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In Situ Analysis of Greek Ceramics and an Iberian Amphora by XRF: Eva Klabin House Museum

Análise *In Situ* de Cerâmicas Gregas e Ânfora Ibérica por XRF: Museu Casa Eva Klabin

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ABSTRACT

The Eva Klabin House Museum (MCEK) in Rio de Janeiro houses ancient artifacts of significant cultural value. This study employed X-Ray fluorescence (XRF) to analyze four pieces: three Greek ceramics and an Iberian amphora. Analyses were conducted in situ with a portable Bruker Tracer III device (rhodium target, 40 kV, 35 µA, 60-second acquisition time). In the Iberian amphora, calcium (Ca), iron (Fe), and lead (Pb) were detected. The high Ca content suggests prolonged submersion encrustations, while the predominant Fe indicates iron-rich clay. Among the Greek ceramics, the situla contained Ca, Fe, and cobalt (Co), with Co raising concerns about potential later pictorial interventions or forgery. The Archaic Greek vase and the smaller vase showed similar compositions, dominated by Fe and Ca, suggesting the use of iron oxide pigments and possible calcite or gypsum in the clay. Trace elements such as manganese, zinc, and strontium provided additional insights into clay origin and production techniques. The results highlight the need for complementary analytical methods. These methods would help determine clay sources, pigments, and manufacturing processes more precisely, while underscoring the importance of non-destructive analysis for preserving museum collections.

keywords greek ceramics, iberian amphora, *in situ* analysis, elemental composition, pigments, clay refinement, cultural heritage

RESUMO

O Museu Casa Eva Klabin (MCEK), no Rio de Janeiro, abriga artefatos antigos de grande valor cultural. Este estudo utilizou fluorescência de raios X (XRF) para analisar quatro peças: três cerâmicas gregas e uma ânfora ibérica. Análises foram realizadas *in situ* com um equipamento portátil Bruker Tracer III (alvo de ródio, 40 kV, 35 µA, 60 segundos de aquisição). Na ânfora ibérica, detectou-se cálcio (Ca), ferro (Fe) e chumbo (Pb). O alto teor de Ca sugere incrustações por submersão prolongada, enquanto o Fe predominante indica argila rica em ferro. Entre as peças gregas, a situla apresentou Ca, Fe e cobalto (Co), sendo o Co um possível indicativo de intervenção pictórica posterior ou falsificação. O vaso grego arcaico e o vaso menor exibiram composições similares, dominadas por Fe e Ca, sugerindo uso de pigmentos de óxido de ferro e possivelmente calcita ou gesso na argila. Elementos traço como manganês, zinco e estrôncio forneceram dados adicionais sobre origem da argila e técnicas de produção. Os resultados destacam a necessidade de métodos complementares para precisar a origem das argilas, pigmentos e processos de fabricação, reforçando a importância de análises não destrutivas na preservação de acervos.

palavras-chave cerâmicas gregas, ânfora ibérica, análise *in situ*, composição elementar, pigmentos, refino de argila, patrimônio cultural

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Introduction

The analysis of paintings presents significant challenges due to their complex composition, which typically includes multilayered mixtures of inorganic and organic materials with both crystalline and amorphous structures. While X-ray fluorescence (XRF) is a valuable non-invasive technique, it may not always provide sufficient information on its own. In such cases, complementary methods like portable X-ray diffraction (XRD) become essential; however, the use of portable XRD for the non-invasive analysis of paintings remains relatively limited (Mendoza-Cuevas et al., 2023).

Over the past decades, various spectroscopic techniques have been employed in non-invasive investigations of valuable paintings using portable systems. These methods have proven instrumental in supporting restoration and conservation efforts, as well as in addressing historical questions related to attribution and provenance. Nevertheless, there is often a critical need to examine the stratigraphy of pigment layers to determine specific painting techniques, an analysis that must also be performed without physically altering the artwork (Mendoza-Cuevas et al., 2023).

The study of artworks and historical artifacts is essential for preserving cultural heritage, enabling a deeper understanding of their origin, production techniques, and conservation status. In this context, Archaeometry plays a fundamental role by providing scientific methods for material analysis and monitoring restoration processes. Among the available techniques, X-ray fluorescence (XRF) stands out for its non-destructive nature and ability to perform *in situ* analyses using portable equipment (Gerodimos et al., 2022; Liritzis & Korka, 2019).

