Distribution and consumption of fluorite and translucent beads in the Iberian peninsula from 6th to 2nd millennia BC*

José Ángel Garrido-Cordero, Carlos P. Odriozola, Ana C. Sousa, Victor S. Gonçalves and João Luís Cardoso

ABSTRACT

Translucent minerals were valued in prehistoric societies for their rarity and socially used as highly symbolic elements. This work addresses the use and nature of Iberian translucent beads. We present the results of chemical (Raman spectroscopy, portable X-ray fluorescence, X-ray diffraction and visible (Vis)/near-infrared (NIR) spectroscopy) and contextual analyses and provide a review of the archaeological literature on the manufacture and use of translucent items during Iberian Late Prehistory. A total of 54 translucent beads from 47 sites, primarily burials, were analyzed; 33 were made from fluorite, while the remaining 21 were made of diverse translucent minerals (calcite, quartz and different silicates). The scarcity of translucent items in the archaeological record, the regional and supraregional scale of its exchange, and its recursive association to other valuables in singular contexts reinforces the idea that their owners/wearers enjoyed a high status.

Key words: Late Prehistory; Iberia; Beads; Fluorite; Translucent minerals; Neolithic; Copper Age; Bronze Age; Raman spectroscopy; XRD; NIR.

RESUMEN

Los minerales translúcidos fueron apreciados por las sociedades prehistóricas por su rareza, y fueron utilizados como elementos altamente simbólicos. Éste trabajo aborda el uso y la caracterización de las cuentas translúcidas de la península ibérica, mediante análisis químico (espectroscopia Raman, espectrómetro de fluorescencia de rayos X portátil, difracción de rayos X y espectroscopia en región visible y en infrarrojo cercano) y contextual, junto a una revisión de la bibliografía arqueológica sobre la producción y uso de adornos y elementos translúcidos durante la Prehistoria Reciente ibérica. Un total de 54 cuentas de 47 yacimientos, mayoritariamente funerarios, han sido analizadas; 33 fueron trabajadas en fluorita, mientras que las restantes 21 fueron realizadas en distintos minerales translúcidos (calcita, cuarzo y varios silicatos). La escasez de adornos translúcidos en el registro arqueológico, su escala regional y suprarregional de intercambio, y su asociación recurrente a otros elementos de prestigio en contextos singulares refuerzan la hipótesis del alto estatus de sus poseedores/portadores.

Palabras clave: Prehistoria Reciente; Península ibérica; Cuentas; Fluorita; Minerales translúcidos; Neolítico; Edad del Cobre; Edad del Bronce; Espectroscopia Raman; DRX; Espectroscopia en región visible y en infrarrojo cercano.

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Dpto. de Prehistoria y Arqueología. Universidad de Sevilla. C/ Doña María de Padilla s/n. 41004 Sevilla. Spain. E-mail: jgarrido8@us.es
https://orcid.org/0000-0002-9585-7917; CPO e-mail: codriozola@us.es https://orcid.org/0000-0002-4411-2528

UNIARQ. Centro de Arqueología da Universidade de Lisboa. Alameda da Universidade. 1600-214 Lisboa. Portugal. ACS e-mail: sousa@campus.ul.pt https://orcid.org/0000-0003-2709-3967; VSG e-mail: vsg@campus.ul.pt https://orcid.org/0000-0002-8120-5192

Universidade Aberta / Centro de Estudos Arqueológicos do Concelho de Oeiras, Câmara Municipal de Oeiras. Rua da Escola Politécnica. 1269-001 Lisboa. ICArEHB (Interdisciplinary Center for Archaeology and Evolution of Human Behaviour), Universidade do Algarve. E-mail: joao.cardoso@uab.pt https://orcid.org/0000-0003-2234-2266


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1. INTRODUCTION

In prehistoric Europe, personal adornments were made from a very diverse repertoire of rocks and minerals where green, black and white hues overwhelmingly dominate the archaeological record. However, beside this colors, transparent and translucent items of different shades stand out as rare and special items, evidencing the importance of color (Sahlins 1976; Jones and MacGregor 2002) and brightness (Gaydarska and Chapman 2008) in raw material selection and the development of a complex symbology during Late Prehistory.

