Non-Destructive Micro-Chemical and Micro-Luminescence Characterization of Jadeite

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Abstract: Jadeite was greatly appreciated by pre-Hispanic cultures in Mesoamerica. Despite its importance, knowledge of its mining sources was lost after the Spanish conquest. In the 1950s the only known jadeite deposits in Mesoamerica were found in the Motagua River Fault (MRF), Guatemala. The aim of this study is to present a methodology that is appropriate for the study of archeological jadeite objects using non-destructive spectroscopic and micro-ion beam analysis techniques. This methodology has been applied to perform mineral, elemental, and luminescence characterization of five jadeite samples from the MRF, with white, lilac, and green colors. Fourier-transformed infrared spectroscopy and X-ray diffraction analysis confirmed the presence of jadeite, albite, and omphacite as the main mineral phases in the samples. Elemental maps using particle-induced X-ray emission (PIXE) with a nuclear microprobe and elemental concentration analysis from individual mineral grains using micro-PIXE coupled with micro-ionoluminescence (IL) allowed the detection of minor feldspar, titanite, and grossular mineral contents. Distinctive features from the mineral, elemental, and luminescence characterization have been found that allow the identification of these five jadeite samples.

Key words: jadeite, PIXE, Ionoluminescence, FTIR, XRD

INTRODUCTION

Jadeite (NaAlSi₂O₆) is a hard and dense monoclinic metamorphic pyroxene that forms under high-pressure and low-temperature conditions (5–11 kbar, 150–400°C), which are normally found in tectonic subduction zones with fluids (Johnson & Harlow, 1999). Due to these special conditions, only a few geological sources exist around the world (Howard, 2001).

Jadeite can be found in solid solution with other pyroxenes: omphacite [(Ca,Na)(Mg,Fe²⁺,Al)Si₂O₆], aegirine (NaFe³⁺ Si₂O₆) and hedenbergite (CaFeSi₂O₆) among them. Jadeite rocks can also present a large amount of associated mineral species (Hauff, 1993), including those related to blueschist facies that originate from basalt rocks, like albite (NaAlSi₃O₈), titanite (CaTiSiO₅), and garnets. Owing to variations in formation processes, jadeites from different sources—and even sources within the same area—present a diverse mineral and elemental composition (Harlow et al., 2011; Flores et al., 2013).

By virtue of its color, hardness, high damage resistance, and a less brittle nature in comparison with other stones, jadeite was greatly treasured by pre-Hispanic cultures, and was traded all around Mesoamerica over a span of two millennia (Roca Cogordan, 2012). Because of the difficulty in carving such a hard rock and the scarcity and remoteness of its sources, jadeite symbolized prestige and was mainly reserved for the political and religious elite (Kovacevich, 2011).

As the Spaniards were primarily interested in gold and silver, the millenary tradition of jade carving perished soon after the Spanish conquest in the early 16th century, and knowledge of the location of its mining sources, as well as its manufacturing technology, was lost.

The only known jadeite deposits in Mesoamerica were found during the 1950s in the Motagua River Fault (MRF), located at the boundary between the North American and Caribbean tectonic plates in eastern Guatemala (Hargett, 1990; Taube et al., 2004).

Common analytical techniques employed in the study of jadeite material are petrography, scanning electron microscopy (SEM) and X-ray powder diffraction, which yield a complete mineral characterization (Harlow et al., 2011; Wang et al., 2012). However, the invasive nature of these techniques makes them inappropriate for the study of precious archeological artifacts. The aim of this paper is to present a methodology that is appropriate for the study of such archeological jadeite objects, using non-destructive spectroscopic and micro-ion beam analysis (micro-IBA) techniques. As an application of this methodology, we perform mineral, elemental, and luminescence characterization of five jadeite samples from the MRF, with colors ranging from white, lilac, and light to dark green.

