to the microscope. In contrast, the electronic signal produced by CL-EPMA passes by selective filters red, green and blue. Each panchromatic image provides the intensity of luminescence of the minerals in respect to the energy range of the applied filter. The final color of CL through this method is a result of an additive color system. These selective filters enable color generation, however at the expense of intensity (Steele, 1990). Probably some luminescent chondrules in CL-OM do not appear in CL-EPMA due to the low intensity signal suppressed by RGB filters.

In order to summarize color trends in a quantitative way by defining the "color index" using the ImageJ software, the whole image was selected, as well as the mean color values and standard deviation record for the red, blue and green pixels. The color index (CI) is the relative proportion of blue pixels to red pixels in digital images. According to Akridge et al. (2004) the color index increases as a function of petrologic type as forsterite is destroyed and feldspar crystallizes in response to metamorphism. Table 2 displays the comparison between the Color Index of Bishunpur in literature and the Color Index that was found on the CL-EPMA image.

Table 2
Blue to Red Color Index (ratio of the blue pixels to red pixels in digital images) for Bishunpur Meteorite. Values of CI obtained by CL-EPMA and CL-OM in this work and the CL-OM available in literature.

Bishunpur	Red		Green		Blue		Color Index
LL 3.1	Mean	σ	Mean	σ	Mean	σ	Blue/Red
CL-OM Akridge *et al.* (2004)	46	48	42	34	38	37	0.82
CL-EPMA (from this work)	28	71	21	60	23	65	0.82
CL-OM (from this work)	27	32	24	25	34	30	1.25

In order to investigate the lack of color using the electronic microscopy system, the sample was submitted to higher electron beam energy, increasing the voltage from 15 KeV to 20 KeV and decreasing the beam current from 70 nA to 7 nA. This new condition was applied according to Reed (2005), who claims that excitation of CL is not very sensitive to the beam accelerating voltage, but sometimes it is advantageous to use a relatively high value (at least 20 kV). It is because this enables the electrons to penetrate the non-luminescent damaged surface layer. The result from these analytical changes was just the appearance of yellow inside the chondrule, as shown in Figure 3c.

5. Conclusion

By far, this is the best CL analytical condition at EPMA to correlate with the results obtained by CL-OM is 20 keV aceleration voltage, 7 nA beam current, and 1 µm of beam diameter.

Although the acquired colors do not fully match in both methods, as well as the mean values of pixels in each filter, the color index revealed to be equivalent in CL-OM (Akridge *et al.*, 2004) and CL-EPMA from this work. The CI of CL-OM is far from the mean value 0.82. However, the standard deviation values of the measure are very high, leading us to conclude that this evaluation method can be an aid to study the thermal metamorphism, but it is not definitive for the meteorite classification.

In our efforts to extend cathodoluminescence measurements of meteorites to the electron microscopy field, we have achieved results that revealed the possibility of equivalence of both technique methods on the petrological classification of meteorites in the near future. The correlation of cathodoluminescence signals in the Bishunpur meteorite, by using optical microscopy and electron microprobe, offers considerable scope for more studies and new applications in the Meteoritic Science, since only OM has been used to observe this kind of radiation. However, such approach faces technical challenges, since the electron microscopy provides an electronic signal which must be converted to a color signal.

This research will continue in order to further refine and confirm our findings. Therefore, the next steps will be to find a reproducible protocol, whose major challenges are: reaching the best analytical condition at CL-EPMA and establishing just one ratio among the L-RBG imaging filters.

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