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The muffle: an innovation around silver refining at Castel-Minier in the 14th century

Muffle: Inovace kolem rafinace stříbra v Castel-Minier, 14. stol.

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Abstract: Castel-Minier, situated in the French Pyrenees, was a centre for silver production between the 13th and 15th centuries. Its location on the outskirts of one of the largest European silver production centres, it was not traditionally associated as a place of innovation. However, the discovery of ceramics dating back to the second half of the 14th century that resemble muffles questions this assumption. Through an archaeological and experimental study, we gain a better understanding of the cupellation structure, while an archaeometric survey of the material from experiments and archaeological excavations sheds light on the use of muffles and the cupellation structure.

Abstrakt: Castel-Minier, situovaný ve francouzských Pyrenejích, býval centrem produkce stříbra mezi 13. a 15. stoletím. Díky jeho umístění na okraji jednoho z největších evropských výrobních center stříbra se o něm tradičně neuvažovalo jako o místu inovací. Avšak objev keramiky podobající se mufliím pocházející z druhé poloviny 14. století tento předpoklad zpochybňuje. Archeologickou a experimentální studií získáváme lepší znalosti o kupe-lačním procesu, zatímco archeometrický průzkum materiálu z experimentů a archeologické vykopávky vrhají světlo na použití mufli a kupelaci.

Key word: muffle, innovation, refining silver, cupellation, Castel-Minier, archeological experimentation, archaeometry

INTRODUCTION

The Castel-Minier site, situated in the French Pyrenees (Fig. 1) was a centre for silver production. By the early 14th century, it had become the foremost mine in the kingdom of France (Bailly-Maître et Benoit 1998). However, despite its prominence, it was relatively small in size and located on the outskirts of a European mining and metallurgical centre where significant advancements in mining and metallurgy occurred both before (Menant 1987) and during the late Middle Ages (Suhling 1976). Bringing to light innovative techniques was not the aim of the archaeological excavation carried out in the mine and workshops of Castel-Minier. However, the discovery of a blower dated to the early 16th century identical to that described in Book IX of the *De Re Metallica* (Agricola 1556) cast doubt on this aspect. This discovery shows that the workshop had access to the best technology of that time despite its remote location (Téreygeol and Heckes 2012). It appears that some elements of high technicity can also be observed in the earlier phases of exploitation. This could be explained by the proximity between Castel-Minier and the mining activities of the Aragonese Crown. Instances of pierced ceramics, an open fireplace and some litharge cakes reveal a previously unseen structure of refining for the Middle Ages. Archaeological experimentations followed by modelling and analysing in the field and the laboratory provide the key to understanding this apparatus with the muffle as the centrepiece.



Fig. 1. Localisation of Castel-Minier and Melle.
Obr. 1. Lokalizace Castel-Minier a Melle.

THE MUFFLE

The muffle is a ceramic component that served a dual purpose: it protected the work area within the furnace from the surrounding fuel and provided a controlled atmosphere. In the existing literature, a muffle is primarily associated with assay furnaces with a floor. However, only two authors, Ercker in his *Probierrbuchlein* in 1556 and the anonymous author of the *Schwazer Bergbuch* (1556) connected the muffle with an open fireplace (Gauthier 2012). The first description of

a muffle dates back to the early 16th century in the *Probi-erbuchlein* from 1524. In this book, the ceramic piece is referred to as a muffle and the manufacturing process of the muffle is described, involving the modelling of the ceramic as a tile (Anonymous 1524, 95–96). In 1540, Biringuccio (Biringuccio 1540) did not provide a specific name for the object, calling it *archetto*, but the accompanying illustration identifies it as a muffle. This might provide a clue to the origin of the object because twenty years earlier in the *Probi-erbuchlein* it had an actual name (muffle in the illustration). Later, in 1556, Agricola also described the object and how it was made (Agricola 1556, 227–228). He used the Latin word *tegula* by analogy with the form of the object. Agricola did not use a new technical word but gave a precise description of the manufacturing process. In his treatise on ore and assay, Lazarus Ercker confirmed a similar method of production as described in *Probi-erbuchlein* (Ercker 1574, 360). He referred to the ceramic piece by its current name: *die Muffel*¹. These four authors all concurred on how the muffle is used in a furnace and on the analogy to a half-round ridge tile.

