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APRESENTAÇÃO

A Nona Edição do Congresso Ibérico de Arqueometria (CIA IX) decorreu em Lisboa de 26 a 28 de Outubro de 2011 nas instalações da Fundação Calouste Gulbenkian. A proposta e compromisso da organização deste evento foi feita pelo Grupo de Geoquímica Aplicada & Luminescência no Património Cultural (GeoLuC) (IST/ITN), dois anos antes na Assembleia Geral da Sociedad de Arqueometría Aplicada al Patrimonio Cultural (SAPaC), e foi aceite por unanimidade.

Com esta decisão, a SAPaC consolida uma linha de actuação, cujo objectivo é difundir e fomentar a colaboração entre os grupos de investigação arqueométrica que trabalham na Península Ibérica. Este objectivo viu-se reforçado e reflectido na composição dos novos órgãos sociais dirigentes da SAPaC, eleita durante a celebração do IX Congresso em Lisboa, que incorpora deste então investigadores portugueses e espanhóis, sendo presidida pela Doutora M. Isabel Dias (IST/ITN, Portugal).

As Actas que aqui se apresentam são uma prova tangível da via integradora desta IX edição do Congresso, verificando-se existir equilíbrio numérico entre os trabalhos apresentados por grupos de investigação portugueses e espanhóis, evidenciando-se mesmo um incremento de projectos em que participam conjuntamente investigadores dos dois países, mostrando o grande interesse que desperta a Arqueometria, em si mesma de natureza interdisciplinar, e os objectivos comuns partilhados pela comunidade científica ibérica.

Definitivamente, este Congresso constituiu um ponto de encontro dos investigadores da disciplina, tendo contribuído para a troca de experiências e o aprofundar de conhecimentos nas diversas metodologias e técnicas aplicadas à caracterização do nosso património histórico e cultural.

A publicação dos trabalhos do CIA IX nos *Estudos Arqueológicos de Oeiras* (EAO), órgão científico do Centro de Estudos Arqueológicos do Concelho de Oeiras/Câmara Municipal de Oeiras, constituiu uma oportunidade única e vantajosa para ambas as partes, já que esta inédita parceria entre uma entidade vocacionada para a investigação e uma Câmara Municipal permitiu uma sinergia de interesses quanto aos custos da publicação deste número e a sua adequada distribuição nacional e internacional. A escolha de uma revista periódica constituiu sem dúvida, a melhor opção, para a garantia de uma divulgação adequada. E a revista sobre a qual recaiu a escolha, prontamente homologada pelo Senhor Presidente da Câmara Municipal de Oeiras, Dr. Isaltino Morais, responde sem dúvida àquele requisito: além de constituir uma referência no panorama editorial nacional em matéria de publicações arqueológicas, com 18 números publicados desde 1991, mantém permuta com cerca de 200 revistas periódicas especializadas, todas de

Arqueologia e Património Arqueológico, especialmente de Espanha, França, Itália, Alemanha, Polónia, Reino Unido, Mónaco e Marrocos, para além de Portugal, incluindo as publicações mais importantes produzidas naqueles países.

Esperamos, deste modo, com a publicação deste volume, ir ao encontro dos interesses de todos os participantes do CIA IX, de todos os que contribuíram com os seus trabalhos para a excelente qualidade deste volume, dos interesses dos associados da SAPaC, dos munícipes de Oeiras, e da comunidade científica nacional e internacional no domínio da arqueometria e da arqueologia.

Pela Comissão organizadora do CIA IX, Presidência da SAPaC
e comissão editorial deste volume dos Estudos Arqueológicos de Oeiras,

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EARLY IRON AGE POTTERY PRODUCTION IN WESTERN POLAND. AN ARCHAEOMETRIC PERSPECTIVE

Manuel García-Heras[1], Fernando Agua[1], Juan F. Conde[1], Urszula Kobylińska[2] & Zbigniew Kobyliński[2]

Abstract

Early Iron Age settlement of the Polish "Old Country" region is a relatively well documented period from recent archaeological fieldworks. Functionally distinct sites, such as strongholds, ceremonial places and cemeteries have been found, covering the chronology between the sixth and the fifth century BC. The results of an archaeometric study undertaken with the aim of approaching the production technology and providing some insights into the probable local or non-local provenance of the main pottery types found in such sites are here reported. Selected pottery samples and modern raw clays from three almost exactly coeval but functionally distinct sites (the stronghold of Starosiedle, the ceremonial place of Kozów, and the cemetery of Sękowice) were characterized by different techniques, such as thin-section petrography, X-ray diffraction (XRD), scanning electron microscopy (SEM) and X-ray fluorescence (XRF) spectrometry. Resulting data indicated the sharing of a single technological tradition among the three sites with no particular specialization concerning the functionality of each site. This tradition is characterized by the general use of non-calcareous illitic clays, which showed a high presence of kaolinitic clay minerals in the case of Starosiedle pottery and a high content of chlorite clay minerals in the case of pottery from Kozów. All the pottery types were fired under predominantly reducing conditions at relatively low temperatures between 700 and 750 °C.