Pigments are materials historically employed to add color and visual expression to objects and architectural structures. In antiquity, they were extensively used in the decoration of walls, sculptures, and various architectural elements. Over the centuries, however, such surfaces have been subjected to natural degradation processes, including wind and sand erosion, solar radiation, rainfall, and fluctuations in climate. These factors have contributed to the fading, peeling, and loss of pigment layers, as well as the deterioration of decorative features. The restoration and protection of these pigment layers are essential not only for preserving the aesthetic characteristics of ancient structures but also for safeguarding their historical and cultural value. These efforts aim to recover and maintain the original appearance of heritage objects, ensuring the transmission of their cultural significance to future generations (Ma et al., 2025).

Fundamentally, an X-ray fluorescence (XRF) experiment begins with the excitation of the sample by a primary X-ray beam. The elements within the sample absorb the incident photons and subsequently undergo electronic decay, emitting secondary radiation known as characteristic fluorescence. This emitted radiation, referred to as the secondary beam, is then detected and analyzed based on its energy. The data acquisition system converts the resulting electrical pulses into a fluorescence spectrum, typically displayed as a graph of intensity versus wavelength (or energy). From this spectrum, it is possible to obtain qualitative and semi-quantitative information about the elemental composition of the sample, as well as perform chemical mapping and other compositional analyses (Nascimento-Dias et al., 2017).

X-ray fluorescence (XRF) spectroscopy has become a widely used technique for the characterization of materials in the fields of cultural heritage and archaeology. Its non-destructive nature, fast data acquisition, simple setup, and portability make it particularly suitable for preliminary analyses of historical artifacts. One of its key advantages is the ability to perform elemental analysis without any physical contact with the object, a feature that is highly valued in conservation practices. Consequently, XRF has become a standard tool among heritage preservation professionals.

Handheld and portable XRF devices further expand its applicability by enabling *in situ* analyses under a wide range of environmental and field conditions, solidifying the technique's place in the analytical repertoire of heritage science (Chiti et al., 2024).

Moreover, XRF is especially effective in the study of painted surfaces and pigment layers due to its non-invasive character, high spatial resolution, and capacity to simultaneously collect data from both surface and subsurface layers. The technique also allows for the detection of minor and trace elements, which can offer significant insights into the geographical origin of raw materials, the historical context of the object, and the production technologies employed. These capabilities are directly relevant to the fields of conservation and restoration, where such information guides informed decision-making (Harth, 2024; Klisińska-Kopacz et al., 2023).

In this context, the Eva Klabin House Museum represents a significant artistic collection in Brazil, housing artworks and objects that follow a classical collecting model. Eva Klabin, born in São Paulo in 1903, dedicated much of her life to building an artistic and cultural legacy. During the 1960s and 1970s, she expanded her collection, created conditions to house it, and initiated its cataloging process. In the 1980s, she formally established the Eva Klabin Foundation to preserve her collection and promote cultural activities. In 1990, a year before her passing, the foundation was officially established and donated to the city of Rio de Janeiro, becoming a reference center for art, history, and culture (Migliaccio, 2007; Silveira, 2023).

Given the cultural significance of this collection and the sensitivity of the artifacts involved, this study applies portable X-ray fluorescence (XRF) for the *in situ*, non-destructive analysis of selected pieces. The primary aim is to investigate their elemental composition, providing insights into the materials and techniques used in their production, and supporting future conservation and restoration efforts through scientific documentation and characterization.

Materials and methods

In this study, we applied the X-Ray fluorescence (XRF) technique to analyze a mini amphora and three ceramic vessels from the Eva Klabin House Museum collection. These pieces date back to the Classical period and were produced in Apulia (Magna Graecia) in the second half of the 4th century BCE (Migliaccio, 2007), as shown in Figure 1.

Figure 1 - Greco-Roman Works: (a) Athens Krater; (b) Miniature Vase; (c) Amphora; and (d) Southern Italy Situla.

(a) (b) (c) (d)

Figure 1(a) presents an Attic column-krater, a representative work of Archaic Greek ceramics, produced in Athens between 560 and 530 BCE. This krater exemplifies the decorative tradition of the time, featuring black-figure designs on an orange background, with purple details and incised lines. It has vertical handles supporting small plaques, characteristic of this type of vessel, and measures 28.5 cm in height and 38 cm in width. On its body, the metopes depict a feline and a swan, common motifs in Archaic Greek iconography, while the rim and handle plaques are adorned with female busts, possibly carrying religious or mythological significance. Crafted from wheel-thrown and polychrome-painted ceramics, this krater was used in Ancient Greece for mixing water and wine during symposia, potentially reflecting symbolisms of fertility, protection, and Dionysian worship. Although its authorship remains unknown, its stylistic features link it to Athenian workshops of the Archaic period, making it an important piece for the study of Greek ceramics (Migliaccio, 2007).

