First experimental reconstruction of an Angkorian iron furnace (13th–14th centuries CE): Archaeological and archaeometric implications

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Abstract
The region around Phnom Dek in Northern Cambodia represents a vast metallurgical landscape with evidence for mining and smelting of iron spanning over a millennium of smelting activities (7th to 20th centuries CE). Research conducted over the past decade in this region has expanded our understanding of the resources, production, technological evidence and furnace structures used during the Angkor period (11th c.-14th c.). In December 2018, a first experimental attempt at reducing iron ore mined from the historic source at Phnom Dek was conducted at the Ecole Française d'Extrême-Orient (EFEO) Center in Siem Reap/Angkor (Cambodia). The experimental research was designed to achieve several interconnected objectives. The aim of our study is not only to address technical questions relative to the metallurgical traits of the furnace and the metallurgical process employed during the late Angkor period, but also to provide new data to clarify the interpretations resulting from the provenance and iron dating analyses that assist in interpretations relating to production and consumption practices. Both the archaeological evidence and ethnographic descriptions were combined to reproduce the experimental furnace. This paper summarizes the preliminary results and provides information on the late Angkorian smelting process and on the accuracy and variabilities of the archaeometric data including provenance and iron dating.

Keywords: Iron, Angkorian furnace, Archaeometallurgy, Experimental smelt, Integrated experimental research

1. Introduction

The Phnom Dek region in northern Cambodia is a vast metallurgical landscape used by iron smelters for over a millennium (7th to 20th centuries CE) (Figure 1). Research about this region has highlighted the scope of iron production, its impact on the development of Khmer history as well as details about local smelting practices. Surveys conducted by European mining and civil engineers during the 19th and 20th c. documented the presence of multiple slag mounds and iron ore sources including the largest at Phnom Dek (the "Iron Mountain"), while French government expeditions generated the initial records of the Kuay people, an ethnic minority who worked iron here since at least the 16th c. (Aymonier, 1900; Dufossé, 1934; Hansen et al., 1876; Harmand, 1876; Khin, 1988; Moura, 1874; Saladin and Fuchs, 1882). More recently, extensive field research and multidisciplinary analysis of metallurgical by-products and iron crampons from temple restorations in Angkor have successfully revealed shifting qualities/preferences of iron products and an increased reliance on metal sourced from the Phnom Dek region (Hendrickson et al., 2017; Leroy et al., 2017). This included shifts in production intensity between the 11th and 15th c. showing correlations with the expansions and ultimate decline of Angkor (Hendrickson et al., 2017; Hendrickson and Leroy, 2020). Reconstruction of smelting practices necessary to refine these regional-scale studies has been hampered by the lack of archaeological furnaces and the incomplete record of Kuay techniques as a potential analogue for earlier systems in the Phnom Dek region (Dupaigne, 1987; Lévy, 1943; Pryce et al., 2014). Excavation of three furnace bases at Tonle Bak (13th-14th c.) 2km south of Phnom Dek (Hendrickson et al., 2019) provides the first opportunity to begin modelling the operation and products of an Angkor period installation using experimental methods.

This paper summarizes the preliminary results of an experimental smelt conducted at the Ecole Française d'Extrême-Orient (EFEO) Center in Siem Reap/Angkor in December 2018 (Experimental Archaeology festival) as part of the IRANGKOR research program. Our work represents a first attempt to reduce ore mined from the historic source at Phnom Dek with the goal of testing hypotheses on the metallurgical traits of the furnace and the smelting process as well as providing information on the accuracy and variabilities of archaeometric data including provenance and direct dating of iron products. While issues of design and furnace charging became apparent, the results from this initial test are a significant step for using experimental research to expand on future studies of past metallurgical practices and modern interpretations.