Archaeologically, the muffle remains little known and there are no examples of it before the second half of the 16th century. The oldest muffle was discovered by Gerd Goldenberger during a survey at Muggenbrunn in Schwarzwald (Goldenberger et al. 1996). It consisted of two sherds from the back of a single muffle. The ceramic paste of these fragments was rough and thick (+/- 1cm, as seen in the picture on p118). An impressive collection of muffles dating back to the late 16th century was unearthed during the excavation of Oberstockstall in Austria. This ensemble comprises seventeen muffles with eleven of them archaeologically complete. According to the drawings, the pieces were crafted by turning². The author of the study referred to the work of an alchemist, although the materials used evoked various ore and metal processes.

It appears that the muffle existed in Central Europe from the early 16th century, as indicated by a combination of historical and archaeological evidence. In their edition of the *Probi-erbuchlein*, Sico and Smith question the invention of

the muffle furnace: “the muffle furnace used for cupelling [...] obviously represents the apex of development over many years”. (p180), but they do not provide further details. It is possible that the muffle previously existed as suggested by the work of Labuda (1997).

At Castel-Minier, we uncovered a batch of nine sherds (Fig. 2). After reassembly, we identified them as muffle fragments. The archaeological context dated the pieces to the second half of the 14th century. The sherds are pierced and the holes were made from the outside of the ceramics before heating. The largest specimen was used to determine the overall shape, which is similar to a half-round ridge tile. The thickness of the paste is between 5 and 7 mm. The base of the arch measures approximately 17 cm and the opening under the muffle bow is about 7 cm. Each fragment has a flat edge used to support the object. We observed two types of muffle ends – one flat and one rounded. The underside of the fragment shows traces of turning but no evidence of use as a glaze or glassy spots. These various indicators provided information about the production method, which we followed in the experiment. A piece of clay was placed on a potter’s wheel used for turning a hollow bottomless cylinder. The cylinder was then removed with a wire that left a flat mark on the edge of the cylinder, while the turning produced a rounded mark on the top. Before heating, the cylinder was cut lengthwise to obtain two muffles with two types of edges: flat and rounded.

In our opinion, the morphology is not sufficient to definitively characterise these pieces. Therefore, we decided to observe the diffusion of the lead in a cross-section of some of the sherds. We assumed that it would be possible to observe the evolution of the lead concentration³. Near the liquid metal inside the muffle, there must be a higher concentration of lead than on the outside of the muffle since it is only in contact with the lead vapour expelled through the holes of the muffle⁴. We conducted a chemical analysis using X-ray fluorescence⁵, which yielded a clear result (Fig. 3). From the inside to the outside of the ceramic, the

1 The term ‘Die Muffel’ is used by Ercker in the first edition of the Beschreibung... of 1574: https://books.google.cz/books?id=r0NdAAAACAAJ&printsec=frontcover&hl=cs&source=gbs_ge_summary_r&cad=0#v=onepage&q&f=false.

2 In Oberstocksthal, we highlight the use of a half pot also used as a muffle (von Osten 1998, 150–151) as mentioned by Biringuccio (1540), Ercker (1574) and Barba (1640).

3 Prior to this study, a test was conducted using a Portable X-Ray fluorescence (pXRF) on a sherd, and we observed a doubling of the lead concentration from the outside to the inside of the muffle (0.2%massPb on the outside and 0.5%massPb on the inside). Additionally, we performed a pXRF analysis on kitchen pottery found on the same layer, and the lead concentration remained unchanged on both sides of the sherd (0.04%massPb).

4 We have previously demonstrated the relevance of such an approach in characterising crucible fragments for lead metallurgy (TÉREYGEOL F. and CASTRO C.(2008): La metalurgia prehispanica de la plata en Potosi – In: Cruz P. et Vacher J.-A. (eds), *Mineria et Metalurgia en los Andes del sud desde tiempos prehispanicos al siglo XVII* - 11-28) or for brass metallurgy (TÉREYGEOL F. (dir) (2014) : *Etudes analytiques des vestiges métallurgiques de la fouille du site le Clos Paul à Charleville-Mézières (08) et Expérimentation sur les techniques de production du laiton au III^e s. ap JC*, tapuscrit – In : Cellule archéologique départementale de l’Aisne - 51).

