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Short communication

Litteras arena conspergere. Uncovering blotting sands on the Portuguese Inquisition documents ☆

M. Nunes [a], G. Wanzeller Martins [a], J. Sarraguça [h], F. Olival [b,c], P. Moita [a,d], Scott G. Mitchell [e], A. Claro [f], T. Ferreira [a,g,*]

[a] *HERCULES Laboratory / IN2PAST, University of Évora, Portugal*
[b] *CIDEHUS, University of Évora, Portugal*
[c] *History Department, Social Sciences School, University of Évora, Portugal*
[d] *Geosciences Department, Sciences and Technology School, University of Évora, Portugal*
[e] *Instituto de Nanociencia y Materiales de Aragón (INMA-CSIC/UNIZAR), Consejo Superior de Investigaciones Científicas-Universidad de Zaragoza, Spain*
[f] *CHAM- Centre for the Humanities, College of Social and Human Sciences, NOVA University of Lisbon, Portugal*
[g] *Chemistry and Biochemistry Department, Sciences and Technology School, University of Évora, Portugal*
[h] *Independent researcher, Portugal*

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ABSTRACT

Historical and archival research focused on the Portuguese Inquisition documents (1551–1800) uncovered the use of *arena* known as blotting sands, a writing accessory used to hasten ink drying. We present in this work the first systematic study combining image analysis, SEM/EDS and μ-Raman techniques, statistics and chemometrics to characterise the blotting sands used by the Portuguese Inquisition and hypothesise their provenance. Iron-titanium and iron oxide minerals categorised as texturally mature sands are the dominant species, consistent with sediment extraction from fluvial or beach contexts and later processed. Chemometrics unveiled time period trends by clustering the samples according to morphology and composition data. This work constitutes a groundbreaking step towards uncovering this intricate writing tool.

1. Introduction and research aim

The handwriting pursuit demanded various writing tools, including blotting sands *(arena)*, much in vogue from the 15th century onwards [1,2]. These sands were sprinkled on the written surface while the ink was fresh, increasing the liquid ink surface area and thus promoting a more rapid drying process.

Typically, blotting sands were any solid material whose grain size fell within the sand category (0.06–2.00 mm, from very fine to very coarse, respectively) [3], composed of minerals grains (e.g., quartz, iron oxides and mica) and other substances such as painted glass or organic materials like gums, wood, or bones [1,2].

One of the first definitions of blotting sands was found in a 17th-century English dictionary [4]. *Areya de escrever* is also mentioned in a Portuguese dictionary from the 18th century [5]. Nevertheless, our team's historical and archival research uncovered the use of blotting sands in earlier periods. The Convent of Christ in Tomar (Portugal), for example, was already buying "three-quarters of black sands for writing" by 1550 [6].

Unfortunately, blotting sands were overlooked in the last century, and almost no literature is available; therefore, little is known about their usage, chemical nature, and processing techniques to obtain them. Some of the few published works include Reissland et al. [1], who investigated blotting sands collected from various documents from the 16th to the 19th centuries and showed that different materials, namely minerals, metal fillings and glass, were selected. A little later, a comprehensive study [2] of Southwest Germany and Northern Switzerland blotting sands, comprising five centuries (14th-19th), uncovered once more that minerals, metal fillings and stained glass, amongst others, were employed with exciting correlations between the type of materials and geographical

☆ *Litteras arena conspergere* (authors' translation: «Throwing dust or sand to the letters»). Pereira, Bento, Florilegio dos modos de falar… Paulo Craesbeeck (Lisbon, 1655), p. 14.

* Corresponding author.
E-mail address: tasf@uevora.pt (T. Ferreira).