Keywords: Pottery, Technology, Production patterns

Resumen

El poblamiento de la Primera Edad del Hierro en la región de la "Vieja Polonia" es un periodo relativamente bien documentado a partir de recientes excavaciones arqueológicas. Se han hallado yacimientos funcionalmente distintos como lugares fortificados, lugares de culto y necrópolis, con una cronología entre los siglos VI y V a.C. Este trabajo presenta los resultados de un estudio arqueométrico realizado con el propósito de determinar la tecnología de producción y la posible procedencia local o no local de los principales tipos de cerámica hallados en estos yacimientos. Se ha caracterizado una selección de muestras cerámicas y arcillas actuales procedentes de tres yacimientos casi exactamente coetáneos pero funcionalmente distintos (el lugar fortificado de Starosiedle, el lugar de culto de Kozów y la necrópolis de Sękowice), mediante las siguientes técnicas: observación petrográfica con lámina delgada, difracción de rayos X (DRX), microscopía electrónica de barrido (MEB) y fluorescencia de rayos X (FRX). Los resultados indicaron que los tres yacimientos compartieron una única tradición tecnológica sin mostrar una determinada especialización en relación a su funcionalidad. Esta tradición se caracteriza por el uso de arcillas ilíticas no calcáreas, con una mayor presencia de caolinita en el caso de la cerámica de Starosiedle y un contenido más elevado de clorita en el caso de la cerámica de Kozów. La cerámica analizada se coció en condiciones predominantemente reductoras a temperaturas relativamente bajas entre 700 y 750 °C.

Palabras clave: Cerámica, Tecnología, Patrones de producción

1 – INTRODUCTION

Early Iron Age settlement of the Polish "Stary Kraj" (The Old Country) region is a relatively well documented period from recent archaeological excavations. Functionally distinct sites, such as strongholds, ceremonial

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places and cemeteries have been found, covering the chronology between the sixth and the fifth century BC. Pottery repertoires unearthed in fieldworks show pronounced differences from the typological point of view, despite the similar chronology and small distances between the sites. However, from a technological point of view, the pottery is not so different since black or black/brownish are the predominant colours either in the body or on the surfaces of most of the potsherds. In addition, three main surface treatments or appearances have been observed: coarse or rough, smoothed, and polished/burnished. Some vessels, in which the complete shape could be reconstructed, have shown different treatments on distinct parts of the container or even between the external and internal sides. Accordingly, an archaeometric study was set out with the aim of approaching the production technology of the main pottery types and providing some insights into the probable local or non-local provenance of pottery. The study is part of a programme of pottery characterization which is carrying out currently through bilateral projects between the Institute of History (CSIC, Spain) and the Institute of Archaeology and Ethnology (PAN, Poland) (GARCÍA-HERAS *et al.*, 2008, in press).

The Polish "Old Country" region is located within the current western Polish Lubuskie Province, which is close to the German border (Fig. 1). To carry out the study, a set of pottery samples was selected from three almost exactly coeval but functionally distinct sites: the stronghold of Starosiedle, the ceremonial or cult site of Kozów, and the cemetery of Sękowice. Samples of modern raw clays were also taken for comparison from the surroundings of Starosiedle and Kozów sites.