5 After sample preparation, we conducted a chemical analysis using X-Ray Fluorescence (XRF). The beam was delivered through a RU-200HB rotating anode from Rigaku, operating at the molybdenum K α energy (17.5 keV). Since we worked in grazing incidence, the beam impinged the surface of the sample over 600 x 30 μm^2 (width x height). XRF spectra were recorded using a Vitus-Silicon Drift Detector (SDD) from Ketek GmbH with a 50 μm step in height and an acquisition time of two minutes. Integrated peak intensities were recorded for every element of interest.

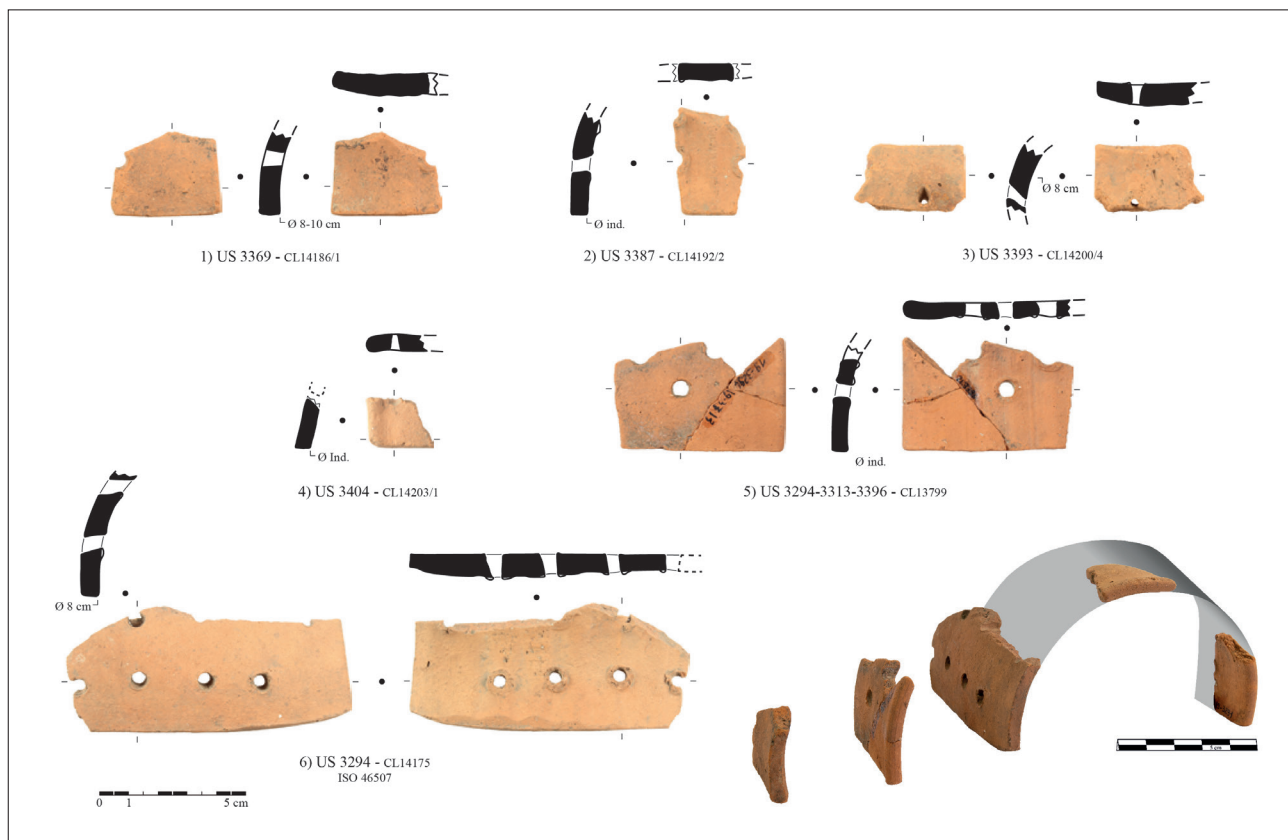


Fig. 2. Muffle ceramics found in Castel-Minier in 2020.

Obr. 2. Muflová keramika nalezená v Castel-Minier v roce 2020.

amount of lead rapidly decreased on the first 3mm. It was minimal in the middle of the paste and this initially excluded the presence of lead in the clay. The amount of lead then increased on the outside edge of the sherd. This concentration of lead was lower than that on the inside edge, thereby indicating the source of lead pollution.