Mineral characterization of the samples was carried out by means of Fourier-transformed infrared (FTIR)
spectroscopy and X-ray diffraction (XRD). As FTIR and XRD on solid samples only allowed us to determine major mineral content, particle-induced X-ray emission (PIXE) elemental maps obtained with a scanning nuclear microprobe was used to identify grains from minor mineral content. Afterwards, punctual analysis by micro-PIXE and micro-ionoluminescence (micro-IL) techniques implemented simultaneously with an ion microprobe provided characteristic elemental concentrations and luminescence spectra from individual mineral grains in the sample. Backscattered electron (BSE) images coupled with scanning electron microscopic energy-dispersive X-ray spectroscopy (SEM-EDS) elemental analysis were used to confirm the mineral identification done by the previous techniques, as BSE images with SEM-EDS offer complementary information for better identification of individual mineral grains.

IBA techniques have a long history in cultural heritage studies (Dran et al., 2004). The use of these techniques provides several advantages, including: multi-elemental analysis with low detection limits (in the µg/g range) by means of PIXE, with a high X-ray yield for the identification of light elements (Verma, 2007), the ability to perform non-destructive and non-invasive analyses in air (Calligaro et al., 2000), and the generation of an intense IL signal.

IL produced in minerals mainly originates from structural defects and specific elemental impurities found in the material (Townsend, 2012). As such, IL is complementary to PIXE, providing information useful for identification of the emitting mineral (Calvo del Castillo et al., 2007). The IL signal also provides information on the condition of the crystal structure and the ionization state of its impurities. Thus, IL is sensitive to formation and alteration processes of minerals (Yang et al., 1997; Götze, 2002).

The results presented here can also provide some insight in the interpretation of luminescence spectra obtained from jadeite samples, as there are disagreements between authors in the scarce literature available on jadeite luminescence (Dopfel, 2006; MacRae & Wilson, 2008; Ruvalcaba-Sil et al., 2008; Tuncer Arslanlar et al., 2011; Pichon et al., 2015).

Distinctive features from all three physical properties have been found that allow the identification of these five jadeite samples. Mineral, elemental, and luminescence characterization of the analyzed samples are presented for future studies.

**MATERIALS AND METHODS**

The five analyzed jadeite samples shown in Figure 1 were obtained from the Mesoamerican Jade Museum in San Cristobal de las Casas, Mexico, who acquired these specimens from the MRF zone in Guatemala. The museum has catalogued the Jungle (J) and Light Green (LG) varieties as easily accessible, and the White (W), Lilac (Li), and Moon (M) varieties as rare. They present a granular crystalline structure and diverse colors from white, lilac, and light to dark green. The samples were cut into a 20 × 15 × 2 mm³ flat cuboid shape, and one of their faces was polished by rubbing bauxite (aluminum hydroxide) particles with a 600 caliber diamond polishing disc, giving a flat polished surface for analysis. All of the analyses in this study were performed at room temperature and no further treatment was applied on the samples.

The identification of main mineral content in the samples was carried out by FTIR spectroscopy using an Alpha Bruker spectrometer (Bruker Corporation, Massachusetts, USA) equipped with a reflectance module, with a range from 400 to 4,000 cm⁻¹, and a 4 cm⁻¹ resolution. The area of analysis was 5 mm². FTIR spectra obtained were compared with mineral reference spectra from the Ostrooumov database (Ostrooumov, 2007).

XRD measurements were done using a Bruker D8 Discover diffractometer, equipped with a copper anode fixed at a grazing angle and a 2θ range from 10 to 90° with a 0.02⁰ step size. To improve mineral detection on solid samples, three diffractograms were acquired for each sample. Mineral identification was carried out by comparative matching based on peak position and relative intensity of peaks from equivalent planes using the PDF 4 software (ICDD, 2016).

Two-dimensional (2D) elemental distribution maps and quantitative results from punctual analyses were obtained in vacuum with the Oxford Microbeams OM150 type scanning electron microscopy-energy-dispersive X-ray spectroscopy (SEM-EDS) elemental analysis were used to confirm the mineral identification done by the previous techniques, as BSE images with SEM-EDS offer complementary information for better identification of individual mineral grains.

