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EARLY VITREOUS MATERIALS FROM SPAIN

Chronological and Compositional Variability in the Bronze Age Beads from the Alicante and Albacete Provinces (about 1650–900 cal BCE)

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ABSTRACT

This research constitutes a systematic archaeological and archaeometric study on ornamental vitreous material from Spain dated to the Late and Final Bronze Age (LBA–FBA, about 1650–900 cal BCE). Our multi-analytical approach combines morphometric, textural, and chemico-mineralogical analyses with typo-chronological and spatial investigations to diachronically quantify the distribution of the beads and highlight how their recipes vary over time. The analyses were performed on a set of 17 beads coming from four archaeological sites in the Alicante (Cabezo Redondo, Cueva de las Delicias, and Peña Negra) and Albacete (El Amarejo) provinces. We were able to document the first mixed-alkali glassy faience ever recorded in Spain and the oldest Egyptian blue bead discovered in western Europe. Additionally, we discovered that plant-ash glasses seem to predominate in this area during the LBA and up to the early FBA. It is only during the FBA (tenth to ninth century BCE) that the earliest natron glasses start to appear, and they show the iron-rich and the aluminum-cobalt variants already observed in numerous beads from European and Mediterranean contexts dated from the tenth to the eighth century BCE.

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During the Late Bronze Age (LBA), the Mediterranean world was an interconnected hub of cultures, trades, and technological innovations.[1] Multiple research projects carried out on the beads and pendants from Italy, France, the Aegean, central Europe, and the eastern Mediterranean clearly evidence that this is also a key period for the

1 Iacono and others 2022.

history and technological development of vitreous materials.[2] A series of seminal archaeological and archaeometric studies allowed researchers to correlate specific chronological phases of the European Bronze Age (BA) and Early Iron Age (EIA) to well-defined compositional variations, which were attributed either to local production centers or to long-distance trade networks.[3]

Despite the abundant research carried out on the BA vitreous materials unearthed in the central and eastern Mediterranean regions, little is known regarding the technology and composition of the earliest protohistoric ornaments in vitreous materials from the Iberian Peninsula. In order to fill this gap, 17 monochrome and polychrome beads coming from four archaeological sites in the area surrounding Alicante and Albacete (namely, Cabezo Redondo, Cueva de las Delicias, El Amarejo, and Peña Negra) were selected based on provenance, typology, stratigraphy, dating, physical characteristics (e.g., color, opacity, texture, conservation state), and context of discovery (burial or settlement) to offer a preliminary overview of the vitreous materials which were circulating in southeastern Spain between the LBA and the Final Bronze Age (FBA) (locally about 1650–900 cal BCE). This is a period of major transformations: first with the disintegration of the Argaric culture and the appearance of new central places in its periphery, and later—about 1250 cal BCE—with a widespread rarefaction of the sites.[4] In the southeast of the peninsula, this crisis was followed by an expansive phase dated between the end of the tenth and the beginning of the ninth century BCE, which is especially evident at the bottom of the valleys and near the coast and reflects a very dynamic system of object circulation and cultural transfer via terrestrial, fluvial, and maritime routes.

The analytical investigations on pre-Roman vitreous materials from Spain are unfortunately still limited. Among the oldest objects analyzed before the current work, we can mention one single bead from Gatas (Turre, Andalusia), phase 4 (1700–1500 cal BCE);[5] 14 from the FBA site of Peña Negra, phase I (tenth century to 750/725 cal BCE); and 25 from the Phoenician site of La Fonteta, phases I (about 725–700 BCE), II (about 700–650 BCE), and VI (about 580–560 BCE).[6] However, the elemental analyses carried out on the beads from these last two sites were acquired by means of surface portable X-ray fluorescence (pXRF), which is not always sufficient to obtain meaningful data for ancient vitreous materials. Hence, the 17 beads investigated in this paper represent a reference assemblage of BA ornaments in vitreous materials from Spain. The multi-analytical study of these objects is of utmost relevance for disclosing information about the raw materials, the production technologies, and the compositional recipes which were circulating in this phase in the Iberian Peninsula, also in comparison with what is known about objects outside the peninsula. But what is even more important is that they can offer novel insights on the long-distance trade networks connecting the Mediterranean Sea during the late second millennium BCE and the early first millennium BCE, a crucial period of Spanish later prehistory and protohistory.

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2 Among others, Artioli and Angelini 2013; Conte and others 2019; Henderson 1988; Karatasios and Triantaphyllidis 2014; Paynter and Jackson 2022; Purowski, Kępa, and Wagner 2018; Varberg, Gratuze, and Kaul 2015.

3 See the synthesis in Angelini, Gratuze, and Artioli 2019; Lončarić and Costa 2023.

4 Lull and others 2013.

5 Henderson 1999.

6 Martínez Mira and Vilaplana Ortego 2014.

MATERIALS AND METHODS

Analytical techniques

Objects were observed for traceological markers, photographed, and sampled with a stereoscopic microscope (SM), an Olympus SZ61-TR, equipped with a Bresser MikroCamII 12 megapixel digital camera and operated by MikroCamLabII software. The images were recorded at multiple focal planes and reconstructed using the focus stacking software Helicon Focus.

The detached chips (dimensions on average 500–700 x 500–700 μm) were embedded in epoxy resin, ground with silicon carbide paper (1200–4000 mesh), and polished with diamond suspension (5 and 1 μm) in the laboratories of the Dipartimento di Geoscienze, Università degli Studi di Padova (Department of Geosciences, University of Padua, Italy), to obtain a flat and smooth cross-sectional surface. Samples were analyzed with an FEI Quanta 200 environmental scanning electron microscope (ESEM) equipped with tungsten filament and an Xplore 30 (Oxford Instruments) energy dispersive (EDS) X-ray detector at the Centro Analisi e Servizi per la Certificazione (CEASC; Center of Analyses and Services for Certification) at the University of Padua. Imaging in secondary (SE) and backscattered electrons (BSE) was used to study the texture and the weathering processes, and to map and quantify mineral inclusions related to coloring/opacifying agents, relics from the batch, and newly formed phases. Point and areal analyses were performed at standard operating conditions of 20 kV voltage, chamber pressure of 0.53 Torr, spot size of 1–5 μm, and a duration of 30 seconds per point/areal acquisition. All the semiquantitative EDS analyses are normalized at 100% by default. The detection limit is variable but in the order of 0.1 wt %, except for elements with a low atomic number or with overlapping peaks.

After SEM-EDS analyses, the vitreous phase of 20 samples (plus 1 zoning) and a selection of inclusions were investigated by electron probe microanalysis (EPMA) to obtain chemical data with very high precision and accuracy. The instrument used was the JEOL 8200 Super Probe with five wavelength-dispersive spectrometers (WDS) at the Dipartimento per le Scienze della Terra, Università degli Studi di Milano Statale (Earth Sciences Department of the University of Milan). Eighteen elements were analyzed: Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, Mn, Fe, Co, Cu, As, Sn, Sb, and Pb. Corning glass standards A and B were analyzed before each session (results in **Table 2**) to evaluate the accuracy (values) and the precision in terms of reproducibility of the data (Standard Deviations), which turned out to be consistent with those recorded in previous experimental rounds with the same instrument.[7]

Working conditions were set at 15 kV accelerating voltage and 5 μA sample current; spot size was varied according to the textural characteristics of the samples: for the transparent glasses and the interparticle glass in sample AM-BV-29, we opted for 5 measures with beam defocused at 10 μm to reduce alkali loss. Opaque samples with homogeneously dispersed crystalline inclusions were investigated with 4–6 measures at 5 μm spot size; since opaque glasses in this set were all heavily weathered, we didn't need any additional point at 10 μm spot size to evaluate the sodium volatilization rates. Mineral inclusions were analyzed with a 1 μm probe diameter to reduce the contribution of the glassy matrix to the acquired data. The minimum detection limit of this technique is approximately 0.03% for most of the analyzed oxides.

7 For details, see Bettineschi and Angelini 2022, 2023.

Sites and materials

The archaeological sites in which the analyzed materials were discovered are reported in **Figure 1**. In this section, a brief synthesis on the archaeological contexts and the consistency of the finds in each site will be shortly provided. **Table 1** summarizes the main archaeological and physical data related to the analyzed samples, including provenance, chronology, typology, state of conservation, dimensions, color, weight, archaeological data on the label, and the number and location of the samples detached. For ease of reference, it also includes the analytical code of each sample, and a set of relevant notes. **Plate 1** presents photographs of all the analyzed samples.

Cabezo Redondo

Cabezo Redondo is a settlement located on a prominent hill in the municipal district of Villena (Alicante), approximately 50 km from the Mediterranean coast. All the vitreous materials belong to the second phase of occupation, whose structures are located over the summit of the hill and on its western slope. A set of over 50 radiocarbon dates offers the chance to precisely define the lifespan of this second phase between about 1650 and 1300/1250 cal BCE, largely coinciding with the local LBA.

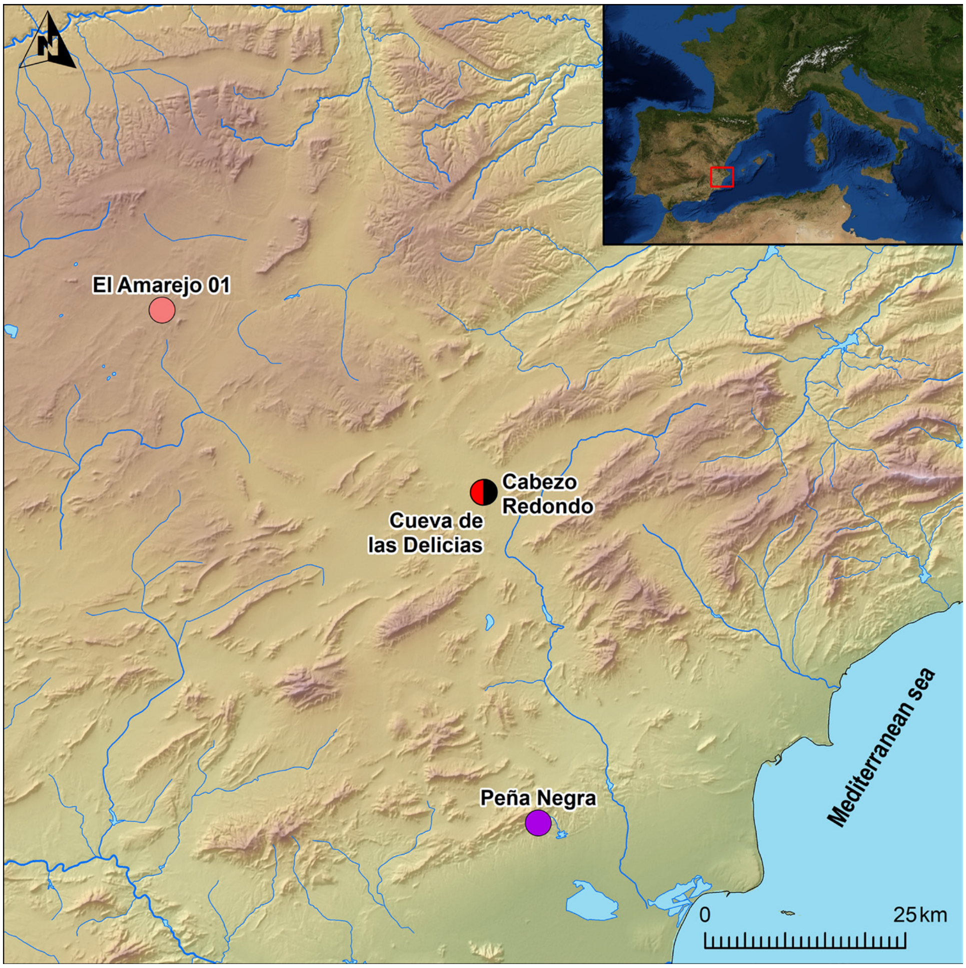


FIG 1. Map of the sites where the beads were found. (Map: G. García Atiénzar)