Figure 1(b) presents a miniature vase painted orange and black, belonging to the same Greco-Roman collection as the other artifacts analyzed in this study. The medieval amphora from the Iberian Peninsula, see Figure 1(c), with no identified author, is a glazed terracotta piece measuring 16.50 cm in height and 10.50 cm in width. It has two handles and features a wine-colored glaze decoration, depicting a central lion and geometric patterns on the neck. Its production belongs to the context of medieval Iberian ceramics, characterized by a fusion of Christian and Islamic influences. Beyond its utilitarian function, the piece carries symbolic meanings associated with strength and protection, reflecting the aesthetics and iconography of the period. Its study contributes to a better understanding of medieval artistic techniques and influences in the region (Migliaccio, 2007).

Figure 1(d) shows the Southern Italy situla, dated to the second half of the 4th century BCE. It features two vertical appendages on the rim, formed by three roundels arranged in a triangle. Its decoration is elaborate, with palmette ornaments, rows of egg patterns between lines, and black dots. Among the representations, a winged Eros stands out, facing left, holding a plant or flower in his right hand. Beside him, there is a woman in profile facing left but turning to the right. She is dressed in draped clothing, wears a headdress and a necklace, and holds a fan in her left hand and a musical instrument on her right, identified as a dancer. Made of wheel-thrown and polychrome-painted ceramic, the vessel measures 25 cm in height and 18 cm in width. While its specific authorship remains unknown, it originates from Apulia, Magna Graecia (Migliaccio, 2007).

To guide the analysis, specific points on the surface of each artifact were selected based on visual distinctions such as color, texture, and decorative detail. Each measurement point was named according to its position or morphological feature (e.g., “lion’s back”, “below handle”, “decorated rim”, “black region on the neck”), allowing precise documentation and comparison across the studied areas. The number of analyzed points varied according to the size and complexity of each piece, totaling 21 points for the Athens Krater, 12 for the Situla, 6 for the miniature vase, and 12 for the Amphora.

All spectra were labeled following a standardized file-naming system that included object identification and surface location (e.g., `dorso_leao_parece_vidro.pdz`, `bojo_embaixo_da_alca.csv`), facilitating the association between elemental composition and visual observation. This naming strategy also enabled data traceability throughout the interpretation process and supported spatial correlation with visual features.

The analyses were conducted using a Bruker Tracer III portable device, equipped with a rhodium (Rh) target, operating at a voltage of 40 kV, a current of 35 µA, and an acquisition time of 60 seconds. The device includes an internal calibration system provided by the manufacturer (Bruker), which ensures proper performance for qualitative analysis. As the objective of this study was to identify the elements present rather than determine their concentrations, no external quantitative calibration was required.

Results and discussion

Based on the analysis conducted, the main elements detected in the XRF spectra of the Athens Krater were calcium (Ca) and iron (Fe). Additionally, elements with lower-intensity peaks and trace elements were identified, including aluminum (Al), silicon (Si), phosphorus (P), potassium (K), titanium (Ti), chromium (Cr), manganese (Mn), copper (Cu), zinc (Zn), lead (Pb), rubidium (Rb), strontium (Sr), and zirconium (Zr). Figure 2 shows some of the characteristic XRF spectra obtained for the Athens Krater.

In Figure 2(a), the XRF spectrum and the image of the orange region behind the tiger are presented. The most intense peaks correspond to calcium (Ca) and iron (Fe), indicating the use of ochre pigments and the presence of carbonates such as calcite ($CaCO_3$) or gypsum ($CaSO_4 \cdot 2H_2O$). The detection of trace elements such as manganese (Mn), zinc (Zn), and strontium (Sr) suggests specific characteristics of the clay and possible refining techniques (Gianoncelli et al., 2021; Papadopoulou et al., 2007).

In Figure 2(b), corresponding to the black region of the horse, Fe predominates, possibly associated with black-colored iron oxides (Fe_3O_4), accompanied by Mn and Ca. These elements indicate black pigments of mineral origin, such as manganese oxide (MnO_2) and “black bone” ($Ca_{10}(PO_4)_6(OH)_2$).