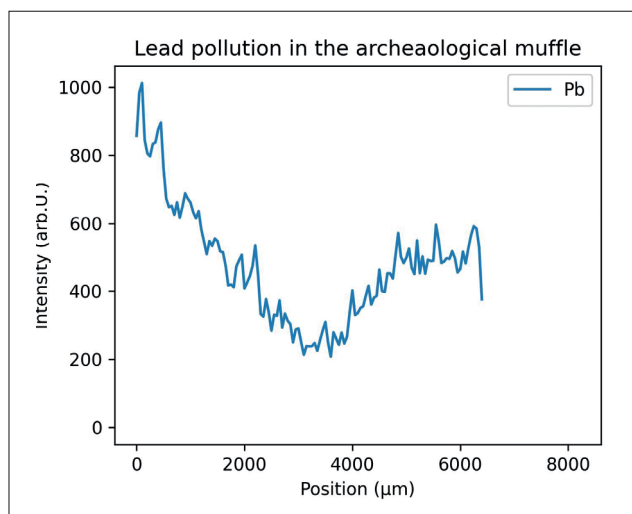


Fig. 3. Intensity of the lead pollution in the thickness of the archaeological muffle (CL14200).

Obr. 3. Relativní obsah olova na řezu muflí (CL 14200).

Based on the morphology of the fragments and the chemical signature of lead in the ceramics, we can therefore identify these sherds.

In addition, we have amassed a significant collection of litharge cakes from the 14th and 15th centuries at Castel-Minier (10.6 kg for 263 fragments). For this study, we only considered the elements found in the immediate vicinity, i.e. 68 fragments with a mass of 4.3 kg. Ten of these objects were sufficiently preserved to identify the shape of a bowl (Fig. 4). Eight of them are round, while two are oblong. Each litharge cake can fit underneath the previously defined muffle. The diameters of those objects are between 6.5 cm and 20 cm. Six of them have diameters between 11 cm and 16 cm.

We also discovered an open fireplace at ground level within the castle, located in the immediate vicinity of the litharge cakes and muffle fragments. This open fireplace is located in a building measuring approximately 50 m² and is positioned against an overhanging rock. Among the corpus of eight open fireplaces found in Castel-Minier between the late 12th and 15th centuries, this particular open fireplace is unusual. First, it is much larger, covering 10,000 cm², whereas the others are between 3,500 and 6,000 cm². In addition, there are no reddish marks on it. There is no drainage pit to prevent water accumulation, which was present in six out of seven cases. Finally, the soil around the structure had



Fig. 4. Litharge cake found in Castel-Minier.
Obr. 4. Lithargit naležený v Castel-Minier.

a high concentration of lead easily identifiable by its grey to white colouration. The surface containing lead extends over an area of 7 m² with a thickness approaching 1cm. A survey with a portable X-Ray fluorescence (180 analysis on 8 m²) was used to map the lead level (Fig. 5)⁶. The results reveal that the lead concentration is high around the open fireplace (up to 20 % mass of lead), and is lower within it (from 0 to 7 % mass of lead). This supports our assumption that this is a cupellation structure.

The hypothesis of an open fireplace with a muffle for silver refining is supported by three pieces of evidence: the spatialisation, the shapes and the synchronicity of the shards, the litharge cakes and the open fireplace. This hypothesis is consistent with the writings of Biringuccio and Ercker as well as the relevant iconography. In *De la Pirotechnia*, Biringuccio specified this after presenting the assay furnace “*Anchora sono alcuni che usano di fare tal saggio senza fornelli, & senza hauer tanti ordini sol con uno pignato bucarato*” (Biringuccio, 1540,

fol.108v), but concluded that it was not the best practice. Ercker, on the other hand, described how a muffle is used in an open fireplace in Lower Saxony (Ercker 1574). Additionally, one of the illustrations in *Schwazer Berbuch* shows a refiner working on a fireplace rather than a furnace (Anonymous 1556).

To test the robustness of this hypothesis, we conducted an experiment complemented by an analytical approach.

THE EXPERIMENT

Methods

The experiment was conducted in the archaeological experimental platform of Melle in France (Fig. 1).

A professional ceramist, François Peyrat, produced several muffles using the previously discussed turning technique. An open fireplace was also constructed according to the dimensions of the archaeological one. A layer of industrial bone ashes was added to the open fireplace as a floor (Fig. 6).



Fig. 6. Experimental set-up for the cupellation.
Obr. 6. Archeologický experiment – kupelace.

