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EDITED BY
Ofir Katz,
Dead Sea and Arava Science Center, Israel

REVIEWED BY
Robert C. Power,
University College Dublin, Ireland
Karina Chueng,
Rio de Janeiro State University, Brazil

*CORRESPONDENCE
Vincenza Ferrara
✉ vincenza.ferrara@arkeologi.uu.se

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Lab waste as hidden treasure. Early results of phytolith analysis from Iberian prehistoric post-ORA pottery powder

Vincenza Ferrara[1,2]*, Robin von Allmen[1],
Maria Fernanda Salame González[1] and Elena Revert Francés[3]

[1]Uppsala Archeobotanical Group (UAG), Department of Archaeology, Ancient History and Conservation, Uppsala University, Uppsala, Sweden, [2]Department of Human Geography, Stockholm University, Stockholm, Sweden, [3]CRC 1070 ResourceCultures, Institute of Pre- and Early History and Archaeology of the Middle Ages and Modern Times, University of Tübingen, Tübingen, Germany

Initially designed to explore cultural interactions between Phoenicians and local communities in the Iberian Peninsula during the 1st millennium BCE, the work presented in our paper expanded beyond traditional Organic Residue Analysis (ORA), by attempting phytoliths extraction from post-ORA pottery powder. The aim was to assess whether such a methodological integration may enhance the understanding of past correlations between environments and material cultures in terms of pottery making and function, use of plant resources, food preparation, cultural practices, intercultural exchanges and networks. Despite working with limited sample sizes (∼1 g per sample), phytolith analysis successfully revealed distinct environmental signatures across different archeological contexts, illustrating the valuable contribution of plant biogenic silica studies within and beyond archeological research. Furthermore, an unexpected positive result in terms of hue detection during the laboratory procedure underscores the research practice as scientific discovery process. By combining archeological, botanical, and chemical perspectives, our study showcases how phytoliths research can extend beyond traditional boundaries and complement established methodologies, reinforcing the necessity of interdisciplinary dialogue and multi-disciplinary approaches to archeology.

KEYWORDS

biogenic silica, blue, dicot phytoliths, Early Iron Age, Iberian Peninsula, organic residue analysis, Phoenicians, wood ash

1 Introduction

During the 1st millennium BCE, the Mediterranean emerged as a highly intertwined region, characterized by extensive connections, movements, exchanges of goods, plants, animals, people, and ideas. Such a dynamic interplay resulted in a flourishing and significant historical period. Human mobility, along with the cultural heritage it carried, offers an interesting perspective for studying the evolution and dynamics of past societies.

The people archaeologically referred to as Phoenicians originated in Canaan, a region in the eastern Mediterranean that encompasses present-day Lebanon as well as the coastal areas of Syria and Israel. They navigated across the sea to establish long-lasting settlements, where they blended with the local populations, in Sicily, Tunisia, Sardinia, Ibiza, Spain, to name a few examples (Aubet, 1994; Delgado and Ferrer, 2007; López-Ruiz, 2021). The Iberian Peninsula, particularly Spain, served as a melting pot of diverse cultures and traditions, bearing significant influence from the Phoenicians, especially in the early 1st millennium BCE.

To explore the cultural entanglements between Phoenicians and local communities in the Iberian Peninsula during the 1st millennium BCE, within the framework of the project "*Cultural entanglements in the Lower Guadalquivir—Interacting ResourceCultures and socio-cultural change in the South of the Iberian Peninsula*" (University of Tübingen), pottery samples were collected in 2023 for Organic Residue Analysis (ORA) from three archaeological sites in Andalusia and Extremadura (southern Iberian Peninsula) by one of the authors of this paper. The primary objective of the ORA was to identify some not-native compounds potentially linked to the Phoenicians or Mediterranean trade more in general, as well as to observe changes before and after the arrival of these settlers in the ninth century BCE.

Each sample weighed between 1 and 2 g and was collected from the interior surface of the pottery fragments and vessels. The analyses were conducted in the ORA laboratories at the University of Tübingen, and the findings are published elsewhere (Revert Francés, 2024; Revert Francés and Toscano, in prep[1]). However, ORA can encounter challenges in detecting plant oils, depending on the methods used (Drieu et al., 2025). Consequently, knowledge on plant remains from pottery residues could be limited if relying only on this method.

Recognizing the importance of interdisciplinarity and the value of approaching the same research question from multiple, different but integrated methodological approaches, we decided to conduct phytolith analysis on the pottery powder left from the previously done ORA process. Several authors have already shown the feasibility of phytolith analysis on sediments and/or residues from vessels and pottery, in order to obtain information on the use and cultural practices connected to those artifacts (e.g., food preparation and/or storage, Hart, 2011; Saul et al., 2013; Wang et al., 2022; Debels et al., 2024; Santiago-Marrero et al., 2024), and combined residue and phytolith analysis (Gong et al., 2025). The post-ORA samples available in the case presented here weighed only approx. 1 g, which could have represented a significant limitation for the phytolith extraction process, as it usually may require a higher amount of material depending on the context (e.g., 3.5 g, cf. Mazuy et al., 2024). Despite such limitations, the analysis proceeded, paving the way for novel work in the field of phytolith extraction from archaeological pottery powder after Organic Residue Analysis. Furthermore, four of the post-ORA samples were selected for pollen analysis.

Our paper is structured as follows. The section Data and Methods presents the archaeological context and key characteristics of the three post-ORA samples used for phytolith analysis, as well as the initial four post-ORA samples chosen for eventual integrated pollen analysis. The results obtained from our laboratory and microscopic analysis are described in the Results section, alongside the report of an unintended discovery of blue pigment in one of the samples, which underwent the first step of the pollen extraction protocol (i.e., acetolysis, cf. Erdtman, 1960). Our results are further discussed in the framework of the evidence gathered from the ORA analysis performed before on the same samples, and within broader considerations about how phytolith analysis, when integrated in a multi- and interdisciplinary research practice, can provide relevant insights to better understand local cultural practices.