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Table 1
Secretaries and period of activity, number of volumes and samples collected. Five 50-year periods were defined for the statistical analysis.

period	secretary | activity	nr. inspected volumes	nr. samples
1551–1600	Domingo Simões (DS) | 1569 - 1579	1	7
1601–1650	Simão Lopes (SL) | 1617 - 1631	7	10
	Diogo Velho (DV) | 1636 - 1675		6
1651–1700	José Cardoso (JosC) | 1681 - 1700		3
1701–1750	José Coelho (JC) | 1700 - 1723	18	13
1751 - 1800	Jacome Esteves Nogueira (JEN) | 1723 - 1761		23
	António Batista (AB) | 1761 - 1769		8
	Manuel Ferreira de Mesquita (MFM) | 1772 - 1794		7

regions. A detailed classification system of blotting sands was established by then.

Under the safeguard of the National Archive of Torre do Tombo (ANTT), the Portuguese Inquisition documentation is considered the most relevant archive of its kind in Europe. It comprises documentation and correspondence from the general supervisory body, namely the General Council of the Holy Office (GC), to the Tribunals of Évora (TE), Lisbon (TL), Coimbra (TC) and Goa (India) [7,8]. Ecclesiastics with previous experience in the Tribunals' work served one at a time as GC secretaries, being in charge of all the paperwork related to the Inquisition's activities. The substantial daily life records of the Inquisition created a considerable amount of documentation, justifying the massive use of blotting sands to fasten the inks' drying process.

The goal of this work was to characterise for the first time blotting sands found in Portugal and particularly in an established institution like the Inquisition, during a significant period of its activity (16th-18th centuries). We also intended to investigate relationships between the materials used over time. Besides, blotting sands contributed to deepening the knowledge of the writing tools used for centuries, especially while iron gall ink was in vogue. It also can pave the way to design new strategies for conserving written heritage.

2. Materials and methods

2.1. Sampling

Representative sampling was carried out in the correspondence sent by the GC, gathered from nine volumes belonging to TE (ANTT, IE, L°41, 42, 44–48, 72, 631), three to TL (ANTT, IL, L°153, 155, 158), and fourteen to TC (ANTT, IC, L°21, 22, 24, 25, 27–35, 680), all compiled by chronological order. The number of selected volumes for each Tribunal depended on the conservation status, meaning that the book could be handled, and the availability of ink with lacuna to be sampled.

Blotting sands were collected per folium, linking each sample to a particular secretary and time period (Table 1). Seventy-seven samples were collected, comprising each 2 to 447 grains, with ca. 70% above 10 grains. The sampling procedure is available in the Supplementary Material (SM.1.1.).

2.2. Characterisation techniques and image analysis

An approach based on microscopic and spectroscopic (OM, SEM/EDS and μ-Raman) techniques was used to uncover the blotting sand grains' size, morphological features and mineral chemistry. Statistic and chemometric analyses were conducted on the obtained data aiming to study possible correlations and evaluate the different secretaries' clustering in time.

A full description of the analytical instrumentation, methodology and statistical and chemometric analyses, datasets and procedures is available in the SM.1.2. section.

3. Results and discussion

3.1. Grain size and morphological features

The grains vary in size from very fine (0.06 to 0.12 mm) to coarse (0.50 to 1.00 mm), but most samples (86–98%) fall into fine (0.12 to 0.25 mm) and medium (0.25 to 0.50 mm) ranges [3]. The grain size distribution (based on the Feret diameter (D_F), described in SM.1.2.2.) per the 50-year periods defined in Table 1 is presented in Fig. 1 (and per century in Fig. SM.2.1.1). The generic evaluation points out a very similar behaviour for all the time periods, with a symmetrical distribution and a near-constant median.

A maximum around $D_F = 0.3$ mm was observed in all distributions, followed by slight shoulder-like features, suggesting a size distribution convolution from populations of different characteristics. This aspect is particularly prominent in the samples from the late 18th century, with an evident fraction of smaller-sized grains. As far as we know, only Milke [2] published data on blotting sands' grain size. Similarly, he observed very homogeneous grain size ranges (mostly 0.2–0.4 mm) with a narrow distribution for comparable materials.