⁶ The portable X-Ray Fluorescence (pXRF) instrument (Niton XI3t 900 GOLDD+) is designed for metal, alloy, ore and soil analysis. It operates with a maximum tension of 50kV and an intensity of 0.1 mA, providing a total power output of 2W. The X-ray generating tube is air-cooled, with a silver anode and a 0.127 mm thick beryllium exit window. The spot had a diameter of 3mm.

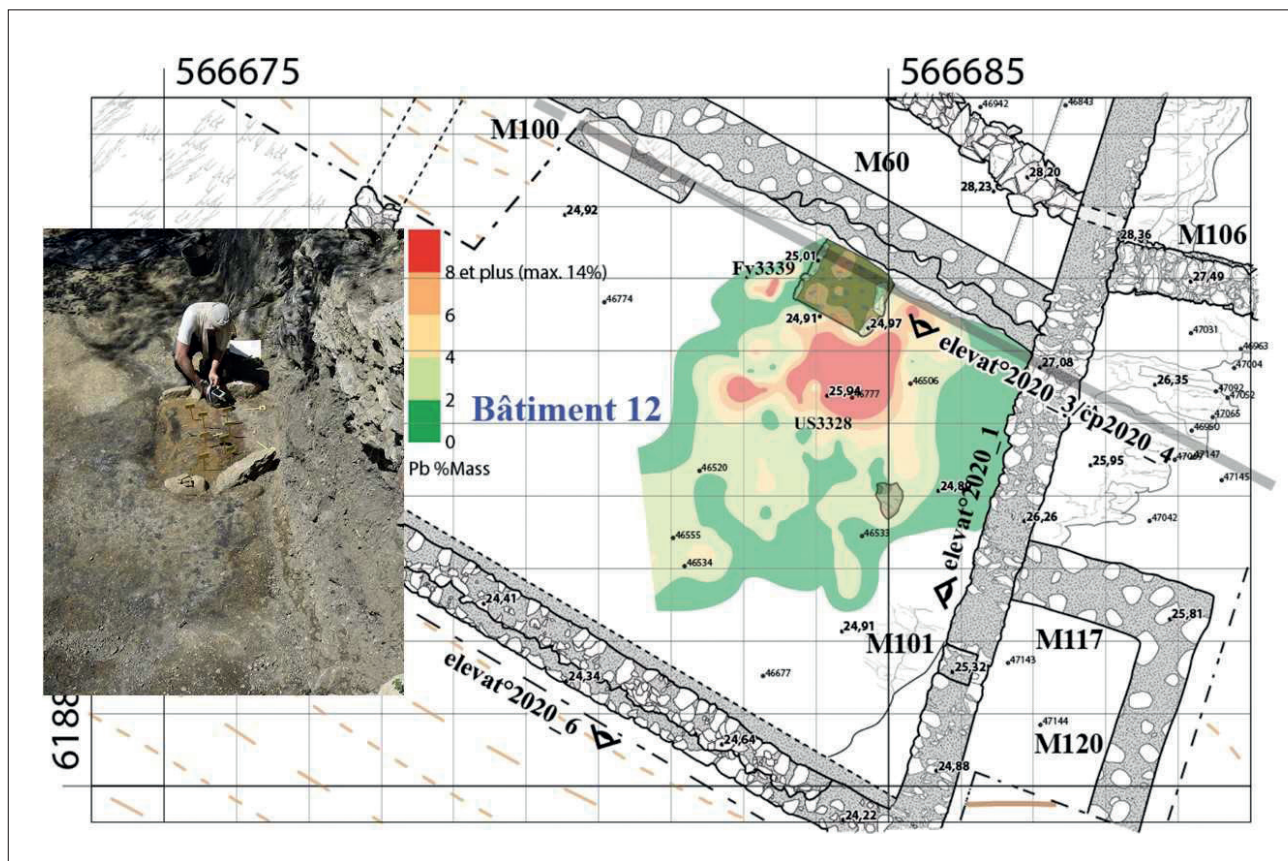


Fig. 5. Map of the lead pollution around the open fireplace in Castel-Minier (pXRF survey).
 Obr. 5. Mapa znečištění olovem kolem otevřeného topeniště v Castel-Minier (průzkum pXRF).