2 Data and methods

Phytolith analysis was conducted on three samples made of post-ORA pottery powder (Table 1) from the archaeological sites of Peñalosa, Tejada la Vieja, and Hacienda, located in the modern-day province of Huelva, in southwestern Spain (Figure 1).

Peñalosa (excavated in 1990) is the oldest of these sites, primarily dating to the Late Bronze Age, with an earlier, less-documented occupation during the Chalcolithic period. The excavated area of this settlement, featuring six pit houses, contained typical Late Bronze Age pottery, with the exception of a small fragment of wheel-made Phoenician red-slipped and burnished ware. Excluding this fragment, the assemblage could confidently be dated to the last centuries of the 2nd millennium BCE. However, the presence of this fragment, along with silver slag, prompted excavators to assign a date to the settlement in the ninth to eighth centuries BCE (García Sanz and Fernández Jurado, 2000). The possibility of the Phoenician fragment being an intrusive artifact cannot be entirely ruled out. Considering this and the otherwise uniform characteristics of Peñalosa, a dating range between the twelfth to eighth centuries is proposed here. From this site, phytolith analysis was performed on 1.003 g of post-ORA pottery powder recovered from the inner wall of a body sherd of a small burnished patera (i.e., bowl; **sample PE008**).

Tejada la Vieja is a fortified settlement, with its defensive walls dating to the late ninth century BCE. Covering approximately 6.4 ha, almost the entire area appears to have been densely constructed. The settlement seems to have been abandoned in the fourth century BCE. It is possible that the establishment of Tejada la Vieja is linked to the abandonment of Peñalosa, situated merely 3 km away (Fernández Jurado, 1987). Phytolith analysis was done on 1.048 g of post-ORA pottery powder from the inner wall of a rim sherd of a coarse storage jar, found in an archeological excavation in 1981 (**sample TV003**).

The Hacienda site (excavated in 2023) is located beneath the old Treasury Building in the city of Huelva. Recent renovation works on the building facilitated archaeological excavations, uncovering remains of the city's harbor dating back to the Early Iron Age. The excavation and analysis of the recovered materials are still in progress. 1.076 g of post-ORA pottery powder from the inner wall of the upper part of a coarse storage jar with one handle was analyzed for phytolith content (**sample HA002**).

Samples for ORA analysis, conducted in the ORA laboratories at the University of Tübingen, were extracted by one of the authors of this paper (Revert Francés, 2024; Revert Francés and Toscano, in prep (see text footnote 1)) in 2023 from the inner surface of the sherds using a drill, according to a standard method (Heron et al., 1991; Charters et al., 1993; Regert, 2011). ORA sampling method

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[1] Revert Francés, E., and Toscano, C. (in prep.). Organic Residue Analysis in Iron Age Settlements of Huelva: Insights from Peñalosa, Hacienda, and Tejada la Vieja.

TABLE 1 Samples for phytolith analysis.

Name	Weight of initial powder (g)	Sampled part of the vessel	Site	Chronology	Weight of final sediment after phytolith extraction (g)
PE08	1.003	Body sherd of a small burnished patera	Peñalosa	ca. 12th–8th cent. BCE	0.015
TV003	1.048	Rim sherd of a coarse storage jar	Tejada la Vieja	9th–4th cent. BCE	0.010
HA002	1.076	Upper part of a coarse storage jar with one handle	Hacienda	8th–6th cent. BCE	0.053