**Figure 1.** The five jadeite samples analyzed. The areas irradiated during micro-particle-induced X-ray emission and micro-ionoluminescence analysis at IF-UNAM are marked. Red squares indicate the areas of backscattered electron images shown in Figure 5. W, white; Li, lilac; M, moon; J, jungle; LG, light green.
nuclear microprobe setup from CTN-IST, Lisbon (Alves et al., 2000). A 2 MeV proton beam focused to a $3 \times 4 \, \mu m^2$ irradiation area and a 100 pA current was used. X-rays were collected by a Bruker AXS X-Flash 4030 X-ray detector placed at an angle of 135° from the beam direction. A 50 µm thick Mylar filter was placed on this single X-ray detector to reduce the intense X-ray signal from the Si matrix and thus improve the detection of minor elemental content; this filter also limited the detection of elements to those heavier than Mg.

Specific areas of the samples were analyzed by micro-PIXE and micro-IL techniques under vacuum conditions with the new ion microprobe installed at the Physics Institute’s (IF-UNAM) NEC 3 MV tandem pelletron laboratory. A 3 MeV proton beam focused to a $6 \times 10 \, \mu m^2$ irradiation area and a 300 pA current was used. The proton beam irradiation area used was similar to that obtainable in external nuclear microprobe setups (Calligaro et al., 2000).

In IF-UNAM’s ion microprobe, PIXE was carried out using a two X-ray detector array, both placed at an angle of 135° from the beam direction: low-energy X-rays were detected using a Ketek (KETEK GmbH, München, Germany) AXAS-A SDD, 30 mm$^2$ active area, 8 µm thick Be window. High-energy X-rays were collected with a Gresham Si(Li) detector, 80 mm$^2$ active area, 12 µm thick Be window with a 60 µm thick Al filter in front of the detector, used for attenuation of low-energy X-rays. Because of the importance to identify sodium in jadeite and albite studies, no filter was used in the low-energy detector, allowing the detection of elements down to Na. Standard sediment reference materials from NIST (SRM 2704 and SRM 2711) were also irradiated under the same conditions, and elemental concentrations were calculated using the GUPIXWIN software (Campbell et al., 2010). The elemental detection limits of this system are close to 1,500 µg/g for light elements (Na–P) and from 10 to 100 µg/g for most other elements.

Light emitted by the samples was collected with an Ocean Optics (Ocean Optics Inc., Florida, USA) 74UV-series collimating lens placed at an angle of 150° from the beam direction and connected via a 50 cm long and 0.7 mm thick optical fiber to an Ocean Optics HR4000 spectrometer with a 5 µm entrance slit and a HC-1 grating. The spectral resolution of the whole system was measured at 3 nm. The acquired IL spectra were corrected for the spectral response of our system. Absorption from the optical fiber caused the efficiency of our system to fall below 10% in the high-energy ultraviolet (UV) spectrum, giving our system a useful spectral range from 270 to 900 nm. A 20 s spectral integration time was used. An optical microscope provided visual observation of the area of analysis.

The micro-IL spectra obtained were smoothed using a Fast Fourier Transform (FFT) filter with a 0.01969 cutoff frequency. Spectral deconvolution was carried out using the Gaussian curve fitting function from OriginPro 8.5 data analysis software (OriginLab Corporation, Northampton, MA, USA). As IL is similar to other more established luminescence techniques such as photoluminescence, cathodoluminescence, and thermoluminescence (TL), the experience from these techniques and their large data bases were used for analyzing the spectra obtained (Pallon et al., 1997; Townsend, 2012).

A Hitachi TM 3030 Plus tabletop scanning electron microscope (Hitachi, Ltd., Tokio, Japan) operated at 18 kV was used to acquire BSE images coupled with SEM-EDS analysis of the areas analyzed previously by micro-PIXE and micro-IL.

**Results**

**Mineral Identification**

FTIR analyses shown in Figure 2 provided the identification of albite in the white sample, jadeite in the moon sample, a combination of albite and jadeite in the lilac and jungle samples, and jadeite with uncertain presence of omphacite in the light green sample, which displays two strong vibrational bands at 960 and 1,060 cm$^{-1}$ (Mei et al., 2003).