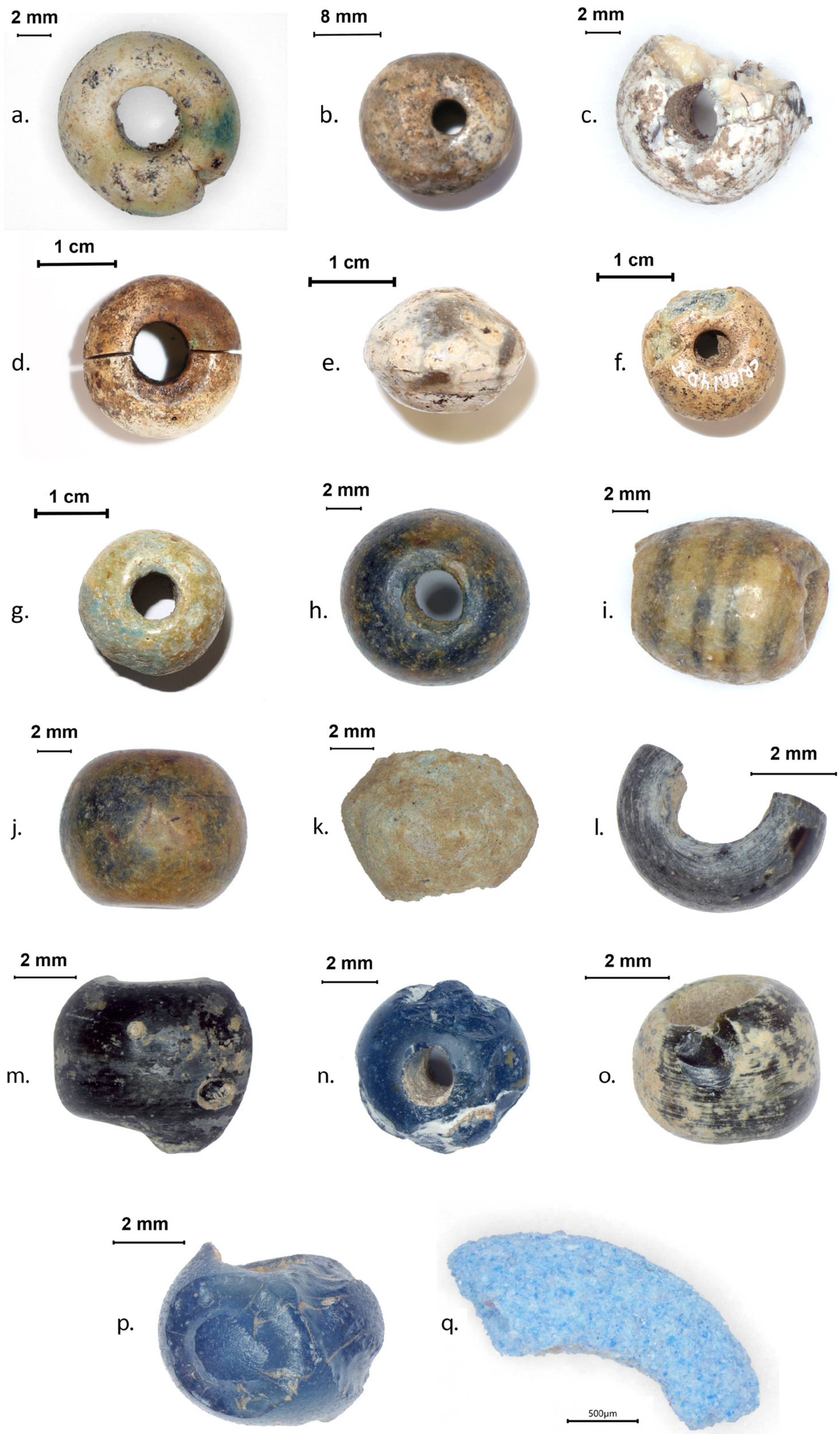


PLATE 1. Multifocal stereomicroscope images of the beads sampled ordered according to Table 1. (a) CR-AV-25; (b) CR-GV-2255; (c) CR-AAF-003 M and Bi; (d) CR-GV-85; (e) CR-BS-555; (f) CR-GA-498; (g) CR-GA-544; (h) CD-GV-03; (i) CD-BoD-04 Bi and V; (j) CD-GV-02; (k) AM-BV-29; (l) PN-AN-80; (m) PN-GN-978; (n) PN-O878 B and Bi; (o) PN-GV-87; (p) PN-OB-13; (q) PN-DEB-15. (Photos and processing: C. Bettineschi)

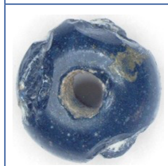
TABLE 1. List and Description of the Beads Sampled. (Table: C. Bettineschi)

Image	Code	Provenance	Period	Absolute Chronology	Typology (descriptive)	Material	Conservation	Dimensions (mm)	Color	Munsell Color Code	Weight (g)	Archaeological data on label	Sampling	Number of samples	Notes
	CR-AV-25	Cabezo Redondo	LBA	3240 ± 30 BP cal BCE 2σ 1607-1581 (4.4%) 1545-1433 (91.0%)	annular	glass	strongly weathered with limited areas with well-preserved glass	Ømax: 10.1; Øhole: 3.8; h. max: 5.6	turquoise-green (well preserved); whitish (weathering)	well preserved: GLAY2 5/10 (greenish gray)	0.525	CR'17, 29/VI, CORTE1, elem. adorno 1, inv. 25, VE 17108	hole	1	Big longitudinal fracture.
	CR-GV-2255	Cabezo Redondo	LBA	3140 ± 60 BP cal BCE 2σ 1518-1260 (95.1%) 1240-1235 (0.4%)	short globular with oval section	glass	strongly weathered with limited areas with preserved glass	Ømax: 18.1; Øhole: 3.7; h. max: 13.5 (roundish side)	green (well preserved); whitish (weathering)	well preserved: GLAY1 4/10GY (dark greenish gray)	4.733	CR198, 23/7, corte XXID, UE2255, X = 1'10; Y = 4'60; Z = –0'54	hole	1	The hole on one side is oval rather than round. There is abundant round porosity; flux lines and deformations (rod-forming); weathering and opalescence.
	CR-AAF-003 M and Bi	Cabezo Redondo	LBA	3110 ± 30 BP cal BCE 2σ 1442-1286 (95.4%) 3130 ± 30 BP cal BC 2σ 1496–1474 (4.9%) 1461–1371 (65%) 1355–1298 (25.5%)	globular, possibily with a white thread decoration (or weathering?)	glass	very weathered	Ømax: 10.9; Øhole: 2.2; h. max: 8.1	white-yellowish to opalescent (weathering); amber (well preserved)	external: WHITE N/9.5; internal: WHITE 10YR 9/2 (pale orange-yellow)	0.595	CR'03 22/07, VE003001, pasta vitrea	fragments already detached	2	Reattached in a historical restoration. Note: The alleged white thread decoration does not show any mineral opacifier in SEM-BSE.
	CR-GV-85	Cabezo Redondo	LBA	3200 ± 30 BP cal BCE 2σ 1513-1417 (95.4%)	short globular	glass	broken in half, outside very weathered but in section remains of well-preserved glass	Ømax: 21.2; Øhole: 7.4; h. max: 13.2	opaque green–turquoise (well preserved); whitish (weathering)	well preserved: GLAY1 5G 6/1 (greenish gray)	6.125	CAB'10, CR10, 28-IX, VE100715-85, marfil1 (cuenta)	hole	1	
	CR-BS-555	Cabezo Redondo	LBA	3550 ± 55 BP cal BCE 2σ 2034-1741 (94.8%) 1709-1700 (0.6%)	biconical, with possible spiral decoration on one side	glass	very weathered	Ømax: 18.6; Øhole: 2.6; h. max: 14.2	green (decently preserved); white-yellowish (weathering)	GLAY1 4/5G 4/1 (graysh green)	4.347	C. REDONDO, n.cat. 035-0555	a micro-chip from the maximum expansion where the best-preserved glass is located	1	The alleged spiral decoration is only on one side of the bead; it would have 3 lines, from around the hole to the maximum expansion; most probably it's just an evidence of flux lines.

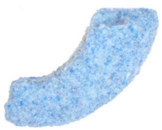
(Contd.)

Image	Code	Provenance	Period	Absolute Chronology	Typology (descrip-tive)	Material	Conserva-tion	Dimensions (mm)	Color	Munsell Color Code	Weight (g)	Archaeo-logical data on label	Sampling	Number of samples	Notes
	CR-GA-498	Cabezo Redondo	LBA	1650–1250 cal BCE	short globular	glass	very weathered, fractured, with limited remains of well-preserved glass under a very thick patina	Ømax: 18.6; Øhole: 4.3 ; h. max: 12.3	dark blue (well preserved); white-yellowish (weathering)	GLAY2 2.5/10B (bluish black)	4.520	C. REDONDO, n.cat. 035-0498	a micro-chip from the fracture where the best-preserved glass is located	1	
	CR-GA-544	Cabezo Redondo	LBA	1650–1250 cal BCE	short globular	glass	partly weathered and frac-tured, but with major remaining of well-preserved glass under a thick patina	Ømax: 19.3; Øhole: 13.2; h. max: 5.2	light blue	GLAY2 6/10BG (green-ish gray)	5.995	C. REDONDO, n.cat. 035-0544	hole	1	
	CD-GV-03	Cueva de las Delicias	LBA-FBA	1600–1000 cal BCE	short globular	glass	well-preserved glass, sometimes under a thick brown patina	Ømax: 10.9; Øhole: 3.1; h. max: 8.2	dark blue	GLAY1 5G 2.5/1 (green-ish black)	1.264	C008-0003	hole	1	Very bubblish; detaching mark and some residues of a fired textile thread seemingly present. Looking at the detached chips, the original color seems to be transparent blue.
	CD-BoD-04 Bi and V	Cueva de las Delicias	LBA-FBA	1600–1000 cal BCE	barrel-shaped with spiral thread	glass	well-preserved glass, sometimes under a thick brown patina	Ømax: 7.9; Øhole: 3.1; h. max: 9.1	dark green (body); white (decora-tion); brown (patina)	body: GLAY1 4/N (dark gray); decora-tion: WHITE 2.5Y 8.5/2 (pale yellow)	0.544	C008-0004	hole	2	Looking at the detached chips, the original color of the body seems to be transparent green.
	CD-GV-02	Cueva de las Delicias	LBA-FBA	1600–1000 cal BCE	short globular	glass	well-preserved glass, sometimes under a thick brown patina	Ømax: 11.6; Øhole: 2.5; h. max: 9.5	dark green-ish–blue (body); brown (patina)	GLAY1 5G 3/2 (very dark graysh green)	1.736	C008-0002	hole	1	Looking at the detached chips, the original color of the body seems to be transparent light blue.

(Contd.)

Image	Code	Provenance	Period	Absolute Chronology	Typology (descriptive)	Material	Conservation	Dimensions (mm)	Color	Munsell Color Code	Weight (g)	Archaeological data on label	Sampling	Number of samples	Notes
	AM-BV-29	El Amarejo 01	LBA-FBA	1700–950 cal BCE	biconical	glassy faience	weathered, rough, but well-preserved material is still present inside	Ømax: 7.9; Øhole: 3.5; h. max: 6.1	greenish–light blue	GLEY1 5G 7/1 (light-greenish gray)	0.261	AM1, '21, VE 109.29, 30/09/21, n. inv. 1	hole	1	Irregular hole edges; surface weathered and corroded.
	PN-AN-80	Peña Negra	FBA	10–9th cent. BCE	annular	glass	fractured in half, opalescent patina	Ømax: 5.2; Øhole: 2.3; h. max: 3.2	very dark greenish/black	GLEY2 2.5/5PB (bluish black)	0.094	PN-15980	sectioned the smallest fragment	1	Flux lines (rod forming); detaching mark; abundant big, round bubbles. Looking at the detached chips, the original color of the body seems to be transparent green.
	PN-GN-978	Peña Negra	FBA	10–9th cent. BCE	long oblate? globular?	glass	fragmented in 3 pieces; opalescent but well-preserved material is still present	Ømax: 6.5; Øhole: 2.5; h. max: 6.8	very dark greenish/black	GLEY2 2.5/5PB (bluish black)	0.36	PN-15978	sectioned the smallest fragment	1	Flux lines (rod-forming); detaching mark; abundant big, round bubbles. Looking at the detached chips, the original color of the body seems to be transparent green.
	PN-O878 B and Bi	Peña Negra	FBA	10–9th cent. BCE	short globular with profiled eyes	glass	blue glass well preserved; white decoration weathered and almost completely gone	Ømax: 7.9; Øhole: 2.0; h. max: 5.8	dark blue (body); white (decoration)	body: GLEY2 2.5/5PB (bluish black); decoration: GLEY1 8/N (white)	0.421	PN-7878	hole (blue); decoration (white)	2	Flux lines; abundant big, round bubbles. Looking at the detached chips, the original color of the body seems to be transparent dark blue. Shrinking marks in the area where the white decoration was applied.
	PN-GV-87	Peña Negra	FBA	10–9th cent. BCE	short globular	glass	only minor chipping	Ømax: 4.7; Øhole: 2.2; h. max: 3.6	very dark greenish/black	GLAY1 2.5 5GY (greenish black)	0.085	PN87; 7-8-11, a4, cuenta vitrea con otra bronzo, n. 15680	hole, near the fractured edge	1	Flux lines (rod forming); two detaching markers on the oppisite sides. Looking at the detached chips, the original color of the body seems to be transparent green.

(Contd.)

