



Decoration composition of Iberian Iron Age ivory artifacts identified by no-destructive chemical analyses

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Received: 23 February 2018 / Accepted: 26 December 2018
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Abstract

In this work, five unique Iberian Iron Age handles have been studied. The use of portable X-ray fluorescence (pXRF) and Fourier transform near-infrared (FT-NIR) spectroscopy no-destructive techniques has allowed us to identify two raw materials (amber and tin) used to decorate the ivory handles. Due to the finesse of their inlaid decoration and the value and exoticism of the material employed for their manufacture, these ivory objects are really exceptional Iberian archaeology findings. Interestingly, it has been possible to determine that tin was used as an adherent material to fix the inlaid small pieces of amber in the handles. The obtained results allow the better understanding of the manufacturing processes, areas of production, and accessibility to exotic materials of valuable objects during the Iberian period.

Keywords Ivory handles · Amber · Tin · No-destructive analyses · Iberian culture · Middle Iron Age

Introduction

Iberian Iron Age artifacts made by worked hard animal materials have been studied from the taxonomic, typological, technological, and functional points of view following the methodological approach developed by Camps-Fabrer (1974, 1979) and Averbouch and Provenzano (1998, 1999). In this work, we have analyzed some unique Iberian ivory artifacts, especially their decoration, employing no-destructive analytical methods.

Iberians is the term that classical sources use to name the native pre-Roman inhabitants of the western Mediterranean region occupying the Iberian Peninsula during the 6th and

the 1st century BC. It is widely accepted that the Iberians developed a high level of urbanization, complex and complementary economic strategies (agricultural practice, metallurgy, livestock breeding, crafts, etc.), varying scales of trade and exchange, funerary rituals, elite levels of society, and independent socio-political territories based on hierarchical urban societies (Bonet-Rosado and Mata-Parreño 2014; Ruiz Rodríguez and Molinos 1998).

When a bone, antler, or ivory artifact is not heavily modified, a correct study of its dimensions and physical properties and a microscopic examination allow us to differentiate between these raw materials (Camps-Fabrer 1974; MacGregor 1985; Rodet-

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Belarbi and Van Ossel 2009). However, during the last decades, different methodologies have been applied to identify animal remains, as well as the impact of depositional and post-depositional processes upon them. Some authors have carried out mineralogical and chemical analyses of ivory, bone, and antler in order to better understand the manufacturing processes and preservation conditions of objects made by these raw materials (O'Connor 1984; MacGregor 1985). Numerous archaeometric studies have been realized to determine ivory origins. Edwards et al. (2005) have employed FT–Raman spectroscopy ivory analyses to observe differences between mammoths and ancient elephants and modern Asian and African elephants. Roth and Bortolaso (2008) have developed the INCENTIUS project, with the aim to distinguish the origin of ivory using microscopic, spectroscopic, and spectrometric analytical methods. Similar studies have been conducted, in order to explore the origin of some Copper Age ivory objects found in the Iberian Peninsula (Schuhmacher and Banerjee 2012; Schuhmacher et al. 2013; Nocete et al. 2013). Also, carbon, nitrogen, oxygen, and strontium isotope analyses have been used in order to better determine the geographical origin of post-medieval ivory found in Amsterdam (Rijkelijhuizen et al. 2015).

For ancient antler objects, some authors have proposed a microscopic examination to detect the presence, size, and orientation of osteons, circumferential lamellae, and interstitial lamellae (Paral et al. 2007). Also, no-destructive micro-PIXE/PIGE analyses have been employed to study about 150 objects made of different bone materials coming from various archaeological sites and belonging to different periods spanning from the Paleolithic to the modern ages (Muller and Reiche 2011).

On the other hand, few studies have examined the composition of adherent and decorative materials. We can point out the case of bone hinges from Gallo–Roman times in which an adhered dark substance was identified. This substance was investigated through Fourier transform infrared (FTIR) spectroscopy and gas chromatography with mass spectrometry that led to the result that it was birch pitch (Mazuy et al. 2014). It is an organic raw material used in handles of bone and lithic artifacts as well as to fix coral inlays.

In this work, five unique Iberian Iron Age handles have been studied as part of a wider bone, ivory, antler, and horn object collection (Blasco-Martín 2015; Mata-Parreño et al. 2017 and 2018) (Fig. 1). Due to the finesse of their inlaid decoration and the value and exoticism of the material employed for their manufacture, these ivory objects are really exceptional and unique through the Iberian Iron Age archaeological record. The five ivory handles are very similar in shape and style although they are not identical. All these objects were found during fieldworks in different Iberian settlements and necropoleis (Fig. 2). Similar objects were found at the archaeological sites of La Serreta (Alcoi-Cocentaina-Penàguila, Alicante) (exhibited in Museu Arqueològic Municipal Camil Visedo Moltó, Alcoi), El Puntal dels Llops

(Olocau, Valencia) (Bonet-Rosado and Mata-Parreño 2002), and San Antonio (Calaceite, Teruel) (Pallarés 1965) but they have no decorations or just a single metal disc in the heel.

The aim of this study is to identify the material of the inlaid decoration in some of the ivory handles as well as the dark substance surrounding the rabbits. The identification of these materials will give an important input to better understand the manufacturing processes, the existence of specialized craftsmen who work with hard animal materials like ivory, and the distribution dynamics of these unique handicrafts.

The inlaid decoration is an ornamental technique rarely documented among the Iberians. It can only be found in the case of jewelry such as in some typologies of fibulas and rings (de la de la Bandera 1986), although the incrustated material has almost never been preserved. In some annular fibulas, the rabbits are located in the bow (Cuadrado 1957) as can be observed in fibulas from La Tène culture where rabbits are found in the bow and in the edge (Navarro 1970). A fibula from La Serreta with a piece of bone at the ends of the spring is known, as well (Grau and Reig 2002–2003). Small pieces of bone, vitreous paste, stone, or amber that potentially could be part of these objects have been found, but since they are isolated findings, linking them to any jewelry was not possible. For example, some bone piriform and circular pieces (tomb 70 of Coimbra del Barranco Ancho) were related to the handles found in the same site (Iniesta et al. 1987; García Cano et al. 2008). However, the size and shape of these pieces do not fit with the rabbits of the two handles found in this necropolis (Fig. 1(6)); therefore, we believe that they would have been part of the inlaid decoration of some other objects.

Within the five studied handles, two of them (Fig. 1(4, 5)) preserve small incrustations made of unidentifiable raw materials. The presence of a dark substance around the incrustation that seems to act as a glue on the surface of the ivory was detected macroscopically. Therefore, no-destructive analytical methodologies such as Fourier transform near-infrared (FT-NIR) spectroscopy and portable X-ray fluorescence (pXRF) analyses were employed to identify the nature and composition of the inlays.

When it was feasible, the handles were systematically analyzed in their different components: ivory, inlays, and dark substance. Furthermore, NIR spectra have been obtained from an amber modern necklace and an Iron Age amber bead from Solana del Castell (Xàtiva, Valencia) (Pérez Ballester 2006), to observe possible spectral resemblance between the incrustations found in some handles and amber.