XRD measurements confirmed all of the minerals identified by FTIR, and it also detected the presence of diffraction peaks associated with other minerals and phases. For the white sample, apart from albite, a calcian albite phase (plagioclase) was clearly observed, and jadeite was observed in one of the diffractograms taken. For the moon, jungle, and light green samples, a calcian–ferroan variety of jadeite was also observed, in which CaFe$^{2+}$ substitutes for NaAl in the jadeite–hedenbergite (Jd–Hd) join.

As FTIR and XRD on solid samples only allowed the identification of major mineral phases, 2D PIXE elemental maps (Figs. 3, 4) together with punctual quantitative results obtained from nuclear microprobe analysis suggest the
presence of minor mineral phases associated with the minerals previously identified. For the white and lilac samples, the presence of alkali (potassium bearing) and plagioclase (calcium bearing) feldspar solid solution was observed. Also, areas with elemental concentrations for both calcium and titanium from 16 to 19 wt% have been associated with titanite (CaTiSiO$_5$), an accessory mineral found in both feldspars and pyroxenes (Ng et al., 2016).

In the green samples, the association of calcium with iron rich areas confirms the presence of a calcian–ferroan
jadeite (Jd–Hd), as identified by XRD. The Ca and Fe rich areas in the jungle sample are also correlated with higher Mn content. The moon sample was very homogeneous, with smooth variations on its Ca and Fe content that were barely noticeable in the elemental maps.

SEM-EDS analysis shown in Figure 5 confirmed the minerals detected with elemental PIXE analysis. This analysis also identified the presence of grossular garnet \([\text{Ca}_3\text{Al}_2(\text{SiO}_4)_3]\) in the white and lilac samples, a mineral that has been associated with jadeite (Wang et al., 2012).

### Elemental Composition by Micro-PIXE

Simultaneous micro-PIXE and micro-IL analysis were done using IF-UNAM’s ion microprobe. As no elemental maps were available with this microprobe system, the selection of irradiation areas for analysis was based on the visible color emitted by IL, as observed through the optical microscope.

In the white and lilac samples, the elemental concentrations obtained by micro-PIXE analysis suggested the presence of alkali feldspars, plagioclases, and grossular as shown in Table 1. The presence of intermixed jadeite and albite grains smaller than the \(6 \times 10^{\mu m^2}\) irradiation area, shown in Figure 5, and the similarity in elemental composition of the two minerals did not allow us to clearly distinguish between them using elemental information from micro-PIXE.

Table 2 presents the elemental concentrations obtained for the green samples. This analysis provided the identification of Ca and Fe poor and rich areas that suggest the presence of jadeite and Jd–Hd phases. The presence of omphacite was also identified in the dark green areas of the light green sample. The color variation of the samples from white, to light and dark green has been directly related to an increment in the iron concentration detected by PIXE in these samples, and with omphacite in the light green sample.

### Luminescence Emission by Micro-IL

Different irradiated areas of the white and lilac samples presented characteristic IL emission spectra as shown in Figure 6. Analysis points W1, W2, Li1, and Li2 show an intense UV emission with peak maxima between 330 and 340 nm. Previous to the efficiency correction of our spectra, small green and red emissions were also observed at 490 and...
680 nm, respectively. In these points jadeite, albite, and alkali feldspars were identified by means of micro-PIXE elemental analysis and confirmed by SEM-EDS. Intense UV luminescence has been reported for alkali feldspars (García-Guinea et al., 2007), and white and purple jadeite has been reported to display an intense violet emission (Pagel et al.,

### Table 1. Micro-Particle-Induced X-Ray Emission Elemental Concentrations for the White and Lilac Samples Obtained with 3 MeV Protons at IF-UNAM.