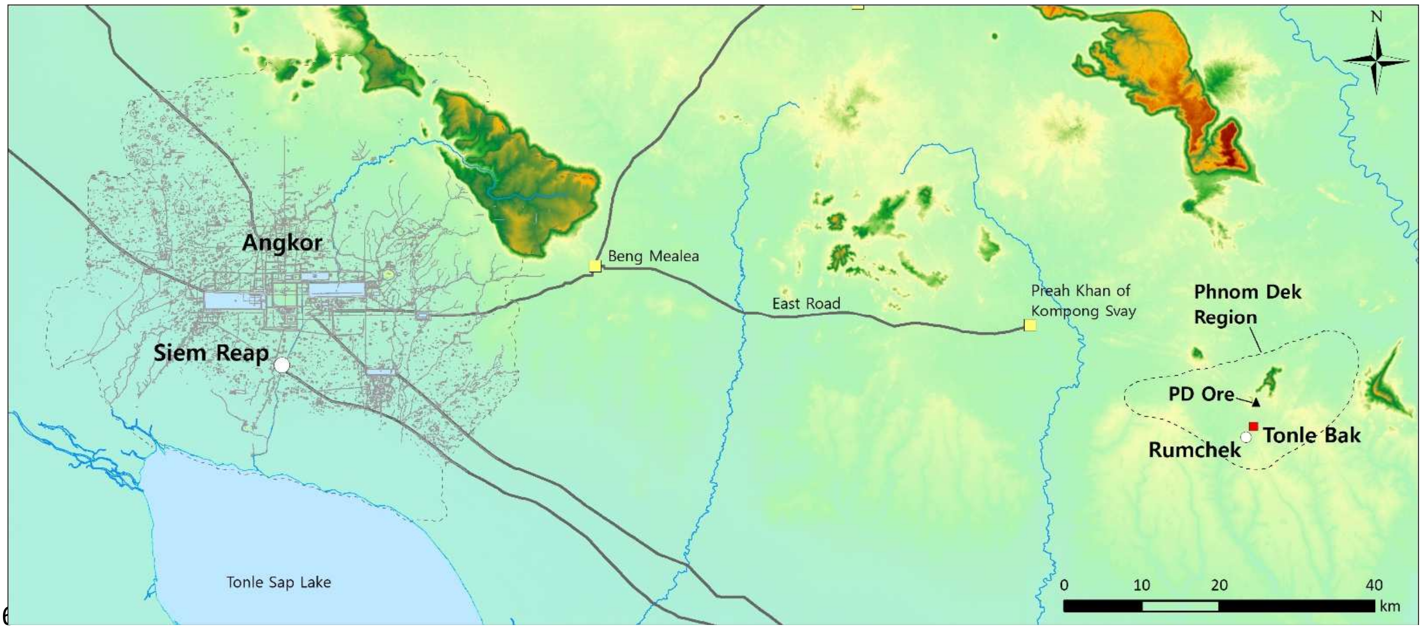


Figure 1: Location of the Phnom Dek (PD) area, the place of collect of the iron ores for this experimental research and the archaeological sites mentioned in the text.

2. Archaeological and Archaeometric Questions

Before examining the results of the experimental smelt, it is necessary to identify some of the key questions that arise from the archaeometric approaches (e.g., provenance and radiocarbon dating analyses) we use to interpret production and consumption practices based on the archaeological record.

Provenance investigation can be based on the investigation of the chemical relationships between the ores, the smelting slag and slag inclusions (SI) entrapped in the iron artefacts. Previous research has demonstrated the possibility of identifying consistent signatures associated with a particular smelting process or geographic origin by defining a combination of oxides (mainly the non-reduced compounds - NRCs-) or oxides ratios. Determining and comparing the chemical signatures in ores and/or slag is an essential step in attempts to establish provenance (Buchwald and Wivel, 1998; Coustures et al., 2003; Leroy et al., 2012; Charlton, 2015). Direct correlation of chemical signatures, especially those based on the major elements, can be complicated by the fact that the NRC ratios can be highly scattered due to the chemical influence of parent materials such as furnace components and fuels (Blakelock et al., 2009; Charlton et al., 2012). Our experimental contribution aims at helping to test and specify these patterns in the case of the Angkorian technology. As the resulting experimental bloom was not hammered to produce an iron product, this study is restricted to the patterns of slag composition resulting from the bloomery smelting process.