Image	Code	Provenance	Period	Absolute Chronology	Typology (descriptive)	Material	Conservation	Dimensions (mm)	Color	Munsell Color Code	Weight (g)	Archaeological data on label	Sampling	Number of samples	Notes
	PN-OB-13	Peña Negra	FBA-EIA	surface (no context)	short globular with profiled eyes	glass	body good; decoration totally lost	Ømax: 5.9; Øhole: 2.1; h. max: 4.5	dark blue	Munsell 22, 5PB 3/10	0.203	PN19-V-13	hole	1	There's only a negative impression where the profiled-eye decoration was originally located and several markers of shrinking, indicating the different temperatures of the two glasses during the application of the decoration.
	PN-DEB-15	Peña Negra	FBA	10–9th cent. BCE	short cylindrical (disc bead)	Egyptian blue	fractured, only a quarter remains	L1: 2.5; L2: 1.0; L3: 1.0	blue	Munsell 22, 2.5PB 8/6	0.001	PN15-II-H-4072-15	sectioned in half	1	

The material record of Cabezo Redondo is extraordinary in quality and variety and testifies to regular contacts with the southeast and the Meseta, but also with areas outside the peninsula, both toward the Atlantic and with the Mediterranean.[8] Among these luxury products, we can mention gold tutuli with comparisons in Catalonia and Portugal, but also silver, glass, and ivory. These high-status goods are present in Cabezo Redondo and other contexts in the Levant of the Iberian Peninsula and have been suggested to reflect the activation, around 1500 cal BCE, of a series of minor circuits integrated in exchange networks between northern Italy, Corsica, Sardinia, and the Gulf of Lyon.[9]

The site has yielded a total of eight potential glass beads, all moderately to heavily weathered, out of which seven were selected and sampled for this study. An additional green biconical bead (catalogue 035-0555) was also observed in stereomicroscopy but turned out to be made of stone (likely steatite), without any original glaze layer, so it was not considered in this study. The archaeological contextualization—including typology, chronology, detailed recovery context, and comparisons of each piece—was recently published by Virginia Barciela González and colleagues; given the space limits, the reader is kindly referred to that work for further specifics.[10]

From a material perspective, the seven beads sampled can be positively identified as true glass; unfortunately, the weathering makes it hard to recognize traceological or production markers in this set. It is worth noting that two beads seem to be decorated with a thread in contrasting color (apparently white on brown and on blue-green glass), now completely transformed into the same yellowish tint as the body. In the case of the bead CR-AAF-003, it was possible to sample both a portion of the body and of the alleged decoration (CR-AAF-003 M and Bi). Electronic imaging has demonstrated without a doubt the absence of mineral opacifiers which would be expected in a white or yellow thread: if a decoration was originally there, it was certainly of transparent glass, a solution which is, however, rather uncommon in LBA and FBA ornaments. Unfortunately, while the glass of the body still preserves non-corroded areas, that of the alleged decoration is completely altered. Sample CR-BS-555 is an irregular biconical bead seemingly characterized by a helicoidal thread running along half of the body, from one perforation to its maximum expansion. In this case, the alleged thread can be more probably interpreted as a residual and highly equivocal trace of flux lines connected to the rod-forming process. Despite the severe weathering, this hypothesis is supported on one hand by the lack of coeval comparative specimens (as far as the authors are aware) and on the other by stereomicroscopy observations (**Fig. 2a**), which point in this interpretive direction, even in the absence of definitive proof.

Cueva de las Delicias

Cueva de las Delicias (Villena) is a funerary complex comprising a series of burial caves which remained in use for about six centuries (1600–1000 BCE). Ten beads were discovered at the site, and they were first interpreted as green stone.[11] Nine of them were later correctly recognized as weathered glasses.[12] From the chronological point of view, the association of the beads with metal objects suggests a chronology in the

8 Soler García 1987; Hernández Pérez, García Atiénzar, and Barciela González 2016.
9 Kristiansen and Larsson 2005.
10 Barciela González, García Atiénzar, and Hernández Pérez 2021.
11 Soler García 1981.
12 Barciela González 2008, 2015.

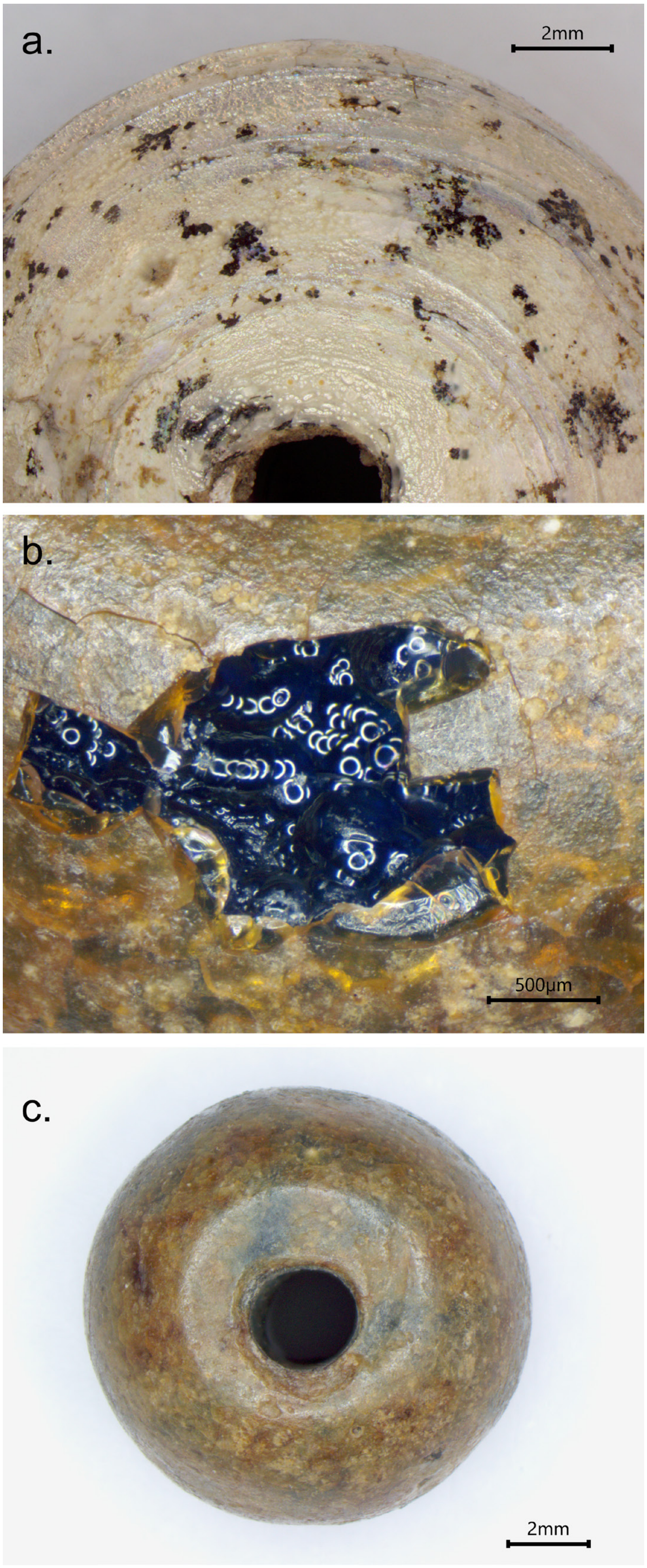


FIG 2. Multifocal stereomicroscope images: (a) CR-BS-555, alleged decoration interpreted as evidence of flux lines; (b) CD-GV-03, thick yellowish patina that covers the unweathered dark-blue glass visible in the center of the image; (c) CD-GV-02, use and wear traces. (Photos and processing: C. Bettineschi)

range of LBA–FBA.[13] Recently, a belt buckle and other related metal objects from the same site have also been assigned to an FBA chronology. Similar pieces have been documented in Chalcolithic megaliths reused in this same period. It is the case of La Sabina 49 (Granada), where a belt buckle was found associated with a blue glass bead which is now lost.[14]

Out of the nine beads discovered, only three were available to be analyzed during this study and they were all sampled. From the typological point of view, the best connotated specimen is the dark-green barrel bead with a helicoidal thread in white glass, CD-BoD-04, while the other two occurrences have generic types: very dark glass and short, globular shape. Barrel beads with helicoidal decoration appear in Southern Italy during the local Middle Bronze Age (MBA) 3 (1450–1330 BCE) and become more common in Italian and European contexts during the Recent Bronze Age. However, they are typical of the FBA, when they were abundantly found in Italy and north of the Alps, especially in Switzerland, Germany, Austria, and France.[15] The majority of them have a light-blue to blue hue and a mixed-alkali or plant-ash composition. Strict comparisons in very dark glass can be found in Sardinia (one dark-blue specimen in the Giants' tomb of Perda 'e Accuzzai, Villa San Pietro, Cagliari, Italy) with a chronology between the thirteenth and the end of the twelfth century BCE, but also from the Kermorvan peninsula (Le Conquet, Brittany, France), and from different sites in Alsace (France), all dated within the limits of the local FBA2 (eleventh to tenth century BCE).[16] In other caves in Alicante (Cova d'En Pardo), there is sure radiocarbon evidence of burials in the period between 1250 and 1000 cal BCE. Combining these pieces of evidence, we can tentatively suggest a similar date for the beads from Cueva de las Delicias.

Despite being covered by a thick layer of yellowish patina (**Fig. 2b**), the beads from Cueva de las Delicias show interesting marks related to use and wear. Looking around the perforation of CD-GV-02, one can recognize rubbing traces and a series of small chipping marks, both concentrated on one half of the perimeter of the hole (**Fig. 2c**). The distribution of these traces indicates the position where the string was typically resting and generated tension. Also, the presence of bead-on-bead wear implies that these ornaments were surely worn for a certain amount of time before being intentionally deposited as grave goods.

El Amarejo

The site El Amarejo 01 is a megalithic burial located in Bonete (Albacete). The structure was originally covered by an earthen and gravel tumulus and built with vertical stone slabs which create two separate spaces (room 1 and room 2) and an access corridor.

The human remains consist of a total of 3,019 skeletal elements, with a minimum number of five adult individuals (probably three males and two females) buried in room 1, and five individuals (four adults and one infant) in room 2. The remains of another individual were recovered on the outside, in what is probably the access to the burial chamber itself.

The grave goods are scarce but include luxury objects such as a spherical gold bead, a two-sided awl in bronze, and a bronze earring. There are also several beads in

13 Simón García 1998.
14 Lorrio Alvarado 2008; Pernas García 2012.
15 Bellintani and Angelini 2020; Angelini and Olmeda 2018.
16 Plouin, Koenig, and Gratuze 2012; Cherel, Gratuze, and Simon 2018.

stone and shell, one in lignite, and one in vitreous material, along with the fragment of a bracelet, a small group of handmade pottery sherds, and a fragment of flint blade.

The grave goods, especially the metallographic composition of the awl and the technology of the gold bead, reveal an advanced BA chronology, most probably after 1500 cal BCE (LBA) given the percentage of tin documented in the awl and the technology applied in the gold bead (possibly the lost-wax process). However, we cannot rule out an early beginning supported by the presence of the flint blade fragment that typologically would belong to the Chalcolithic period.

As already mentioned, in terms of vitreous materials, the burial yielded only one light-blue bead (previously unpublished) that was identified as glassy faience during this study thanks to stereomicroscopic observations (also later confirmed by SEM imaging). From a typological perspective, it is a light-blue biconical bead, very similar in shape and dimension to the faience occurrences of the EBA unearthed throughout Europe.[17] Its classification as glassy faience, however, supports a later dating, starting from about 1700–1650 BCE onward, as testified by the conical buttons and the annular, cylindrical, segmented, and short-biconical beads recovered in northern and central Italy in contexts dated to the local MBA 1A (1700/1650–1550 BCE).[18] While the biconical type is generally rather rare in Europe, a few occurrences in glassy faience are known up to the FBA, such as the specimens from Boiron[19] and Hauterive-Champreveyre.[20] The bead, thus, is of little help in restricting the wide chronological range attributed to the burial site. Radiocarbon dating is currently in progress on the human remains to further refine the timespan of the depositions and hence of the grave goods discovered.

Peña Negra I

The protohistoric site of Peña Negra (Crevillent, Alicante) is one of the most outstanding sites in the southeast of the Iberian Peninsula. The first interventions date back to the decade 1976–1987, under the direction of Alfredo González Prats.[21] In 2014 research was resumed as part of a multidisciplinary project.[22] The first occupation of the area refers to the FBA, in the so-called Peña Negra I horizon, dated approximately from the tenth century cal BCE to 750–725 BCE, although the site reaches a fully urban character only in the EIA (Peña Negra II). Its abandonment occurs toward the third quarter of the sixth century BCE. Besides ivory working, testified by multiple finished pieces (fragmented) and matrices or scraps, the site yielded several beads in carnelian and vitreous material, as well as abundant copper-based objects.[23] Additionally, historical excavations demonstrated the presence of a metallurgical workshop producing different types of weapons, ornaments, and tools, some of them for daily use, but others made for export—evidence of commercial contacts with Atlantic Europe and the Mediterranean.[24]

All the beads analyzed during this research were found in the settlement, and most frequently in the FBA levels of Sector II; this is an area of dwellings, craft activities,

17 Sheridan, Eremin, and Shortland 2004; Tite, Shortland, and Angelini 2008.
18 Santopadre and Verità 2000; Bellintani and others 2005.
19 Angelini and Olmeda 2018.
20 Henderson 1988.
21 González Prats 1990.
22 Lorrio Alvarado and others 2020.
23 Camacho Rodríguez and others 2022; Lorrio Alvarado and Torres Ortiz 2022.
24 Ruiz-Gálvez Priego 1990.

and dumps with a dense human occupation that begins in the FBA and extends throughout the EIA.[25] Particularly, the dark-green beads PN-AN-80, PN-GN-978, and PN-GV-87 (unpublished) were all recovered in 1987 within an important metallurgical dump of the FBA. The profiled eye bead PN-O878 was recovered during the campaigns of 1983–1985 in an area of circular dwellings belonging to the oldest phase of the FBA occupation, similarly to the Egyptian-blue specimen PN-DEB-15 (unpublished). Finally, PN-OB-13 (unpublished) comes from recent surface surveys in Sector V, lacking any stratigraphic association. However, it is identical in shape and color of the body to PN-O878, even if traces of the thread are no longer present here due to aggressive weathering processes.