Apart from the long-known social value of amber (see Odriozola et al. 2019 for a review), it was only very recently that other translucent raw materials have attracted scholars' attention. Such is the case of rock crystal (Garrido-Cordero 2015; Morgado et al. 2016) or fluorite (Cardoso et al. 2012; Goemaere et al. 2013; Garrido-Cordero et al. 2020).

Iberian translucent beads are rare, and when recorded they were generally classified as quartz or rock crystal with no further analysis for most of the 20th century (i.e., Leisner and Leisner 1943, 1965; García and Spahni 1959; Almagro and Arribas 1963). However, besides quartz varieties, there are many more minerals that are not usually considered in the case of translucent beads (calcite, opal, fluorite, etc.) or even some sheet silicates that when thinned to a certain level are capable of transmitting light (Baysal 2017: 6-7).

This paper focuses on the characterization of a remarkable set of translucent items from Iberian Late Prehistory (6th to 2nd millennia BC), mostly in the form of personal ornaments. The chemical and contextual analysis of the beads inventoried here throws new light on the use and social significance of translucent personal adornments, a subject on which little research has been yet carried out, and allows to approach its distribution and consumption among Late Prehistory's communities. It signifies a great advance in this field, as this paper furnishes additional and revised information to a previously published report (Garrido-Cordero et al. 2020). Due to the important presence of fluorite mineral in the composition of this ornaments and to recent works dealing with the use of this mineral in Late Prehistory, this paper also attempts to highlight the role of fluorite in Iberia's Late Prehistory.

1.1. Archaeological background

Translucent ornaments have been recorded among different contexts and chronologies in European Late Prehistory. Fluorite personal adornments are recorded from the Upper Paleolithic to the Bronze Age in Western Europe (Goemaere et al. 2013), and from the Late Neolithic to the Bronze Age in the Iberian peninsula (Pozo et al. 2002; Cardoso et al. 2012). However, despite fluorite's relatively abundant natural occurrence in Western Europe and the Iberian peninsula, it was rarely used during Late Prehistory.

Although small if compared to other raw materials (Vermeersch et al. 1990; Delye et al. 2011), fluorite consumption in Europe, particularly in Belgium and France, seems to increase from 5th to 3rd millennia BC. During the 2nd millennium BC, however, fluorite continued to be used occasionally for personal adornments (Demakopoulou et al. 1996: 23; Warmenbol 2001). Fluorite bead distribution pattern is associated to the main outcrops of fluorite occurring in the Central Massif and the Pyrenees, suggesting a local exploitation and a regional scale distribution (Roscian et al. 1992: 233-234; Polloni 2008; Hauzeur and Cauwe 2012: 39).

In Iberia, the first evidences for translucent adornments are related to Early and Middle Neolithic contexts of Southern Spain, in which pendants made of calcite are recorded (Navarrete 1976; Carmona et al. 1999; Goñi 2004; Ramos 2004). Unlike other parts of Europe, no personal adornment made from fluorite is known in Iberia before the 4th millennium BC. Although most of the sites lack radiocarbon and contextual dating, the use of fluorite most likely ranges from the late-4th to mid-3rd millennium BC. Beside this, a few cases might point to a continued use under the Argar culture during the 2nd millennium BC in south-east Iberia. So far, only the two fluorite beads from Tomb 111 at Fuente Álamo have been documented in that period (Pozo et al. 2002; Schubart et al. 2006). Quantitatively, since Bensaúde recorded in the 1880's the first Iberian fluorite bead in Cova da Moura (Cardoso et al. 2012), a total of 8 beads have been registered on Iberia (Blanco and Rothenberg 1981; Gomes et al. 1998; Cardoso et al. 2012; Ribeiro and Loureiro 2015)1.

2. MATERIALS AND METHODS

A total of 47 sites with translucent beads and a total number of 87 items are reported in this work (Fig. 1, see SF1, SF2 and SF4 for contextual information of the studied material) after exhaustive research involving the whole Iberian peninsula. However, due to difficult access to some collections, only a set of 53 beads (SF1, Fig. 2) have been analyzed by means of Raman spectroscopy, and additionally by one or more of the following techniques: X-ray diffraction (XRD), portable X-ray fluorescence (p-XRF) and VIS/NIR

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spectrometry (see SF1 for a detail on techniques applied to each specimen).