Studies of iron production systems are also complemented by recent developments in direct dating of iron alloys through radiocarbon analysis. The principle of 14C dating of iron relies on the presence of carbon atoms in the metal structure (Fe_3C) originating from the CO-rich atmosphere generated by the wood charcoal used as fuel during the smelting process. Thus, the radiocarbon age of the alloy is related to the radiocarbon age of the wood charcoal and more or less shifted from the reduction event according to the age of the fuel. Nevertheless, we can assume that this effect is reduced by the fact that when burning an entire trunk or a collection of trees, the outer rings of the trunk and branches, i.e. the youngest wood, outweigh the oldest heartwood (Dee and Ramsey, 2014). Precise calculation of this wood-age offset is impossible but may be estimated by anthracological studies of the charcoal remains, by analogy with ethnographic cases if they exist or by experimental reconstitutions. In 2011, some of the authors carried out an experimental smelting (Leroy et al., 2013; S. Leroy et al., 2015b), involving carbonated ore (siderite) and young branch wood (≤ 10 yrs old), whose purpose was to investigate a potential ageing effect of the carbonated ore on the radiocarbon date of the metal produced (Oinonen et al., 2009). The final iron date falls in the last 10 years range of the wood charcoal showing the predominance of the fuel in the production of a CO-rich atmosphere in the furnace. Nevertheless, the "old wood effect" in iron dating was not directly investigated and it seemed obvious to integrate this aspect in the experimental research.

Within these broader archaeometallurgical issues, the experimental smelt seeks to enhance our understanding of Cambodian iron production through several interconnected objectives. First, the evidence from the Tonle Bak excavation is used to tease out the technical parameters underlying the smelting process employed during the 13th c. The archaeological data are still limited and many questions remain about the Angkorian furnace characteristics such as about the exact number and placement of tuyères, wall height, quantity of raw materials used per operation, type of the ventilation, and duration of a single smelt. Given that furnace walls in the Cambodian procedure were destroyed to remove the bloom, there is no certainty of finding new data on these elements in the future and an experimental approach remains the best way to test any structural assumptions. Second, investigation into the products and waste from the experiment will be used to determine how they were manufactured (e.g., redox conditions, technical gestures). Incremental feedback between archaeological and archaeometric observations are combined to provide new evidence about the furnace operation. Third, chemical and radiocarbon dating investigation of this experimental assemblage can help to provide new data to clarify the interpretations resulting from the provenance and dating analyses within the context of the Angkorian technology.

Finally, the experiments at the EFEO center are designed to provide not only a better understanding of the technologies used in medieval Cambodia, but to also fulfill important community and educational objectives. These experiments are an excellent opportunity for training and knowledge transfer and serve as an unparalleled medium to help young Khmer archaeologists and students understand the long-forgotten technologies of their past and how to recognize them in the field.

3. Archaeological and Archaeometric Evidence: The Late Angkor Period Smelting Process

Research conducted over the past decade has expanded our understanding of the resources, technological materials and furnace structures used in the Phnom Dek region (Hendrickson et al., 2017; Hendrickson and Leroy, 2020; Pryce et al., 2014). By using this localised evidence in our experimental smelt we have a greater opportunity to replicate and inform on patterns seen in the archaeological record.

3.1 Resources

Phnom Dek is one of several ore bodies in the region. It became a preferred source during the Angkor period and specifically during the 13th c. (Hendrickson and Leroy, 2020; Leroy et al., 2017). The composition of this ore was first chemically determined by French specialists as a massive hematite ore deposit containing two types of minerals. The first type is a mix of magnetite (Fe_3O_4) and hematite (Fe_2O_3), rich in iron (>70_{wt}%), while the second type is composed of limonite (mainly composed of goethite FeOOH) with a lower iron content (30-45_{wt}%) (Caillère, 1972; Saladin and Fuchs, 1882). Recent analysis of the iron ores in the frame of IRANGKOR confirm these observations and will not be detailed in this article. No ore processing area was found on site but small roasted ore fragments found on Tonle Bak suggest that the size of ore particles added to the furnace was approximately 1–2 cm in diameter. For the time being, no data are currently available on the identification of the charcoal species used for the fuel, but ongoing studies will provide first results in the region in the near future.