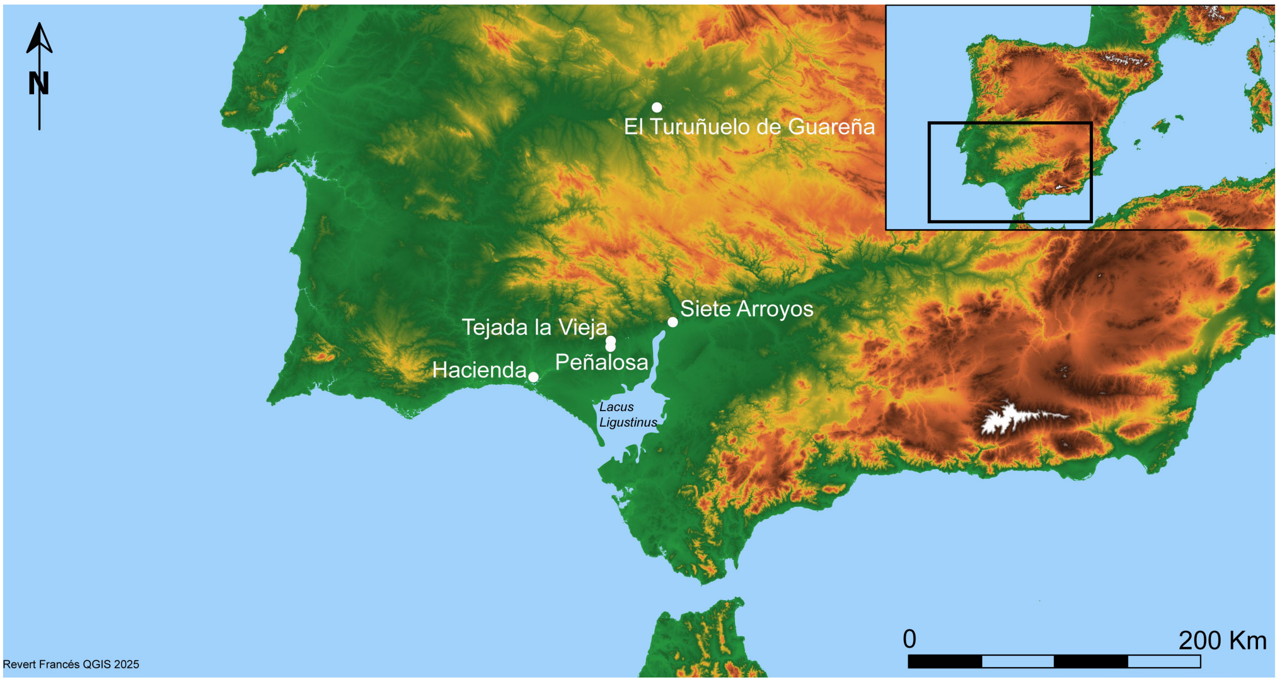

FIGURE 1
Map of the southwest of the Iberian Peninsula with the archeological sites named in the map. The drawing of the Lacus Ligustinus is based on the research of Oswaldo Arteaga (Arteaga and Roos, 1992; Arteaga et al., 2016).

foresees the removal of a first layer of material from the artifact, due to potential contamination from soil and/or the handling process after excavation, and the use for Organic Residue Analysis of only the powder obtained from drilling of the subsequent layer, located just below the first (e.g., Rageot et al., 2019; Spiteri et al., 2025).

After ORA analysis, the same samples, with identical weight, were brought in 2024 at the Paleobiology Laboratory (Department of Earth Sciences, Uppsala University) to attempt phytolith analysis by the Uppsala Archeobotanical Group on these post-ORA pottery samples.

Typically, ORA samples weigh between 1 and 2 g. When sampling for ORA, to prevent further damage to the ceramics, no surplus material was collected for potential integrative analyses. Consequently, in the case presented in this paper, we worked with particularly small samples suitable for phytolith analysis. Moreover, such a low amount of post-ORA pottery powder available per sample (approx. 1 g) could not allow to conduct both phytolith and pollen analysis on the same material. For such a reason, we selected four new post-ORA pottery residue samples for an initially planned pollen analysis (Table 2). These samples come from the archeological sites of Siete Arroyos (Seville), Hacienda (Huelva), and Casas del Turuñuelo de Guareña (Badajoz), in southwestern Spain.

Siete Arroyos is a burial site dating to the Early Bronze Age. The excavation and analysis of the archeological materials are ongoing (Bartelheim et al., 2025). From this site, pottery powder from a vessel and soil from its interior were sampled.

The excavation of the archeological site of Casas del Turuñuelo de Guareña (hereafter referred to as El Turuñuelo) is currently underway and is expected to continue in the coming years. The site presents a complex archeological narrative: evidence has been found of a banquet, the sacrificial slaughter of 41 equids alongside other animals such as cattle and pigs, and a deliberate conflagration that abruptly ended its use (Rodríguez González and Celestino, 2017, 2019; Ibora Eres et al., 2023).

TABLE 2 Samples for intended pollen analysis.

Name	Weight of initial powder (g)	Sampled part of the vessel	Site	Chronology
SA002_L2	1.000	Bottom of a coarse pottery bottle	Siete Arroyos	19th−17th cent. BCE
SA_Bod003	1.002	Soil sample from inside the vessel SA002_L2	Siete Arroyos	19th−17th cent. BCE
HA012	1.019	Bottom of a coarse storage jar	Hacienda	8th−6th cent. BCE
TR016	1.011	Body sherd of a coarse vessel	El Turuñuelo	5th cent. BCE

Phytolith extraction was conducted at the Paleobiology Laboratory (Department of Earth Sciences, Uppsala University), according to Mazuy et al. (2024) protocol, which allows the extraction of phytoliths from samples characterized by low biogenic silica content. This protocol foresees the deflocculation of the samples with magnetic stirring and the separation of its fine fraction via sieving (200 μm). Carbonates are then removed by hydrochloric acid treatment and organic matter with potassium hydroxide in a hot bath. In the resulting solution, phytoliths are then extracted by heavy liquid flotation and further cleaned from remaining organic matter through boiling in hydrogen peroxide.

The phytoliths resulting from the extraction were identified, counted and interpreted at the Department of Archaeology, Ancient History and Conservation (Uppsala University), using a light microscope at ×400 magnification. A minimum of 200 diagnostic morphotypes were identified and categorized into plant taxonomic groups according to the International Code for Phytolith Nomenclature (ICPN; Neumann et al., 2019) and the PhytCore online database (Albert et al., 2016). For each sample, we calculated each morphotype's relative abundance and analyzed their assemblages in terms of the ratio of inflorescence to culm-leaves morphotypes, long to short cell phytoliths, Dicotyledonous to Poaceae morphotypes (cf. Ferrara, 2024 for details about how to calculate these indices).

The ratio of inflorescence to culm-leaves phytoliths indicates the amount of ELONGATE DENDRITIC phytoliths (from inflorescence parts of grasses) to ELONGATE ENTIRE and ELONGATE SINUATE (from culms and leaves; Piperno, 1988; Tsartsidou et al., 2007; Delhon et al., 2020), and can be informative about the presence of spikelets and/or straws in an assemblage. ELONGATE phytoliths, produced by grasses from the Poaceae family (Twiss et al., 1969; Fredlund and Tieszen, 1994; Piperno, 1988; Ball et al., 2001; Neumann et al., 2019), can be distinguished among ELONGATE ENTIRE morphotype from the stems and ELONGATE SINUATE from the leaves, while ELONGATE DENDRITIC from the inflorescence of the plant (Rosen, 1992; Ball et al., 1999; Portillo et al., 2006; Albert et al., 2008).