The ivory handles

The five studied handles are laminar artifacts with a rounded heel. They were found in different Mediterranean archaeological sites (Figs. 1 and 2 and Table 1). The handle called EC

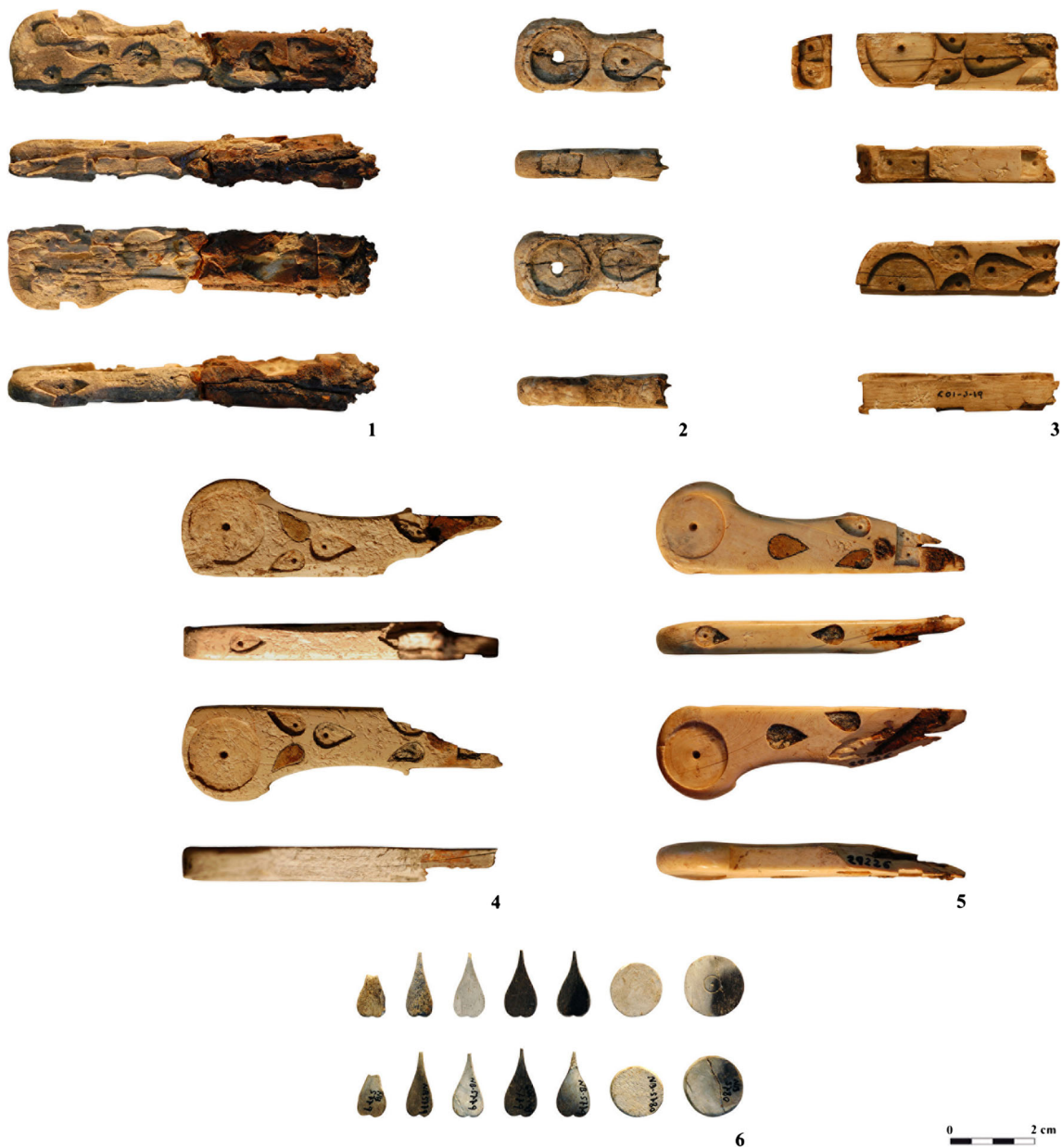


Fig. 1 The studied ivory handles, from 1, El Cigarralejo (Mula, Murcia); 2, Necropolis Coimbra del Barranco Ancho (Jumilla, Murcia); 3, Settlement Coimbra del Baranco Ancho (Jumilla, Murcia); 4, La Serreta

(Alcoi-Cocentaina-Penàguila, Alicante); 5, Turó del Montgròs (El Brull, Barcelona); 6, bone pieces from tomb 70 of Coimbra del Barranco Ancho (Jumilla, Murcia)

was found in El Cigarralejo (Mula, Murcia); CBA1 and CBA2 in Coimbra del Barranco Ancho (Jumilla, Murcia); LS in La Serreta (Alcoi-Cocentaina-Penàguila, Alicante); and TM in Turó del Mongròs (El Brull, Barcelona).

All of them have lanceolate and circular decorations made by employing a technique called inlay. Rhomboidal (EC), scutiform, and rectangular decorations (CBA1 and CBA2) can be observed in some of the ivory handles. Moreover, in some of the rabbits, the incrustated material has been preserved. A small hole can be observed where incrustations are not preserved. In handle CBA2, a circular mark around the small hole, left by the tool employed to extract the ivory and fix the

inlays, is clearly detected. Handles EC, LS, and TM partially preserve the iron blade.

The most ancient handle is part of the grave goods of a tomb dated to 350–325 BC and it was found in El Cigarralejo (EC) necropolis, but it is badly preserved (Cuadrado 1987). In this artifact, there are lanceolate and circular rabbits in both faces and a rhomboidal rabbit on the side (Fig. 1(1)).

In Coimbra del Barranco Ancho, two handles were discovered, CBA1 in the necropolis and CBA2 in the settlement (García Cano 1997). Both objects belong to the 4th and 3rd centuries BC. The handle from the necropolis (CBA1) was

Fig. 2 Mediterranean area of Spain. Location of the Iberian Iron Age sites where the ivory handles were found



related to a small mirror handle (García Cano 1997) and it has two rabbits in both faces, a lanceolate and a circular in the heel and a rectangular one on the side. The circular rabbits have an engraving along its circumference and a central hole that perforates the whole object being the only one with this characteristic (Fig. 1(2)).

The handle CBA2 has a circular rabbit in both faces, a lanceolate one between two small pairs of the same shape, other two rectangular, one scutiform, and an incomplete rectangle in the back of the heel. In this piece, the circular marks left by the used instrument are clearly observed (Fig. 1(3)).

La Serreta (LS) handle (Grau 1996) was found in a settlement area constituted by several domestic units abandoned at the beginning of the 2nd century BC. It is decorated in both faces with

nine small lanceolate rabbits, where two of them preserve the incrustated material and other two circular rabbits can be seen in the heel and a lanceolate one in the back of the piece. On one side, a rectangular rabbit employed to fix the ferrule to maintain the blade is preserved. A dark substance can be observed in all the lanceolate rabbits and around the inlays (Fig. 1(4)).

Finally, the handle of Turó del Montgròs (TM) was found inside the *phylacteria* (casemate) of the fortress in a 3rd century BC chronological context (Molist and Rovira 1986-1989). It is very similar to the handle found at La Serreta (LS), being the only difference related to the presence of one lanceolate rabbit on the side of the handle. The TM handle preserves two incrustations on one side, while a dark substance around the inlays and the rabbits was also detected (Fig. 1(5)).

Table 1 Description of the studied objects

ID	No.	Collection	Site	Context	Chronology	Length (cm)	Width (cm)	Depth (cm)
EC	T294	MAIEC	El Cigarralejo	Funerary	4th BC	7.8	1.9 (max)/1.2 (min)	0.85
CBA1	1104-3	MAMJM	Coimbra del Barranco Ancho	Funerary	4th–3rd BC	2.1 (inc)	1.09 (max)/0.83 (min)	0.53
CBA2	J19	MAMJM	Coimbra del Barranco Ancho	Settlement	4th–3rd BC	4.6 (inc)	1.3 (inc)	0.8
LS	2096	MAMCVM	La Serreta	Settlement	3rd–2nd BC	8	2.4	0.9
TM	29226	MAC-Barcelona	Turó del Montgròs	Settlement	3rd BC	7.45	2.2 (max)/1.3 (min)	0.8

Materials and methods

Measured samples

The five ivory handles (El Cigarralejo, EC; Coimbra del Barranco Ancho, CBA1 and CBA2; La Serreta, LS; and Turó del Mongròs, TM) have been analyzed by running pXRF and NIR measurements in different points of the objects (Table 2). Furthermore, a modern amber necklace (MAN) and an Early Iron Age amber bead from Solana del Castell (SC) have been analyzed to be compared with the spectra of the inlaid decoration of the handles.

