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POUR L'ETUDE DES CÉRAMIQUES MÉDIÉVALES  
ET MODERNES EN MÉDITERRANÉE

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MEDITERRANEAN CERAMICS PROCEEDINGS**

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# THEMES

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## **THEME 1 | Ceramics in Wrecks and Underwater Discoveries**

Discoveries in the wrecks are generally left as isolated studies within the general scientific research and publications on ceramics. During building constructions and municipal infrastructural works ceramics are unearthed randomly; but can reveal important information if they can be studied in context of urban consumption. Conversely, underwater discoveries often provide a snapshot of associations of production for import or export as well as pottery used daily by sailors. These ceramic lots provide us with important information about chronologies and trade flows.

## **THEME 2 | Architectural Ceramics**

Architectural ceramic decoration (glazed brick, mosaic-tiles, tiles, bacini) in different regions of the Mediterranean at different periods will be included in the program of the congress.

## **THEME 3 | Kilns, Workshops and Productions**

Archaeological excavations constitute the essential source of information for the study of ceramics throughout the Mediterranean. In the context of this congress, it is important to discern the historical developments and possible relationships that can exist between the various workshops, both in manufacturing techniques, the nature of new products, or the transfer of know-how that can highlight both the relations between the hinterland of the Mediterranean region, such as Iran as well as those between different regions of the Mediterranean itself.

## **THEME 4 | Pottery in Anatolia (from the Byzantine period until the Ottoman period)**

The Medieval period of Anatolia is a time of great demographic and cultural change. Various kingdoms and communities have lived or have succeeded in Anatolia and left their mark. (Byzantine, Seljuk, Armenian, Georgian, the Venetian and Genoese colonies, Syriac populations, Umayyad, Abbasid, various Arab dynasties of Syria and Iraq, the invasion of the Mongols and Timurids, different Pre-Seljuk dynasties, Seljuks and post Seljuks, the Crusaders, the Ayyubids, the Mamluks, the Knights of Rhodes, the Ottomans, etc.)

## **THEME 5 | Import / Export**

Imports and exports of pottery and tiles in medieval and modern times will be included in the program of the congress. It will be interesting to try to see, in the context of imports / exports, the relationship between the quality of ceramics exchanged according to the demand of the social classes and their consumption habits. Similarly, the stylistic influences that can result from these imports and exports between the various regions of the Mediterranean is still a subject of research rewarding to discover.

## **THEME 6 | New Discoveries**

Results of new research and discoveries will highlight workshops or unknown productions, new techniques and/or technology transfer.