Raman spectroscopy was performed using a portable BWTEK iRaman Plus device. The laser diode operated with a wavelength of 785nm produces a power of up to 420mW in the laser port. Filters were not used to reduce the power of the laser. The selected range of the measurement spectrum was between 150 and 3300 cm\(^{-1}\) with a high efficiency quantum CCD detector. The selected measurement accuracy was 4 cm\(^{-1}\). The measurement conditions, as regards the laser power and integration time have varied from one object to another in order to obtain the best possible Raman signal.

Chemical composition was measured by an Oxford Instrument XMET-7500 p-XRF equipped with a Rh tube, a silicon drift detector (SDD), and an automatic 5-position filter changer. Quantification was obtained after 3 analysis on a 9mm\(^2\) spot using the SOILS-LE program based on fundamental parameter (FP) method.\(^2\)


\(^2\) This method is the most appropriate when no standardized method is available or when a large number of elements have to be analyzed (Beckhoff et al. 2006: 403).
Fig. 2. A sample of the beads analyzed in this paper (bead photographs can be seen in SF4). In colour in the electronic version.
XRD analysis was performed using a Panalytical X’Pert Pro 0/0 diffractometer equipped with Cu Ka source (1.5406 Å) operating at 45 kV and 40 mA. A PixCel detector was used and the data were collected on transmission mode with a 2D detector. Patterns were obtained using a step width of 0.053° 20 between 5° and 70° 20 and a counting time of 155 s per step at ambient temperatures. An incident beam PreFIX module with X-ray mirror for Cu radiation was used to allow non-destructive analysis.

An ASD Terraspec Halo full range spectrometer measuring the visible and near infrared regions (350-2500 nm) was used to record the reflectance spectra. The instrument uses a CCD and two Peltier cooled InGaAs sensors mounted over a Goertz post dispersive monochromator and three gratings. The device performs mineral identifications automatically by identifying the best peak match between the library and the recorded spectrum.

3. RESULTS AND DISCUSSION

3.1. Fluorite beads

Fluorite accounts for an exceptionally simple vibrational structure that exhibits a single $T_{2g}$ Raman active band at ca. 320 cm$^{-1}$ (Gee et al. 1966; Srivastava et al. 1971; Keramidas and White 1973; Alencar et al. 2016). 11 of the Raman analyzed beads (SF1) show the fluorite diagnostic $T_{2g}$ Raman band at ca. 320 cm$^{-1}$ (Fig. 3a). However, the pale pink beads (CMR-436, AGCI-2011.154.278, AGCI-985.51.617, FA-T111-DJ83773 and FA-T111-VI-1) accounts for additional bands to the $T_{2g}$ Raman active band at lower and higher frequencies (Fig. 3b). These additional bands may be caused by a) the accommodation of Y, Sr, Ba ... and REEs in substitution of Ca (Sverdrup 1968; Cherniak et al. 2001; Tu and Sievers 2002; Chen and Stimets 2014; Lenz et al. 2015); b) by radiation-induced defects (Alencar et al. 2016); c) by the incor-

Fig. 3. Raman spectra in the diagnostic region, A. set of samples that shows solely the fluorite diagnostic $T_{2g}$ Raman band at c. 320 cm$^{-1}$ (El Pozuelo 7 bands above 322 cm$^{-1}$ might be ascribed to monazite $A_g$ and $B_g$ modes (Errandonea et al. 2015); B. set of samples that shows the fluorite diagnostic $T_{2g}$ Raman band at c. 320 cm$^{-1}$ and additional bands. AGCI: Anta Grande da Comenda da Igreja, CMR: Casa da Moura; FA: Fuente Álamo.
poration of lead in the structure causing the switch of the $T_{2g}$ band to ca. 465 cm$^{-1}$ (Kumar et al. 2019), d) by the common intergrowth of monazite (Deng et al. 2017) peaking at lower and higher frequencies (Errandonea et al. 2015) or c) by the interference pattern (ripple) generated by the edge filters in luminescent samples (Vandenabeele and Edwards 2018: 327-328).