Our samples comprise mainly single-mineral grains, although multi-mineral grains were also found. Representative images of blotting sands from two secretaries in the 18th century are shown in Fig. 2 (additional SEM micrographs and discussion on the grains' morphology and surface texture can be found in Fig. SM.2.1.2. and after).

The impact marks frequently detected on the grains' surface with the observed morphological prevalence as rounded and well-rounded shapes are commonly associated with medium-to-long-distance transport in an aqueous environment [2,9].

ImageJ® was used to estimate roundness (R) and circularity (C), morphological/shape parameters (SM.1.2.2.), with most samples (88%) broadly integrating the well-rounded class ($0.72<R<0.82$) and presenting high sphericity ($0.70<C<0.90$) [10,11]. As observed for the D_F distributions (Fig. 1), C and R parameters also suggest multimodality due to the coalescence of different grain distributions composing the samples, with only slight variations observed over time (detailed data in Table SM.1. and Fig. SM.2.1.3.).

In sum, the results on the morphology showed that the blotting sands seem to be texturally mature, meaning well-rounded sediments caused by abrasion during transport, and are consistent with those reported in the literature [2].

3.2. Mineral chemistry

Overall, samples present similar composition regardless of the time period and secretary. The combined use of SEM/EDS and Raman spectroscopy revealed that samples are composed in decreasing frequency of occurrence and amount by Fe-Ti oxides (ilmenite), Fe oxides (e.g. haematite, chromite), silicates (e.g. almandine, quartz, zircon), and Ti oxides (rutile and anatase), and a group comprising other oxides and phosphates. Heavy minerals

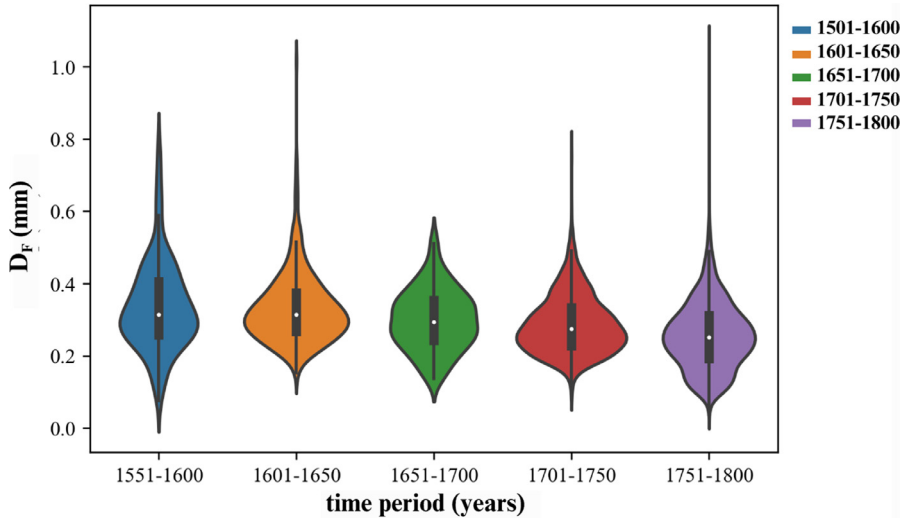

Fig. 1. Violin plots for grain size distribution per time period.

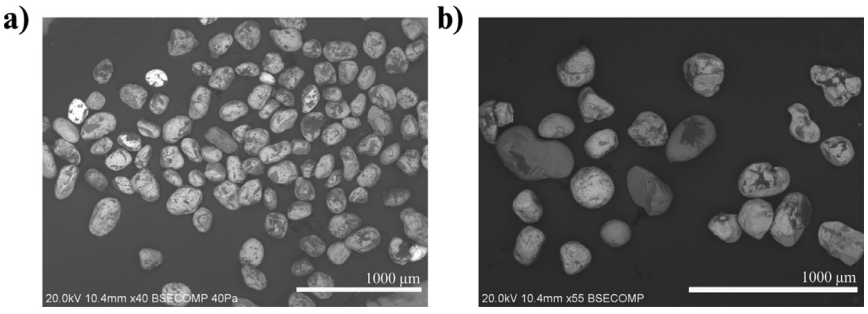

Fig. 2. Blotting sands from: a) José Coelho, 1722; b) António Batista, 1767.

thoroughly dominate the samples' composition in contrast to the examples mentioned in the literature, which included different materials and minerals [1,2,12].