**THEME 5 | Import / Export**

**TEMA 5 | İthalat / İhracat**

|  |     |
|--|-----|
| <b>Antonio ALFANO</b> _____  | 11  |
| Le Produzioni di Età Islamica (Prima Metà XI Secolo) Alla Villa del Casale di Piazza Armerina (Enna, Sicilia): Il Contesto della Fornace Presso le Terme Meridionali   |     |
| <b>Susana GÓMEZ, Jacinta BUGALHÃO, Helena CATARINO, Sandra CAVACO, Catarina COELHO, Jaquelina COVANEIRO, Isabel Cristina FERNANDES, Ana Sofia GOMES, Maria José GONÇALVES, Isabel INÁCIO, Marco LIBERATO, Constança dos SANTOS</b> _____ | 21  |
| El Verde y Morado en el Extremo Occidental de Al-Andalus (Siglos X al XII)   |     |
| <b>Irina GUSACH</b> _____  | 31  |
| Art Ceramics from Iznik Found in the Ottoman Fortress Azak   |     |
| <b>Neus SERRA VIVES</b> _____  | 37  |
| La Cerámica del Pozo N° 23 del Convento de los Capuchinos (Palma)  |     |
| <b>Henri AMOURIC, Lucy VALLAURI</b> _____  | 47  |
| Marseille et le Levant Ottoman : Flux (?) et Poussières d'Echanges Entre XVII <sup>e</sup> -et XX <sup>e</sup> S.  |     |
| <b>Julia BELTRÁN DE HEREDIA, Núria MIRÓ I ALAIX</b> _____  | 61  |
| La Cerámica Oriental de Siria y Egipto en Barcelona  |     |
| <b>Jeannette Rose ALBRECHT</b> _____   | 73  |
| La Rupture Nasride, Un Nouvel Apport Oriental pour la Céramique Andalouse ?<br>Problématique des Influences Dans les Décors  |     |
| <b>Svetlana BILIAIEVA</b> _____  | 85  |
| Anatolian Imports of Pottery on the Territory of Ukraine in Medieval and Early Modern Period   |     |
| <b>Javier LARRAZABAL, Paulo DORDIO, Beatriz BÁEZ GARZÓN</b> _____  | 91  |
| Contexto Cultural de los Púcaros Hispánicos Entre los Siglos XVI y XVII:<br>Un Ensayo de Identificación de Producciones y Diacronías a Partir de las Colecciones del Monasterio de Santa Clara-a-Velha de Coimbra                        |     |
| <b>Mariya MANOLOVA-VOYKOVA</b> _____   | 103 |
| Import of Middle Byzantine Pottery to the Western Black Sea Coast: An Overview   |     |
| <b>Sergey BOCHAROV, Andrey MASLOVSKI, Nikita IUDIN</b> _____   | 117 |
| The Impact of Ceramic Imports on Ceramic Manufacturing in the Cities of the North-Eastern Black Sea Region in the Late XIII-XIV Centuries  |     |
| <b>André TEIXEIRA, Joana BENTO TORRES</b> _____  | 127 |
| Abastecimiento Cerámico de la Alcázar Seguer Portuguesa: Las Rutas Comerciales del Mediterráneo y del Atlántico en el Norte de África (Siglos XV-XVI)  |     |
| <b>Sandra CAVACO, Jaquelina COVANEIRO</b> _____  | 139 |
| Evidência das Relações Comerciais do Porto de Tavira (Portugal) Através da Cerâmica  |     |
| <b>Susana GÓMEZ MARTÍNEZ, José Rui SANTOS</b> _____  | 145 |
| Évora y Mértola (Portugal): Convergencias y Divergencias en las Rutas de Comercio de Cerámica del Garb Al-Andalus (Siglos X al XII)  |     |
| <b>Elvana METALLA</b> _____  | 151 |
| Céramiques Médiévales Provenant des Fouilles Récentes de L'amphithéâtre de Durrës (Albanie)  |     |
| <b>Marisa TINELLI</b> _____  | 157 |
| Polychrome Lead-Glazed Ware in Medieval Salento (Apulia): Production, Trade and Use  |     |
| <b>Iryna TESLENKO</b> _____  | 169 |
| Ceramic Import and Export of Crimea at the Final Stage of the Genoese Domination in the Black Sea Region   |     |