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<tr>
<th></th>
<th>Ab</th>
<th>Ab–Pl</th>
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<th>Pl</th>
<th>Kfs</th>
<th>Ab/Jd</th>
<th>Grs</th>
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<td>6.9</td>
<td>7.5</td>
<td>10.2</td>
<td>4.6</td>
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<td>Mg (%)</td>
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<td>—</td>
<td>—</td>
<td>0.4</td>
<td>—</td>
<td>0.7</td>
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<tr>
<td>Al (%)</td>
<td>10.4</td>
<td>11.0</td>
<td>10.8</td>
<td>9.3</td>
<td>15.1</td>
<td>12.7</td>
<td>13.3</td>
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<tr>
<td>Si (%)</td>
<td>32.8</td>
<td>26.9</td>
<td>18.1</td>
<td>30.0</td>
<td>26.3</td>
<td>29.0</td>
<td>15.7</td>
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<td>Ca (%)</td>
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<td>3.86</td>
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<td>5.71</td>
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<td>Fe (%)</td>
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<td>0.09</td>
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<td>0.09</td>
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<td>0.02</td>
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<td>—</td>
<td>212</td>
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<td>—</td>
<td>—</td>
<td>212</td>
<td>114</td>
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<td>K (µg/g)</td>
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<td>778</td>
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<td>Cr (µg/g)</td>
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<td>Mn (µg/g)</td>
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<td>—</td>
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<td>Ni (µg/g)</td>
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<td>—</td>
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<td>—</td>
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<td>Cu (µg/g)</td>
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<td>Zn (µg/g)</td>
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W, white; Li, lilac.
Average uncertainties are 5 and 15% for major and trace elements respectively.
Naming nomenclature for suggested minerals is found in Figures 3 and 4.

*Mineral was not clearly identified.

### Table 2. Micro-Particle-Induced X-Ray Emission Elemental Concentrations for the Moon, Jungle, and Light Green Samples Obtained with 3 MeV Protons at IF-UNAM

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<th>J3</th>
<th>LG1</th>
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<td>Al (%)</td>
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<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Zr (µg/g)</td>
<td>104</td>
<td>—</td>
<td>204</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>

M, moon; J, jungle; LG, light green.
Average uncertainties are 5 and 15% for major and trace elements, respectively.
Naming nomenclature for suggested minerals is found in Figures 3 and 4.

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2000; Tuncer Arslanlar et al., 2011), although to our knowledge this characteristic spectra has not been previously reported for white jadeite and albite.

Elemental analysis of point W4 suggests the presence of plagioclase feldspar. The IL spectra obtained from this area displays an intense peak at 598 nm, which does not agree with that reported for plagioclases at 570 nm (Götze, 2012). We have not been able to identify a mineral related to jadeite or albite that possesses an elemental composition similar to that obtained from analysis point W4 and emits an intense luminescence emission around 598 nm. An explanation may be found in a shift to higher wavelengths observed in lunar plagioclases shocked at high pressures (Gucsik et al., 2005). This emission band could also indicate the presence of small grains from an unknown mineral phase that has not been identified with the applied techniques.

The presence of grossular (from analysis points Li3 and W3) was also confirmed by its luminescence spectra in Figure 6 showing its characteristic Mn$^{2+}$ band at 588 nm, with only grossular from the white sample displaying the additional V$^{2+}$ band at 719 nm (Pagel et al., 2000; Gaft et al., 2013b).

Gaussian deconvolution of the IL spectra from point W1 in Figure 7a displays additional blue emissions around 420 and 468 nm. Luminescence centers for feldspars have been well characterized by multiple authors; the bands shown in Figure 7a have been assigned using the luminescence data for feldspars collected by Götze (2012). The UV band found at 3.9 eV (320 nm) has only been reported by TL studies (Krbetschek et al., 1997), and is attributed to defects associated with sodium (Garcia-Guinea et al., 1999).