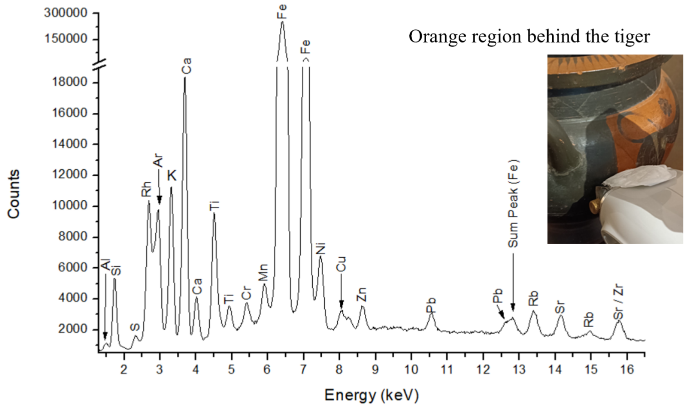
In Figure 2(c), which depicts a black region without decorative details, the detected elements are similar to those in Figure 2(b), but with slightly lower intensities for Ca and K, reinforcing the uniformity in pigment application. Finally, in Figure 2(d), showing the tiger’s back (side B), Fe and Ca once again predominate, suggesting consistent use of the same pigment mixture throughout the piece. The presence of Rb, Sr, and Zn serves as a potential provenance marker for the clay (De Bonis et al., 2018; Gianoncelli et al., 2021).

The black pigment may also contain carbon-based materials such as carbon black or charcoal, which cannot be detected by XRF, requiring complementary analyses to confirm.

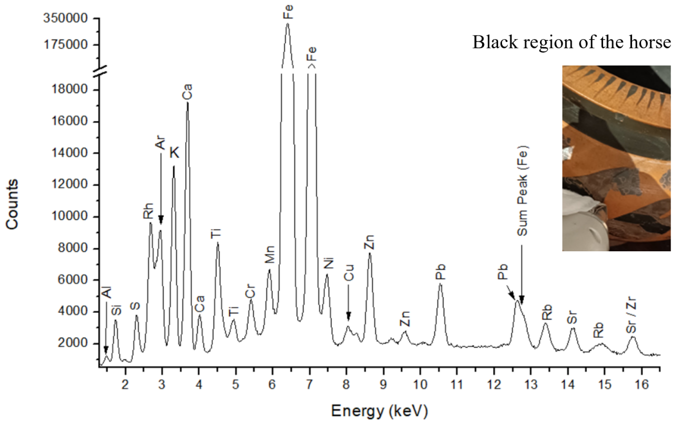
Given the remarkable similarities in their elemental compositions, the miniature vase was examined in direct comparison with the Athens Krater, allowing for a more comprehensive assessment of potential shared origins, production methods, and raw material sources. The striking correspondence between their XRF spectra indicates that both artifacts may have been produced using similar clays and pigments, possibly within the same cultural and technological context. Consequently, the analytical interpretations and considerations applied to the Archaic Greek vase are equally relevant to the miniature vase. Figure 3 present representative XRF spectra illustrating this close compositional relationship.

Figure 2 - Characteristic XRF spectra and corresponding photos of the Athens Krater: (a) orange region behind the tiger; (b) black region of the horse; (c) black region without details; (d) tiger's back, side b.

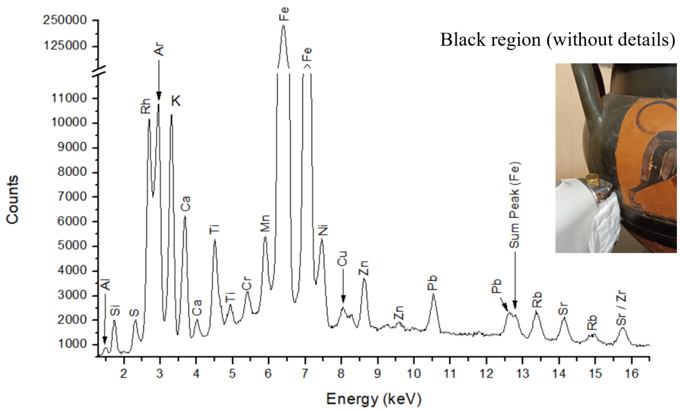
(a)



(b)



(c)



(d)