EC handle was analyzed in seven different decoration points: in the surface of the ivory and near to the preserved iron blade (Fig. 3 and Table 2). CBA1 was measured in both faces in the lanceolate decoration and in the circular decoration (Fig. 4 and Table 2). CBA2 was analyzed in eight different points along the rabbets and on the surface of the ivory in all the faces of the artifact (Fig. 5 and Table 2). LS is a quite well-preserved handle and eight different points were measured along the incrustations and the dark substance (Fig. 6 and Table 2). Also, TM is a well-preserved artifact and ten points were measured in the inlays and the surface of the ivory (Fig. 7 and Table 2).

As mentioned above, the modern amber necklace (Fig. 8) and the archaeological amber beads (Fig. 9) were analyzed in three different points.

Experimental part

The samples were photographed and analyzed at a macroscopic and microscopic level. We used a Nikon D3300 AF-P DX 18-55mm DSLR camera, with macro lens Micro-Nikon 60mmf/2.8D model and a digital microscope (DM). Near-infrared (FT-NIR) spectroscopy and portable X-ray fluorescence (pXRF) were employed to identify the material composition present in the decoration of the handles. The analytical techniques employed in this work were adequately validated, employing standard reference and sample control materials, in order to obtain accurate results, and the instrumentation and parameters used are described in the next sections.

pXRF analysis

X-ray fluorescence spectra were directly obtained using a portable S1 Titan energy-dispersive X-ray fluorescence spectrometer by Bruker model (Kennewick, WA, USA) equipped with a rhodium X-ray tube and an X-Flash® SDD detector. For the instrument control, the S1RemoteCtrl (Geochem-trace programme) was used. In addition, S1Sync software by Bruker was employed to measure the content of CaO, P₂O₅, Fe, Sr, and Sn. For spectra processing, the ARTAX software by Bruker was also used (Gallelo et al. 2016, 2017). The certified material, Bone Ash NIST 1400, was used as a standard reference for evaluating the accuracy of the pXRF data obtained for CaO, P₂O₅, Fe, and Sr.

FT-NIR analysis

A Fourier transform near-infrared spectrometer, multipurpose analyzer (MPA) by Bruker (Bremen, Germany), equipped with an integrating sphere and a fiber optic probe, was employed for the acquisition of diffuse reflectance spectra. This instrument is equipped with a NIR source, a quartz beamsplitter, and a PbS detector. For instrumental and measurement control as well as for data acquisition, Opus 6.5 software by Bruker was used.

The ivory handles were directly measured in different points by using an optical fiber probe to obtain NIR spectra by diffuse reflectance (DR-NIR). Spectra were recorded in Kubelka–Munk units, in the 14,000–4000 cm⁻¹ spectral region, using a resolution of 4 cm⁻¹ and accumulating 50 scans per spectrum. The background spectrum was acquired from the closed integrating sphere using the same instrumental conditions to those employed for the samples (Cascañt et al. 2017). To evaluate the significance of the obtained spectra, the modern amber necklace (MAN) and the archaeological amber bead (SC) were employed as sample controls.

OM and DM analysis

The studied artifacts were macroscopically observed employing an optical microscope SMZ (Nikon) and a digital

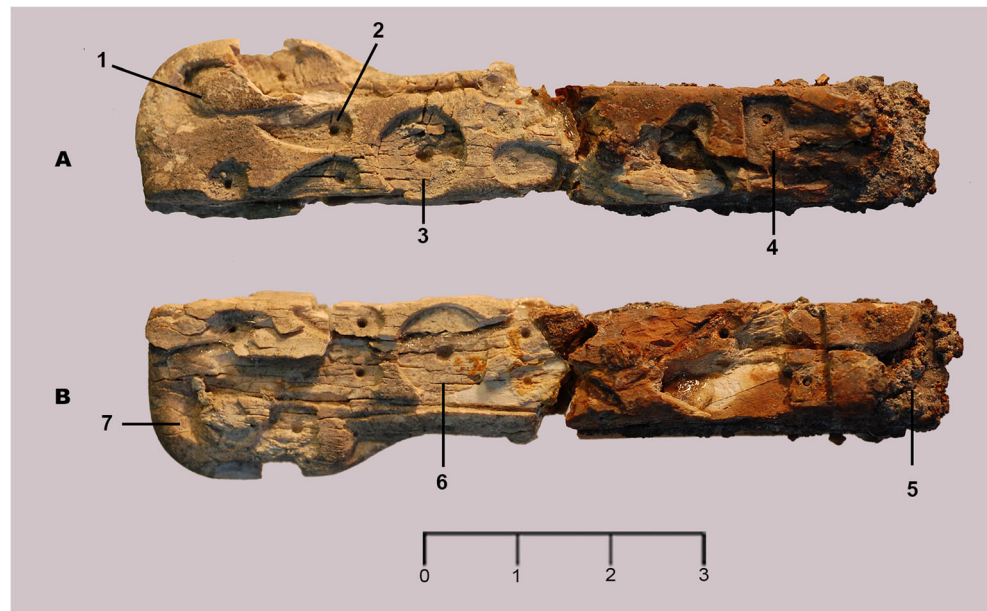
Table 2 Ivory handle reading points and no-destructive analysis employed. pXRF results. Concentration of elements in the analyzed points is expressed as percentages (%)

Samples	Description	pXRF	NIR	CaO (%)	P ₂ O ₅ (%)	Fe (%)	Sr (%)	Sn (%)
EC.1	Side A: ivory part	X	X	39.2	7.14	0.22	0.27	0.04
EC.2	Side A: ivory lanceolate rabbit	X	–	17.2	6.75	0.10	0.26	0.34
EC.3	Side A: ivory circular rabbit	X	X	16.3	5.00	0.11	0.23	0.08
EC.4	Ivory rectangular rabbit with iron oxide	X	–	8.13	3.85	21.6	0.32	4.54
EC.5	Side B: ivory with iron oxide	–	–	12.1	3.13	21.6	0.34	0.78
EC.6	Side B: ivory circular rabbit	X	X	29.9	3.32	0.12	0.24	0.05
EC.7	Side B: ivory flat part	–	X	–	–	–	–	–
CBA1.1	Side A: ivory circular rabbit	–	X	–	–	–	–	–
CBA1.2	Side A: ivory lanceolate rabbit	–	X	–	–	–	–	–
CBA1.3	Side B: ivory circular rabbit	–	X	–	–	–	–	–
CBA1.4	Side B: ivory lanceolate rabbit	–	X	–	–	–	–	–
CBA2.1	Side A: ivory lanceolate rabbit central hole	X	X	18.0	8.72	0.12	0.08	0.86
CBA2.2	Side A: ivory lanceolate rabbit central hole	X	X	19.0	8.61	0.13	0.07	0.64
CBA2.3	Side A: ivory flat part	X	X	26.0	16.4	0.11	0.10	0.46
CBA2.4	Side C: ivory flat part	X	X	28.5	11.0	0.27	0.07	0.20
CBA2.5	Side B: ivory circular rabbit central hole	X	X	21.0	8.17	0.15	0.09	<LOD
CBA2.6	Side B: ivory lanceolate rabbit central hole	X	X	17.7	9.39	0.11	0.09	0.39
CBA2.7	Side D: ivory rectangular rabbit between the holes	X	X	23.1	13.3	0.10	0.10	0.70
CBA2.8	Side D: ivory flat part	X	X	29.2	19.9	0.19	0.10	0.09
LS.1	Side A: ivory circular rabbit	X	X	33.0	24.9	0.10	0.03	0.02
LS.2	Side A: lanceolate incrustation	X	X	9.51	4.69	0.17	0.02	18.4
LS.3	Side A: trace of dark substance lanceolate rabbit	X	X	12.4	7.50	0.21	0.04	12.1
LS.4	Side A: ivory flat part	X	X	52.4	37.0	0.18	0.04	0.34
LS.5	Side B: ivory circular rabbit	X	X	30.3	21.7	0.13	0.03	0.38
LS.6	Side B: lanceolate incrustation	X	X	16.8	8.46	0.21	0.01	16.2
LS.7	Side B: ivory flat part	X	X	50.4	35.6	0.15	0.03	0.08
LS.8	Side B: ivory flat at the top part	X	–	7.25	3.10	6.08	0.02	0.09
TM.1	Side A: ivory circular rabbit	X	X	28.1	20.5	0.13	0.02	<LOD
TM.2	Side A: lanceolate incrustation	X	X	11.4	6.85	0.22	0.01	15.0
TM.3	Side A: lanceolate incrustation	X	X	10.0	6.07	1.32	0.02	9.61
TM.4	Side A: ivory rectangular rabbit between the holes	X	X	17.4	11.9	0.57	0.02	12.3
TM.5	Side A: ivory lanceolate rabbit	X	X	24.4	16.8	1.36	0.03	0.78
TM.6	Side B: ivory circular rabbit	X	X	15.5	8.18	0.10	0.02	1.42
TM.7	Side B: traces of dark substance lanceolate rabbit	X	X	18.9	13.9	0.16	0.02	11.2
TM.8	Side B: traces of dark substance lanceolate rabbit	X	X	8.75	4.97	0.17	0.02	9.99
TM.9	Side B: ivory flat part	X	–	43.4	29.8	0.15	0.02	0.02
TM.10	Side C: traces of dark substance lanceolate rabbit	–	X	–	–	–	–	–
MAN.1	Modern amber necklace	–	X	–	–	–	–	–
MAN.2	Modern amber necklace	–	X	–	–	–	–	–
MAN.3	Modern amber necklace	–	X	–	–	–	–	–
AA.1	Archaeological amber bead from Solana del Castell	–	X	–	–	–	–	–
AA.2	Archaeological amber bead from Solana del Castell	–	X	–	–	–	–	–
AA.3	Archaeological amber bead from Solana del Castell	–	X	–	–	–	–	–