From a typological perspective, profiled eye beads (also known as ring-eye beads) are among the most common and long-lasting types recovered in EIA contexts. In central Italy, their appearance is dated in EIA1 (about 950–800 BCE), even if their spread reaches its climax in EIA2 (800–720 BCE) with a progressive decrease during the Orientalizing phase (720–670 BCE).[26] They appear in two main variants, with yellow or white decoration; however, in many occurrences the thread is lost, making it impossible to determine its original color. This is most probably due to a combination of compositional and technological factors. Looking at the beads from Peña Negra, we could infer that the application of the decoration occurred when the body was already partially cooled, preventing a good sintering between the two glasses. This is evident in the shrinking marks currently visible in both samples PN-OB-13 and PN-O878 (**Fig. 3a, b**), a characteristic they share with several other beads of the same type recovered in Spain and abroad. Profiled eye beads are also known in Slovenia, from Novo Mesto (tenth to ninth century BCE),[27] and in the Iberian Peninsula, for example, from the necropolis of Las Cumbres, Cádiz (about 800 BCE).[28]

The three other beads from the site are characterized by very simple shape (irregularly annular or short globular) but very dark glass. They can thus be attributed to the so-called "black" glasses, one of the classes which emerge in the transitional period from the tenth to the eighth century BCE. Despite appearing black when massive, the color of these glasses is either very dark green or very dark blue. They are widespread in Europe (France, Poland) and in the Mediterranean world (Portugal, Spain, Tunisia, Turkey), being particularly common in Italy. Though appearing similar, they have been shown to belong to a series of distinct compositional classes.[29]

During sampling, it was possible to clarify that the "black" beads from Peña Negra are all made of very dark green glass (**Fig. 4a**). Additionally, both the eye beads and the black beads from Peña Negra show oriented bubbles, flux lines, and one or more raised thread-ends (**Fig. 3a**), which are sure evidence that they were produced by rod-forming, using the winding technique.

In the recent investigations of the site, a tiny fragment of a short cylinder bead in Egyptian blue was also recovered (PN-DEB-15; **Fig. 3c**) within levels of the LBA (tenth to ninth century BCE). A recent review of the production centers for this material

25 González Prats 1990.
26 Yatsuk and others 2023; Koch 2022.
27 Šmit, Laharnar, and Turk 2020.
28 Palomar, Peña-Poza, and Conde 2009.
29 Conte and others 2018; Van Ham-Meert and others 2019; Gomes 2021; Koch 2021.

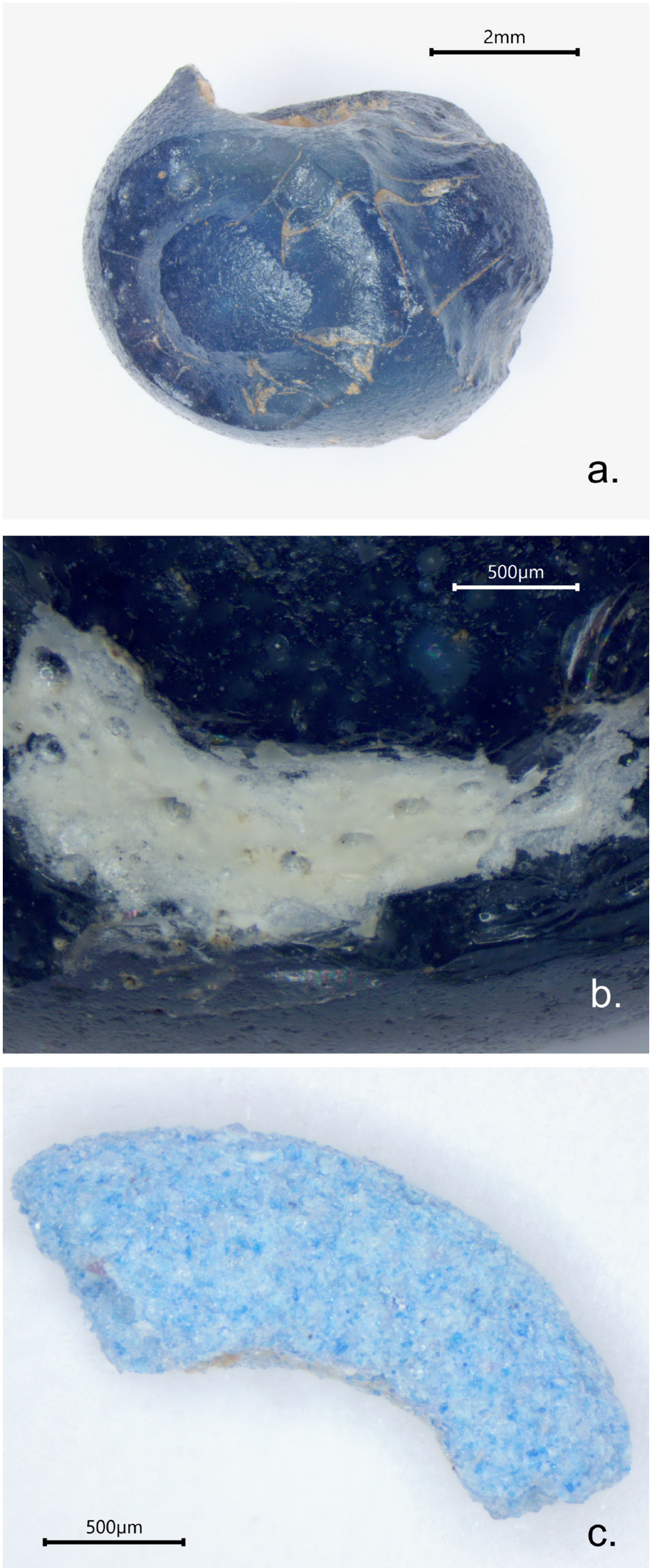


FIG 3. Multifocal stereomicroscope images: (a) PN-OB-13, showing shrinking marks corresponding with the lost decoration and raised thread-end; (b) PN-O878, detail of the remaining decoration; (c) PN-DEB-15, Egyptian blue bead. (Photos and processing: C. Bettineschi)

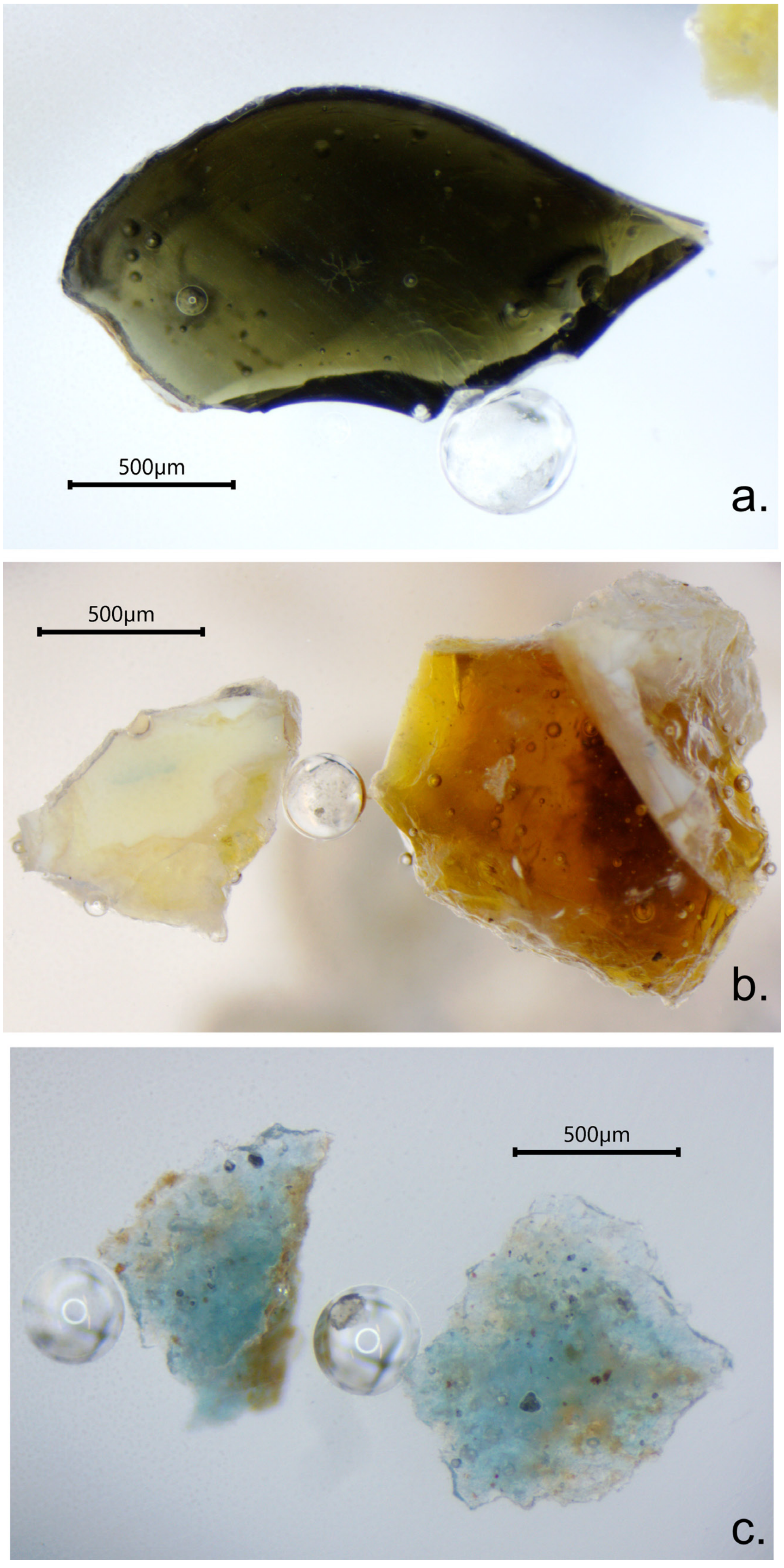


FIG 4. Stereomicroscope images of the samples: (a) PN-GN-978, the radial cracking in the center is due to the presence of a silica grain; (b) CR-AAF-003, amber glass with the alleged white decoration; (c) Glassy faience AM-BV-29, showing a mix of vitreous and mineral phases and a uniform light-blue color. (Photos: C. Bettineschi)

evidenced that during the BA and IA, workshops and semifinished products were only found in the eastern Mediterranean and in the Near Eastern regions, with the westernmost occurrence in Tiryns, Greece.[30] Westward of Greece, Egyptian blue beads are extremely rare prior to the Roman period. We can cite a scarab discovered at the site of La Fonteta (Guardamar del Segura, Alicante, Spain) from phase III (second half of the seventh century BCE), and a scaraboid and square bead from phase VI (580–560 BCE).[31] There is also a long cylindrical bead found in Vinha das Caliças 4 (Beja, Portugal), a sixth-century BCE necropolis.[32] Additionally, an Egyptian blue bead dating to the eighth century BCE was excavated at the site of Runnymede, in southern England.[33] In France, Germany, Switzerland, Austria, and Italy, no Egyptian blue beads were ever recovered—at least up to now—from the LBA or EIA phases. For the time being, the bead from Peña Negra is the earliest known Egyptian blue ornament discovered in the western Mediterranean.

RESULTS

Glassy matrix

The major and minor elements in the amorphous phase of the analyzed samples are reported in **Table 2** as weight percent (wt %) of the oxides. The chemical analyses show a wide range of compositions and a non-negligible number of weathered glasses ($Na_2O + K_2O < 4\%$), which include all opaque whites (CR-AAF-003Bi, CD-BoD-04Bi, PN-0878-Bi) and three out of seven transparent green/blue glasses from Cabezo Redondo (CR-GA-498, CR-BS-555, CR-GV-2255) (**Figs. 5, 6**). To avoid any confusion, compositional data related to altered samples will not be discussed further or plotted in the next diagrams; they will only be considered for textural observations and for the identification of the mineral opacifiers.

In general terms, silica contents range from 59.6% to 67.7% in glasses and reach the peak value of 70.9% in the glassy faience sample from El Amarejo. Soda goes from 6.1% to 19.9%, potash from approximately 0.2% to 11.6%, lime from 1.2% to 5.7%, magnesia from 0.4% to 7.1%, and alumina from 1% to 6.5%.