A representative sample from José Coelho (1710) is shown in Fig. 3a. The Fe-Ti oxide grains constitute the most significant fraction. Fe oxide, Ti oxide, and silicates can also be observed. EDS point analysis for this sample with the minerals' frequency of occurrence is presented in the SM (Fig. SM.2.2.1.). The Raman spectra for grains from the four major compositional groups are shown in Fig. 3b (corresponding Raman shifts and assignments in Table SM.2.2.1.).

The Raman spectrum for ilmenite (Fig. 3bi) showed spectral features similar to those reported by Wang [13]: a sharp band at 219 cm^{-1}, resulting from the movement of iron ion in the crystal lattice, a weak band at 361 cm^{-1}, and a strong band at 676 cm^{-1} due to the symmetric stretching vibration of TiO_6 octahedra. The spectrum of haematite is depicted in Fig. 3bii. According to the literature, this mineral has seven Raman-active vibration modes ($2A_{1g} + 5E_g$), of which six are recorded in our spectrum [13–15]. Considering the silicates fraction, minerals from the garnet group (mainly almandine and minor occurrence of pyrope and grossular), zircon and quartz were identified. Garnets are a group of nesosilicate with the general formula $X_3Y_2(SiO_4)_3$, ($X = Fe^{2+}$, Mg^{2+}, Ca^{2+} or Mn^{2+}; $Y = Al^{3+}$, Fe^{3+}, Cr^{3+}, Ti^{3+} or V^{3+}), with almandine ($Fe_3Al_2(SiO_4)_3$), pyrope ($Mg_3Al_2(SiO_4)_3$), grossular ($Ca_3Al_2(SiO_4)_3$), amongst others, as end-members [16]. Almandine was confirmed by the EDS analysis of Fe-Si-Al-rich grains (also by crystal habit in the BSE observations) and Raman spectroscopy (Fig. 3biii) with bands at 176, 229, 358, 380, 512, 569, 872, 921, and 1047 cm^{-1}, in agreement with the almandine spectrum available on RRUFF™ database (RRUFF™ code: X050010). The Fe-Al garnet series are mainly characterised by three regions assigned to A_{1g} modes, 950–920 cm^{-1}, 555–560 cm^{-1} and 342–368 cm^{-1} corresponding to the νSi-O stretching, δSi-O bending and $[SiO_4]^{4-}$ rotational modes, respectively [16,17]. Combinations of Mg-Al-Si and Ca-Al-Si elements were also obtained by EDS point analysis in a few grains, suggesting the substitution of Fe by Mg ions in the almandine-pyrope series and by Ca ions in the $Fe_3Al_2Si_3O_{12}$-$Ca_3Al_2Si_3O_{12}$ (almandine-grossular) garnet solid solutions [18,19]. Still, in the silicates fraction, representative spectra of zircon and quartz are exemplified in Figs. 3biv and 3bv, respectively. For the Ti oxides, anatase and rutile were recorded in Fig. 3bvi. Raman spectroscopy was also crucial in identifying minerals like cassiterite, tourmaline, xenotime, and monazite (data not shown) in the smaller compositional defined group.

Multi-mineral grain analysis showed that ilmenite was the host for submillimetre minerals located on grains' margins and fractures (additional discussion in Fig. SM.2.2.2.).

3.3. Grains morphology and elemental composition in time

The multivariate analysis (HCA) on the dataset established (section SM.1.2.4.) correlated shape features and minerals' composition in time, associated with the Inquisition secretaries in duty. The introduction of multivariate analysis (HCA) allowed the characterisation of complex data with a large number of simultaneous descriptors (section SM.1.2.4.).