microscope (DM) Dino-lite mod. An AM7115MZT EDGE (magnification range from $\times 10$ to $\times 200$) equipped with an

optical fiber illuminator and an integral measurement functions software for high-precision images was used.

Fig. 3 Analyzed points on the handle from El Cigarralejo (EC)



Data analysis

Data processing was carried out using in-house written functions employing Matlab (R2016b) from Mathworks (Natick, MA, USA) and Toolbox 8.2 from Eigenvector Research Inc. (Wenatchee, WA, USA) was employed for principal components analysis (PCA).

PCA applied to IR spectra was used as data exploration based on the distance between samples from the ivory, the incrustations, and the dark substance to evaluate the reliability of the spectral differences obtained.

Results and discussion

XRF analysis

Different parts of the ivory handles (EC, CBA1, CBA2, LS, and TM) were measured directly by pXRF. Table 2 summarizes the concentration values expressed in wt% of CaO, P₂O₅, Fe, Sr, and Sn of different artifact parts (circular decorations, lanceolate decorations, and flat parts). CaO and P₂O₅ are the main compounds in ivory (Ca₅(PO₄)₃(OH)), while Sn is the main element around the incrustations, and it was found only in LS and TM and in the dark substance identified in some rabbits, of these two artifacts (Figs. 6 and 7). To make sure that the elements detected from the incrustation and the dark substances are reliable, the ivory surface was analyzed as a blank and the results were compared with those obtained from these two materials.

Six different measure points were taken in EC. The highest levels of CaO were found in points EC.1 (39.2%) and EC.6 (29.9%) which coincide to the ivory handle flat part and the circular decoration respectively. The lowest CaO values belong to the decoration with iron oxidation marks (EC.4) and the top part of the handle also with iron oxidation traces (EC.4) and the top part of the handle also with iron oxidation traces (EC.5). Both EC.4 and EC.5 have high Fe values (around 21%). The highest Sn levels in this object can be observed in the rectangular decoration EC.4 (4.54%). Maybe, the high

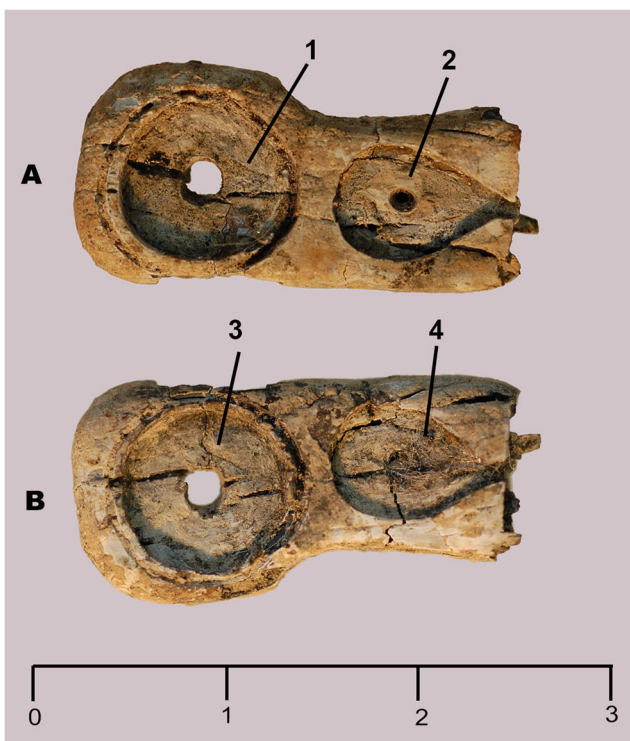
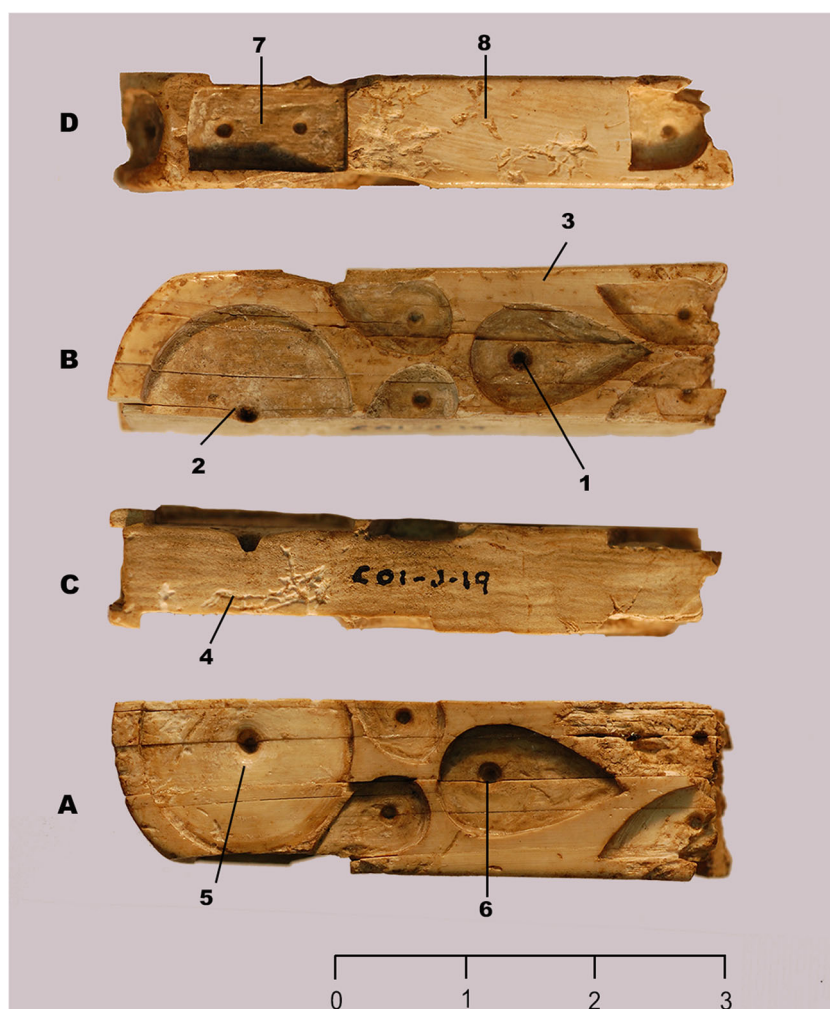


Fig. 4 Analyzed points on the handle from Coimbra del Barranco Ancho (CBA1)

Fig. 5 Analyzed points on the handle from Coimbra del Barranco Ancho (CBA2)



Sn levels in EC are caused by the presence of the dark substance remains which are not clearly visible to the naked eyes.