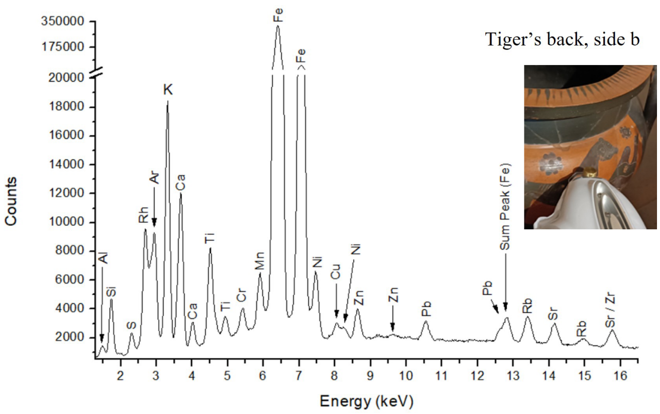
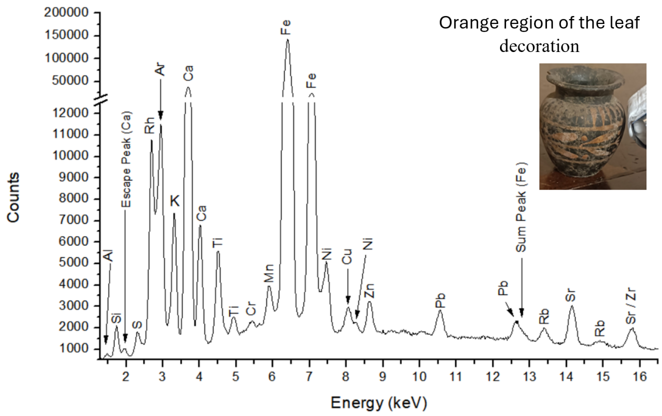
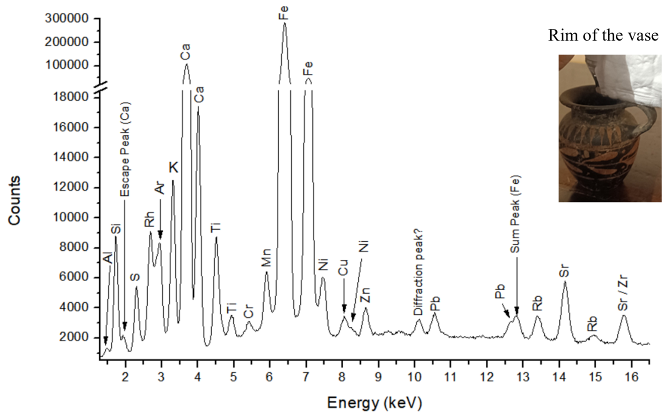


Figure 3 - Characteristic XRF spectra of the Mini Vase: (a) XRF spectrum and photo of the orange region of the leaf; (b) XRF spectrum and photo of the vase rim.

(a)



(b)



In Figure 3(a), corresponding to the orange region of a decorative leaf, the spectral pattern is almost identical to that of the Athens Krater, with iron (Fe) and calcium (Ca) as the dominant elements and traces of manganese (Mn) and zinc (Zn), suggesting the use of similar ochres. In Figure 3(b), at the rim of the vase, iron (Fe) and calcium (Ca) again predominate, accompanied by traces of manganese (Mn) and zinc (Zn). The consistency between these results indicates that the piece was likely produced using clay and pigments comparable to those found in the Krater, possibly within the same technical and production context.

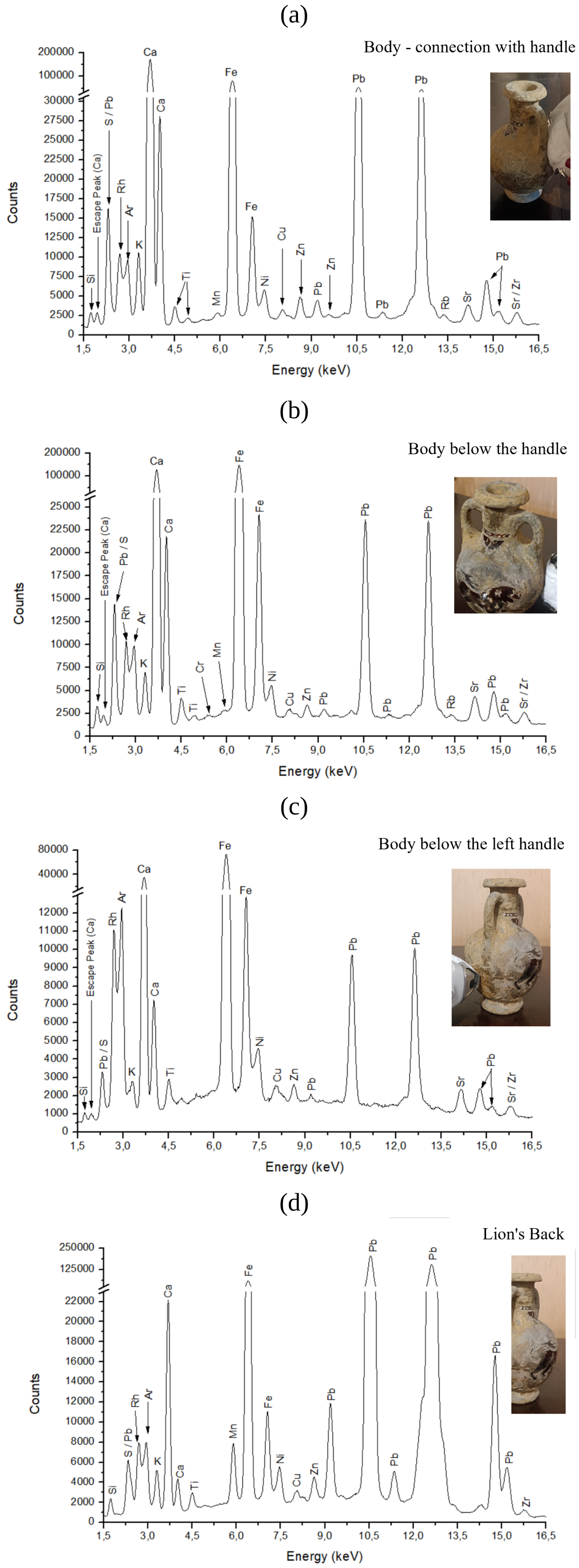
Overall, the strong compositional similarity between the two artifacts supports the hypothesis of a shared origin and manufacturing technique.