The classic plot K_2O vs. MgO (**Fig. 6**) emphasizes the presence of at least three compositional macro-groups, which are essentially related to the type of fluxing agent employed, plus three outliers all originating from the site of Peña Negra (the dark-blue glasses PN-O878-B and PN-OB-13, and the dark-green glass PN-AN-80), which will be presented separately. The first group includes only the light-blue glassy faience from El Amarejo (AM-BV-29). The sample is characterized by a low-magnesium (MgO 0.37%), high-potassium (K_2O 11.6%) (LMHK) composition,[34] also known as mixed alkali, due to the presence of inversely proportional amounts of soda and potash (Na_2O 6.15%). The second set includes all the non-weathered green, brown, and blue glasses from Cabezo Redondo and Cueva de las Delicias, for a total of seven samples (CR-GA-544, CR-AAF-03M, CR-AV-25, CR-GV-85, CD-GV-02, CD-GV-03, CD-BoD-04V). It comprises high-magnesium (MgO 5.4–7.1%) glasses (HMG) that contain relevant levels of potash (2.9–3.5%) and lime (4.9–5.7%) typical of a plant-ash

30 Kovalev and others 2023.
31 Martínez Mira and Vilaplana Ortego 2014; Almagro-Gorbea and others 2022.
32 Costa and others 2022.
33 Hatton, Shortland, and Tite 2008.
34 Angelini and others 2004; Henderson and others 2015; Venclová and others 2011.

TABLE 2. Results of the EPMA Analysis on the Amorphous Phase of the Samples as a Mean of 4–5 Point Analyses Expressed in Oxides Weight Percent (wt %), with Standard Deviation (SD). (Table: C. Bettineschi)

Sample Name	Color	Na_2O		MgO		Al_2O_3		SiO_2		P_2O_5		SO_3		Cl		K_2O		CaO	
		mean	SD	mean	SD	mean	SD	mean	SD	mean	SD	mean	SD	mean	SD	mean	SD	mean	SD
GLASSY FAIENCE LMHK																			
AM-BV-29	light blue	6.15	0.55	0.37	0.11	1.50	0.52	70.86	0.48	0.18	0.05	0.11	0.06	0.36	0.10	11.61	0.53	2.90	0.48
HMG																			
CR-GA-544	light blue	19.68	0.13	7.13	0.07	2.15	0.05	58.63	1.53	0.16	0.00	0.25	0.02	0.92	0.03	3.55	0.13	4.89	0.24
CR-AV-25	green	19.18	0.20	5.65	0.09	1.15	0.03	62.96	0.61	0.16	0.02	0.29	0.03	0.81	0.04	2.90	0.02	4.89	0.05
CR-GV-85	green	19.86	0.32	6.30	0.09	1.27	0.03	61.39	0.35	0.16	0.02	0.18	0.03	0.75	0.02	3.22	0.02	5.64	0.08
CD-GV-02	blue	19.55	0.20	6.19	0.07	1.71	0.04	60.17	0.57	0.18	0.03	0.30	0.01	0.87	0.02	3.19	0.05	5.68	0.02
CD-GV-03	blue	19.02	0.29	6.32	0.05	0.99	0.05	62.32	0.14	0.18	0.01	0.28	0.04	0.82	0.02	3.07	0.05	5.63	0.08
CD-BoD-04V	green	17.75	0.28	5.42	0.20	1.79	0.05	63.97	0.36	0.16	0.03	0.31	0.03	0.79	0.01	3.13	0.07	5.43	0.32
CR-AAF-03M	transparent brown	19.20	0.20	6.11	0.09	1.65	0.01	63.17	0.96	0.16	0.03	0.32	0.05	0.95	0.04	3.03	0.03	5.40	0.04
LMG / NATRON BLACK																			
PN-GN-978	greenish black	18.07	0.19	0.58	0.03	1.13	0.05	66.34	0.88	0.11	0.02	0.54	0.07	0.39	0.03	0.97	0.03	1.20	0.02
PN-GV-87	greenish black	16.06	0.16	0.71	0.10	1.20	0.14	67.86	0.51	0.14	0.06	0.15	0.02	0.36	0.03	0.99	0.02	1.64	0.36
HMLK																			
PN-AN-80	greenish black	17.66	0.29	1.96	0.31	2.41	0.24	63.96	0.61	0.12	0.02	0.63	0.20	0.44	0.02	1.28	0.03	1.83	0.13
PN-OB-13	dark blue	17.38	0.11	4.01	0.04	5.94	0.15	65.51	1.00	0.04	0.03	0.33	0.03	0.42	0.01	0.16	0.01	4.60	0.02
PN-O878-B	dark blue	16.93	0.22	3.21	0.09	6.48	0.13	67.04	0.32	0.04	0.01	0.27	0.02	0.63	0.04	0.26	0.01	3.81	0.02
ALTERED SAMPLES																			
CR-GA-498-zonC	light blue	0.44	0.04	1.20	0.11	1.52	0.01	87.46	0.42	0.02	0.02	0.17	0.02	0.02	0.01	1.18	0.09	0.20	0.02
CR-GA-498-zonD	light blue	0.55	0.01	13.06	1.09	1.74	0.11	70.04	1.94	0.04	0.02	0.23	0.03	0.03	0.01	1.36	0.02	0.12	0.01
CR-BS-555	weathered/green	0.37	0.01	10.54	0.73	2.09	0.03	73.81	1.45	0.01	0.01	0.09	0.02	0.03	0.00	1.52	0.05	0.16	0.01
CR-GV-2255	green	0.62	0.03	20.33	0.43	1.92	0.07	56.36	0.52	0.03	0.01	0.14	0.02	0.10	0.01	0.71	0.08	0.24	0.04
OPAQUE WHITE																			
CR-AAF-03Bi	weathered/ unknown	0.84	0.04	4.85	1.21	3.54	0.04	81.76	1.52	0.02	0.02	0.16	0.05	0.41	0.02	2.58	0.05	0.24	0.01
CD-BoD-04Bi	opaque white	0.65	0.10	2.48	0.41	2.34	0.10	80.35	0.83	0.06	0.04	0.22	0.04	0.85	0.05	0.38	0.02	0.83	0.06
PN-O878-Bi	opaque white	0.06	0.05	0.38	0.04	0.30	0.03	78.72	0.28	0.06	0.01	0.47	0.07	1.01	0.07	0.19	0.05	5.73	0.09
STANDARDS																			
Corning Standard A	colorless	14.01	0.14	2.67	0.10	0.93	0.03	67.72	0.59	0.13	0.01	0.12	0.04	0.10	0.02	2.90	0.01	5.02	0.04
Corning Standard B	colorless	16.75	0.28	1.04	0.05	4.20	0.07	62.88	0.29	0.46	0.03	0.92	0.06	0.17	0.01	1.03	0.03	8.64	0.06

(Contd.)

Sample Name	Color	TiO_2		MnO		FeO		CoO		CuO		As_2O_5		SnO_2		Sb_2O_5		PbO		Total
		mean	SD	mean	SD	mean	SD	mean	SD	mean	SD	mean	SD	mean	SD	mean	SD	mean	SD	mean
GLASSY FAIENCE LMHK																				
AM-BV-29	light blue	0.05	0.02	0.02	0.02	0.46	0.17	0.01	0.01	4.10	0.64	0.01	0.01	0.47	0.20	0.72	0.05	0.01	0.02	99.86
HMG																				
CR-GA-544	light blue	0.05	0.04	0.05	0.02	0.44	0.07	0.00	0.01	1.09	0.02	0.02	0.02	0.01	0.01	0.26	0.04	0.03	0.02	99.30
CR-AV-25	green	0.04	0.02	0.04	0.01	0.33	0.03	0.01	0.01	1.12	0.05	0.01	0.02	0.01	0.01	0.15	0.02	0.00	0.00	99.69
CR-GV-85	green	0.03	0.02	0.06	0.03	0.30	0.03	0.02	0.01	0.05	0.06	0.01	0.02	0.02	0.02	0.20	0.02	0.02	0.02	100.64
CD-GV-02	blue	0.04	0.01	0.05	0.02	0.35	0.05	0.00	0.01	1.10	0.08	0.02	0.02	0.02	0.02	0.16	0.01	0.03	0.02	99.60
CD-GV-03	blue	0.06	0.01	0.06	0.01	0.45	0.07	0.02	0.02	1.18	0.04	0.01	0.01	0.00	0.00	0.20	0.01	0.04	0.08	100.65
CD-BoD-04V	green	0.03	0.01	0.07	0.03	0.46	0.05	0.01	0.01	0.09	0.05	0.02	0.02	0.01	0.01	0.17	0.04	0.01	0.01	99.63
CR-AAF-03M	transparent brown	0.04	0.02	0.04	0.02	0.28	0.03	0.01	0.02	0.02	0.02	0.00	0.00	0.00	0.00	0.18	0.01	0.00	0.01	100.55
LMG / NATRON BLACK																				
PN-GN-978	greenish black	0.25	0.06	0.01	0.01	10.72	0.21	0.02	0.02	0.01	0.03	0.01	0.01	0.00	0.00	0.02	0.03	0.00	0.00	102.05
PN-GV-87	greenish black	0.20	0.04	0.01	0.01	10.76	0.33	0.01	0.02	0.00	0.00	0.01	0.01	0.01	0.01	0.05	0.02	0.01	0.01	101.99
HMLK																				
PN-AN-80	greenish black	0.30	0.06	0.02	0.02	9.32	0.21	0.01	0.02	0.03	0.02	0.05	0.04	0.00	0.00	0.07	0.04	0.00	0.00	102.32
PN-OB-13	dark blue	0.15	0.03	0.27	0.02	0.68	0.02	0.05	0.06	0.01	0.02	0.01	0.02	0.00	0.00	0.02	0.02	0.01	0.02	101.16
PN-O878-B	dark blue	0.14	0.04	0.16	0.04	1.10	0.06	0.03	0.03	0.02	0.03	0.00	0.00	0.02	0.02	0.03	0.04	0.02	0.03	101.28
ALTERED SAMPLES																				
CR-GA-498-zonC	light blue	0.01	0.01	0.02	0.03	0.36	0.03	0.01	0.02	0.52	0.03	0.02	0.02	0.01	0.01	0.05	0.02	0.00	0.00	93.19
CR-GA-498-zonD	light blue	0.01	0.02	0.03	0.03	0.42	0.06	0.01	0.01	1.03	0.35	0.00	0.00	0.02	0.01	0.11	0.03	0.01	0.01	88.81
CR-BS-555	weathered/green	0.03	0.02	0.02	0.04	0.69	0.02	0.00	0.00	1.34	0.17	0.00	0.00	0.00	0.00	0.12	0.02	0.01	0.01	90.82
CR-GV-2255	green	0.02	0.01	0.04	0.01	0.43	0.07	0.00	0.01	0.90	0.05	0.01	0.02	0.03	0.04	0.04	0.02	0.02	0.02	81.94
OPAQUE WHITE																				
CR-AAF-03Bi	weathered/ unknown	0.07	0.02	0.04	0.02	0.56	0.05	0.00	0.00	0.03	0.03	0.01	0.01	0.00	0.01	0.17	0.03	0.02	0.02	95.30
CD-BoD-04Bi	opaque white	0.03	0.02	0.04	0.03	0.68	0.10	0.01	0.02	0.21	0.02	0.01	0.01	0.02	0.01	1.24	0.08	0.02	0.03	90.40
PN-O878-Bi	opaque white	0.05	0.05	0.01	0.01	0.19	0.03	0.01	0.01	0.06	0.04	0.05	0.04	0.03	0.03	5.11	0.88	0.01	0.01	94.25
STANDARDS																				
Corning Standard A	colorless	0.85	0.08	0.85	0.12	0.92	0.14	0.18	0.01	1.14	0.12	0.02	0.02	0.16	0.03	1.88	0.08	0.03	0.02	99.63
Corning Standard B	colorless	0.12	0.03	0.24	0.02	0.27	0.04	0.02	0.02	2.81	0.06	0.02	0.03	0.03	0.03	0.48	0.04	0.43	0.08	100.51

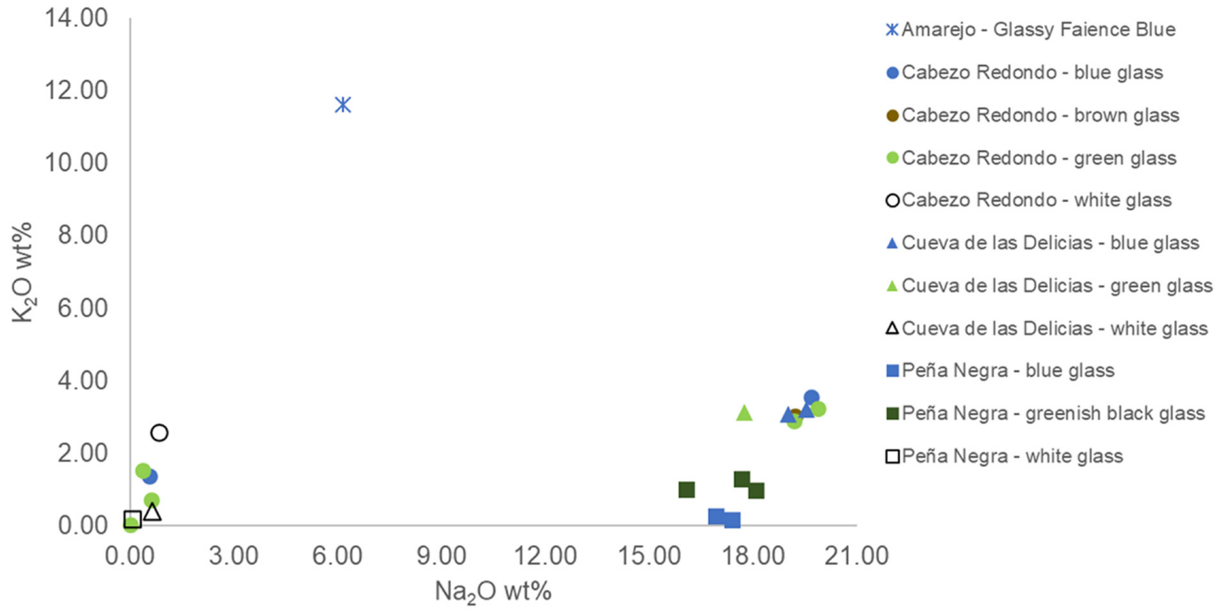


FIG 5. Na_2O vs. K_2O content in the glassy matrix of the analyzed samples, including weathered glasses. (Graph: C. Bettineschi)