|  |            |
|--|------------|
| <b>Rodrigo BANHA DA SILVA, André BARGÃO, Sara FERREIRA, Filipe OLIVEIRA</b>  | <b>175</b> |
| Between Mediterranean and the Ocean: Lisbon's Pottery in a Transitional Period in the Late Middle Ages   |            |
| <b>Vassilis AYANNIDES</b>  | <b>181</b> |
| Céramiques de la Faïencerie de Sarreguemines Dans l'Île de Chio  |            |
| <b>Vladimir Y. KOVAL</b>   | <b>185</b> |
| Spanish Ceramics in Medieval Bolgar  |            |
| <b>J. VROOM, M.W. van IJZENDOORN</b>   | <b>197</b> |
| Splashed Ware: A Little-Known Byzantine Glazed Ware from the Aegean (12 <sup>th</sup> -13 <sup>th</sup> C AD)  |            |
| <b>Anastasia G. YANGAKI</b>  | <b>203</b> |
| Observations on the Pottery (5 <sup>th</sup> -Early 20 <sup>th</sup> C. AD) from the Theopetra Cave, Thessaly (Greece)   |            |
| <b>THEME 6   New Discoveries</b>   |            |
| <b>TEMA 6   Yeni Keşifler</b>  |            |
| <b>Gülğün YILMAZ</b>   | <b>211</b> |
| Ayasuluk Tepesi Kazılarında Bulunan Kazıma ve Baskı Dekorlu Sırsız Seramikler  |            |
| <b>Matteo Gioele RANDAZZO, Antonio ALFANO, Paolo BARRESI</b>   | <b>219</b> |
| Production and Distribution at the Roman Villa del Casale Between the Early and the Later Middle Ages. A Preliminary Re-Evaluation of the 'Gentili's Findings' |            |
| <b>Pamela ARMSTRONG</b>  | <b>231</b> |
| Some Byzantine and Ottoman Pottery Types Circulating in Anatolia and the East Mediterranean  |            |
| <b>Effie ATHANASSOPOULOS, Kim SHELTON</b>  | <b>241</b> |
| The Sanctuary of Zeus at Nemea, Greece: The Medieval Deposits (12 <sup>th</sup> -13 <sup>th</sup> Centuries A.D.)  |            |
| <b>María del Cristo GONZÁLEZ, Valentín BARROSO, Yasmína CÁCERES, Jorge DE JUAN, Consuelo MARRERO, Pedro QUINTANA</b>   | <b>249</b> |
| Formas Azucareras y Otros Repertorios Cerámicos en el Ingenio de Agaete: la Industria del Azúcar en Gran Canaria (Islas Canarias) Entre los Siglos XV al XVII  |            |
| <b>Juan Manuel CAMPOS CARRASCO, Javier BERMEJO MELÉNDEZ, Lucía FERNÁNDEZ SUTILO, Elena LOBO ARTEAGA, Noelia RUIZ PINTO</b>                                     | <b>257</b> |
| El Puerto Colombino de Palos de la Frontera (Huelva, España): Un conjunto alfarero de los ss XV-XVI  |            |
| <b>Galina GROZANOVA</b>  | <b>269</b> |
| Dark Age Pottery Complex from the Village of Kapitan Andreevo, NE Thrace   |            |
| <b>Murat DURUKAN</b>   | <b>277</b> |
| A Recently Discovered Ceramic Dump and the Localization of the Lost Aulai Settlement in Cilicia  |            |
| <b>Z. Tuğçe GÜNGÖR</b>   | <b>285</b> |
| Pisidia Antiokheia'sından Pişmiş Toprak Ritüel Kaplar  |            |
| <b>Evi KATSARA</b>   | <b>297</b> |
| Byzantine Glazed Pottery from Sparta (12 <sup>th</sup> to 13 <sup>th</sup> Centuries AD): Observations in the Light of New Archaeological Finds                |            |
| <b>Florence PARENT, Akila DJELLID, Farid CHALAH</b>  | <b>311</b> |
| Entre Production et Consommation : Place des Martyrs à Alger (Résultats Préliminaires)   |            |
| <b>Platon PÉTRIDIS</b>   | <b>339</b> |
| <i>De la Réserve à la Vitrine</i> : La Mise en Valeur de la Céramique Byzantine en Grèce   |            |
| <b>Elena PEZZINI, Viva SACCO</b>   | <b>347</b> |
| Le Produzioni da Fuoco a Palermo (IX-XI Secolo)  |            |