The amphora's XRF spectra reveal calcium (Ca), iron (Fe), and lead (Pb) as the main elements, alongside silicon (Si), potassium (K), titanium (Ti), chromium (Cr), manganese (Mn), copper (Cu), zinc (Zn), rubidium (Rb), strontium (Sr), and zirconium (Zr) as minor or trace elements. The high calcium (Ca) levels are linked to calcareous encrustations from prolonged submersion, while iron (Fe) is the primary component of the clay body (Cámara et al., 2017). The manufacturing process likely followed ancient ceramic recipes involving purified and tempered clay, with inclusions such as quartz, feldspar, or calcite (Mommsen, 2001).

In the pictorial layer, calcium (Ca) may derive from pigments such as white bone ($Ca_{10}(PO_4)_6(OH)_2$), calcite, or gypsum; Fe is linked to ochres (yellow, brown, and red); and Pb indicates the presence of pigments like lead white, massicot, or lead red. The detection of copper (Cu) suggests possible use of malachite, azurite, Egyptian green, or Egyptian blue, requiring further analytical confirmation. Some characteristic XRF spectra of the Amphora are shown in Figure 4.

In Figure 4(a), the body near the handle shows high calcium (Ca) from calcareous incrustations, with iron (Fe) as the main clay component; in Figure 4(b), lead (Pb) appears alongside calcium (Ca) and iron (Fe), indicating lead-based pigments; in Figure 4(c), the composition mirrors previous points, confirming consistency; and in Figure 4(d), manganese (Mn) joins calcium (Ca), iron (Fe), and lead (Pb), possibly linked to umber pigment.

Figure 4 - Characteristic XRF spectra of the Amphora and corresponding photos: (a) body – connection with the handle; (b) body – underneath the handle; (c) body – underneath the handle (left side); (d) lion's back.



However, when evaluating the spectra of the Southern Situla, the main elements detected were calcium (Ca), iron (Fe), and cobalt (Co). Additionally, elements with lower-intensity peaks and trace elements included aluminum (Al), silicon (Si), phosphorus (P), potassium (K), titanium (Ti), chromium (Cr), manganese (Mn), copper (Cu), zinc (Zn), lead (Pb), rubidium (Rb), strontium (Sr), and zirconium (Zr).

The low-intensity and trace elements found can be attributed to minor components commonly present in natural clays and are therefore frequently observed in trace element analyses of ancient ceramics (Hall & Minyaev, 2002; Romano et al., 2006; Tsiachri et al., 2018). The spectra obtained by XRF pose significant interpretative challenges, as the results reflect the overlap of elements detected in the pictorial layer, underlying layers, and those present in the clay used in the object's fabrication (Hall & Minyaev, 2002).

The intense iron (Fe) peaks, one of the main constituents of clays, suggest the use of ochre pigments in the pictorial composition. The identification of calcium indicates the possible presence of white compounds such as calcium carbonate ($CaCO_3$), gypsum ($CaSO_4 \cdot 2H_2O$), or white bone ($Ca_{10}(PO_4)_6(OH)_2$).

Figure 5 presents the characteristic XRF spectra of the Southern Italy Situla. In Figure 5(a), the black background region shows an XRF spectrum dominated by iron (Fe), calcium (Ca), and cobalt (Co). The presence of cobalt is atypical for the period and suggests the possible application of anachronistic pigments. In Figure 5(b), corresponding to the core of a lateral flower, the same elements are detected, though with variations in intensity, indicating consistency in the pigment composition across different decorative areas. In Figure 5(c), representing Eros' chest, the pattern of dominant iron (Fe), calcium (Ca), and cobalt (Co) is repeated, supporting the hypothesis of uniform pigment use throughout the piece. In Figure 5(d), the skirt of the female figure displays a similar elemental distribution, further confirming this homogeneity.