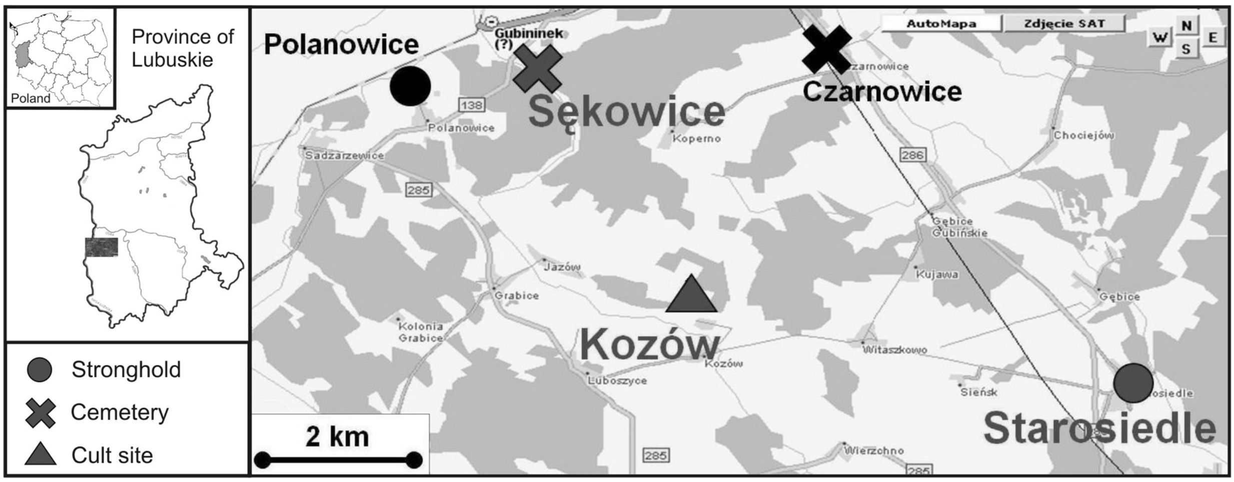

Figure 1 – Some maps showing the location of the Lubuskie Province and the known archaeological sites in the region. The three sites of the study are highlighted.

The oldest one is the cult site of Kozów, which was excavated between 2001 and 2004 by a team led by the IAE-PAN. This important ceremonial site, connected with the cult of "sacred spring", has been identified as the place of discovery in 1882 of the famous Greco-Scythian golden hoard, known in literature as the "Vettersfelde hoard" (NEBELSICK & KOBYLIŃSKI, in press), and should be dated at *c.* 500 BC, the final phase of the Hallstatt D period. The stronghold of Starosiedle, somewhat later than the site at Kozów, was first excavated between 1920 and 1923 by Schuchhardt (1926) and re-excavated between 2001 and 2004 by a German-Polish team led by the IAE-PAN (KOBYLIŃSKA, KOBYLIŃSKI & NEBELSICK, 2008). Finally, the latest of the three sites, the cemetery of Sękowice, was excavated by the regional museum of Zielona Góra and its graves was mostly dated to the second half of the fifth century BC and, consequently to the Hallstatt D/La Tène A period

(LEWCZUK, 2004). The site of Kozów is culturally affiliated to the so-called Białowice (Billendorf) Group, whereas the sites of Starosiedle and Sękowice are affiliated to the Górzyca (Göritz) Group. Chronological differences between them are, however, less than 100 years. Roughly speaking, the pottery ensembles unearthed in these three sites are dominated by large storage vessels, sometimes with ornament of cordons, notched rims or incised decorations; bowls of various sizes, generally plain with smoothed or polished/burnished surfaces or decorated with incisions; and handle dippers with incised decorations most of the times. Large storage vessels are more numerous in the stronghold, whereas the amount of bowls is higher in both the cult site and the cemetery. A specific form, known previously mostly from cemeteries, but unexpectedly found in great amounts in one of the wells of Kozów, were small black polished bowls with *omphalos* (navel) in the bottom, evidently used for special purposes, contrary to other forms of bowls and to large cooking and storage vessels from the settlement at Starosiedle. Their provenance has been of special importance for understanding the socio-cultural context of the site. Number of other vessels in the material from Kozów was minimal. In the case of Sękowice, the pottery ensemble was dominated by decorated amphorae used as cremation urns but, because of their museum value, they could not be sampled for the present study.

The archaeometric study here undertaken has been useful to reconstruct technology and production patterns of pottery of Early Iron Age communities settled in Western Poland since, up to now, little was known about pottery-making of such chronology in this region.

2 – MATERIALS AND ANALYTICAL TECHNIQUES

2.1 – *Pottery and raw clay samples*

A total of 39 potsherds (15 from Starosiedle, 14 from Kozów, and 10 from Sękowice) plus 3 modern raw clay samples (2 from Starosiedle and 1 from Kozów) were selected to undertake the study. The sampling of potsherds encompassed the three main typological shapes and the three surface appearances mentioned above. The two clay sediments from Starosiedle were gathered approximately 1.5 km NW from the site, within an area valued by the modern villagers as a source of good clay, whereas the clay sediment from Kozów was taken from one of the clay levels of the archaeological excavation. Clay samples were modelled into small briquettes in the laboratory and, after dried at 80 °C for 120 min, they were fired in an electric tubular kiln up to 850 °C, with a constant flow of nidron (N_2/H_2 90:10 vol %) to simulate the predominant non-oxidising conditions observed in most of the potsherds. After an annealing period of 120 min, the temperature was raised at a heating rate of 10 °C/min. Maximum temperature was only maintained for 15 min. Then, the kiln was left to cool down slowly in a closed condition.