IL spectra obtained from the green samples are shown in Figure 8. They present broad UV (with peak maxima between 330 and 355 nm), green (peak maxima around 560 nm), and red (peak maxima around 700 nm) characteristic emission bands that have been previously reported for jadeite (Pagel et al., 2000; Ruvalcaba-Sil et al., 2008; Pichon et al., 2015). The zoning of luminescence obtained within the same sample identifies sequences of crystallization during rock formation (Sorensen et al., 2006). No luminescence was detected from omphacite found in the light green sample.
Luminescence activators found in jadeite still lack a thorough study, those shown in the Gaussian deconvolution of Figure 7b are based on a previous IL analysis on jadeite samples (Ruvalcaba-Sil et al., 2008). This study assigned the UV emission band to Ce$^{3+}$ impurities emitting at 330 nm, and the green emission around 580 nm to Mn$^{2+}$ impurities substituting for Na$^+$. This same study assigned the red band around 700 nm to a combination of Al$^{3+}$, Ti$^{4+}$, and Ca$^{2+}$ impurities, whereas studies carried out at AGLAE have linked this red emission to trace amounts of manganese (Pichon et al., 2015). Spectral deconvolution of the jungle and light green samples found an additional UV emission band at 3.95 eV (313 nm), which to our knowledge has not been reported for jadeite.

Proton beam irradiation caused an exponential decay of IL signal in the UV emission for the white and lilac samples and in the UV and red emissions for the jungle and light green samples. Figure 9 shows this decay in the light green jadeite sample, with spectra acquired every $10^7$ ions/cm$^2$ (or 100 s) up to a total fluence of $2.5 \times 10^{18}$ ions/cm$^2$ (21 min). The formation of a band at 480 nm after an ion fluence of $5 \times 10^7$ ions/cm$^2$ may relate this emission to the formation of new defect centers in the crystal structure (Townsend et al., 2007).

**DISCUSSION**

Minerals identified in the samples are presented in Table 3. This table displays how the mineral identification obtained using the proposed methodology is in good agreement with the minerals identified by SEM-EDS.
Despite variability in the elemental concentrations observed from different analysis points within the jadeite samples, a tendency was observed in the average concentration of iron and manganese present in the jadeite and albite grains of the analyzed samples, as shown in Figure 10. Although not being conclusive, this tendency may provide a distinctive marker that allows identification of these five samples from an elemental point of view.

Characteristic IL spectra were also obtained from each of the five samples. The band at 716 nm from grossular in the white sample provides a clear marker differentiating it from the lilac sample. In the green samples, the overall low luminescence intensity and absence of a band at 320 nm (as observed from spectral deconvolution) are characteristics found only in the moon sample. Only the jungle sample displayed an area with a pure green emission.

The applied methodology offers advantages that prove valuable for provenance studies (Tykot, 2004) of jadeite archeological artifacts. Some of these advantages include the capability to identify major and minor mineral content, the low elemental detection limits (down to trace element concentrations), and the sensitivity to formation and alteration processes of minerals offered by IL, a technique that has found some success in provenance studies (Lo Giudice et al., 2009; Nagabhushana et al., 2012). The ability of this methodology to perform punctual analyses offers a unique advantage, as recent studies have concluded that the characterization of individual mineral grains found in heterogeneous objects provides better results in provenance studies (Re et al., 2011; Niespolo et al., 2014).

The scarce amount of samples analyzed in this study does not allow positive identification of physical properties that can be used as markers to discriminate between different sources found in MRF jadeite, as required for provenance studies. However, the unique characteristics found in the IL spectra, together with the elemental tendencies observed across the five samples, provide encouragement for a complete study on a broad range of jadeite specimens.

**Table 3.** Minerals Identified in the Five Samples by Means of the Different Non-Destructive Techniques, and with Scanning Electron Microscopic Energy-Dispersive X-Ray Spectroscopy (SEM-EDS) Analyses for Comparison.