The rounded Fe-Ti oxides (ilmenite) and Fe oxides (e.g. haematite) grains dominate in all periods (Fig. SM.2.3.1.). The resulting scores map and respective dendrogram using only the three first principal components of PCA (29.4%, 19.3%, and 13.6% explained variance, respectively) are presented in Fig. 4.

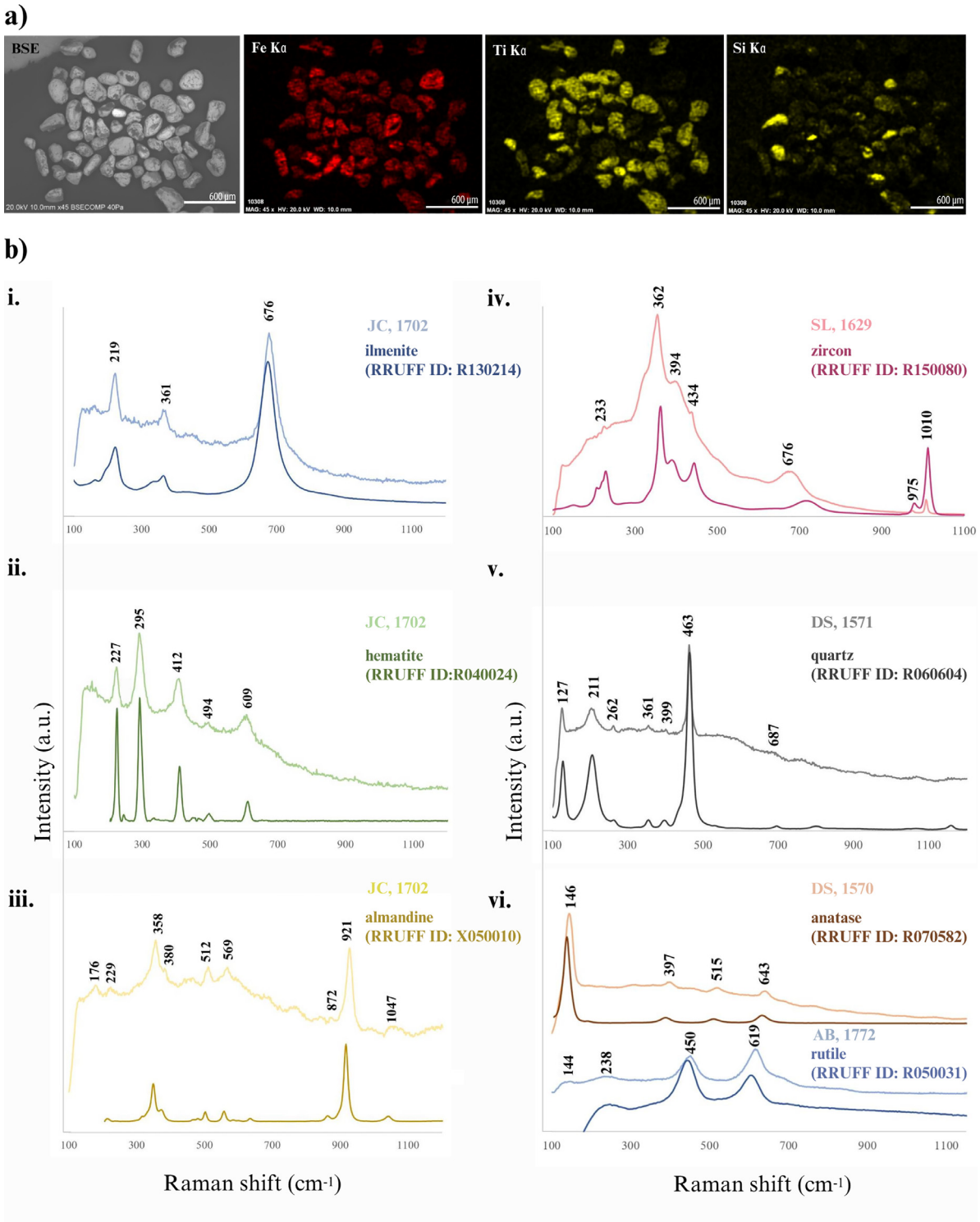

Fig. 3. a) sample from José Coelho (1710) with BSE image and Fe Kα, Ti Kα, and Si Kα EDS elemental maps; b) Raman spectra (lighter colours) of samples from different secretaries and comparative spectra from the database RRUFF^{TM} (darker colours): i, ii, and iii) José Coelho (1702); iv) Simão Lopes (1629); v) Domingos Simões (1571); vi) Domingos Simões (1570) (above) and António Batista (1772) (below).

The dendrogram's structure from the scores of the PCA analysis (Fig. 4a) shows the formation of 5 small clusters, with 4 samples not joining any cluster. In two larger clusters (JEN, brown cluster; JC, red cluster), the correlation was only established amongst samples from a unique secretary, well located in time, with JC samples spanning the first decade of the 18th century and the JEN's, two later decades (1736–1757). Nevertheless, two other samples from each secretary did not fall into the previous clusters and integrated a new cluster representing the time span from 1705 to 1738 (violet cluster).

On the other hand, 3 out of 5 samples from the second half of the 18th century are clustered together (yellow cluster). The separation between this cluster and the samples from the first half of the 18th century is clearly observed along the PC1 scores axis (Fig. 4b). One sample from the late 18th century (MFM (1787)) is at very negative values of PC1 and PC2 scores. Actually, it is