The ratio of long cell to short cell phytoliths can provide information on the grass composition in terms of age and phenological phase, thus we can infer if local vegetation was formed by young or mature grasses (Delhon et al., 2024). Furthermore, when it comes to assemblage analysis of grass short cell phytoliths, it is possible to distinguish among the following three Poaceae subfamilies, represented by these specific phytolith morphotypes: RONDEL (Twiss et al., 1969; Fredlund and Tieszen, 1994; Piperno and Pearsall, 1998; Barboni and Bremond, 2009), TRAPEZOID (Barboni and Bremond, 2009) and CRENATE (Twiss et al., 1969; Fredlund and Tieszen, 1994; Barboni et al., 2007) as representative of Pooideae (C3 grasses growing in temperate climates); SADDLE (Piperno, 2006; Madella et al., 2016) and RONDEL in association with them (Bamford et al., 2006; Barboni and Bremond, 2009) as indicators of Chloridoideae (C4 grasses in dry and warm environments); BILOBATE (Twiss et al., 1969; Fredlund and Tieszen, 1994; Barboni and Bremond, 2009), POLYLOBATE (Twiss et al., 1969; Fredlund and Tieszen, 1994; Neumann et al., 2019) and CROSS indicating usually Panicoideae (C4 grasses in warm and wet contexts).

Comparing Dicots vs. Poaceae morphotypes can inform on the different origins of the phytoliths (trees or shrubs vs. grasses). The following morphotypes are attributable to dicotyledonous: SPHEROID (Bozarth, 1992; Alexandre et al., 1997; Albert et al., 1999; Runge, 1999; Delhon et al., 2003) and BLOCKY (Tsartsidou et al., 2015; Boixadera et al., 2016; Ntinou and Tsartsidou, 2017; Burguet-Coca et al., 2020; Kraushaar et al., 2021; Tencariu et al., 2022).

The chemical preparation of samples to extract pollen was performed according to the acetolysis protocol (Erdtman, 1960), which is the standard chemical treatment to enhance pollen morphological features and remove non-pollen organic matter. Acetolyzed samples were analyzed under a bright-field microscope, Nikon Eclipse Ni, with a 40× objective.

3 Results

Phytoliths were present in all the three samples and in good preservation state, despite the initial low amount of pottery powder processed (Figure 2). In all the three samples, there is a predominance of short cell vs. long cell phytoliths (PE08 53.5% vs. 21%; TV003 45% vs. 36%; HA002 64.5% vs. 18%). All the three samples show, however, overall high percentages of dicot phytoliths as well: PE08 14.5%, TV003 10%, HA002 10.5% (Table 3), reflected by the Dicotyledonous/Poaceae index values (Table 4), thus attesting the presence of residues from dicot plants.

If the three samples show similarities in such respect, there are important differences among them.

HA002 is the sample with the highest number of short cells (64.5%) when compared to the others, and these include also phytolith morphotypes attributable to C4 Panicoid grasses (BILOBATE 1.5% and POLYLOBATE 0.5%; cf. Out and Madella, 2016). HA002 also has a slight presence of SPHEROID ECHINATE phytoliths (0.5%), attributable to palm trees (Neumann et al., 2019, 195–196).

On the contrary, TV003 is the sample that presents the highest percentage of long cell phytoliths among the three, 36%, of which 7.5% is formed by ELONGATE DENDRITIC morphotypes