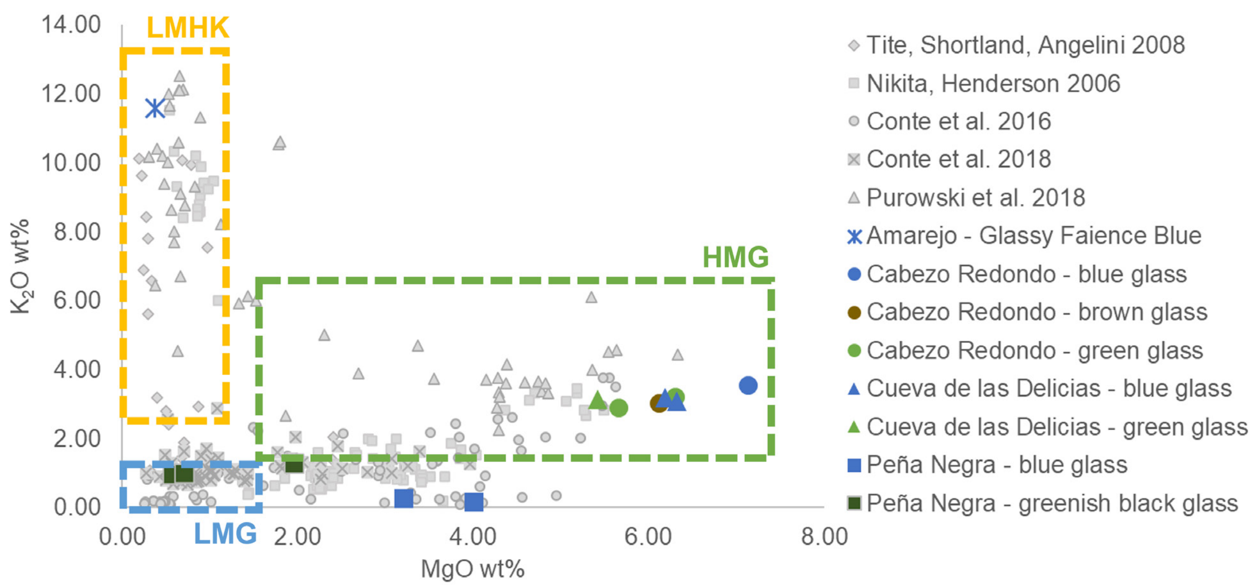


FIG 6. MgO vs. K_2O content in the glassy matrix of the analyzed samples and in a selection of samples from other sites. Data from Tite, Shortland, and Angelini 2008; Nikita and Henderson 2006; Conte and others 2016; Conte and others 2018; Purowski, Kępa, and Wagner 2018. Average boundaries for the compositional macro-groups are also reported. (Graph: C. Bettineschi)

flux coming from eastern Mediterranean regions.[35] The third cluster incorporates two very dark-green samples from Peña Negra (PN-GN-978, PN-GV-87), whose composition can be framed in the class of low-magnesium glasses (LMG), showing K_2O in the order of approximately 1%, low MgO (on average 0.6–0.7%) and CaO (always < 2%), but also very high levels of FeO (about 10.7%). LMG glasses, including the FeO-rich

35 Rehren 2008.

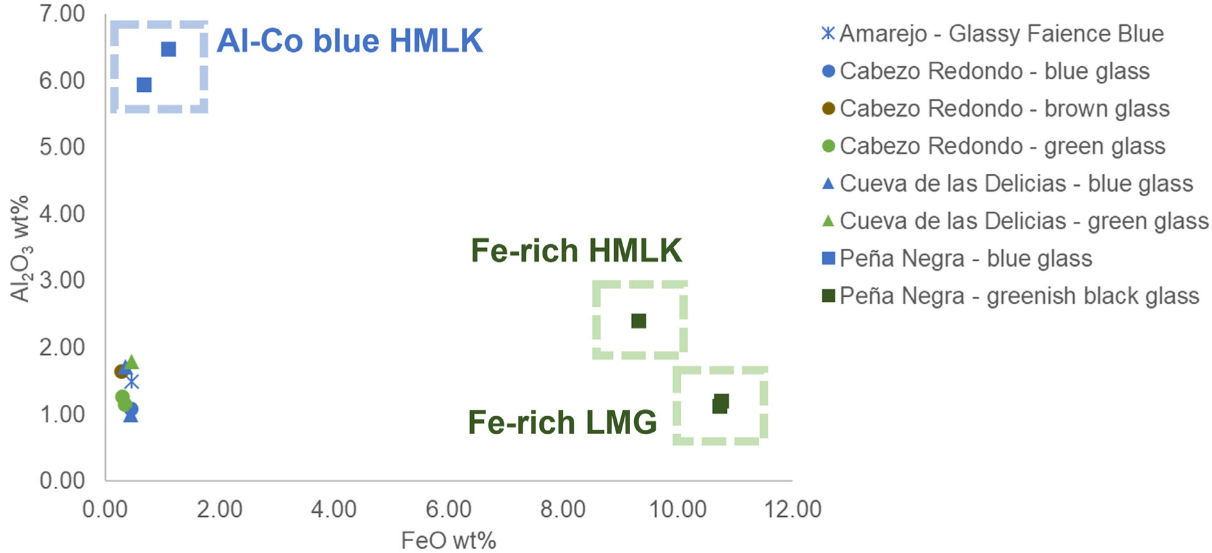


FIG 7. FeO vs. Al_2O_3 content in the glassy matrix of the analyzed samples, highlighting the three main compositional variants in the Peña Negra assemblage. (Graph: C. Bettineschi)

variant (**Figs. 6, 7**), were all produced using natron as fluxing agent.[36] This peculiar LMG subgroup was already recognized in several LBA–EIA sites in Italy, France, and Portugal and is often named "natron-black."[37]

Finally, three glasses have an intermediate HMLK composition with MgO from 2% to 4% and potash in the range of classic natron glasses ($K_2O < 1.5\%$). This class can be further subdivided in two distinct groups:

(1) Iron-rich HMLK glass (**Figs. 6, 7**), which is represented in this assemblage only by the very dark green bead PN-AN-80 with MgO 2%, K_2O 1.3%, CaO 1.8%, Al_2O_3 2.4%, and FeO 9.3%. The composition is, indeed, very similar in major, minor, and trace elements to the Fe-rich LMG glasses from the same site, with only a minor enriching in MgO and Al_2O_3. It seems, thus, very likely that this sample was also produced with natron and very impure sands rich in Mg- and Fe-bearing minerals (pyroxenes and amphiboles).
(2) Al-Co blue HMLK glass (**Figs. 6–8**), the recipe associated with the two dark-blue profiled eye beads from Peña Negra PN-O878-B and PN-OB-13, is characterized by very high amounts of alumina (here 6–6.5%) and by the use of cobalt as ionic chromophore (CoO 0.03% and 0.05%, respectively). In previous studies, this composition has been possibly recognized (with some minor residual doubts) as natron-based glass enriched in alumina, magnesia, and cobalt oxide during the coloring process by means of cobaltiferous alums from the Egyptian oases.[38]

Considering the moderate to advanced surface weathering of all the beads, the color of the glass was determined during sampling from the detached micro-chips (see

36 Jackson and others 2018; Degryse, Scott, and Brems 2014.
37 Conte and others 2016; Gratuze 2009; Gomes 2021.
38 Gratuze 2009; Conte and others 2016; Shortland, Tite, and Ewart 2006.

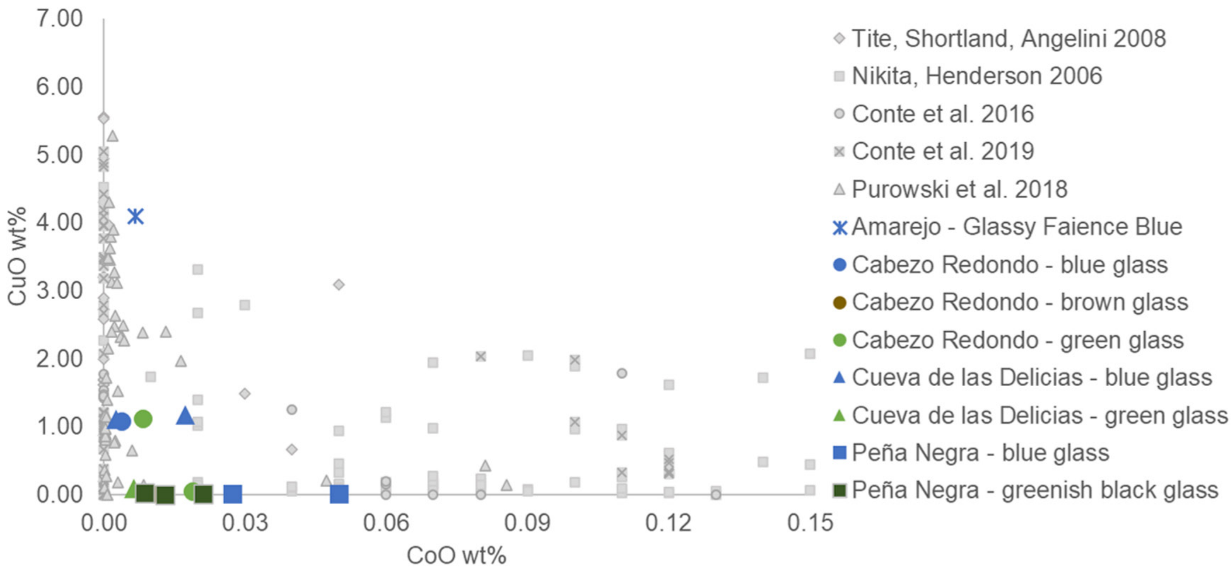


FIG 8. CoO vs. CuO content in the glassy matrix of the analyzed samples and in a selection of samples from other sites. Data from the sources in the caption to FIG. 6. Note that the EPMA detection limit for CoO is around 0.03%. (Graph: C. Bettineschi)

Figure 4). In general terms, the set comprises mostly transparent green and blue beads (with either a light or a very dark tone), one transparent brown/amber sample (CR-AAF-03M), and three opaque white glasses (CD-BoD-04Bi, PN-O878-Bi, and CR-AAF-03Bi, which was later recognized as a transparent glass of unknown color during SEM investigations, as noted above).

The ratio CoO/CuO (Fig. 8) shows that all seven transparent blue and green glasses from Cabezo Redondo and Cueva de las Delicias, except CR-GV-85 and CD-BoD-04V, are colored with copper (CuO in the range from 1% to 1.2%). SnO_2 is aways under detection limit in all the glasses (< 0.03 wt %); however, it reaches 0.47 wt % in the glassy faience from El Amarejo, with a correspondingly higher CuO (4.1%). The Cu/Sn ratio (8.9) agrees with the use of a Cu alloy with 10% of tin, which is perfectly compatible with the composition of the ancient bronze objects produced in the timespan considered. On the contrary, cobalt exceeds the detection limits only in the two dark-blue samples from Peña Negra (PN-O878-B, PN-OB-13).

The amber bead CR-AAF-03M (see Figure 4b) as well as the green samples CR-GV-85 and CD-BoD-04V do not contain any ionic chromophore in significant concentration (see Figures 7, 8). This is a typical feature of transparent brown glasses, whose color has been widely attributed to the Fe-S complex (FeO 0.28% and SO_3 0.32%).[39] Likewise, the green sample CR-GV-85 probably derives its color from FeO (0.3%), but under different redox conditions. Finally, iron is surely the chromophore responsible for the very dark greenish-black color of three samples from Peña Negra (PN-AN-80, PN-GN-978, PN-GV-87), which contain FeO in the range 9.3–10.8% (see Figure 7).