|  |            |
|--|------------|
| <b>Ágnes KOLLÁTH</b> _____   | <b>357</b> |
| Sgraffito Decorated Ceramics from Ottoman Hungary (16 <sup>th</sup> -17 <sup>th</sup> Centuries)   |            |
| <b>Margherita FERRI, Cecilia MOINE</b> _____   | <b>365</b> |
| Abitudini Conviviali tra Veneto ed Emilia Romagna nel Trecento   |            |
| <b>Jean-Christophe TRÉGLIA, Rosemary Le BOHEC, Riccardo LUFRANI,<br/>Laurent MAGGIORI</b> _____  | <b>387</b> |
| Jérusalem. Céramique Mamelouke du Couvent Saint-Etienne  |            |
| <b>Aránzazu MENDÍVIL-UCEDA</b> _____   | <b>391</b> |
| En la Marca Superior de Alandalús. La Cerámica Andalusí de Saraqusta, Al-Madina-L-Bayda  |            |
| <b>Vincenzo CASTALDO, Giuseppe MOLLO</b> _____   | <b>395</b> |
| La Ceramica a Bande della Campania Interna: Riesame delle Evidenze e Nuovi Dati da Pago del Vallo di Lauro   |            |
| <b>Giacomo CESARETTI</b> _____   | <b>401</b> |
| Pesaro, North-Western Adriatic Coast: Medieval and Renaissance Pottery from the Site of Via Dell'abbondanza  |            |
| <b>Ricardo Costeira da SILVA</b> _____   | <b>405</b> |
| Late 16 <sup>th</sup> Century Glazed Ceramics from Coimbra (Portugal)  |            |
| <b>Alexandra GASPAR, Ana GOMES</b> _____   | <b>411</b> |
| Cerâmicas do Século XIV Na Alcáçova do Castelo de S. Jorge, Lisboa, Portugal   |            |
| <b>Konstantina GEROLYMOU</b> _____   | <b>419</b> |
| Artefacts of the Ottoman Past: Clay Tobacco Pipes from Messinia (Greece)   |            |
| <b>Susana GÓMEZ MARTÍNEZ, Massimo BELTRAME, Fernando BRANCO CORREIA,<br/>José Rui RIBEIRO DOS SANTOS, António CANDEIAS, José MIRÃO</b> _____   | <b>423</b> |
| Comparative Provenance Analysis of Common, Painted and Glazed Islamic Pottery (X-XIII Century) from the Cities of Mértola and Évora (Portugal) Using a Portable Hand Held Xrf Spectrometer |            |
| <b>Rumyana KOLEVA</b> _____  | <b>429</b> |
| Pottery from a Middle Byzantine Period Settlement Near Zlatna Livada, Bulgaria   |            |
| <b>Viva SACCO</b> _____  | <b>433</b> |
| Ceramica con Decorazione a <i>Splash</i> da Palermo (Fine X – Prima Metà XI Secolo)  |            |
| <b>Asil YAMAN</b> _____  | <b>439</b> |
| Late Roman Pottery from Bathhouse VI of Arycanda (Lycia)   |            |
| <b>Miguel BUSTO-ZAPICO, Mario MENÉDEZ-MIRANDA, Marcos BOUZA ARECES,<br/>José Avelino GUTIÉRREZ GONZÁLEZ</b> _____  | <b>449</b> |
| Archaeometric Analysis of Asturian Pottery in XVI-XVII Centuries   |            |

# ARCHAEOLOGICAL ANALYSIS OF ASTURIAN POTTERY IN XVI-XVII CENTURIES

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## **Resumen**

*Ofrecemos en este trabajo los resultados de los primeros análisis arqueométricos realizados sobre producciones cerámicas elaboradas en Asturias (España) entre principios del siglo XVI y mediados del siglo XVII. Se han seleccionado 10 muestras pertenecientes a las tipologías más representativas de la cerámica asturiana de esta época. Las muestras han sido estudiadas utilizando diversas técnicas de análisis: Fluorescencia de Rayos X (XRF), para la cuantificación elemental de los componentes de cada muestra y Glow Discharge Time of Flight Mass Spectrometry (GD-ToFMS) para conocer los elementos minoritarios presentes. Los datos aportados nos permitirán caracterizar y conocer desde un punto de vista físico-químico las producciones que se elaboraban en Asturias a finales de la Edad Media.*