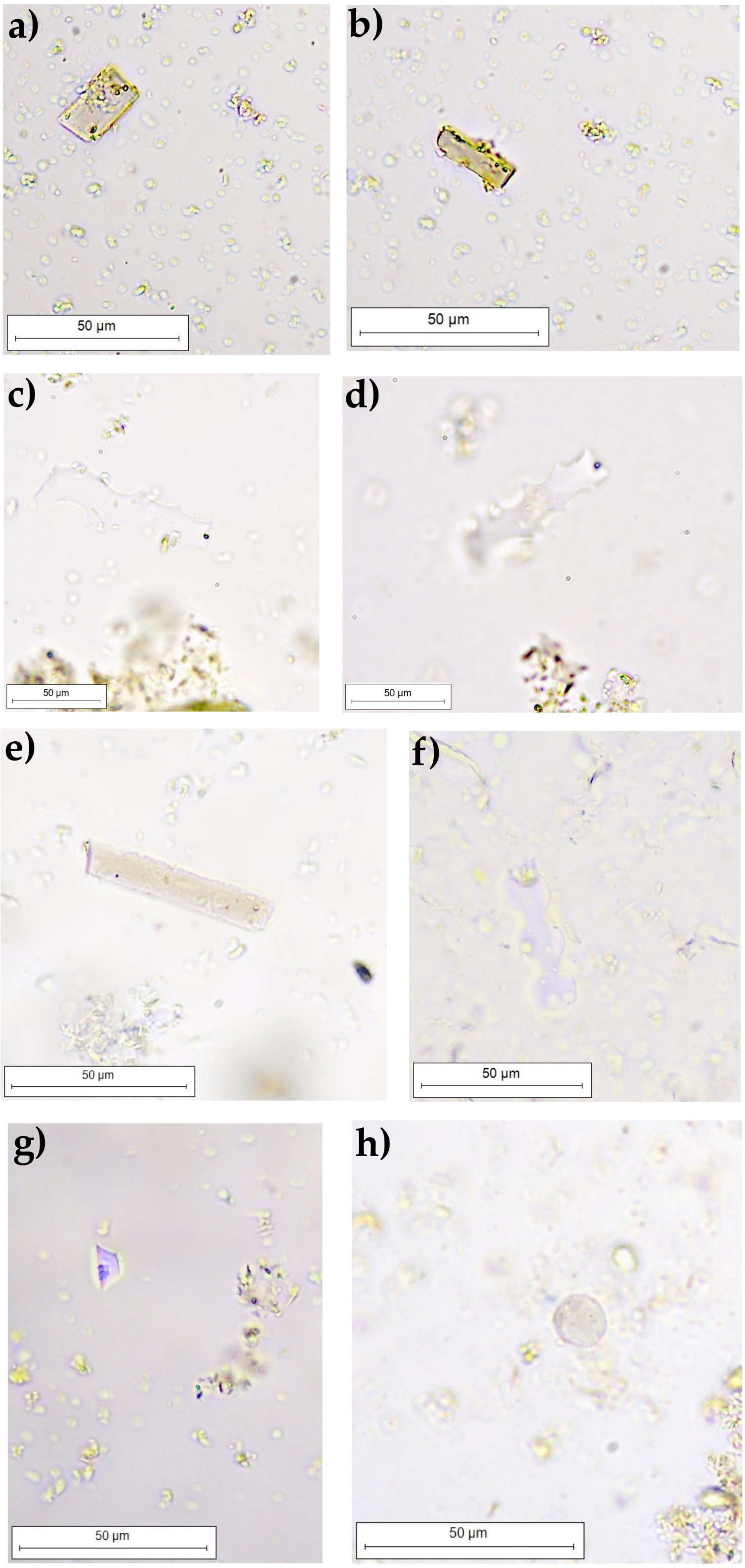

FIGURE 2
Examples of phytoliths extracted from the samples: **(a)** BLOCKY (PE08); **(b)** BLOCKY, side view (PE08); **(c)** ELONGATE DENDRITIC (TV003); **(d)** ELONGATE DENDRITIC, side view (TV003); **(e)** ELONGATE ENTIRE (HA002); **(f)** POLYLOBATE (HA002); **(g)** RONDEL (TV003); **(h)** SPHEROID PSILATE (TV003).

TABLE 3 Phytolith morphotypes: absolute counts and frequency (%) per sample (PE08—TV003—HA002).

Phytolith morphotype	PE08		TV003		HA002	
	Absolute count	Frequency (%)	Absolute count	Frequency (%)	Absolute count	Frequency (%)
Spheroid psilate	7	3.5	2	1	9	4.5
Spheroid echinate	0	0	1	0.5	1	0.5
Acute bulbosus	6	3	5	2.5	9	4.5
Blocky	22	11	17	8.5	11	5.5
Bulliform flabellate	16	8	13	6.5	4	2
Elongate entire	33	16.5	57	28.5	33	16.5
Elongate sinuate	5	2.5	0	0	0	0
Elongate dendritic	4	2	15	7.5	3	1.5
Bilobate	0	0	0	0	3	1.5
Polylobate	0	0	0	0	1	0.5
Crenate	1	0.5	0	0	13	6.5
Rondel	82	41	83	41.5	92	46
Trapezoid	24	12	7	3.5	21	10.5
Total	**200**	**100**	**200**	**100**	**200**	**100**

(produced in the inflorescence parts of a plant and/or in crop by-product, cf. Santiago-Marrero et al., 2024). Moreover, when this morphotype has been identified at the microscope and categorized, it was possible to attest that the recognized Elongate dendritic phytoliths (see example in Figure 2) belonged to C3 wild grasses and/or cereals and not to Panicoids (*sensu* Ball et al., 1999; Lu et al., 2009; Madella et al., 2014; Neumann et al., 2019). Elongated dendritic phytoliths have not been found in such high percentages in the other two samples (PE08 2%; HA002 1.5%). The phytolith assemblage of TV003 contains a small percentage of Spheroid echinate as well (0.5%).

Phytolith morphotypes percentages and assemblage analysis are presented for all the three samples in Tables 3, 4, and visually shown in Figure 3.

Results from pollen analysis did not attest the presence of pollen in the samples analyzed. However, after the first preprocessing step of the pollen acetolysis protocol (water bath 90 °C with Sodium hydroxide 10% for 5 min), the sample TR016 showed the presence of a vivid blue compound (7.5B7/8 or 7.5B8/8; Figure 4).

4 Discussion

Evidence from phytolith analysis provides integrative information to the ORA results that, when interpreted within the broader archeological context of the pottery remains analyzed, can provide new insights into the local environment, plant use in terms of dietary, cultural and symbolic practices, allowing us also to make assumptions about intercultural influence and exchange.

TABLE 4 Phytolith morphotypes assemblage analysis per sample (PE08—TV003—HA002).

Assemblage analysis (morphotypes ratio)	PE08	TV003	HA002
long cell	21	36	18
short cell	53.5	45	64.5
Index long cell/short cell	**0.4**	**0.8**	**0.3**
DICOTS	14.5	10	10.5
POACEAE	53.5	45	64.5
Index D/P	**0.3**	**0.2**	**0.2**
Elongate dendritic	2	7.5	1.5
Elongate entire + Elongate sinuate	19	28.5	16.5
Index inflorescence/culm-leaves	**0.11**	**0.26**	**0.09**

Despite the modest number of samples, within a comparative perspective, the results from phytolith analysis show a high amount of dicot phytolith morphotypes from wood and bark in all the three samples.