Beside these features in the diagnostic region, a set of bands appear in the frequency-shift region between 1000 and 2500 cm$^{-1}$ that are the result of the 785 nm laser induced photoluminescence (PL) (Fig. 4). PL bands have been formerly proposed as mineral characteristic (Chen and Stimets 2014), therefore the match between fluorite reference PL spectra and the here recorded would support the identification of all the analyzed specimens as fluorite.

Therefore, based on 1) the presence of the $T_{2g}$ Raman, 2) the coincidence of the PL of the specimens with that of fluorite, and 3) the chemical composition; fluorite is proposed as the main mineral phase for these 11 beads (SF3).

Even though 15 of the Raman analyzed beads (Lapa do Bugio LB-0011, LB-0012; Olival da Pega OP-21815B, OP-21815A; Anta Grande da Comenda da Igreja AGCI-985.51.670; Tituaria TIT-988.16.42; Gruta da Marmota MRO/S-25; São Paulo 2 SP2-5743; Penedos de São Miguel PSM-112; Leceia LC93-FA-C3; El Pozuelo 1; El Pozuelo 5; Los Gabrieles 6; and Cau de l’Olivar 16477-7 and 16477-17) lack the $T_{2g}$ band, their chemical composition and PL spectra (Fig. 4c) suggests fluorite as the main mineral phase for these 15 beads. XRD analysis have been performed on a selection of this set of 15 beads and on Anta Grande do Zambujeiro’s ME-3760. Beads from Leceia LC-93-FA-C3 (see Cardoso et al. 2012: 39, fig. 4), Gruta da Marmota MMT-26 and Anta Grande do Zambujeiro ME-3760 matches the standard XRD fluorite pattern (ICDD PDF card 1-77-2251) (Fig. 5a), while El Pozuelo 5 (Fig. 5b) matches that of fluorite (ICDD PDF card 1-1274).

Although only 4 beads have been submitted to XRD analysis, we might argue that this set of 16 beads

![Fig. 4. Raman spectra in the frequency-shift region between 1000 and 2500 cm$^{-1}$ showing the typical fluorite PL spectra: A. set of samples lacking the $T_{2g}$ Raman band; B. set of samples that shows additional bands to the $T_{2g}$ Raman band. LC: Leceia, LB: Lapa do Bugio, OP: Olival da Pega 1, AGCI: Anta Grande da Comenda da Igreja, TIT: Tituaria, MRO: Gruta da Marmota, SP2: São Paulo 2, PSM: Penedos de São Miguel, CMR: Casa da Moura; FA: Fuente Álamo.](https://doi.org/10.3989/tp.2020.12256)
are most likely fluorites, based on the Ca content and absence of bands that could be assigned to calcite and the PL bands.

### 3.2. Calcite translucent beads

Raman spectra of specimens AGCI-2011.54.73 (Anta Grande da Comenda da Igreja), AL-35-G11 (Alapraia), T3-179.91 (Trigache 3) and 16477-18 (Cau de l’Olivar d’en Margall) registered bands at 284, 710 and 1085 cm\(^{-1}\) assigned to calcite external \(E_g\) mode, and internal \(E_g\) and \(A_1g\) modes (Sood et al. 1981; Chen and Stimets 2014); the Halo device found a match with calcite for Cueva de la Pulsera, Abrigo 6 de la Araña, Cueva de los Mármoles MA-528, Alapraia AL-35-G11, Cau de l’Olivar d’en Margall 16477-18, and Cabeço da Ministra 220-1 and 220-2; and XRD analysis for Buraca da Moura de Rexaldia MMTN-2215-1 matches that of calcite. Consequently, these 7 beads are classified as calcites.

Additionally, apart from their chemical composition, translucent beads from Calatras IV (ID-220), Trigache 3 (T3-179.91) and La Veguilla I were also published as made of calcite (Natividade 1903; Leisner and Leisner 1965; Villalobos García 2015).

### 3.3. Silica-based translucent beads

9 translucent beads with a silica-based chemical composition have been identified as quartz varieties and different types of sheet silicates (SF1). Different quartz varieties were determined by Raman spectroscopy for 4 beads: transparent quartz (rock crystal) for AGCI-985.51.618 (Anta Grande da Comenda da Igreja), tridymite for MMA-6862\(^3\) (São Paulo 2), citrine quartz for 2006.23.4 (Vidigal 2) and QAP-199 (Quinta do Anjo). Additionally, ARQ-VNSP-964.004 (Vila Nova de São Pedro) transparent bead chemical composition is compatible with that of quartz (SF3).