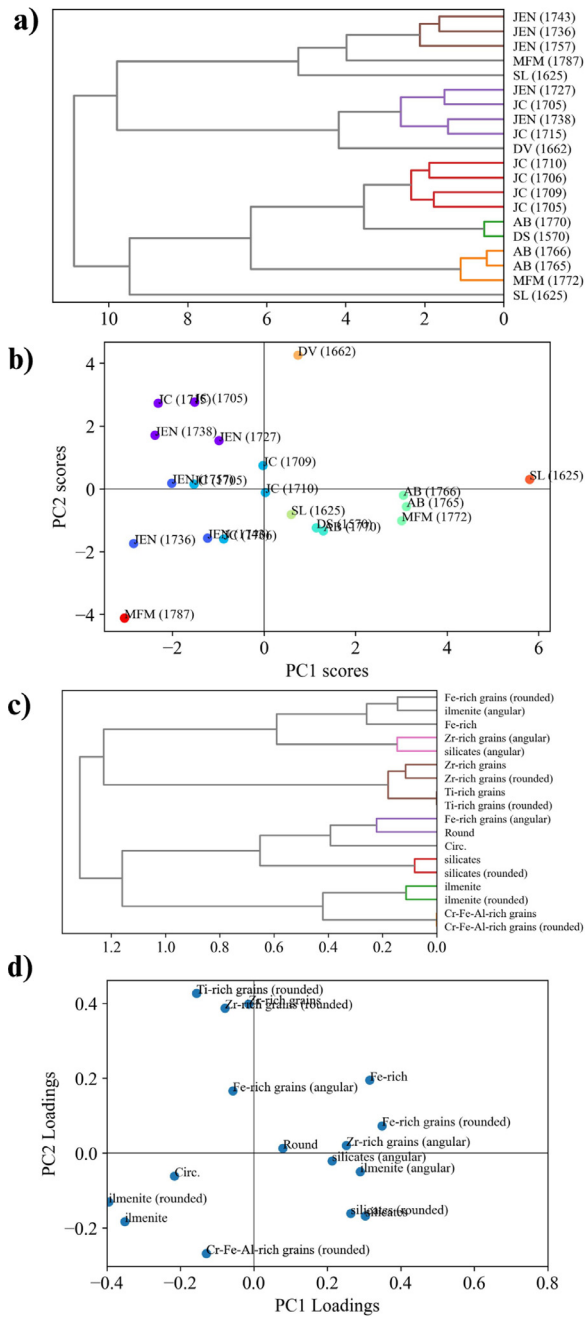

Fig. 4. a) HCA dendrogram for the scores in PC1 and PC2; b) Scores for PC1 and PC2 in the PCA analysis (points coloured highlighting the groups obtained by HCA); c) HCA dendrogram for the loadings in the 3 PCs used in the analysis; d) Loadings for PC1 and PC2 in the PCA analysis.

substantially different from the former 18th-century samples since it includes Cr-Fe-Al-rich grains (6.9% relative frequency of occurence), only observed also in the JEN's (1710) (1.2% relative frequency of occurence).

Samples from the 17th century showed a more dispersed distribution in the scores map (Fig. 4b). SL (1625) (orange point) and SL (1625) (light green point) are very close in PC2, although distant in PC1. This behaviour may be due to zircon (angular) in one sample and silicates (rounded) in the other. Besides, DV (1662) is very close to SL (1625) (green point) in PC1 and distant in PC2 scores due to compositional differences to what angular ilmenite and rounded silicate grains may have contributed. However, it should be noted that this time period would require a larger number of samples for a more consistent characterisation.

The influence of the descriptor variables in the scores' distribution is shown in Fig. 4c. The relative fraction of ilmenite grains negatively correlates to the fraction of Fe-rich grains. Furthermore, a positive correlation is observed between the rounded-shaped Ti-rich grains and the Zr-rich grains (overall and rounded shape).

3.4. A glimpse into the past – provenance & processing technologies

Due to the blotting sands' similarities - the small grain size range, high textural maturity and limited mineralogical variability - proposing a geographical origin can be complex but not impossible. Firstly, morphology and composition suggest that sands derive from deposits with similar provenance. Heavy mineral concentrates occur naturally in placers like beach deposits, stream channels or the mouth of rivers. Grain-to-grain collisions in an aqueous