<table>
<thead>
<tr>
<th>Techniques/Samples</th>
<th>White</th>
<th>Lilac</th>
<th>Moon</th>
<th>Jungle</th>
<th>Light Green</th>
</tr>
</thead>
<tbody>
<tr>
<td>FTIR</td>
<td>Ab</td>
<td>Ab, Jd</td>
<td>Jd</td>
<td>Ab, Jd</td>
<td>Jd, Omp*</td>
</tr>
<tr>
<td>XRD</td>
<td>Ab, Pl, Jd*</td>
<td>Ab, Jd, Pl*</td>
<td>Jd, Jd–Hd*</td>
<td>Ab, Jd</td>
<td>Jd, Jd–Hd, Omp</td>
</tr>
<tr>
<td>PIXE</td>
<td>Ab/Jd³, Pl, Kfs, Grs, Ttn</td>
<td>Ab/Jd³, Pl, Kfs, Grs, Ttn</td>
<td>Jd, Jd–Hd</td>
<td>Ab/Jd³, Jd–Hd</td>
<td>Jd, Jd–Hd, Omp, Ttn</td>
</tr>
<tr>
<td>IL</td>
<td>Ab/Jd/Kfs³, Pl³, Grs</td>
<td>Ab/Jd/Kfs³, Grs</td>
<td>Jd</td>
<td>Jd</td>
<td>Jd</td>
</tr>
<tr>
<td>SEM-EDS</td>
<td>Ab, Jd, Pl, Kfs, Grs, Ttn</td>
<td>Ab, Jd, Pl, Kfs, Grs, Ttn</td>
<td>Jd, Jd–Hd</td>
<td>Ab, Jd, Jd–Hd</td>
<td>Ab, Jd, Jd–Hd, Omp, Ttn</td>
</tr>
</tbody>
</table>

FTIR, Fourier-transformed infrared; XRD, X-ray diffraction; PIXE, particle-induced X-ray emission; IL, Ionoluminescence.

Naming nomenclature found in Figures 3 and 4.

*Mineral was not clearly identified.

Could not distinguish between minerals.

![Figure 10.](https://www.cambridge.org/core/coreimages) Manganese and iron concentrations acquired from jadeite and albite mineral grains. Concentrations obtained by micro-particle-induced X-ray emission with 2 MeV protons at Lisbon’s nuclear microprobe and with 3 MeV protons at IF-UNAM’s ion microprobe. Black symbols indicate point analyses and the colored columns present their average.
The simultaneous application of micro-PIXE with micro-IL did not allow us to establish a clear relationship between the IL intensity of the peaks observed and the elemental concentrations of the activators found in the literature, such as cerium, aluminum, or manganese. The fact that no correlation has been found may indicate that these activators are in concentrations below PIXE’s elemental detection limit (Townsend et al., 2007). It may also indicate any of the following (or a complex combination of these factors): there is a presence of additional luminescence activators, the emissions are caused by structural defects in the crystal lattice, unknown luminescence quenchers are present, or that the different crystal orientations within the sample affect polarization of the light emitted (Finch et al., 2003).

The application of time resolved IL analysis could shed new light on the identification of jadeite’s emission centers (Gaft & Panczer, 2013a). It is also believed that this technique could provide additional markers for provenance studies (Czelusniak et al., 2016).

Other improvements can be made to the proposed methodology. The inclusion of scanning mode to IF-UNAM’s ion microprobe would allow for better selection of analysis areas, based on the elemental maps obtained. The application of Raman spectroscopy would provide a good complement for the positive identification of jadeite and its associated minerals (Gendron et al., 2002; Delgado et al., 2015), particularly if used with micro-Raman mapping mode (Petriglieri et al., 2015).

**CONCLUSIONS**

Guatemalan jadeite samples were successfully characterized by means of spectroscopic and micro-IBA techniques, identifying major and minor mineral species that were also found by means of SEM-EDS analysis.

The methodology presented here allows a non-destructive mineral characterization of jadeite samples, together with elemental concentrations and luminescence spectra from individual mineral grains, providing a unique tool for the study of archeological jadeite objects.

This methodology also displayed potential for provenance studies of jadeite samples. A tendency was observed in the average manganese and iron concentrations obtained from the jadeite and albite grains, and singular characteristics were found in the IL spectra from each of the five samples. The study of a broad range of jadeite specimens from all available deposits in the MRF and from archeological sources needs to be performed to allow identification of statistically valid markers that permit the discrimination of jadeite from different sources, and to confirm validity of the proposed methodology in provenance studies of Mesoamerican jadeite.

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**References**