We formed a hollow in the ashes and the silver-lead alloy was placed inside it. The muffle was then placed over the alloy and covered with charcoal. The rear opening of the muffle was consistently covered with charcoal, while the front opening was open or closed with charcoal as per the requirements of the process. We provided forced ventilation through bellows with a 6L air volume. The operator adjusted the blowing power to maintain the temperature and ensure an oxidant atmosphere. The airflow was driven by a tuyere directly towards the front of the muffle.

Cupellation started after heating the structure. When finished, we removed the silver bead (the final product) and the litharge cake. The ashes were cleaned from the open fireplace and a new cupellation could be initiated with the muffle. We made ten attempts as described in Table 1.

The same analyses performed on the archaeological remains were also conducted on the experimental materials, i.e. a pXRF analysis of the soil and an XRF analysis of the muffle.

Tab. 1. List of the different attempts and results (analysed by pXRF).
 Tab. 1. Přehled experimentů a jejich výsledků (analyzováno pomocí pXRF).

| Trial | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10Es |
|--|------|------|------|------|-----|------|-----|------|-----|------|
| Estimation of the silver mass percentage by weight | ~0.1 | ~0.1 | ~0.0 | 10 | 10 | 8,4 | 8,4 | 5,7 | Und | 8,7 |
| Mass of the bullion (g) | 923 | 763 | 745 | 129 | 85 | 92 | 89 | 133 | 122 | 109 |
| Forced ventillation | No | Yes | No | Yes | Yes | Yes | Yes | Yes | Yes | Yes |
| Silver bead – mass (g) | No | No | No | 14 | 10 | 8 | 8 | 6 | 11 | 13 |
| Silver bead - amount of silver (mass percentage) | No | No | No | 95,7 | 89 | 90,5 | Und | 98,6 | 85 | 81 |

RESULTS AND DISCUSSION

The cupellation experiment with a muffle on an open fire-place proved successful. Of the ten attempts, seven were successful based on obtaining a silver bead or enriched bullion⁷. The metal went from an average mass of 108 g and a mass percentage of silver of 8.5 % to a mass of 10 g and a percentage of silver of 90 % at the end of the cupellation. The *silberblick*, which indicates the end of the cupellation, was observed four times. The variation in the outcomes appears to be related to the lack of experience of the experimenters and the absence of forced ventilation in the initial attempts.

The loss of lead amounted to 89 % of the initial alloy's mass of lead. Most of it was absorbed by the ashes. The drop in the percentage of lead in the alloy proves that cupellation with a muffle and an open fireplace is viable. However, this was not a demonstration of the use of such a process at Castel-Minier. Nonetheless, this experiment provided material such as the muffle and litharge cakes. In addition, the experimental litharge cakes (Fig. 7) visually resemble the archaeological cakes, even if the experimental litharge appears green due to the presence of copper traces in the experimental alloy.



Fig. 7. Experimental litharge cake.
Obr. 7. Lithargit z experimentů.

The muffle was used ten times before it broke, and cracks appeared during the initial heating, alongside the turning marks. This indicates the duration a muffle can be used in experimentation and consequently for how long the archaeological muffle was used. This allows us to compare the amount of lead contained in the archaeological shard and the experimental ceramic.

The lead pollution in the muffle follows a three-part curve (Fig. 3) running from the inside to the outside of the muffle. Near the inside edge of the ceramics, the lead pollution is high, up to 750 arb.U. Inside the material, the pollution is

low, four times less than the pollution on the inside edge. The final step is visible on the outside edge of the ceramic. These observations hold true for the archaeological artefact and the experimental one (Fig. 8), although the maximum concentration in the experimental one is slightly lower.

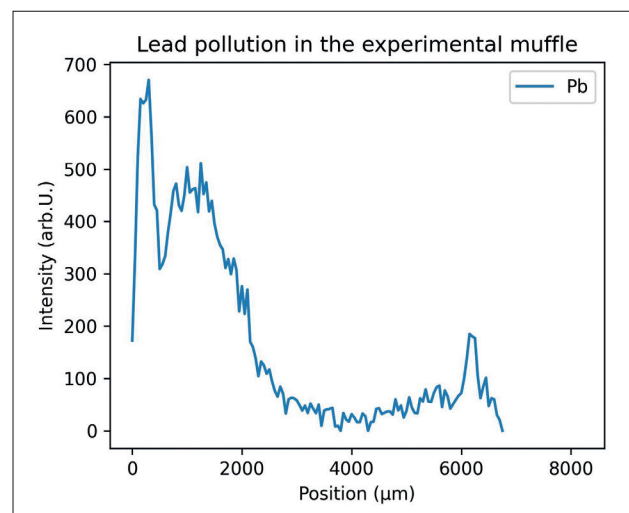


Fig. 8. Intensity of the lead pollution in the thickness of the experimental muffle.