2.2 – *Characterization and statistical techniques*

Both pottery and raw clay samples were analyzed through the following characterization techniques: thin-section petrographic examination, X-ray diffraction (XRD), scanning electron microscopy (SEM) and X-ray fluorescence (XRF) spectrometry.

Thin-sections were cut perpendicularly to the rim of potsherds. Petrographic examinations were accomplished with a Kyowa Bio-Pol 2 polarizing light microscope. Micrographs from thin-sections were recorded with a Leica DFC480 camera. XRD analyses were carried out with a PANalytical X'Pert-MPD unit using Kα of copper radiation (1.54056 Å), under set conditions of 45 kV and 40 mA. Diffractograms were obtained between 2θ = 5-60 °C. SEM observations were undertaken on fresh fractures coated with gold-palladium with a Hitachi S-3400-N microscope (CCHS-CSIC Madrid), using acceleration voltages of 15 kV. Chemical analysis by XRF was carried out by a PANalytical Axios wavelength dispersed X-ray spectrometer equipped with a tube of rhodium. Powder bulk samples for XRD and XRF analyses were prepared by grinding the body of the potsherds, with their most external surfaces removed to avoid contaminations, in an agate mortar. For XRF analyses, pressed boric acid pellets, using a mixture of n-butylmethacrylate and acetone (10:90 wt %) as binding medium, were prepared. Finally, chemical data obtained by XRF were submitted to multivariate statistical techniques such as cluster and principal components analysis (BAXTER, 1994), using the Systat v. 10.2 package. Oxide concentrations were transformed into log base 10 values to compensate for large magnitudes of difference between major and minor elements (BISHOP & NEFF, 1989). Among the 17 oxides determined by XRF, statistical analyses were undertaken using only 10 oxides due to missing values in some of the samples. XRF was conducted on 31 potsherds and the 3 clays.

3 – RESULTS AND DISCUSSION

3.1 – *Thin-section petrographic study*

The petrographic examination of samples from the stronghold of Starosiedle revealed the presence of a single and poorly sorted fabric for all the potsherds, independently of their typology or surface treatment. It was composed of abundant large sub-rounded to sub-angular inclusions disseminated throughout a fine-grained clay matrix. In general, clay matrices showed a high birefringence without evidences of vitrification. They exhibited abundant, long, and elongated macro-pores, sometimes parallel oriented to the surfaces. The clay matrices presented a wide variability of colours from black or grey to black/brownish or grey/brownish transitions. The large inclusions are composed mainly of polycrystalline fragments of granitic rocks, whose mineralogy was quartz, two kinds of feldspars (microcline and plagioclase) and, occasionally, amphibole and mica (biotite) (Fig. 2A). These minerals occurred both as individual crystals (derived from the granitic rock) and as crystals within the granitic rock fragment. The sub-angular morphology and the large size (up to 3 mm in some cases) of granitic inclusions strongly suggest that they could have been intentionally crushed and added to the clay matrix. In addition, some predominantly monocrystalline quartz inclusions not higher than 1.5 mm exhibited very rounded edges (Fig. 2B), which could indicate a glacial origin for clay deposits used as clay base for making this pottery, since such deposits usually show that mechanical rounding.

With the exception of one sample from a large storage vessel from Kozów, in which some organic matter remains were observed, all the potsherds from both Kozów and Sękowice sites presented the same fabric observed in the Starosiedle samples, no matter their typology or surface treatment. However, some small mineralogical differences were detected. The potsherds from Kozów barely showed amphibole crystals, either within the granitic fragments (Fig. 2C) or as individual crystals in the matrix. Moreover, they also presented some chlorite crystals (Fig. 2D), which were only detected in samples from this cult site. As far as Sękowice potsherds are concerned, they also showed large and probably intentionally added inclusions of granitic rocks (Fig. 2E), as well as smaller and very rounded monocrystalline quartz inclusions (Fig. 2F). Amphibole crystals were similar to those observed in the Starosiedle samples.