The detection of cobalt (Co) in all analyzed areas raises three possible scenarios: (i) The vase is a forgery. (ii) The ceramic body is authentic, but the pictorial layer is anachronistic, potentially added in a later period. This hypothesis is supported by the fact that, while the trace elements are consistent with ancient Greek ceramics, the pictorial layer could have been applied either in the late Middle Ages (if smalt was used) or after the late 18th century (when cobalt green became available). (iii) Both the vase and the pictorial layer are authentic, reflecting the legitimate use of cobalt within a specific historical context.

Further analyses, such as Raman spectroscopy and XRD, are required to confirm its origin. Cobalt is present in various green and blue pigments, including cobalt green ($CoO \cdot nZnO$), smaltite ($(Co,Ni)As_{3-x}$), cobalt blue ($CoO \cdot Al_2O_3$), and cerulean blue ($CoSnO_3$).

Noll et al. (1975) discusses the use of aluminum cobalt oxide (Al_2CoO_4) in blue pigments of ancient Egyptian ceramics. The Romans also used cobalt blue (Rapp, 2009). In the Situla, the presence of cobalt (Co) may be related to these ancient applications. However, the cobalt used in antiquity was not in the form of cobalt oxide (CoO), as this compound does not occur naturally (Privitera et al., 2024). To determine the exact origin of the identified cobalt, further analyses such as Raman spectroscopy or XRD are required. Figure 5 presents some of the characteristic XRF spectra of the Southern Italy Situla.

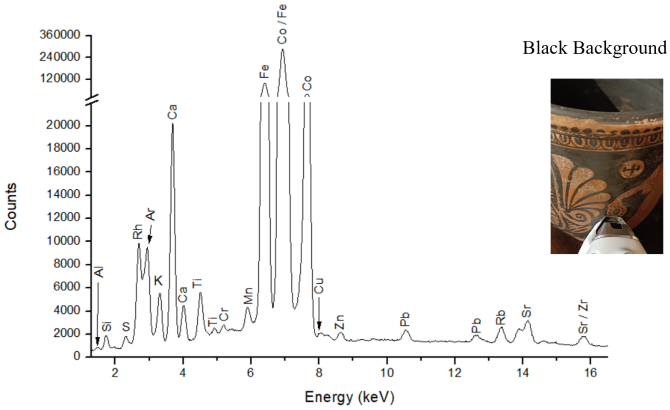
Therefore, the multielemental analysis, Table 1, revealed the presence of the following elements and possible pigments in the analyzed works:

Table 1 - Pigments, pigment distribution, and analyzed works

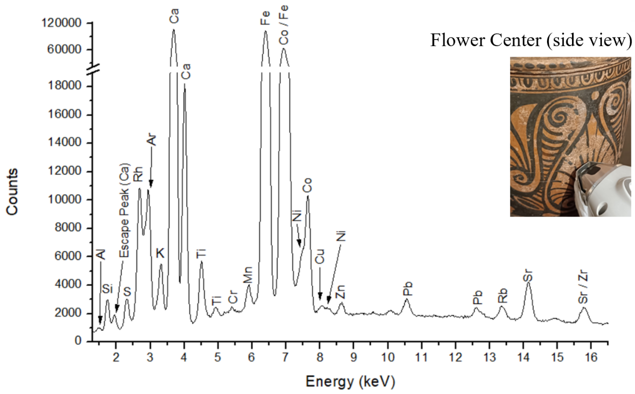
Elements	Pigments	Works
Calcium (Ca)	White bone, white, calcite, gypsum, and black bone	Amphora, Situla, and Athens Krater
Iron (Fe)	Brown ochre, yellow ochre, black iron oxide, and red ochre	Amphora and Situla
Lead (Pb)	Lead white, massicot, lead red, and litharge	Amphora, Situla, Mini vase, and Athens Krater
Cobalt (Co)	Cobalt yellow, cobalt green, enamel, cobalt blue, and cerulean blue	Situla
Manganese (Mn)	Umbra, manganese dioxide	Amphora, Situla, Mini vase, and Athens Krater
Copper (Cu)	Malachite, azurite, Egyptian green, and Egyptian blue	Amphora, Situla, Mini vase, and Athens Krater

Figure 5 - Characteristic XRF spectra of the Southern Italy Situla and corresponding photos: (a) black background; (b) flower core (side); (c) region of Eros' chest; (d) skirt of the dress.