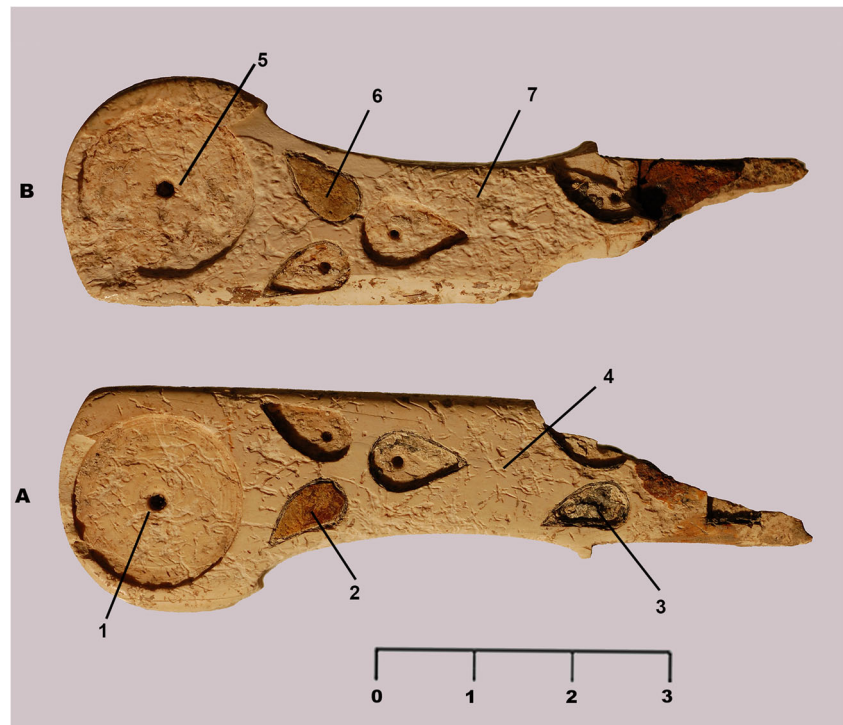
Since CBA1 is a badly preserved and very fragile artifact, pXRF analyses were not carried out due to the possible damage risk. Concerning CBA2, it was better preserved than CBA1 and the analyses were carried out. CaO levels were high in points CBA2.8 (29.2%), CBA2.4 (28.5%), CBA2.3 (26%), CBA2.7 (23.1), and CBA2.5 (21%). Low CaO values were found in the ivory central holes of lanceolate decorations CBA2.6 (17.7%), CBA2.1 (18%), and CBA2.2 (19%). In CBA2.1, Sn values (0.86%) were the highest followed by CBA2.7 (0.70%) and CBA2.2 (0.64%). Also in this case, the higher levels of Sn were related to the dark substance identified by digital microscopy (Fig. 10).

LS handle is one of the best preserved studied objects, and the incrustation fixed in the lanceolate rabbits and the dark substance can be still observed. CaO high levels could be observed in the flat parts of the object corresponding to point LS.4 (52.4%) and LS.7 (50.4%).

Lower CaO values could be seen in the lanceolate incrustation LS.2 (9.5%) and LS.6 (18%) and also in the dark substance in rabbit LS.3 (12.5%). The same points (LS.2, LS.6, and LS.3) have the highest Sn values respectively 18.4% (LS.2), 16.2% (LS.6), and 12.1% (LS.3). La Serreta Sn results make clear that it was employed to fix the inlays.

TM handle is also well preserved and it is possible to observe the lanceolate inlays and the dark substance in some parts. CaO high levels could be observed in the flat part TM.9 (43.4%) and the circular decoration TM.1 (28.1%) and TM.5 (24.4%). Low CaO values could be observed in the dark substance in TM.8 (8.65%) and in the incrustation decoration TM.3 (10%) and TM.2 (11.4%) as well. Sn values are high in TM.2 incrustation (15%), TM.4 flat part between the holes (12.3%), TM.7 dark substance (11.2%), TM.8 (9.99%), and TM.3 (9.63%). As in LS, the TM handle tin results confirm that incrustations and dark substance are related to the presence of this element employed to adhere the inlays.

Fig. 6 Analyzed points on the handle from La Serreta (LS)



NIR analysis

FT-NIR spectra

Figures 11 and 12 show the spectra of the studied ivory points in all the handles EC (a), CBA1 (b), CBA2 (c), LS (d), and TM (e), the analyzed incrustation and dark substance points found in LS and TM, and the amber samples MAN (f) and SC (g), all expressed in Kubelka–Munk units. The averaged NIR spectra are shown in this study without any data pre-processing, in the region between 9000 and 4000 cm^{-1} . These spectra present differences in the intensity and position of some bands, but all them show absorption bands around 7200 and 5220 cm^{-1} related to the stretching–bending combination mode of H_2O species and the stretching overtone of the OH^- of water. In the 5000–5308 cm^{-1} region, the stretching–bending combination mode of H_2O can be observed, in agreement with the previous observations of Ishikawa et al. (1989) who studied a synthetic, carbonate-free, colloidal calcium hydroxyapatite and reported a sharp H_2O combination mode at 5308 and of Stathopoulou et al. (2008) that assign this feature to a surface H_2O species. In addition, a band at 6982 cm^{-1} can be attributed to the stretching overtone of the OH^- (Stathopoulou et al. 2008).

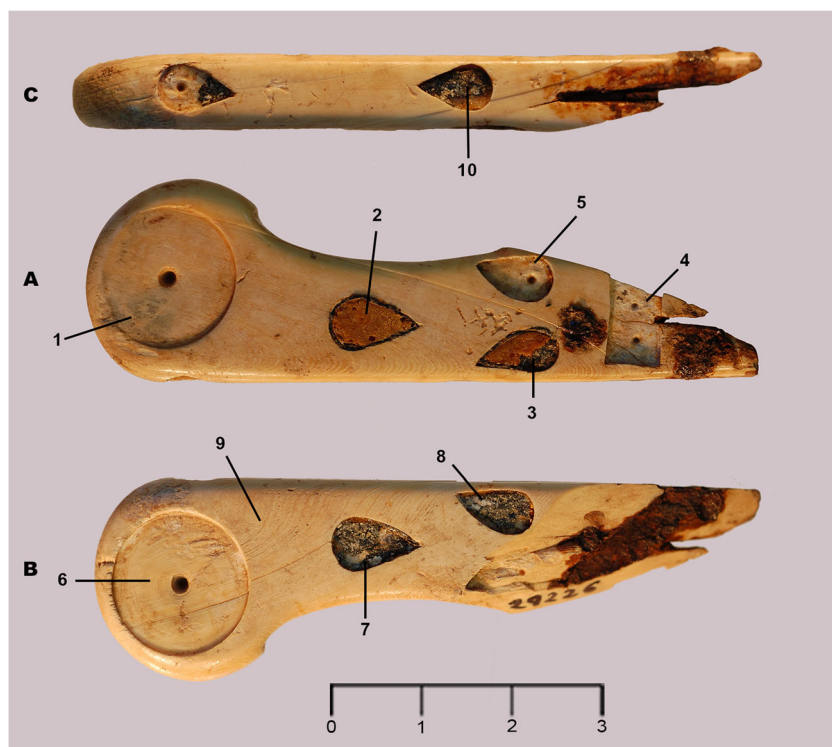
The spectra corresponding to LS handle (Fig. 11(d)) and TM handle (Fig. 12(e)) present the bands corresponding to overtones of mineral and organic compounds at 4353–4343, 5000–5308, 5711, 5868, and 6982 cm^{-1} . The bands in the 4353–4343 cm^{-1} region could be attributed to different natural