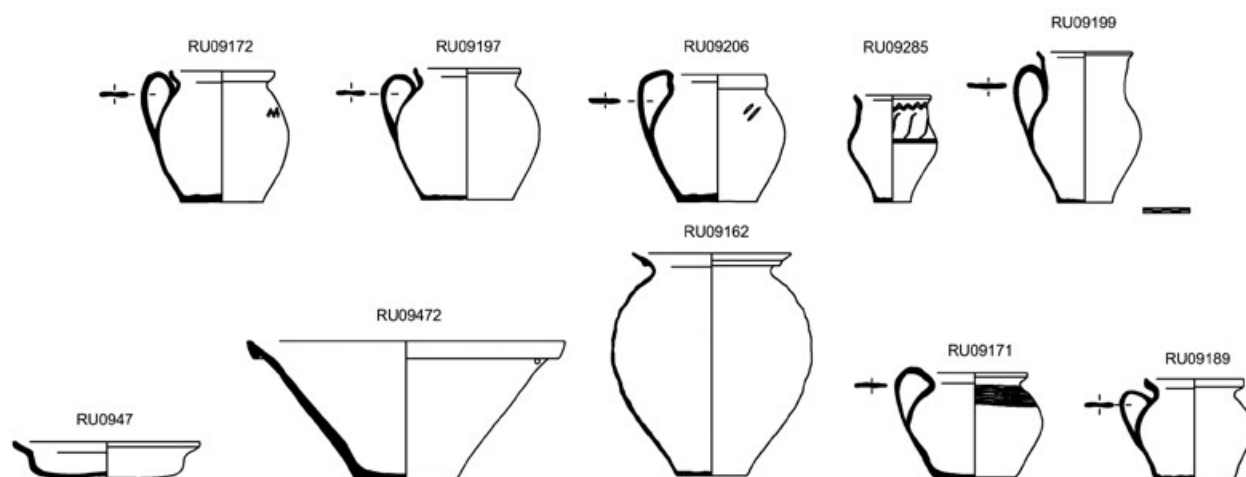
## **1. Introduction**

At the beginning of the modern age, most of the pottery produced and widely used in Asturias (Spain) came from Faro (Oviedo) and Miranda (Avilés). The place of Faro was active from the XI-XII centuries (Fanjul et al., 2013, pp. 2-3) while Miranda, although we do not have reliable data, it is thought that the potters worked as long ago as Late Middle Ages (Feito, 1985, p. 145).

The pottery found at these production sites are the most abundant in any urban archaeological excavation that has taken place in Asturias. Furthermore, these productions sites are the precedent of the *cerámica negra*. Therefore, when the pottery from Faro and Miranda were studied, a key production sites used to understand the Middle and Modern ages in Asturias were investigated. Due to this historical relevance, the archaeometric characterization is used to learn about the production methods in these areas and to define reference for compositional analysis.

## **2. Material and Methods**

In order to characterize the production sites of Faro and Miranda, we have chosen a population of 10 individual pieces of pottery as representative samples (Figure 1). These samples belong to the archaeological excavation of *Casa Carbajal Solís*, dated from the XVI-XVII centuries and they have been samples of different functional, formal and typological studies (Busto Zapico, Gutiérrez González and Estrada García, 2015, p. 466). Based on this previous research, we are able to establish the provenience of our 10 samples and relate them to their production site through macroscopic analysis of the different techno-typological groups (Busto Zapico, 2015, pp. 36-37). Therefore, we will not work with the fragments recovered in the pottery centre and it will be not possible to define Reference Groups, just Paste Compositional Reference Units (Buxeda i Garrigós, Cau Ontiveros, Gurt i Esparraguera and Tuset i Bertran, 1995, 1995).



**Fig. 1** Pottery cases analyzed.

The samples are classified as follows: 6 pieces came from the pottery center of Faro (Busto Zapico, 2015, pp. 39-52). Specifically, the types that are present are as follows: *Puchero II* (RU09172, RU09197), *Puchero III* (RU09206), *Jarrita II* (RU09285), *Jarrita IV* (RU09199) and *Plato II* (RU0947). From Miranda there are only two typologies (Busto, 2015, pp. 52-55): *Vedrio I* (RU09472) and *Cántaro I* (RU09162). Finally, two samples do not reveal clear parallels to any others. They could seem to be related to ancient production sites or a different pottery center not readily known (Busto Zapico, 2015, pp. 55-59). They are *Puchero I* (RU09171) and *Puchero IV* (RU09189). In this study, the objective was to study the most represented Asturian pottery typologies from the XVI-XVII centuries, taking into account the products destined for food processing, as well as their presentation and social consumption.