environment during medium-to-long-distance transport or prolonged wave reworking are likely responsible for the dominant morphology. Considering that the General Council activities were centralised in Lisbon, the sedimentary deposits in the Tagus river, enriched in heavy minerals, are a potential source. Salgueiro et al. [23] reported that considerable ilmenite concentrates were found in the Pliocene deposits at Cruz de Pau region, Seixal (Lisbon), in the Tagus hydrographic basin, resulting from alluvial and aeolian processes.

Some Portuguese beaches can also be considered. Cascalho et al. [9] outlined variable quantities of heavy minerals in Praia Grande (Sintra), where ilmenite, haematite, and magnetite, amongst others, can be found. Moreover, Moura & Pinto [24] described the mineralogical composition of sands from S. Torpe's beach (Sines), where important heavy mineral sands naturally occur, mainly as magnetite, ilmenite and haematite. Considering the physical vicinity of Setúbal and Lisbon regions to Lisbon city, the beaches in these regions were good candidates for supplying Inquisition's blotting sands. However, other national or international areas cannot be excluded.

After extraction, mineral processing was required to prepare samples with these levels of heavy mineral concentration. Common sieving was probably the primary mineral processing technology, followed by mechanical panning (i.e., gravity separation by density) [20,21], already used in the 16th century for gold recovery but also employed for heavy minerals [20,22].

4. Final remarks

The combined multi-analytical, statistical and chemometric study highlighted that samples are classified as sands, mainly medium-sized, broadly integrating the well-rounded class and presenting high sphericity. Shape features are characteristic of texturally mature sediments, probably resulting from medium-to-long-distance transport in aqueous environments. The sands are mainly black and systematically dominated by Fe-Ti and Fe oxide minerals.