Even relatively modest percentages of dicots phytolith morphotypes can be interpreted as meaningful (Carnelli et al., 2004; Tsartsidou et al., 2007), as dicots have a low phytoliths production compared to grasses. Their high number in all the three samples (each of them located in diverse archeological contexts) can thus be interpreted beyond any doubt (cf. Wang et al., 2022 for a similar argument about the interpretation of a high amount of grass phytoliths extracted from pottery residues). First and foremost, such a high amount of dicot phytoliths (when associated with higher percentages of C3 grasses, as in the samples analyzed here) can inform about the local paleoenvironment of the time,

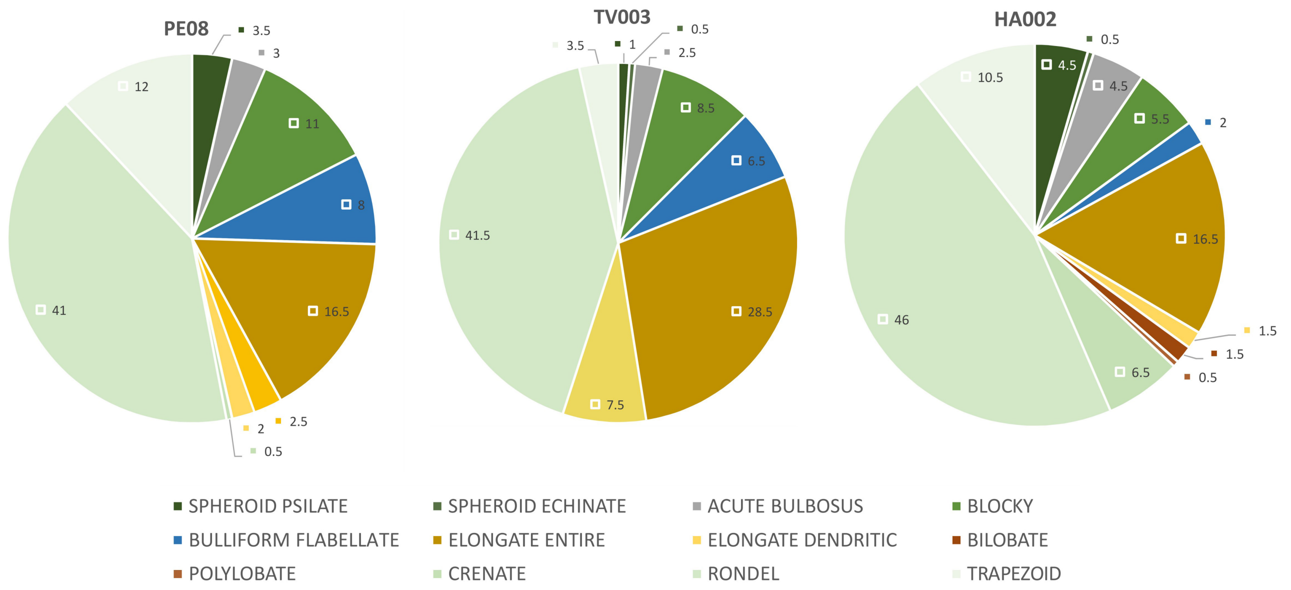

FIGURE 3
Phytolith morphotypes percentages in the three samples PE08, TV003, and HA002.

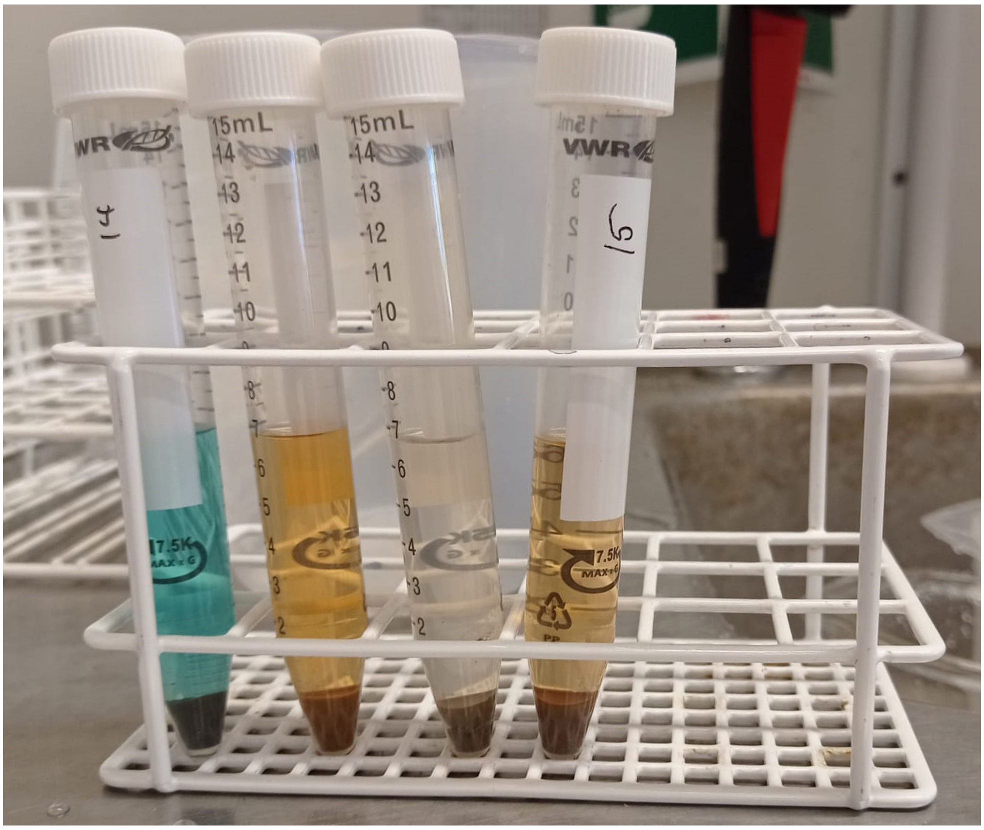

FIGURE 4
Vivid blue compound present in sample TR016 after the first preprocessing step of pollen acetolysis.

characterized by a general temperate climate and related type of vegetation (Neumann et al., 2019). Furthermore, the fact that such a high amount of dicots phytoliths from wood and bark is present in all the three structurally diverse and geographically-chronologically distinct pottery fragments (a small burnished patera—Peñalosa PE08, a rim sherd of a coarse storage jar—Tejada TV003 and an upper part of a coarse storage jar with one handle—Hacienda HA002) can be interpreted as a form of practice common to all the three different sites. One first hypothesis is that such a common practice may have been either local indigenous or brought from outside in the past, and already well-diffused and adopted by that time. Since the three samples belong to three pots different in both size and specific shape, one potential explanation for their common high amount of dicot phytoliths could be that these phytoliths are evidence of wood ashes use as pottery temper or to improve the quality of the clay (Coria-Noguera et al., 2021; Gardner et al., 2025).