Finally, the petrographic examination of modern raw clay briquettes fired in the laboratory showed abundant and detrital quartz inclusions, some of them with mechanical rounded edges as those observed in archaeological potsherds (Fig. 2G-H). In addition, small inclusions of microcline and plagioclase feldspar

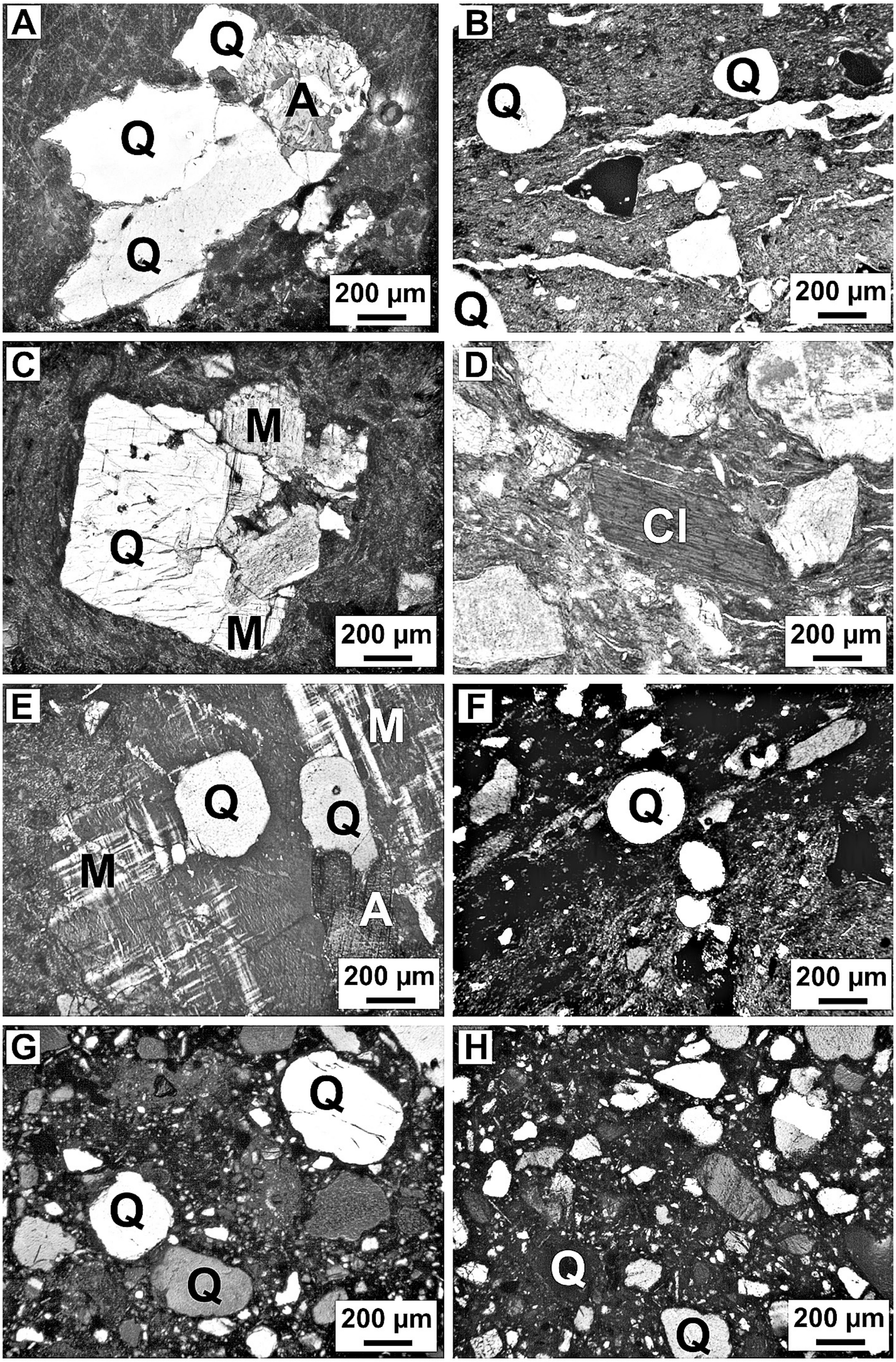

Figure 2 – Thin-section micrographs. Crossed nicols except B and D which were taken with one nicol. A-B) Potsherds from Starosiedle. C-D) Potsherds from Kozów. E-F) Potsherds from Sękowice. G) Raw clay 2 from Starosiedle fired at 850 °C. H) Raw clay from Kozów fired at 650 °C. A Amphibole. Cl Chlorite. M Microcline (K-feldspar). Q Quartz.

crystals were also identified in the raw clays. These observations indicated that there were clays with similar mineralogical features in the region. Consequently, a probable local provenance could be suggested for most of the pottery here analyzed.