A significant morphological-compositional correlation was observed between samples from periods close in time, suggesting that similar blotting sands were used. The geological context of Lisbon city and its outskirts, namely the Sintra and Setúbal regions, include heavy mineral deposits, making them attractive sources, considering that the correspondence between the GC and the three Tribunals was carried out in Lisbon. Moreover, the concentration of heavy minerals in the samples suggests assemblage procedures based on processing technologies (sieving and panning).

What criteria supported the choice of these particular black sands? Besides the proximity with probable availability and reduced costs, the aesthetical purpose must have played a crucial role.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.culher.2023.04.003.

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Margarida Nunes: PhD researcher in Chemistry at the University of Évora (Portugal), holding a funded scholarship by FCT (SFRH/BD/147,528/2019). Her current research focuses on methodologies for the identification, characterisation, and preservation of iron gall ink-containing documents.

Gláucia Wanzeller Martins: PhD researcher in Chemistry at the University of Évora (Portugal). She has been developing her PhD in Heritage Science, working on the characterisation of frescoes paintings. She has a Master's degree in Chemistry from the Federal University of Rio de Janeiro (Brazil) and graduated in Chemistry at the same institution. She is a technician in Chemistry and Textile from the Technology center of Chemical and Textile Industry – SENAI-CETIQT (Brazil). She was a research fellow holding a grant funded by FCT (PTDC/ART-HIS/32,327/2017) in the scope of the IRONIC project. She works in vibrational and electronic spectroscopy at the Federal University of Rio de Janeiro.

Jorge Sarraguça: obtained his PhD in Chemistry from the University of Coimbra and was a pos-doc researcher at the Faculty of Pharmacy of the University of Porto and a visiting researcher at the University of Federal do Maranhão (UFMA), Brazil. His research has covered different areas, such as the application of computational techniques to molecular systems with pharmaceutical applications and biological activity, the integration of computational techniques in analytical chemistry, and the use of multivariate analysis and machine learning techniques in art conservation studies. In the last years, he has been an Assistant Researcher at the Department of Chemistry of NOVA University, School of Science and Technology (FCT-UNL)

Fernanda Olival: Associate Professor at the University of Évora and a specialist in Early Modern History (Iberian Military Orders, Inquisition and the materiality of writing). She is also the director of CIDEHUS, a research centre specialising in social changes in the South from a historical perspective.

Patrícia Moita: obtained her PhD in Geology in 2009 and has been a member of the HERCULES research Unit since 2012. Develops her research in geochemistry, mineralogy and petrology with a current focus on analysis techniques for natural and processed geological materials. Some ongoing topics include granitic magmatic processes, in-situ carbonation processes and CO_2 storage and historical materials mortars, ceramics and polished stones. She has been an Assistant Professor since 2009 at the University of Évora (Portugal) and supervisor of several Master and PhD students. She has participated (as the coordinator or team member) in several research and dissemination projects.

Scott G. Mitchell: Tenured Senior Scientist of the CSIC whose scientific research interests lie firmly between fundamental synthetic chemistry and applied materials science. His principal research area concerns synthesising molecular and nanomaterials with tuneable properties that can be applied to areas as diverse as medicine, home appliances, and cultural heritage conservation.

Ana Claro: Integrated researcher and vice-coordinator of the group Art, History and Cultural Heritage at CHAM – Centre for the Humanities. Her primary research focuses on the study of materials applied to Cultural Heritage, mainly in manuscripts, textiles and paintings. In her research, she also associates the nanotechnology to new conservation and restoration treatments, such as the iron gall ink corrosion through the ongoing project IRONIC.

Teresa Ferreira: Chemistry Assistant Professor at the University of Évora, where she has been involved in Analytical Chemistry, Material Science, and Chemistry applied to Cultural Heritage programs. Her main areas of research have included the study of historical textiles, graphical documents such as photographic items and paper documents presenting several types of pathologies, iron gall inks and their devastating effects, illuminated manuscripts, ceramic tiles, and metal artefacts.