Using the 10 samples, we have conducted different chemical analyses using X-ray fluorescence (XRF) and Glow-discharge time-of-flight mass spectrometry (GD-ToFMS) at the Scientific-Technical Services at the University of Oviedo. From each individual sample, 1g was taken, pulverized, and homogenized by mill RS4494364 (Retsch Mühle, Germany). The preparation of the was pearls done following the standard procedure (alkaline fusion in  $\text{LiBO}_4$  matrix). The sample analysis by XRF was performed using a spectrometer PHILIPS PW2404 (Philips, Germany) equipped with an automatic loader PW2540, and a 4Kw power Rh excitation source. The GD-ToFMS used is an instrument equipped with a pulsed radio-frequency source and a time-of-flight mass analyzer (Nelis et al., 2015, pp. 389, 763-767). The used proto-

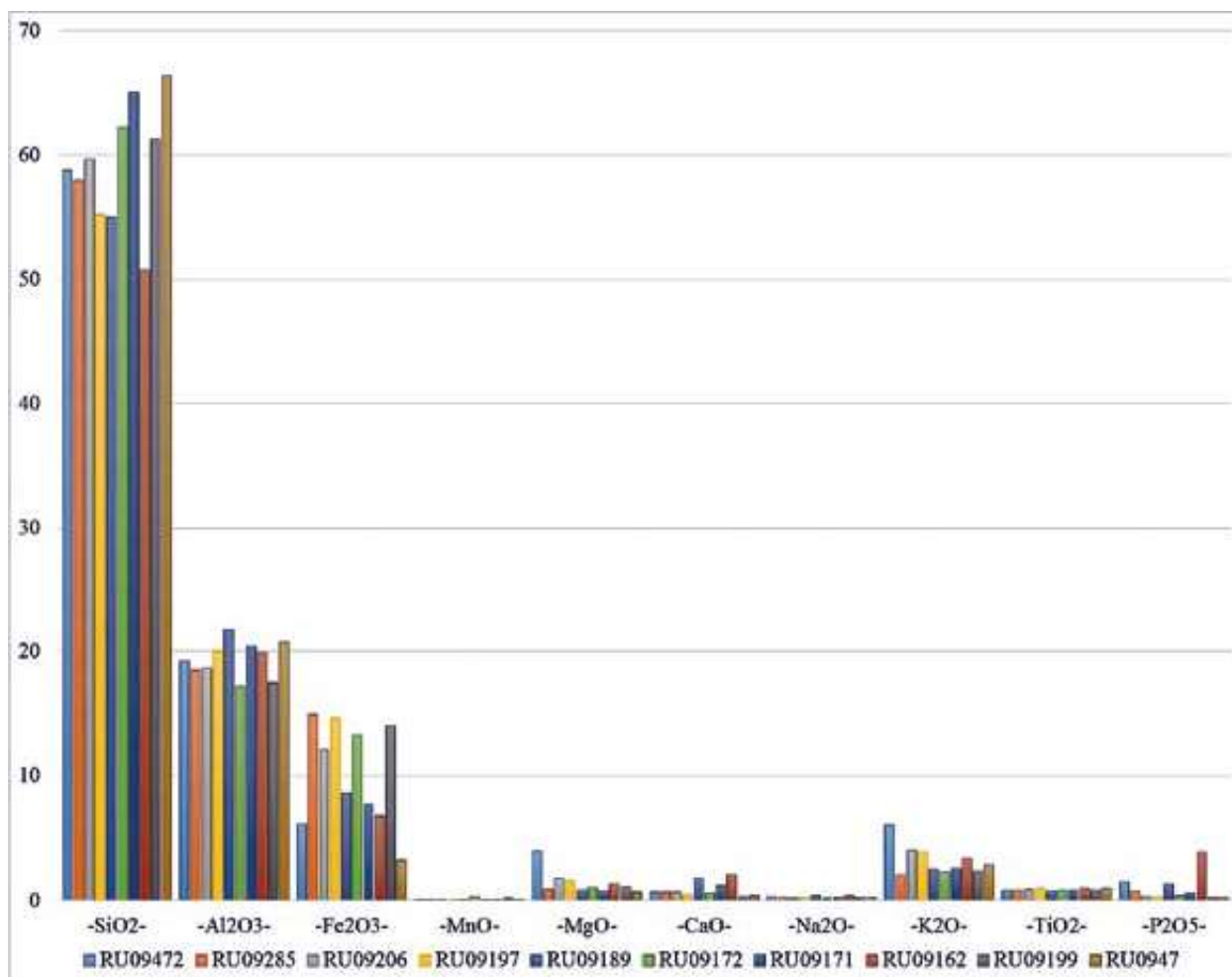
type is composed by a glow discharge (Horiba Jobin Yvon, Longjumeau, France) coupled to a time of flight mass spectrometer with orthogonal geometry (Tofwerk, Switzerland).