Texture and mineral inclusions

In addition to revealing the different patterns of weathering (which will not be discussed here for the sake of brevity), BSE images showed the presence of various

39 Paynter and Jackson 2018; Schreurs and Brill 1984; Angelini, Nicola, and Artioli 2012.

mineral inclusions, comprising coloring and opacifying agents, but also relics from the batch and newly formed phases. The (limited) remaining portions of non-altered glass in the beads from Cabezo Redondo and Cueva de las Delicias are always homogeneous. The only exception is CD-BoD-04, whose dark-green glass shows an even dispersion of colloidal Cu sulfide particles. Due to the sub-micrometric dimension of the inclusions (**Fig. 9a**), it was not possible to precisely determine their shape or their stoichiometry, which would allow us to identify their exact mineral nature. However, the Cu/S wt % ratio derived from EDS data (3.75) suggests them to be most probably chalcocite (Cu_2S). The white glass from the same bead (CD-BoD-04Bi) is equally interesting: as expected, it contains small and finely dispersed aggregates of Ca antimonates (EDS data), which are mostly anhedral and partly dissolved into the (severely weathered) glassy matrix. However, a big lump of euhedral crystallites is also present (**Fig. 9b**) and EPMA determined them to be Na antimonates (mineralogically, brizziite). We interpret this as an evidence of combined *ex situ* addition (Ca antimonates) and *in situ* precipitation (brizziite), where this last occurrence can most probably be associated with the very low CaO (only 0.8%) in the vitreous phase.

The glasses from Peña Negra show only minor signs of weathering on the external surface. Air bubbles are always present. In terms of inclusions, PN-GN-978 contains one residual silica grain (**Figs. 4a, 9c**); and PN-O878-B a few newly formed wollastonite ($CaSiO_3$) crystals close to the outer surface (**Fig. 10a**), which are considered indicators of high temperatures or found to be the result of weathering.[40] All other transparent glasses are very uniform. The white decoration in PN-O878-Bi is opacified by means of euhedral Ca antimonates with rectangular morphology and limited substitutions of PbO (up to approx. 2%) (**Fig. 10b**). BSE imaging suggests that they belong to the orthorhombic variant with formula $Ca_2Sb_2O_7$.[41]

The biconical bead from El Amarejo AM-BV-29 is composed of a comparable quantity of mineral inclusions and an amorphous phase, confirming its preliminary interpretation as glassy faience (**Figs. 4c, 10c**). There is no preserved glaze, and the body of the bead is very uniform, with the interaction layer (IL) not actually distinguishable from the core. The porosity is rather scarce, but several bubbles have significant dimension, up to approximately 60–70 μm. Silica grains constitute the majority of the mineral inclusions, with a dimension from a few microns up to 100–120 μm. Chemical mapping (**Fig. 11**) and EDS and EPMA data also highlight the presence of numerous alkali feldspars, a few augites, and frequent cassiterite (SnO_2) inclusions.

BSE observations of the Egyptian blue bead from Peña Negra PN-DEB-15 demonstrate the total lack of any residual vitreous component. This is most probably a result of weathering,[42] but it could also be—less probably—derived from a complete synthesis of the raw materials into cuprorivaite crystals, the calcium-copper tetrasilicate with formula $CaCuSi_4O_{10}$ which confers the typical blue color to the Egyptian blue frit. From a microstructural perspective, only big, angular silica grains (darker gray in **Fig. 12**) and proportionally fewer cuprorivaite crystals (lighter gray in **Fig. 12**) survive in the sample, within a diffuse porosity (black in **Fig. 12**).

40 Pusch and Rehren 2007.

41 Lahlil and others 2010.

42 Hatton, Shortland, and Tite 2008.

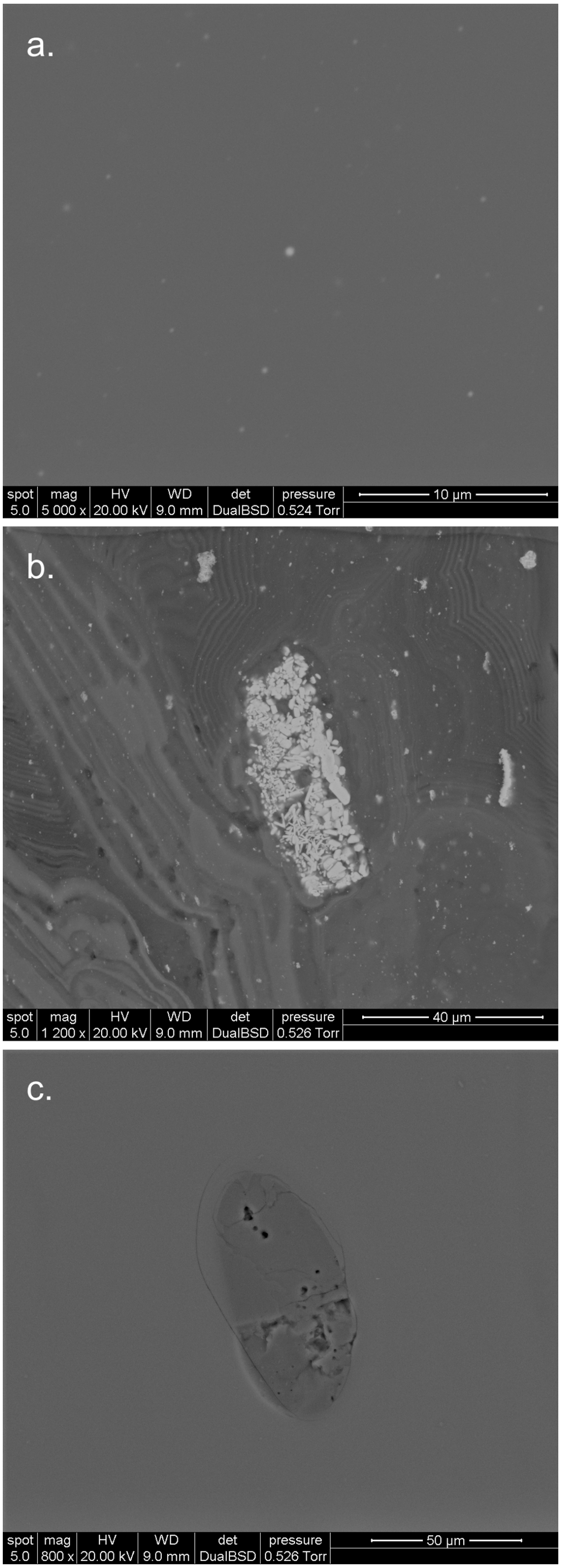


FIG 9. BSE images: (a) CD-BoD-04V, colloidal dispersion of chalcocite particles; (b) CD-BoD-04Bi, opacification via anhedral Ca antimonates and lump of euhedral brizziite crystals; (c) PN-GN-978, with a residual silica grain. (Images: C. Bettineschi)

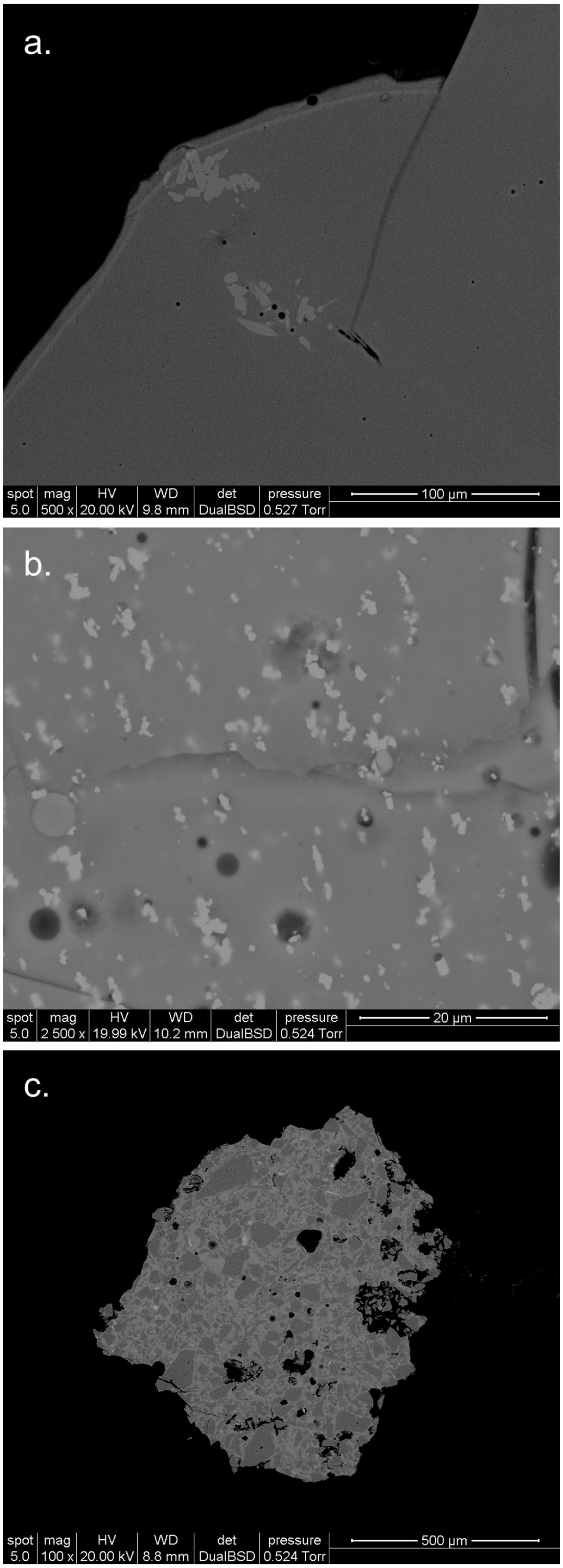


FIG 10. BSE images: (a) PN-O878-B, newly formed wollastonite crystal; (b) PN-O878-Bi, euhedral Ca antimonates opacifiers; (c) Glassy faience sample AM-BV-29, cross-section. (Images: C. Bettineschi)

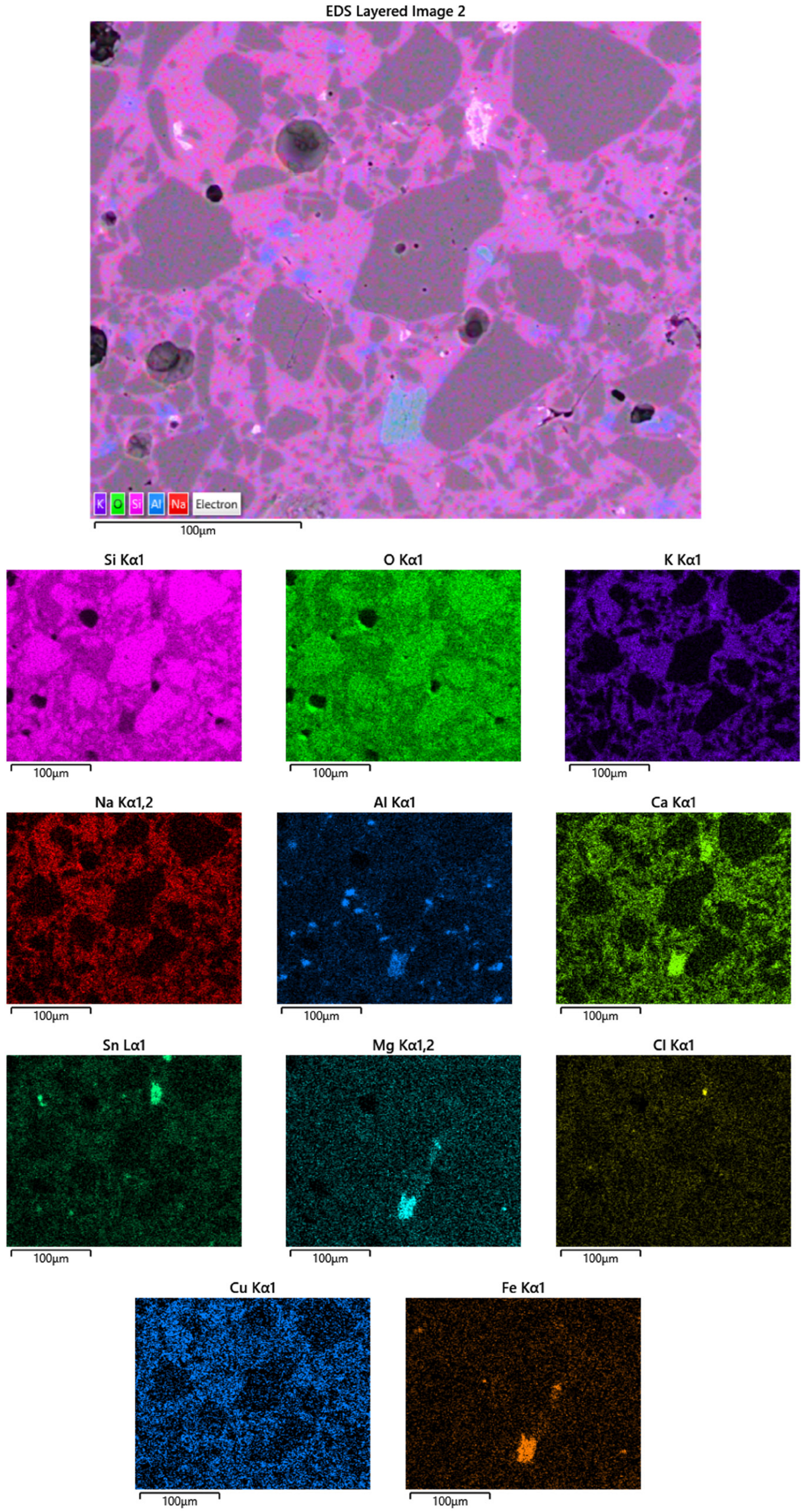


FIG 11. Chemical mapping of the glassy faience AM-BV-29. (Images: C. Bettineschi)