Another possible explanation for the origin of the counted dicot phytoliths might be rituals of light burning of small wood branches to produce smoke and/or fragrance (*sensu* Buonasera et al., 2023). However, this last explanation would apply only to the small patera—PE008, since the other two fragments derive from bigger pots that could not have had such a use.

Preserved in the fabric of pottery (Wallis et al., 2014; Dzhanfezova, 2021), when not in a forest context, higher proportions of dicot phytoliths (especially those derived from wood/bark) have been associated in the literature with ash presence (Delhon, 2010; Dudgeon, 2024). As demonstrated by Albert et al. (2003), phytoliths from wood ashes are highly informative in archeological contexts and Delhon (2010) observed how this type of phytoliths has no specific characteristics indicative of their exposure to high heat (i.e., their supposed blackish color as charred phytoliths). Consequently, the high percentage of dicot phytoliths in all the three samples investigated in this paper could be correlated with the presence of wood ash in the pottery fabric.

One last alternative explanation that might explain such a large quantity of dicot phytoliths in all the three fragments can be the use of wooden tools as part of food processing, for example, to grind or crush plant materials (Portillo et al., 2017, 2024). However, such a hypothetical explanation does not take into consideration the distinctive dimensions and structural characteristics of the three vessels, which differ significantly in size, shape, and—likely—function. Consequently, the probability of an identical use-related phytolith signature across all three samples appears highly unlikely.

We conclude that the most probable explanation of the high number of dicot phytoliths in all the three fragments may be indicative of wood-ash tempering practices of the pottery or its clay improvement; practices that, moreover, appear to be connected with the local culture at the broader regional level (Coria-Noguera et al., 2021). Another element of commonality among the three samples is indicated by the ORA results, which show the presence of beeswax and conifer resin. According to the literature and the specific use we have hypothesized for every single pot, as explained further below, the mixture of beeswax and conifer resin could be interpreted as mastic used for sealing and insulation purposes (Grace, 1996).

The comparison of phytolith analysis and ORA results for every single sample brings further integrative insights as well.

The results of the solvent extraction of lipids from sample **PE08**, published in Revert Francés (2024), show well-preserved beeswax, with a concentration of 334 µg/g of pottery powder. Additionally, a malic acid-rich fruit product was detected, with a concentration of 2 µg/g of pottery powder. Due to the elevated presence of beeswax, we then hypothesize that this small patera may have been used as a lamp (according to the interpretation of Revert Francés, 2024) or to contain some sort of products (e.g., cosmetic scented balms made with natural ingredients, probably fruits), while we exclude the ritual use to create light fragrance smoking (as advanced above, when providing a second interpretative option that could explain the high presence of dicot phytoliths from wood and bark).

In sample **TV003** (rim sherd of a coarse storage jar), the high percentage of long cell phytoliths (36%), of which 7.5% ELONGATE DENDRITIC from C3 wild grasses or cereals (Ball et al., 1999; Lu et al., 2009; Madella et al., 2014; Neumann et al., 2019), may attest the use of the vessel for storage of adult plants and/or crop by-products. The evidence of C3 cereals from the phytolith assemblages may be indicative of their cultivation and use at that time in the area, as reported by other sources (cf. Pérez-Jordà, 2020). From the ORA, a probable presence of ruminant fat, at an exceptionally high concentration of 5,637 µg/g of pottery powder was identified, which could have been used as a preservative for the adult plants and/or crop by-products processed or cooked in some way. A malic acid-rich fruit product (17 µg/g of pottery powder) completed the assemblage of organic compounds found in this coarse jar, whose most probable use could have thus been storage of prepared/processed food of some sort.

Sample **HA002** is derived from the upper part of a coarse jar with one handle. The potential interpretation of its scope as a serving jar is supported by both ORA and phytoliths results. From the ORA, an interesting and well-preserved mixture (470 µg/g of pottery powder) was identified, consisting of animal fat, conifer resin, other plant products (high degraded triterpenes) and wine (tartaric acid: 2.27 µg/g of pottery powder; malic acid: 0.77 µg/g of pottery powder; %TA: 74%, following Drieu et al., 2021), suggesting multiple possible liquid contents for this vessel. In addition, the presence of SPHEROID ECHINATE phytoliths in this sample may be evidence of the palm tree *Chamaerops humilis*, endemic of the Iberian Peninsula and widespread in the site area. Such an assumption is corroborated by the ORA results, which indicate the presence of *Chamaerops humilis* as well, through evidence of triterpenes (which are contained in the palm tree, cf. Taibi et al., 2025). Debels et al. (2024) do not exclude the idea that phytoliths from palm trees could be indicative of certain food products (e.g., palm oil and palm wine, commonly prepared at the time in the area according to Pérez-Jordà et al., 2017). However, since the ORA results provide evidence of highly degraded triterpenes, and SPHEROID ECHINATE phytoliths are also present in sample TV003, we do not exclude the hypothesis that, apart from an indicator of palm wine or oil, the presence of these phytoliths could be an indication of the use of palm wood in pottery-making practices as well. Sample HA002 also contains traces of millets (Panicoideae), proved by the presence of BILOBATE, POLYLOBATE and CROSS morphotypes. Even though millets could not be attested by the ORA on this specific sample (*sensu* Standall et al., 2022), millets were present in the city of Huelva (Pérez-Jordà et al., 2024) and, thus, the liquid content of the jar could have also been porridge or millet-based beverages (cf. Dunne et al., 2022). When compared with sample TV003 (characterized by traces of C3 cereals) and framed within an intercultural perspective emphasizing millet as typical North African crop (Le Moyne et al., 2023) and the city of Huelva as having a long history of Phoenician influence (Pérez-Jordà, 2020; Pérez-Jordà et al., 2024), we could advance the hypothesis that sample HA002 is indicative of a change in local agricultural practices and/or diet, which could then attest cultural exchanges between southern Spanish and North African communities.