### 3. Results

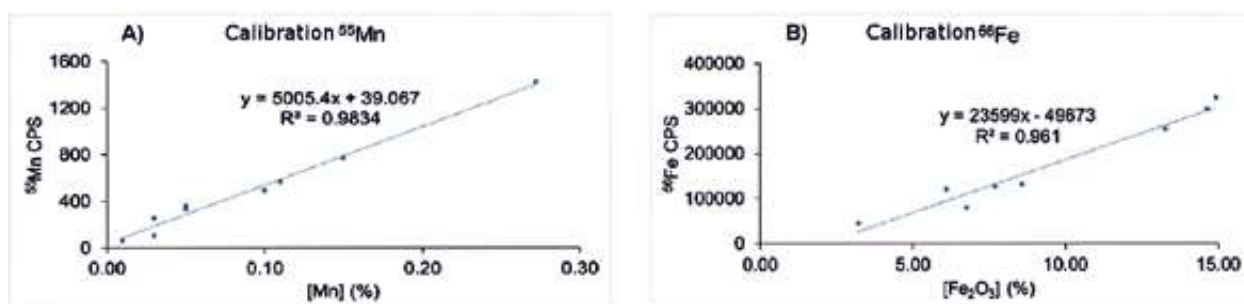
The results obtained by XRF have been used to perform the quantification of the mayor constituents in the samples. Results provided by XRF has been also used to validate the date obtained from GD-ToFMS analysis as it will be discussed later on. Thus, it can be observed how all the analyzed pottery pieces are mainly composed by  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , and to a lesser extent,  $\text{K}_2\text{O}$ . There is no appreciably great variability among the analyzed samples with regard to major components (Figure 2). It is worth noting the low content of calcium oxide, even in the RU0947 sample, as it is discriminant, because the sample is a glazed piece and as such, a high calcareous content is expected.

Furthermore, the results obtained by XRF have been used to corroborate the use of the GD-ToFMS technique for the qualitative analysis of the minor elements. As it can be seen in Figure 3, the signals obtained by the GD-ToFMS technique show a linear correlation when they are represented facing the concentrations obtained by the XRF (reference technique for this type of analysis). This allows us to use the GD-ToFMS signals for minor elements as new factors for the discrimination and characterization of units of compositional reference.

After GD-ToFMS results validation, the different mass spectra (Figure 4) obtained for minor elements shows how the signal, and therefore the



**Fig. 2** XRF quantitative results of the different samples analyzed.



**Fig. 3** A) Calibration curve of the signals obtained through GD-ToFMS for  $^{55}\text{Mn}$ , where “y” is the intensity of the signal per second, and “x” is the concentration obtained by XRF for the Mn. B) Calibration curve of the signals found through GD-ToFMS for  $^{56}\text{Fe}$ , where “y” is the intensity of the signal per second, and “x” is the concentration obtained by XRF for the Fe. In both cases, “R<sup>2</sup>” is the determination coefficient of the analysis by linear regression of the calibration curve.

concentration of the minor elements (e.g. Pt, Au, and Hg), vary significantly between the analyzed samples. It can be observed that all the samples include Au and Pt, but not in the same amount. At the same time, in all cases small signals of Hg are obtained. On the other hand, only five samples contain Bi and

Pb (RU0947, RU09189, RU09206, RU09285, and RU09472). Hence, the qualitative results obtained for minor elements will be used to carry out a statistical analysis including these elements as discriminant factor, looking for a better differentiation between the samples.