resins identified, as oil (4343 cm^{-1}), shellac (4344 cm^{-1}), dammar (4346 cm^{-1}), colophony (4348 cm^{-1}), copal (4349 cm^{-1}), mastic (4352 cm^{-1}), and sandarac (4353 cm^{-1}) (Sciutto et al. 2014). Some additional spectra are identifiable as colophony and mastic peaks, respectively, revealed at 5711 and 5868 cm^{-1} (Sciutto et al. 2014). Natural resin bands are especially marked in the lanceolate incrustations (LS.2, LS.6, TM.2, and TM.3). Also, the archaeological amber bead (SC) and the modern amber necklace (MAN) show the same important bands ranging between 4353 and 4343 cm^{-1} and mastic spectra at 5711 and 5868 cm^{-1} . This could mean that the material composition should be a resin, in particular, amber. Bands between 5000 and 5308 cm^{-1} , as explained above, are related to calcium hydroxyapatite and could be observed in all LS and TM read points, while bands 5711 and 6982 cm^{-1} are related to the combination of vibrations of H–O–H bend and O–H stretch of water.

Data exploration by PCA

PCA was used for exploratory data analysis to observe if the spectral differences between the handle points related to the different materials analyzed (dark substance, incrustation, and ivory) are statistically meaningful. Figure 13(a) presents the score plots for the first and the second main components obtained from PCA for the spectral region from 4600 to 4000 cm^{-1} . These components represent 80.87% of the explained variance, being 68.12% and 12.75% for PC1 and PC2, respectively. Principal components analysis (PCA) has been

Fig. 7 Analyzed points on the handle from Turó del Mongròs (TM)



applied to the whole set of samples and score plots were represented as data points (samples) projected into the PC space.

Magnitude and signs of the loadings from PC1 indicate that the samples plotted in the right area of the graph belong to the incrustation class (LS.6, LS.2, TM.2, TM.3). Again, following PC1, in the central left side of the plot, all the samples belonging to the ivory group are located, while the dark substance (LS.3, TM.7, TM.8, and TM.10) is located between the ivory

and the incrustation group. The major dispersion of the dark substance in the PC is related to the material of contact, being ivory for LS.3 and TM.8 points or incrustation for TM.7 and TM.10 ones.

PCA clearly shows that the spectral window from 4600 to 4000 cm^{-1} (Fig. 13(b)) is marking differences that include the resin bands and this allows us to confirm that the presence of resin could be amber.

Therefore, it can be concluded that NIR spectra data provides a no-destructive, fast, and green tool to identify unknown materials in ivory handle decorations. To avoid any mistakes, the use of PCA, as an exploratory method, allows us to clearly observe spectral differences within materials found in the same artifacts and to compare the different materials present in each handle.

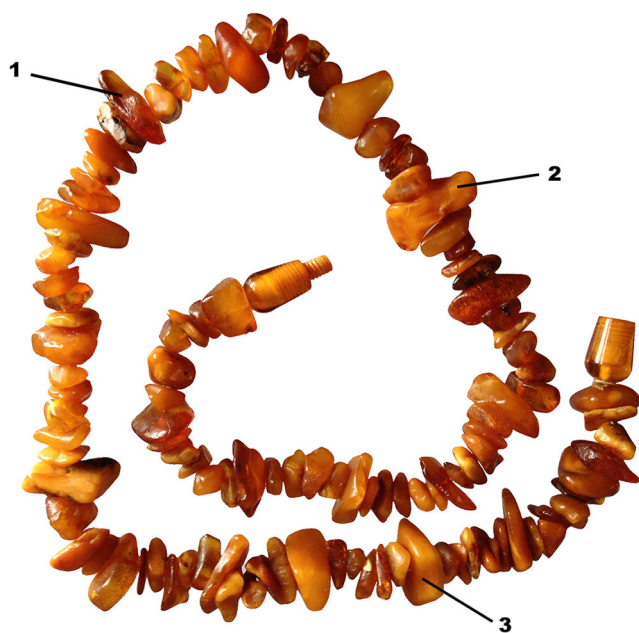


Fig. 8 Analyzed points on a modern amber necklace (MAN)



Fig. 9 Analyzed points on an Early Iron Age amber bead (SC) from Solana del Castell



Fig. 10 Dark substance captured by digital microscopy in CBA2 (point 2)

OM and DM analysis

Optical microscopy and digital microscopy were employed to better visualize the inlays and the rabbets. As can be observed in Figs. 6 and 7, the surface of the incrustation looks like a natural resin in the handles LS and TM, as confirmed by NIR results. In LS and TM (Figs. 6 and 7), the presence of the dark substance at the edge of the inlays that we have identified as tin employed as binder to fix the resin can be observed. Furthermore, as mentioned above, traces of the dark substance interpreted as tin can be observed by digital microscopy in the handle CBA2 (Fig. 10).

Archaeological evaluation and overview

Very interesting results were obtained by pXRF and NIR analysis. pXRF analyses show the presence of Sn, especially in the better-preserved handles LS and TM, although small amount of this metal was detected also in handle CBA2, where traces of tin were observed by digital microscopy in the small holes.

NIR spectra allowed us to identify natural resin bands particularly marked in the lanceolate incrustations. This material is probably amber, and this is confirmed by comparing the modern and ancient amber spectra that present also the most important resin bands observed at $4353\text{--}4343\text{ cm}^{-1}$ (Figs. 11 and 12).

Isolated findings of small amber objects are documented in Iron Age sites: the analyzed bead from La Solana del Castell, the bead from the settlement of Pic dels Corbs (Sagunt, Valencia) (Barrachina 2012), and different beads found in tomb 120 at the necropolis of L'Albufereta (Alicante) (Verdú 2015, 419, Fig. 3477). Also, more than seventy beads were discovered in tombs 26 and 27 (Fernández Flores et al. 2014, 449) in the necropolis of Angorrilla (Alcalá del Río, Sevilla). However, in the Iberian Peninsula, amber pieces belonging to older chronologies are more often found (Rovira i Port 1994, Álvarez-Fernández et al. 2005, Peñalver et al. 2007, García-

Puchol et al. 2012, Murillo-Barroso and Martín-Torres 2012, Murillo-Barroso and García Sanjuán 2013, Vera Vera Rodríguez 2014).

In this study, for the first time, the use of tin to fix amber in the Iberian Peninsula was reported. So far, its use, together with lead, has only been observed as soft soldering for jewelry (Asderaki-Tzoumerkioti 2008; Perea et al. 2010). A thin layer of dark substance has been described as a fixation material on some Mycenaean pieces, although nothing is said about its chemical composition (Mylonas 1983). On the other hand, two fibulas from the 8th to 7th century BC have been studied for the Etruscan world. In their bows, there are incrustations of ivory and maybe amber surrounded with a tin layer. However, XRF analysis was carried out only in one case, confirming the presence of tin (Formigli 2004). An ivory handle was recovered in Chiusi (Italy) by the Soprintendenza Archeologica di Tuscany (Florence), and in this case, radiographs and chemical analyses were carried out detecting the presence of tin (Formigli 2004). Both studies considered the use of tin to enhance the color of the amber, and the same happens when tin is mixed with vitreous paste, obtaining a whitish hue (Vigil Pascual 1969).

In the analyzed handles, tin appears around the amber and in the base of the rabbets. Therefore, we consider that it was used as an adherent material. Discovering that tin was used as an adherent for inlaid decorations is of great importance to better understand the quality of the manufacturing techniques in the Iron Age. Furthermore, we also contemplate the possibility that this manufacturing technique could reinforce the brilliance and the golden color of the amber, as suggested by Formigli (2004) in his study about inlaid amber in the Etruscan jewelry.