Obr. 8. Intenzita znečištění olovem ve stěně experimentální mufle.

The similarity of the curve between the archaeological and the experimental muffle proves that the presence of lead is not due to the manufacturing process or the burial of the sherd.

Hence, the lead pollution is the result of a metallurgic process involving the muffle. The different parts of the curves can be explained by the use of the muffle during the cupellation process. Near the liquid metal inside the muffle, there is a high concentration of lead in the air. The lead then spreads into the ceramics causing pollution. Therefore, the duration of the use of the muffle is not sufficient for the lead to spread throughout the thickness of the ceramic. This is why there is a high concentration of lead near the inside edge of the ceramic but almost none in the middle of the material.

The airflow over the muffle explains the pollution on the outside of the muffle. There are openings at both ends of the muffle, as well as holes throughout the artefact. This allows the air to circulate with a high concentration of lead leading to pollution on the outside of the muffle. However, this pollution is less intense because the concentration in the airflow is lower on the outside than on the inside of the muffle. A 3D representation of the lead pollution on the inside, outside and middle of the experimental muffle shows what appears to be the airflow (Fig. 9).

⁷ There is difference between the supposed mass percentage of silver in the bullion and the actual measurement. This can be explained by the use of an uncontrolled composition of the bullion. The experiment is focused on lead contamination rather than the efficiency of the process already demonstrated in previous papers.