3.2 – *Mineralogical phases and equivalent firing temperatures*

The XRD results were in agreement with petrographic observations and, likewise, they did not show any difference on the basis of typology or surface treatment of pottery. The main phases detected were those associated with granitic inclusions, namely quartz and feldspars (microcline and plagioclase). The secondary phases determined were, on the contrary, those related to the clay matrix. Thus, illite was detected in all the samples. However, some important differences were found. Several potsherds from Starosiedle showed the clay mineral kaolinite (Fig. 3A), whereas some potsherds from Kozów presented chlorite (Fig. 3B). One of the modern raw clays gathered from the vicinity of Starosidle also showed kaolinite (Fig. 3C), whereas the raw clay taken from Kozów also presented chlorite (Fig. 3D). These data therefore suggest two outstanding points. The first one is that pottery from Starosiedle can be characterized by the presence of kaolinite (not detected in samples of any other site) and, the second one, that pottery from Kozów can be characterized by the presence of chlorite (neither detected in samples of other sites). Additionally, kaolinite was only detected in the raw clay from Starosidle, whereas chlorite was only present in that from Kozów, which both suggest a probable local origin from pottery of each site, since both pottery ensembles can be clearly separated from mineralogical data. The XRD determinations confirmed, for the rest, the presence of amphibole in potsherds from Starosiedle (Fig. 3E) and Sękowice (Fig. 3F), which was already identified by thin-section petrography.

The presence of illite, kaolinite and chlorite phases in most of the potsherds studied suggests a relatively low range of firing temperatures for this kind of pottery. Thermal decomposition of kaolinite begins at 550°C, that of chlorite at 650 °C, and that of illite at 850 °C approximately (MAGGETTI, 1982; RICE 1987). The raw clay briquettes from Starosiedle and Kozów fired in the laboratory up to 850 °C did not present kaolinite and chlorite reflections and only some residual reflections corresponding to the illite phase, which means that the pottery analyzed was not fired at a temperature as high as 850 °C. Overall the equivalent firing temperature can be therefore established between 700 and 750 °C as much, since the majority of the samples still presented well defined illite reflections. The presence of weak reflections of kaolinite and chlorite in some samples from Starosiedle (Fig 3A) and Kozów (Fig. 3B) respectively, may indicate that, in some cases, the firing could barely reached 600 or 650 °C. SEM data also agreed with a general low firing temperature since, on the whole, layered microstructures observed can be associated to a non-vitrification state.

3.3 – *Chemical and statistical analyses*

A cluster analysis, not shown here for space reasons, using the centroid clustering method on a matrix of Euclidean distances firstly classified the samples into two groups and three outliers. The same structure showed a principal components analysis undertaken later from a variance-covariance matrix. The compositional distinctiveness of these two groups is illustrated in Fig. 4, which displays sample scores and group membership, with respect to the first two components. Both components summarized 78.2% of the total variation in the data (62.2% the first one and 16.0% the second). The concentration of iron, potassium, manganese and magnesium oxides on the one hand, and titanium and silicon oxides on the other, were key values that revealed the largest separation among the two groups. The plot also shows three unassigned or outlier samples. Means