Both ivory and amber are exotic and unique raw materials. Ivory has a fine, soft, and non-porous texture and a brilliant and whitish surface; moreover, obtaining it from the environment is hard. During the Iron Age, large-size mammals like elephants or hippopotamus were not present in the Iberian Peninsula and obtaining ivory from their tusks was difficult; thus, it was a very valuable raw material. Due to the difficult access to ivory (Mata-Parreño et al. 2017, 2018), the diffusion of this material or even of finished artifacts in the Iberian territory took action maybe by itinerant craftsmen.

Amber had been used since the antiquity to make ornaments due to its workability and because of its beauty, saturated color, and translucency. Moreover, magical and medicinal properties have been attributed to this material (Álvarez-Fernández et al. 2005; Causey et al. 2012). At the moment, we do not know if the origin of the studied amber incrustations is local or foreign. The presence of amber in the Baltic area is well-known but outcrop of Cretaceous amber in the Iberian Peninsula is also documented, especially in the North and the East of the Peninsula and in the Maestrat Basin (Álvarez-Fernández et al. 2005; Peñalver et al. 2007; Najarro et al.

Fig. 11 NIR spectra of the ivory handles EC (a), CBA1 (b), CBA2 (c), and LS (d) in the region between 4000 and 9000 cm^{-1} without data pre-treatment. Identified peaks, 7200 and 5220 cm^{-1} (H_2O ; OH^- of water); 5000–5308 cm^{-1} (H_2O); 6982 cm^{-1} (OH^-); 4353–4343 cm^{-1} (natural resins); and 5711 and 5868 cm^{-1} (colophony and mastic)

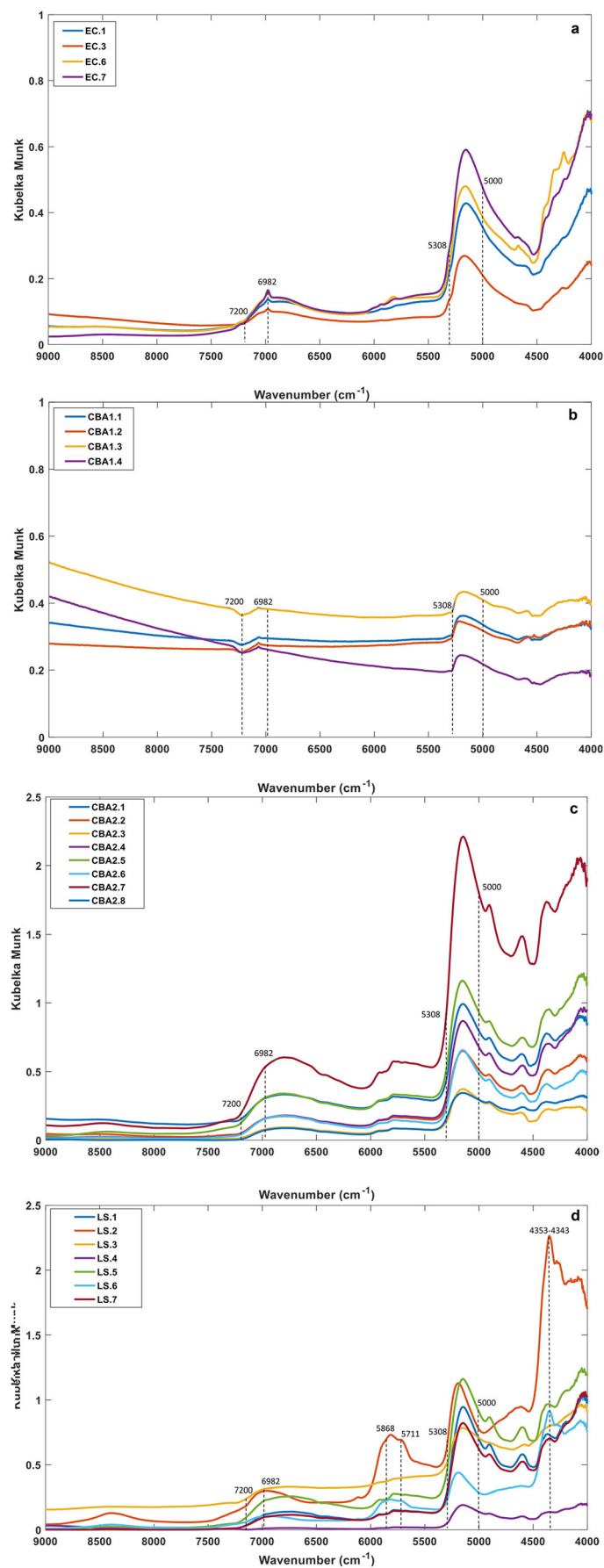
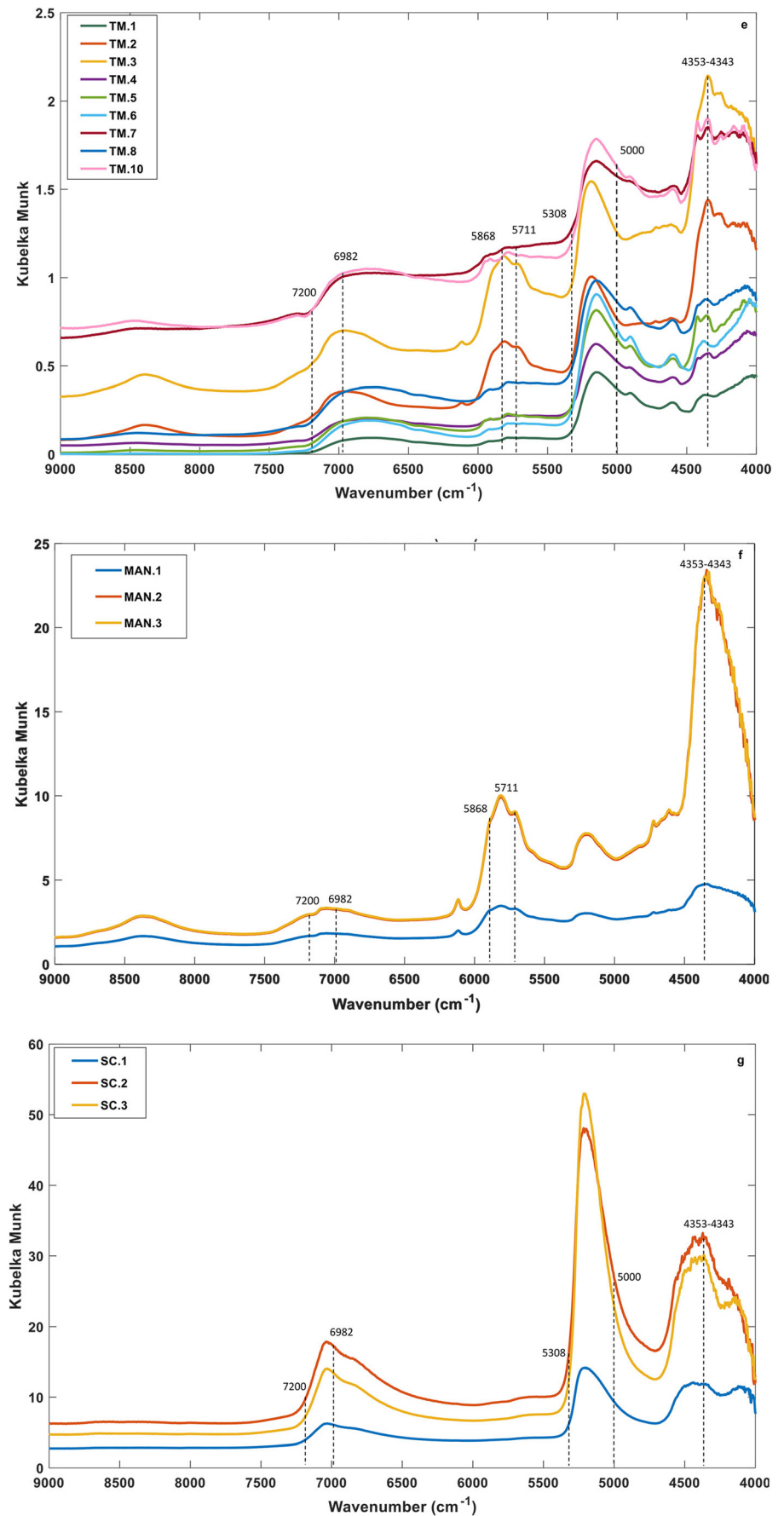