Sheet silicate minerals were determined by VIS/NIR spectroscopy where the Halo device found a match with mica type mineral for Alto da Feteira’s TOP-94, Poço Velho’s IGM-356, Anta Grande da Comenda da Igreja’s AGCI-985.51.684 and Buraca da Moura de Rexaldia’s MMTN-2215-1.

To summarize, according to analyses performed here, we have recorded 33 fluorite beads, 11 calcite beads, 5 translucent quartz beads, and 5 translucent sheet silicate beads. What added to the published translucent beads sums up to a total of 87 translucent beads currently known for Iberia.

### 4. DISTRIBUTION AND CONSUMPTION OF TRANSLUCENT ORNAMENTS IN IBERIA

In Iberia, translucent items were scarce and rare, and appear mostly in the form of finished adornments. To date, unlike Belgium and France, no remains of production or items in the process of being manufactured are known. However, fluorite mineral was found at the 3\(^{rd}\) millennium BC burials of tholos of Cueva del Vaquero and La Emisora sector in Valencina de la Concepción.

The 6\(^{th}\) to 2\(^{nd}\) millennia BC translucent items inventoried here were recovered at burials with exception made to the ones recovered at the 3\(^{rd}\) millennium BC fortified sites of Leceia and Vila Nova de São Pedro.

\(^3\) Raman spectra contain the characteristic quartz bands together with bands peaking at 402 and 412 cm\(^{-1}\) that could be ascribed to high temperature phases of quartz (tridymite) (Kingma and Hemley 1994; Kawano et al. 1994).

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4.1. Consumption

Despite the limited number of radiocarbon dates available for the sites studied here, a first use of translucent adornments must be placed in the 6th to late-4th millennia BC at south Iberia funerary caves, where most of the items are made of calcite (80%) (Tab. 1). During the late-4th to 3rd millennia BC the number of minerals used for adornments increases, with fluorite being the most used (51%). While from the 2nd millennium BC onwards the number of translucent items used for personal adornment decreases drastically being the use of fluorite infrequent (18%). This trend of use recalls of that of greenstones, where all kind of minerals were used until the 3rd millennium BC for beadmaking, moment since which variscite starts to dominate the archaeological record as exotica until the 2nd millennium BC (Odriozola et al. 2016) (Fig. 6). Thus, during the 3rd millennium BC exotica seems to standardize, variscite for green and fluorite for translucent adornment. However, unlike the mean value of translucent items per site from the 6th to the 2nd millennia BC that remains invariable –ca. 1.7–, the mean value of fluorite beads per site increases during the late-4th to 3rd millennia BC to end decreasing in the 2nd millennium BC (Tab. 1).

The higher frequency of appearance of fluorite beads against other translucent beads since the late-4th millennium BC could be due to the physico-chemical properties of minerals, i.e. hardness. Calcite (Mohs scale =3), a low hardness mineral, is the most used mineral until late-4th millennium BC. From then and until the 2nd millennium BC the number of recorded translucent items and minerals increases greatly, being fluorite (Mohs scale = 4), calcite and quartz (Mohs scale =7) the most usual ones. Thus, rock selection might have been driven along the millennia by rock hardness and the development of tools capable of carving and drill harder rocks. We need to bear in mind that the technical skills needed to carve transparent quartz, either mono-crystals or not, into beads together with its high hardness could have shaped raw material choice (Morgado et al. 2016).

<table>
<thead>
<tr>
<th>Millennia BC</th>
<th>Calcite</th>
<th>Fluorite</th>
<th>Quartz</th>
<th>Silicate</th>
<th>Other</th>
<th>N.D.</th>
<th># sites</th>
<th># translucent / # sites</th>
<th># fluorite / # sites</th>
</tr>
</thead>
<tbody>
<tr>
<td>6th – 4th</td>
<td>8*</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>2*</td>
<td>0</td>
<td>10</td>
<td>6</td>
<td>1.7</td>
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<tr>
<td>Late 4th - 3rd</td>
<td>10</td>
<td>34</td>
<td>5</td>
<td>5</td>
<td>0</td>
<td>13</td>
<td>66</td>
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<td>87</td>
<td>49</td>
<td>1.8</td>
</tr>
</tbody>
</table>

Tab. 1. Bead’s mineral frequency and mean values of translucent and fluorite items per site.