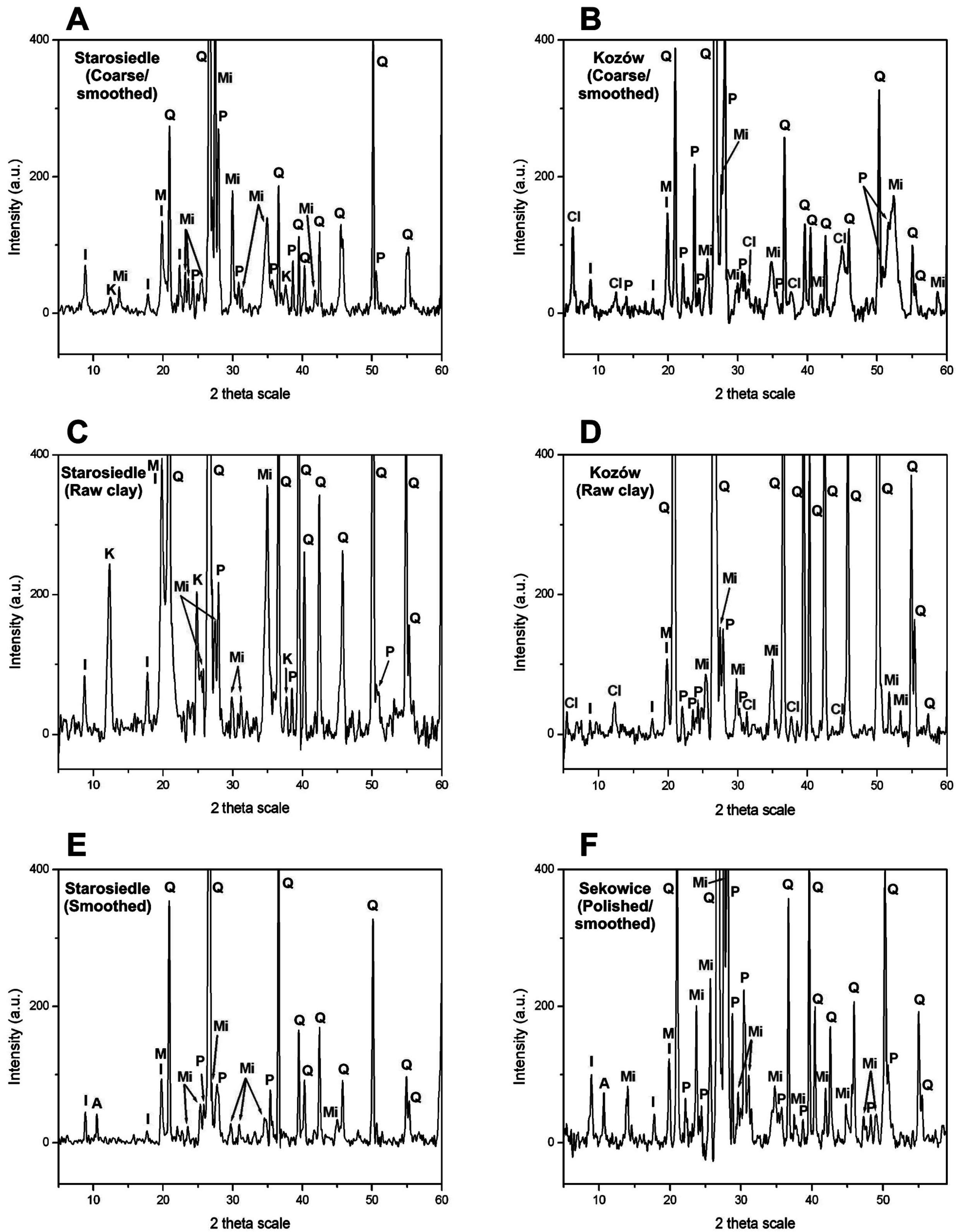

Figure 3 – X-ray diffractograms from potsherds and raw clays. A Amphibole. Cl Chlorite. I Illite. K Kaolinite. M Mica. Mi Microcline (K-feldspar). P Plagioclase (Na feldspar). Q Quartz.

and standard deviations of both groups, as well as composition of the three outliers are presented in Table 1. All the pottery analyzed can be considered as non-calcareous since the concentration of calcium oxide was lower than 5 wt %.

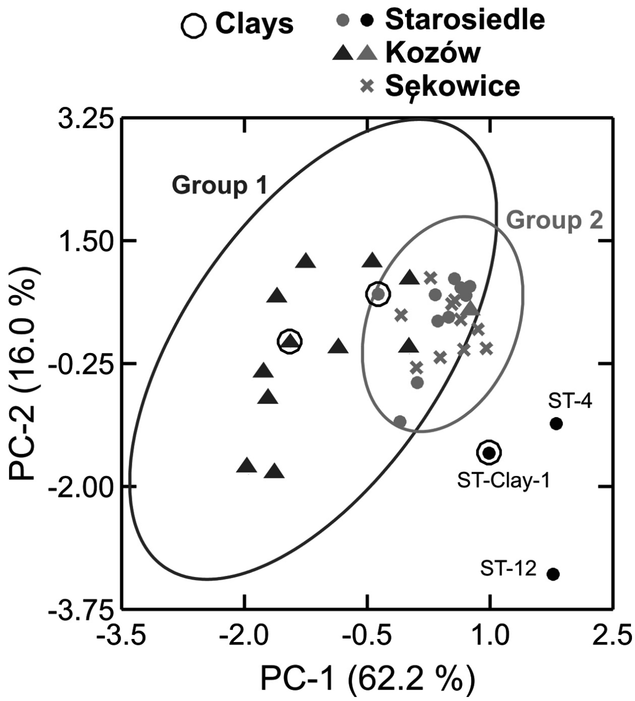

Figure 4 – Plot of the first two components derived from principal components analysis of XRF data (n = 34). The ellipses represent the 95% confidence level for membership in each group. ST Samples from Starosiedle.

Table 1 – Means and standard deviations (SD) of groups derived from statistical analyses of XRF data and composition of outliers (wt %).