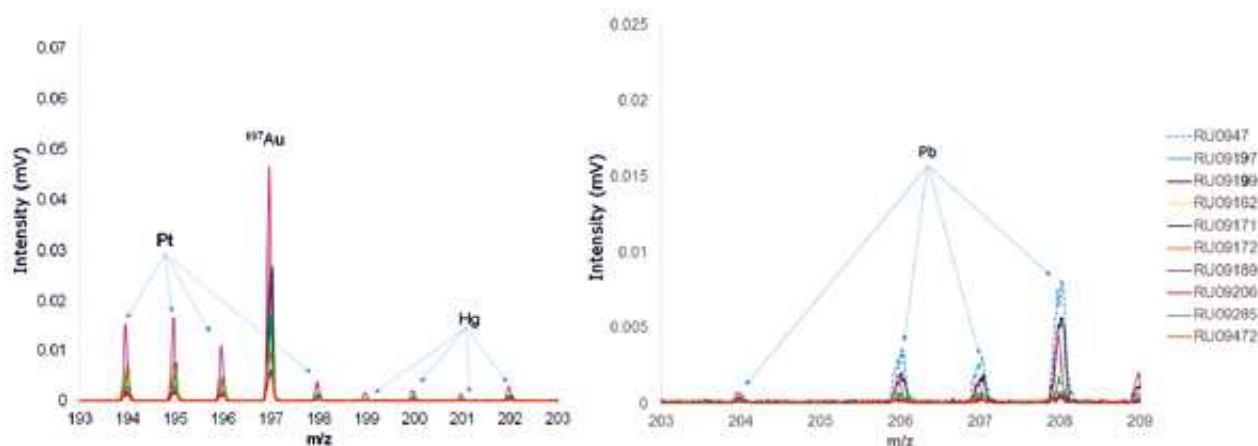


Fig. 4 A) GD-ToFMS Mass spectra for minor elements (Pt, Au, Hg, and Pb) in the analyzed samples.

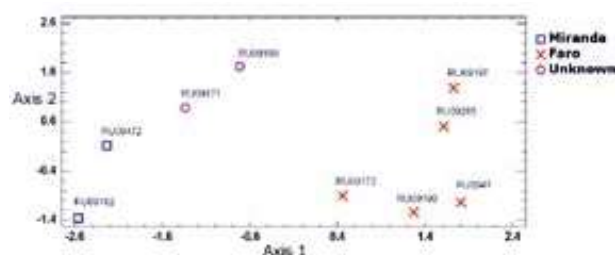


Fig. 5 Result of the discriminant analysis using Fe, Al, Au and Pb as variables.

Joining the results of both analytical techniques (XRF and GD-ToFMS), a discriminant analysis has been performed. The result of the analysis, using the variables (Fe, Al, Au and Pb), due to the better differentiation obtained between the different samples, can be observed in Figure 5. It can be seen that the samples congregate around three different groups or Paste Compositional Reference Units.

#### 4. Discussion

Since the samples analyzed were just 10, the conclusions are a preliminary statement. Even though; this research opens the door to other archaeometric studies in the future. The capabilities of GD-ToFMS technique for the identification of major and minor elements constituting the different samples has been proved. It has been checked the potential capabilities to develop qualitative analysis of the minor elements from the different samples, as the results were validated for the mayor elements based on the concentration results obtained by XRF.

In this context, it is possible to analyze different elements that usually are not studied in this type

of samples due to their low concentration. The minor components could be used as discriminant factor during the classification study upon the different pottery pieces origin. As it has been observed, the discriminant analysis offers better results when these variables are used during the classifying study.

Faro and Miranda production sites are specific Paste Compositional Reference Units. It is worth to notice that it does not seem to exist any chemical difference among the production sites of Faro regarding the common pottery and the glaze one that we have analyzed (RU0947). Therefore, it is probable that the clay was the same, and the differences are due to the production process.

In the same way, a third group seems to be identified. In this group, we found pieces with an older chronology, probably from Late Middle Ages (Busto Zapico, 2015, pp. 49-58). We cannot made a conclusion about this third group of classification but, yo lo quitaría, we can presume different possibilities. In one hand, it could be a different pottery workshop or different productions made around Faro. In the other hand if they are productions from Faro, their chemical differences could indicate that different points of clay caption change between the Middle Ages and the Modern Ages. Other possibility is that the pottery made at the production site of Faro change during that time too. If they are producing in a different manner the composition could be also altered and form this third group.

As previously said the conclusions here presented are preliminary. It is necessary to expand the number of samples, using fragments directly recovered in the pottery centre, and analyze autochthonous clays supposedly used for the production of potteries. Thus, the study could be completed in a reliable manner and

chemical reference groups for the Asturian pottery could be established from the sixteenth and seventeenth centuries.

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