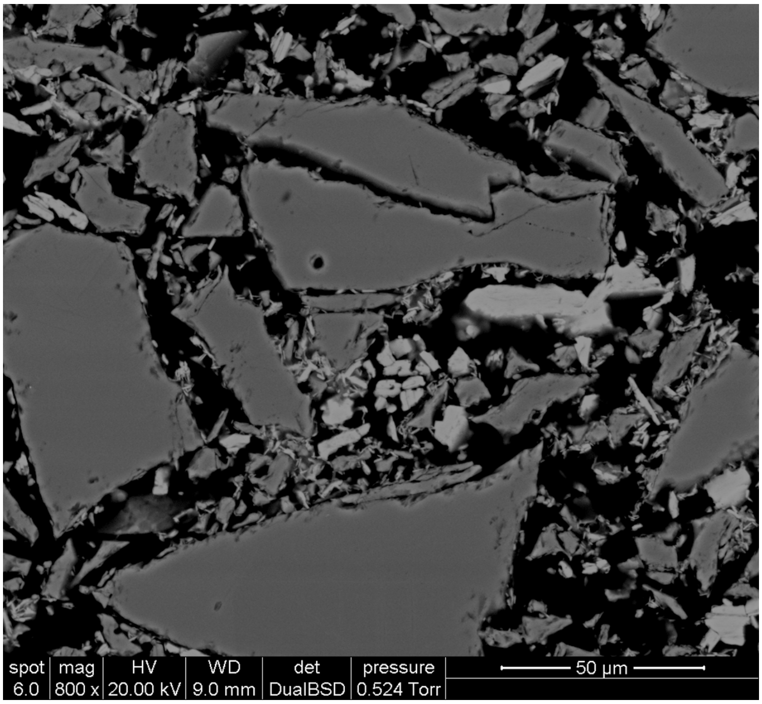


FIG 12. BSE image of the texture of the Egyptian blue bead PN-DEB-15. (Image: C. Bettineschi)

DISCUSSION

True glasses

All seven non-weathered samples from Cabezo Redondo and Cueva de las Delicias were classified as plant-ash glasses based on the soda, magnesia, potash, and lime content. The HMG recipe is typical of the glasses produced using halophytic plant ashes in the Near East and Egypt starting from the second millennium BCE.[43] This composition is extremely widespread in central Europe and in the whole Mediterranean region during the LBA, with occurrences unearthed even in the far north of Europe and throughout Romania, Germany, and Scandinavia.[44]

The samples from Cabezo Redondo and Cueva de las Delicias cover a wide timespan from the end of the seventeenth to approximately the tenth century BCE. In general, they show very low impurity content, suggesting that they are freshly batched glasses or, alternatively, that in recycling, the glassmakers took care to remelt only similar colors together. The alumina content, associated with the very low amounts of FeO and TiO_2, is compatible with very pure or purified sands, but it could also, less probably, be due to a contamination from the quartz-grinding process and from the ashes, or even to an enrichment from the crucible. Most of the beads are colored with copper (with cobalt under detection limits), which is the preferred solution for the majority of the light-blue and green beads from this period. However, one sample is made of HMG amber glass. This color is rather unusual in LMG glasses before the Hellenistic period,[45] but it is especially rare in Europe during the LBA. There is, indeed, a subgroup of plant-ash glasses known as high magnesium brown glasses

43 Turner 1956; Sayre and Smith 1961.
44 Varberg and others 2016; Varberg, Gratuze, and Kaul 2015.
45 Paynter and Jackson 2018.

(HMBG) documented in central and northern Italy during the local RBA and FBA.[46] However, the texture of HMBG glasses is different from that of CR-AAF-03M: while the Spanish sample is perfectly transparent and very homogeneous, HMBG glasses show abundant euhedral crystals of diopside and/or augite and droplets of copper sulfides; the chemistry is also not compatible, due to the higher content of CaO (6–7%) and especially Al_2O_3 (2–3.5%) and FeO (4–5.5%) in the HMBG class. Transparent HMG brown glasses are, however, attested in the well-known glassmaking centers of Amarna and Malkata (Egypt) during the fourteenth century BCE. In the western Mediterranean, the only strict compositional comparison for CR-AAF-03M is an annular bead from the Nuragic Giants' Tomb of Gonnosfanadiga (Cagliari, Sardinia) dated to about 1450–1250 BCE.[47] The chronology is in perfect accordance with the one proposed for the sample from Cabezo Redondo (Beta-332581, 3110 ± 30 BP, 1442–1286 cal BCE, 95.4% 2σ), confirming that Sardinia and southeastern Spain were served by similar maritime circuits probably departing from the far east shores of the Mediterranean during the early second half of the second millennium BCE.

The barrel bead with helicoidal decoration CD-BoD-04 has an HMG composition. This is somewhat unexpected considering that many FBA beads with the same typology recovered from across Europe are mixed-alkali glasses. There are, however, two known exceptions from France, one found on the site of the Kermorvan peninsula (Le Conquet, Brittany) and the other in Eckwersheim (Bas-Rhin), both dated to the eleventh to tenth century BCE.[48] Trace element analyses on those two beads suggest a Near Eastern provenance, which is most probably the case also for our sample based on the similarity of the major element composition. The opaque white decoration is predominantly colored by *ex situ* addition of Ca antimonates, but *in situ* precipitated brizziite was also identified. Brizzite appears sporadically in ancient glasses—most of the time unintentionally, being a consequence of insufficient Ca amounts in the glassy matrix. It was, however, recognized in other white decorations of the FBA and EIA which frequently have low CaO levels, and is also sporadically present both in the Near East and Europe up to late antiquity.[49]

Despite the wide chemical variations, the five beads from Peña Negra dated to the tenth to ninth century BCE are all produced using mineral natron as flux. In Egypt and the Near East, this composition systematically substitutes for HMG glasses starting from the tenth to ninth century BCE and remains the most common recipe used throughout the IA and beyond. The discovery of LMG samples in central and western Europe as early as the tenth to ninth century BCE is uncommon but confirmed by several specimens. Among these early natron glasses, the samples from Peña Negra belong to three different variants—(1) Fe-rich LMG; (2) Fe-rich HMLK; (3) Al-Co blue HMLK—which have already been recognized in Italy as well as Portugal, France, Greece, Hungary, former Yugoslavia, and Czechia.[50] The very dark appearance of Fe-rich LMG and HMLK beads is due to the voluntary addition of iron as a raw material, probably in the form of highly impure sands, rich in heavy minerals. However, the discovery of all the beads from Peña Negra belonging to the Fe-rich compositional variants in the dump of a metallurgical workshop might tentatively suggest

46 Artioli, Angelini, and Polla 2008.
47 Angelini, Nicola, and Artioli 2012.
48 Cherel, Gratuze, and Simon 2018.
49 Muros and Zacharias 2019; Bettineschi and Angelini 2022.
50 Conte and others 2016; Conte and others 2018; Lončarić and Costa 2023.

a connection with metal production, for example, a coloring process triggered by adding a certain amount of slag chunks or powder to the molten batch, as already suggested for HMBG glasses.[51] The bead from Gatas, which was already considered anomalous when first analyzed by Julian Henderson in 1999, remains an open problem.[52] While it can be framed generally in the heterogeneous class of Fe-rich greenish-black glasses, its composition with high K_2O and MgO (both > 2%) and its very early chronology (1700–1500 cal BCE) might tentatively identify it as an ancestor of the later Fe-rich LMG productions, sharing a similar coloring technology.

Al-Co blue HMLK glasses derive their peculiar chemical composition from the use of Egyptian colbaltiferous alums as coloring agent. Beads with a similar composition from the ninth and eighth centuries BCE were discovered in France and Italy and constitute some of the earliest natron glasses ever recorded in western Europe.[53] Their presence in Spain as early as in the tenth to ninth century BCE shows once again how the Iberian Peninsula was directly and precociously integrated in the maritime traffic which reached the western side of the Mediterranean Sea, paving the way to the Phoenician-Punic colonization of southern Spain.

Glassy faience

The bead from El Amarejo was classified as a glassy faience LMHK based on a combination of textural and chemical features. Mixed-alkali vitreous materials are attested in central Europe, Italy, Britain, Ireland, and France starting from the EBA (about 2200–1900 BCE). At the beginning, this composition was reserved for faience beads, but later, as of about 1700/1650 BCE, production of glassy faience ornaments began. While the peak number of occurrences of glassy faience LMHK dates to the period 1700/1650–1450 BCE, they continue to be sporadically found up to the FBA north of the Alps. LMHK faience, glassy faience, and glass objects are considered a local European production, in contrast with the coeval plant-ash vitreous materials which were imported as semi-finished or finished products from Egypt and the Near East.[54]

Judging by the quantity of alumina (1.5%), and especially by the very abundant presence of alkali feldspars and other relics from the batch, sands were certainly used as silica source. The color is due to copper, likely introduced in the form of bronze. The composition is generally consistent with the conical buttons and beads from northern and central Italy which are common during the local MBA1–2,[55] but also with the later Swiss occurrences from the FBA.[56] Unfortunately, the composition of LMHK glassy faience objects is rather variable even in pieces from the same assemblage, possibly due to the production technology, or to the existence of multiple workshops (not yet identified), or even the use of non-standardized processes and raw materials, depending on availability. Even more probable is that we are confronted with a combination of the three. This complicates the identification of chemical markers with chronological significance in these materials and, ultimately, prevents us from assigning with certainty the El Amarejo bead to one or the other phase. However,

51 Artioli, Angelini, and Polla 2008.
52 Henderson 1999.
53 Conte and others 2016; Gratuze 2009.
54 Henderson 1988; Angelini, Gratuze, and Artioli 2019.
55 Tite, Shortland, and Angelini 2008.
56 Henderson 1993; Angelini and Olmeda 2018.

its presence testifies to relevant contacts with Italy and central Europe, marking an important additional, previously unknown locale in the distribution of mixed-alkali vitreous materials.

Egyptian blue

At present, sample PN-DEB-15 is the only published occurrence of an Egyptian blue bead recovered in the western Mediterranean / western Europe with a BA dating. Egyptian blue has a long history as a pigment, but in ancient times it was also employed to produce small ornamental objects. Although this technology was introduced in Egypt and the Near East around the early third millennium BCE, it appears around the Mediterranean only about a thousand years later, when it was essentially confined to Minoan and Mycenean sites. Its discovery in Spain at the turn of the second to the first millennium BCE is a clear indicator of long-distance trade networks.

Due to severe weathering, the microstructural examination evidenced only the presence of abundant silica grains and of small cuprorivaite crystals. The lack of interparticle glass is unfortunately a well-known issue that affects the vast majority of the BA Egyptian blue samples recovered in Greece and the Aegean that have been analyzed to date. This being the case, it is not possible to gain information regarding the raw materials or to suggest a tentative provenance for the bead on a compositional basis. The smaller quantity and dimensions of the cuprorivaite inclusions (max. 25–30 μm) with respect to those of silica account for the very light blue color of the bead. In the literature this has been considered as an indicator that the beads in Egyptian blue are secondary products derived from grinding, shaping, and refiring primary lumps.[57]

CONCLUSION

The results of this research clearly evidence the existence of well-defined chronological trends in the compositional variability of the vitreous materials unearthed in the investigated sites. During the LBA and up until the early FBA, plant-ash glasses not only prevail, but are the only recipe attested in the settlement of Cabezo Redondo and in the funerary cave of Cueva de las Delicias. The only exception is the glassy faience LMHK discovered in the megalithic burial of El Amarejo, whose chronology still needs to be ascertained in further detail (radiocarbon dating on the human remains is in progress). While we still lack provenance analysis—Laser Ablation Inductively Coupled Plasma Mass Spectrometry is expected as the next step of the project—we could ascertain a certain continuity in the glass supply between the post-Agar and the very beginning of the FBA. However, during the tenth to ninth century BCE, natron glasses substitute for the HMG productions, at least from what we can infer judging from the samples of Peña Negra. The site has also yielded an Egyptian blue bead, which is the first ever recorded in western Europe.

Considering this framework, the passage from HMG to LMG glasses appears less gradual in Spain with respect to the situation as it emerged in Italy or France, where, however, the number of the analyzed samples is considerably higher and the geographic distribution of the sites is significantly more pervasive. More data, thus, are strongly required to understand if the diachronic variations evidenced during this work are effectively representative of the whole Iberian Levant or if they are only a reflection of the sampling biases in this work. It is equally crucial to analyze a statistically significant number of beads discovered in Argaric contexts. This will help

57 Tite, Bimson, and Cowell 1984.

elucidate the developmental trajectory of the vitreous material industry in Iberia from the early second millennium BCE.

Despite the aforementioned limitations, this research has yielded valuable insights into the circulation of ancient vitreous materials, contributing to shedding light on a poorly documented region in terms of protohistoric glass studies. Our findings demonstrate a prompt reception by the Iberian Peninsula of the technological innovations introduced in the eastern Mediterranean, evidencing its direct and pivotal role as the end member of the maritime trades connecting the lands and seas of Europe, North Africa, and the Levantine coast throughout the Late and Final Bronze Age.

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