Fig. 6. Types of minerals used by period for beadmaking. A. 6th-4th millennia BC; B. Late 4th-3rd millennia BC; C. 2nd millennium BC. Site numeration corresponds to Fig. 1’s legend. World Hillshade: https://services.arcgis.com/arcgis/rest/services/Elevation/World_Hillshade/MapServer/0 and AWMC coastline and rivers http://awmc.unc.edu/awmc/map_data/.
4.2. Distribution

Fluorite beads distribution area largely fits the Iberian Atlantic seaboard, the Douro and Tagus river basins, with their tributaries, and to a lesser extent to the Guadalquivir estuary and the Guadiana basin (Fig. 6). Although at many sites fluorite might come from nearby places⁴, a regional or supra-regional scale exchange network for fluorite must not be disregard. To this particular, the fluorite beads from the Tagus estuary have been interpreted as exotic elements arriving through exchange networks (Cardoso et al. 2012) either because of the absence of sources anywhere nearby these sites, or either because the estuary itself acted as poles of attraction of highly valued raw materials and items during the late-4th to 3rd millennia BC (Fig. 6). Similarly, Valenciana de la Concepción monumental burial treasures large amounts of exotic and might be acquiring fluorite beads from the closest occurrences at Morón and El Castillo de las Guardas –ca. 80 km– (Calderón 1910) or from supraregional exchange networks.

4.3. Social value

Fluorite beads are scarce in the archaeological record and when found they occasionally appear in outstandingly wealthy monuments associated to exotica such as amber beads. For example, fluorite and amber personal adornment appears together in the 32% of the sites that accounts for fluorite beads. Amber items, considered without doubt exotica, are numerically much more frequent than fluorite and translucent items, 647 amber (Odriozola et al. 2019) against 34 fluorite and 51 non-fluorite translucent beads (SF1). Thus, the low number of fluorite and translucent items in each tomb if compared with amber and other valued rare rocks, and the association of amber and fluorite items, particularly the lavishly decorated beads from Anta Grande da Comenda da Igreja, São Paulo 2 and Gruita da Marmota (Fig. 2), reinforces the idea of singularity and exclusiveness of fluorite and, indeed, all translucent minerals used for beadmaking.

However, the symbolic significance, special meanings and connotations of fluorite might not only stem from the former reasons but also from its materiality and sensorial properties, including its capacity to transmit light and wide palette of unusual natural colors, like pink, violet or blue. Indeed, it seems that the pink and violet shades, the rarer ones, appear in the megaphiliths where individuals of higher rank were buried, for example, at Anta Grande da Comenda da Igreja, Anta Grande do Zambujeiro or Los Millares 12.

Therefore, translucent beads, and fluorite beads in particular, may have been items whose use and possession differentiated certain individuals within their communities, materializing social differences by means of their asymmetric display among the living and the dead. In addition to the former, the association of fluorite beads to other non-local raw material items of great value strongly suggests the high rank or status of those people wearing such items. Tombs 12 and 63 at Los Millares are considered prestigious (Chapman 2008), as their grave goods included pottery with symbolic decoration, unique ivory objects and amber beads (Leisner and Leisner 1943; Chapman 2008; Odriozola et al. 2019). Similarly, the Bronze Age burial of Fuente Alamö’s tomb 111 corresponds to a high-rank young woman with rich grave goods (Pozo et al. 2002; Schubart et al. 2006). Thus, only the kin groups, factions or individuals who were buried in monumental tombs were able to afford or had the right to own personal adornment made out of translucent minerals, particularly of fluorite.

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SUPPLEMENTARY FILES

In the online version of this paper, you can consult the files:

SF1: Inventory of Iberian translucent beads;
SF2: Contexts of the sites studied and reported in the text;
SF3: Portable X-ray fluorescence recorded chemical composition (element %). BDL: Below Detection Limit;
SF4: List and photographs of the items here studied.

⁴ See https://egdi.geology.cz/record/basic/55002b8a-928c-42c7-8483-70810a010851 for fluorite deposits and favorable scores.