Oxide	Group 1 (n = 11)		Group 2 (n = 20)		Outliers (n = 3)		
	Mean	SD	Mean	SD	ST-Clay-1	ST-4	ST-12
Na_2O	0.48	0.22	0.63	0.17	0.29	0.58	0.54
MgO	1.04	0.55	1.39	0.40	0.97	1.16	0.38
Al_2O_3	20.66	3.48	20.49	1.20	14.86	19.59	30.33
SiO_2	70.37	5.18	65.06	1.91	76.05	63.65	56.00
K_2O	2.87	0.56	3.47	0.60	2.28	2.17	1.51
CaO	0.63	0.30	0.80	0.25	0.47	1.44	0.88
TiO_2	1.08	0.25	0.93	0.09	0.69	0.93	0.85
MnO	0.02	0.01	0.06	0.02	0.16	0.24	0.34
Fe_2O_3	2.80	1.11	7.10	0.73	4.18	10.16	9.11
BaO	0.05	0.01	0.07	0.02	0.05	0.08	0.06

The Group 1 is composed of 11 samples and is characterized for having lower contents of sodium, magnesium, potassium, calcium, manganese, and iron oxides than the Group 2. Except one sample belonging to a large storage vessel assigned to the Group 2, all the samples from Kozów, including the raw clay gathered in the site and the potsherd with organic matter remains, were associated to the Group 1. This fact is in agreement with both petrographic and XRD data (presence of chlorite and absence of amphibole in comparison with samples from the other two sites) and implies that pottery from Kozów was probably locally made with a base clay very similar to that taken in this work. On the contrary, the Group 2 is composed of 20 samples and is characterized for having lower concentrations of mainly silicon and titanium oxides than the Group 1. In this Group 2 they are assigned 8 potsherds from Starosiedle, one of the two raw clays gathered in its vicinity, one sample from Kozów and the 10 potsherds analyzed from Sękowice. The pottery from Starosiedle and Sękowice was grouped together, which means that it is not possible to chemically distinguish both productions. This is partially in agreement with both petrographic and XRD data since, even though both productions showed amphibole crystals in thin-section, only potsherds from Starosiedle presented kaolinite by XRD. In any case, the pottery from Starosiedle and Sękowice can be chemically distinguished from the pottery of Kozów. Additionally, the presence within this group of one of the raw clay samples from Starosiedle may indicate that pottery of this settlement was probably elaborated also with local clays.

Finally, one of the two clays together with two potsherds from Starosiedle (a bowl and a large storage vessel) were classified as outliers, which suggest that some of the pottery made in Starosiedle could have been manufactured from other local clays still not sampled or, perhaps, that some minor pottery from Starosiedle were brought from other sites rather than Kozów or Sękowice.

4 – CONCLUSIONS

The archaeometric study of a representative set of potsherds from three almost coeval but functionally distinct sites (the stronghold of Starosiedle, the ceremonial place of Kozów, and the cemetery of Sękowice), has been useful to reconstruct the technology and the production patterns of pottery of Early Iron Age communities settled in the “Stary Kraj” (The Old Country) region of Western Poland. Resulting data indicated the sharing of a single technological tradition among the three sites, despite important formal, stylistic and functional differences of pottery vessels, with no particular specialization concerning the functionality of each site, since no particular differences on the basis of typology or surface treatment of pottery were found. This tradition is characterized by the general use of local non-calcareous illitic clays in which an important amount of crushed polycrystalline fragments of granitic rocks were intentionally added as temper. Deliberate addition of non-plastic granitic fragments is suggested by their large size and sub-angular morphology. Such addition would produce a sort of ceramic composite which would improve mechanical properties of ceramic vessels, thereby increasing their toughness. Most of the pottery analyzed was fired under predominantly reducing conditions at relatively low temperatures between 700 and 750 °C, even though in some cases the presence of kaolinite and chlorite reflections might indicate a lower temperature of 600 or 650 °C as much.

Some compositional differences were determined in the ceramic materials analyzed according to the archaeological site in which they were unearthed. The pottery from Starosiedle showed illitic-kaolinitic clays and the presence of amphibole; the pottery from Kozów exhibited illitic-chloritic clays and the absence of amphibole; whereas the pottery from Sękowice present only illitic clays, without kaolinite or chlorite clay minerals, and a higher presence of amphibole crystals. Overall resulting data indicated a local origin for the pottery of each site and, consequently, the emergence of a pottery production pattern characterized by the presence of differentiated productions in each settlement site but within a single technological tradition. The pottery was produced and consumed in each own settlement and was scarcely distributed to other sites, even to cult sites as that of Kozów.

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