Fig. 12 NIR spectra of the ivory handle TM (e) and the amber samples MAN (f) and SC (g) in the region between 4000 and 9000 cm^{-1} without data pre-treatment. Identified peaks, 7200 and 5220 cm^{-1} (H_2O ; OH^- of water); 5000–5308 cm^{-1} (H_2O); 6982 cm^{-1} (OH^-); 4353–4343 cm^{-1} (natural resins); and 5711 and 5868 cm^{-1} (colophony and mastic)



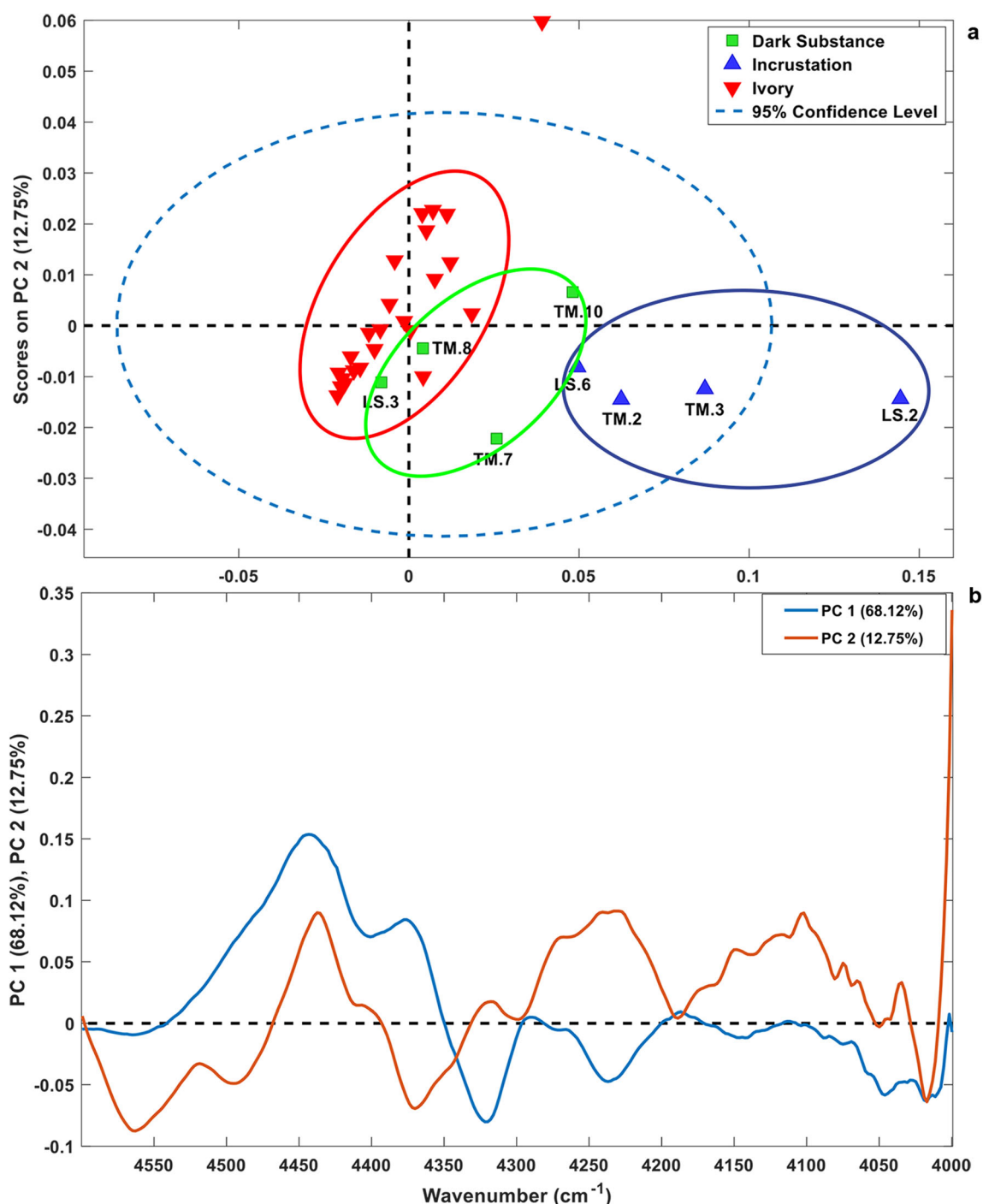


Fig. 13 Score plots for first and second principal components obtained by PCA from NIR spectra of the ivory handle read points, divided in three classes (dark substance, incrustation, and ivory). Loadings of PC1 and PC2 of spectra using the region from 4000 to 4600 cm⁻¹

2009). Therefore, the amber could come from nearby areas. About tin production, different mines are documented in the southeast of the Peninsula (Rodríguez Díaz et al. 2013) and the production of this metal in the region is documented before and during the Roman period (Rovira Guardiola 2005).

The combined use of three rare raw materials like ivory, amber, and tin has not been documented in any other artifact from the Iron Age in the studied area; on the contrary, it has

been found, for example, in Etruscan fibulas (Formigli 2004). Also, the inlay technique is common in the Oriental and Central Mediterranean (Caubet and Gaborit-Chopin 2004), but not in the Western world.

The hypothesis that these objects were made in the same workshop or workshops and acquired by the high-class society could be supported by looking at how the three raw materials were worked and by observing the similarity within the studied

handles in their form, size, functionality, and decoration motifs, something evident especially by comparing EC, TM, and LS. It is possible that these pieces reached the Iberian Peninsula through trade, but they can also be an example of peninsular artisan production. Another possibility points to foreign craftsmen that could have settled in the peninsula.

Finally, it is interesting to observe that the geographical distribution of the studied ivory handles is similar to that of the ivory combs (Mata-Parreño et al. 2017) and maybe these objects' production or trading could be related to the same region.

Conclusions

No-destructive pXRF and NIR analyses in the decoration of ivory handles have revealed unusual results. NIR spectra helped to identify the resin, probably amber, employed as an embedded material in the rabbits, and pXRF results revealed the presence of tin, used as an adherent.

These data increase the value and meaning that come from these handles, made with two exotic materials: ivory and amber. Ivory or bone objects with incrustations from the Iron Age in the Iberian Peninsula are rarely documented. For the first time, this study has revealed the use of amber and the use of an adherent material such as tin that had only been detected as welding; therefore, this study consistently contributes to shed light about manufacturing processes, areas of production, and accessibility to exotic materials of valuable objects in the Iberian culture.

Acknowledgements The authors acknowledge the collaboration of the staff from the museums where the pieces are deposited (Museu Arqueològic Municipal Camil Visedo de Alcoi, Museo de Arte Ibérico El Cigarralejo de Mula, Museo Arqueológico Municipal Jerónimo Molina de Jumilla y Museu Arqueològic de Catalunya-Barcelona) and all the people that have help us through this paper, especially José Pérez Ballester, director of the excavations of Solana del Castell, for his collaboration and Carmen García Rayo for the English assistance.

Funding information Gianni Gallelo acknowledges the financial support of the European Commission (project H2020-MSCA-IF-2015-704709-MATRIX). Agustín Pastor acknowledges the financial support of Generalitat Valenciana (PROMETEO project II/2014/077) and Ministerio de Economía y Competitividad-Feder (project CTQ 2014-52841-P and project CTQ 2012-38635). Consuelo Mata acknowledges the financial support of Generalitat Valenciana (ACOMP2015/256) and Ministerio de Economía y Competitividad (HAR2013-45770-P).

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