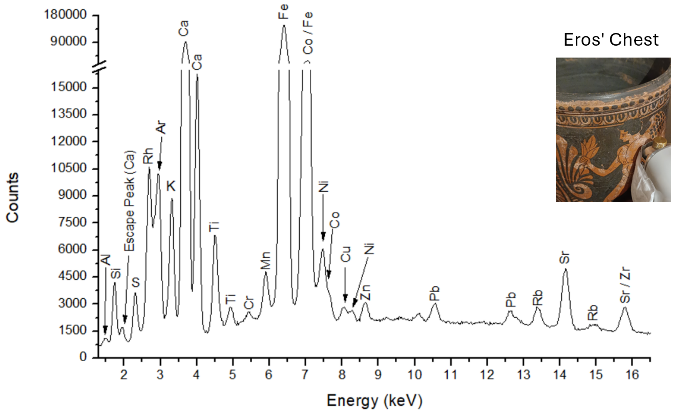
(a)



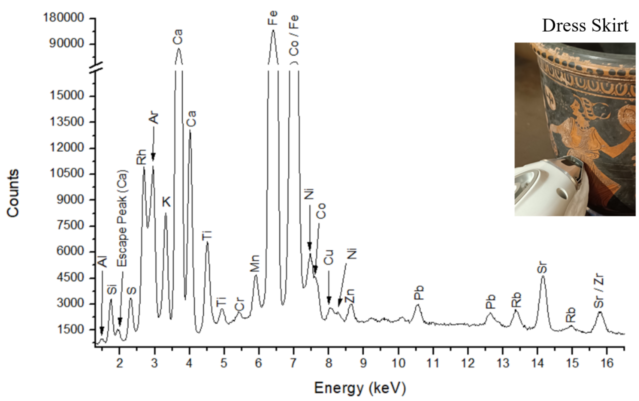
(b)



(c)



(d)



Conclusions

This study demonstrated the effectiveness of X-Ray fluorescence (XRF) *in situ* analysis for ancient ceramics, enabling the identification of the chemical elements present in the pieces from the Eva Klabin House Museum. The analysis revealed important characteristics about the materials used in the manufacturing and decoration of Greek ceramics and the Iberian Amphora, contributing to a better understanding of the techniques employed in different periods and regions.

The presence of elements such as iron (Fe), calcium (Ca), manganese (Mn), and lead (Pb) provided clues about the type of clay used and the pigments applied in the decoration of the pieces. The detection of cobalt (Co), an element uncommon in ancient pigments, raises concerns about potential later interventions or forgery, suggesting the need for complementary analyses to verify its origin. Furthermore, the calcium incrustations observed on the Amphora confirm its long submersion, directly influencing the results obtained.

In addition to providing essential data about the chemical composition of the works, XRF analysis assists in future restoration processes, allowing restorers to better understand the condition of the pieces and make informed decisions about necessary interventions. The study also emphasizes the relevance of using complementary techniques for more precise identification of clay, pigments, and the processes employed in the manufacture and decoration of the ceramics.

In this context, Raman spectroscopy can contribute by identifying molecular structures of pigments—particularly organic compounds and black pigments not detectable by XRF—while X-ray diffraction (XRD) is useful for determining the crystal structure of clays and pigments, helping to distinguish mineral phases with similar chemical compositions. These complementary methods are essential to confirm the nature and authenticity of materials, providing greater precision in pigment and clay identification (Mendoza-Cuevas et al., 2023).

The results obtained reinforce the importance of XRF as a non-destructive tool for the characterization of archaeological ceramics, contributing to provenance, authenticity, and conservation studies. However, for a more precise identification of pigments and manufacturing processes, complementary techniques such as Raman spectroscopy and X-ray diffraction (XRD) may be applied in future studies. Thus, this work not only expands knowledge about the analyzed pieces but also highlights the need for interdisciplinary approaches in Archaeometry.

Acknowledgments

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Author Contributions

J. E. Cavalcante and **R. M. P. S. Borges** an participated in: Conceptualization, Data Curation, Formal Analysis, Investigation, Methodology, Visualization, Writing – original draft. **I. V. N. S. Franzi**, **A. G. Paula** and **E. Azevedo** an participated in: Resources and Investigation. **R. T. Lopes** an participated in: Resources and Investigation. **D. F. Oliveira** an participated in: Validation, Resources, Supervision, Writing- revision and editing.

Conflicts of Interest

The authors certify that no commercial or associative interest in the manuscript represents a conflict of interest.

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