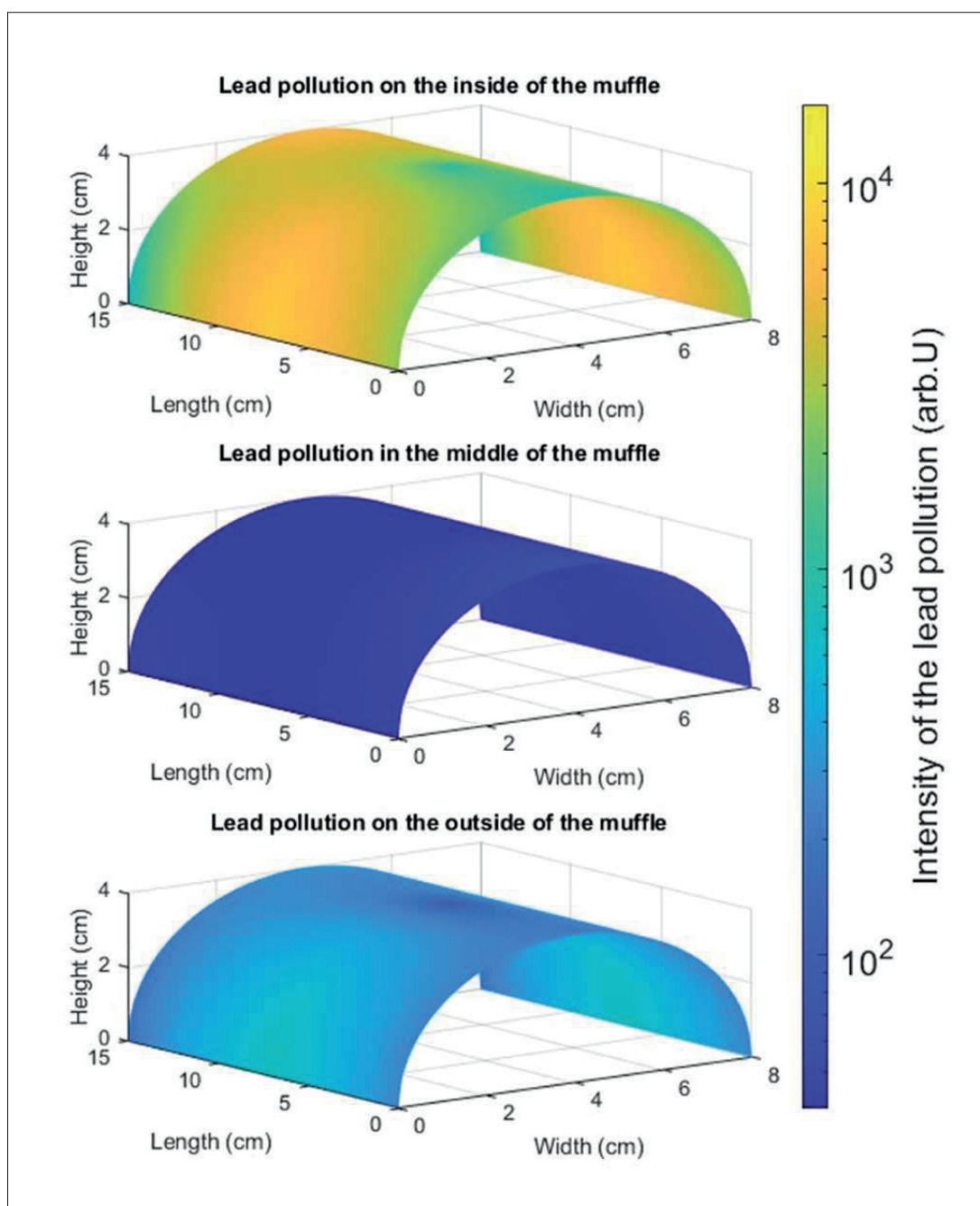


Fig. 9. 3D visualisation of the lead pollution in the experimental muffle.
Obr. 9. 3D vizualizace relativního obsahu olova v experimentální mufli.

SOIL

By analysing the composition of the soil at the archaeological site, we created a map of the intensity of the lead pollution (Fig. 5). The lead pollution is more intense outside the open fireplace than inside. This observation, and the absence of reddish marks, showed that the soil in the open fireplace was protected by something, which supports the hypothesis that the open fireplace was used for cupellation. The ashes placed on the open fireplace serve as thermal protection, with a temperature coefficient of less than

1W/m.K (Zbogar et al. 2005), so they can be regarded as insulating material.

In addition, during the cupellation process, only the layer of ashes became polluted with lead, which was subsequently removed between each cupellation. Therefore, the soil remained relatively unpolluted. The cleaning of the ashes in the open fireplace could explain the high lead pollution outside because the highly polluted ashes were thrown on the ground near the open fireplace, as we also did during the experiment.

Measuring the pollution experimentally proved challenging due to the naturally high concentration of lead in the ground at the experimental platform in Melle, which is also a lead-silver mining site. Therefore, the limited number of experiments conducted was insufficient to create measurable pollution. However, establishing a neutral control point showed the difference in the pollution between the inside and the outside of the open fireplace. The inside of the open fireplace was less polluted than the outside. The experiment further supports the conclusion that the archaeological open fireplace was used as a setting for silver cupellation.

CONCLUSION

The concept of a muffle has been well-documented in literature since the early 16th century. However, the hypotheses formulated following the excavation of a level dating from the second half of the 14th century at Castel-Minier cast doubt on the date of appearance of the muffle. To investigate these assumptions, we devised a protocol for conducting experiments. The implementation of the protocol was successful as we succeed in refining silver thus demonstrating the viability of the process. However, the multi-disciplinary approach (geochemistry, chemical analysis of archaeological materials and experimental archaeology) allows us to draw conclusions about the historicity of this production system. Ceramics identified as muffles are indeed muffles, and the same applies to the cupellation structure. It is evident that cupellation on an open fireplace at ground level was used in Castel-Minier, representing the earliest known evidence of this innovative metallurgical ceramic practice. This also applies to the cupellation process in which a muffle was used. These developments can be observed more than a century later in the metallurgical treatises of the Renaissance.

SUMMARY

Castel-Minier, situated in the French Pyrenees, was a centre for silver production between the 13th and 15th centuries. Despite being located near one of the largest European mining and metallurgical centres, it was not traditionally associated as a place of innovation. However, the discovery of ceramics dating back to the second half of the 14th century that resemble muffles questions this assumption. Through an archaeological and experimental study, we gain a better understanding of the cupellation structure, while an archaeometric survey of the material from experiments and archaeological excavations sheds light on the use of muffles and the cupellation structure.

The study of the concentration of lead was conducted using X-Ray fluorescence. This study concluded that ceramics identified as muffles are indeed muffles. The chemical analysis of the soil on and around the open fireplace by portable X-Ray fluorescence allows to map the lead concentration. The similarity between the archaeology and experiments means we can arrive at a conclusion about the

cupellation structure. The cupellation was done on an open fireplace at ground level, under a muffle.

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