Summarizing, the results of our work indicate clearly that combining phytolith analysis with ORA can be a valuable integrated method to further investigate prehistoric material culture, cultural influences, and gain information about local environmental and climatic conditions, even in the case of using post-ORA samples. Additionally, from the interpretation of our results, it is possible to assume that the phytoliths extracted and analyzed from the post-ORA pottery powder derive quite probably from both the ceramic matrix and their prolonged specific uses. The high potential of phytolith analysis to be informative even in the case of post-ORA residues can thus be attested.

Our results indicate, moreover, that phytoliths extraction from post organic residue analysis is not only a viable integrative method, but also that the ORA chemical pre-treatment (cf. Mottram et al., 1999; Garnier and Valamoti, 2016), being centered on the extraction of organic compounds, has not altered the content of non-organic silica material, thus the integrity of the phytoliths in our samples. We believe that the drilling process for obtaining ORA samples is unlikely to have damaged the phytoliths, as they are smaller than the resulting powder particles, and therefore should remain intact during sampling. The preservation state of the phytoliths found in our samples is excellent (cf. Figure 2), and no broken phytoliths have emerged during the analysis at the microscope. However, a dedicated study and controlled experiments on the impact of such drilling process on phytoliths preservation should be worth pursuing. Another aspect that deserves further investigation is how variations in pottery manufacturing (e.g., kiln temperature, clay fabric, firing technology) may affect the preservation of phytoliths originally embedded in the clay, as well as how higher porosity of the vessel could result in an increased possibility of hosting a greater amount of residues in the matrix of the clay. These aspects are extremely relevant and worth being investigated further. However, they are out of the scope of this paper, since the focus of our work has been on attempting phytoliths extraction and analysis from pottery powder samples already used for Organic Residue Analysis, with the aim to see if such methodological integration performed in the same samples could provide additional information on pottery making and use, as well as of practices related to plant use, food preparation and/or consumption, cultural exchanges and networks.

The vivid blue compound observed in sample TR016 during the pollen extraction process is worth reflection and further investigation. As a color which rarely occurs in natural conditions, particularly in the Iberian Peninsula Early Iron Age context, the possibility of encountering this hue unintentionally is remote. Our opinion is that the chemical reaction given by the sample during the first step of the acetolysis process in the laboratory was key to determine such a specific compound; we believe that the heat during the process could have been the deciding factor in "awakening" the hue. Moreover, the lack of pollen in this sample led to the presumption of the sample being a copper-derived blue pigment. Although, based on Pliny the Elder's description of the production of blue pigments and dyes (Pliny the Elder, 1938, LCL 394: 120–121), the use of woad (*Isatis tinctoria* L.) and even indigo (*Indigofera tinctoria*; Vauquelin et al., 2024) was common at the time (Clark et al., 1993), pollen or phytoliths from this species could be expected to be encountered in the sample. Dyeing with indigo was apparently accomplished early in European prehistory (Kramell et al., 2014). Woad has been the subject of several interesting studies on ancient dye techniques, currently cultivated also as a sustainable source of indigo dye (Hartl et al., 2015). The dye compound indigotin has been identified in many archaeological contexts, from Neolithic Çatalhöyük to contemporary Turkey (Zohary et al., 2012).

This particular sample, from an Early Iron Age site (fifth century BCE), was collected from El Turuñuelo de Guareña, a ritualistic context including animal sacrifices and ceremonial burning, converting the building into a Tumulus. The sample was extracted from a coarseware fragment initially believed to be a crucible. However, this may have been the result of a glassy pigment residue on the internal surface of the fragment, a product of the heating process needed for the elaboration of copper-based pigments, particularly "Egyptian Blue," requiring a heat between 800 ° and 900 ° for its production. Yet, it cannot be discarded the hypothesis that this pigment could be a lapis lazuli ultramarine. However, given the faded nature of the sample before being subjected to treatment, it is more probable that the pigment may be a copper silicate (Pilans, 1913). We can neither exclude the hypothesis that the vivid blue hue was the result of surface decoration of pottery materials at the final stage of production, made with vivianite (cf. Dillian and Bello, 2009, even though its use has not been archeologically attested in Mediterranean countries yet).

In conclusion, the work presented in this paper demonstrates that, without further extraction of samples on already limited material culture artifacts, post-ORA residues can be further processed for phytolith analysis, whose results—when interpreted within an interdisciplinary framework—can be highly informative of the complex relationships between material culture(s) and local environment(s), above all in historical contexts as prehistory, where preservation of other types of evidence may be extremely poor.

Data availability statement

The datasets presented in this study can be found in online repositories. The names of the repository/repositories and accession number(s) can be found at: https://doi.org/10.5281/zenodo.15371479.

Author contributions

VF: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Resources, Visualization, Writing – original draft, Writing – review & editing. RA: Conceptualization, Formal analysis, Investigation, Methodology, Writing – review & editing. MS: Conceptualization, Formal analysis, Investigation, Methodology, Writing – original draft, Writing – review & editing. EF: Conceptualization, Formal analysis, Funding acquisition, Investigation, Methodology, Resources, Visualization, Writing – original draft, Writing – review & editing.

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Conflict of interest

The author(s) declared that this work was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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The author(s) declared that generative AI was not used in the creation of this manuscript.

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