3.2 Technological Evidence

The traditional iron technology in Cambodia involves direct smelting in low furnaces and the production of tap slag. The smelting slag from the surveyed sites, though dated from different periods, share a similar morphology showing an accumulation of different layers of flow shaped by the slag pit (see Figure 2) (Hendrickson et al., 2019). Morphological characteristics show low viscosity, and slag seems to separate cleanly from the bloom. The Reducible Iron Index (see (Charlton et al., 2010)), which provides information on the furnace redox conditions, calculated for a preliminary set of samples from late Angkorian/Kuay sites indicates the incomplete reduction of available FeO (Pryce et al., 2014). Moreover, intensive metallographic analysis of metal produced during the Angkor period revealed that from the early 12th c., the nature of the alloy from the iron-making workshops of the Phnom Dek area, probably including the Tonle Bak sites, is rather homogeneous with low-carbon content (Leroy et al., 2017). Overall, the results are consistent with an iron-making process favoring the production of low-carbon blooms rather than natural steel.

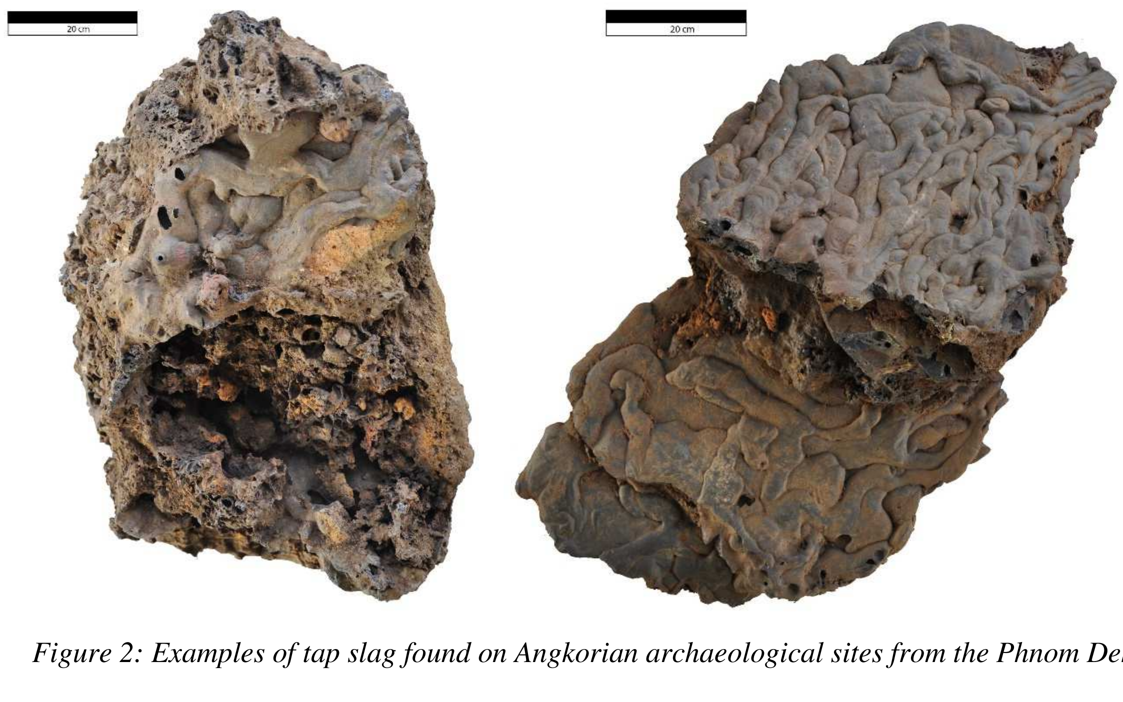


Figure 2: Examples of tap slag found on Angkorian archaeological sites from the Phnom Dek area.

3.3 Furnace Structure

In 2017, excavations at Tonle Bak provided the first evidence for furnace structures dating to the Angkor period (13th-14th c.) (Hendrickson et al., 2019). The three furnaces are rectilinear in shape and approximately 2m long and 1m wide. Only the base of the furnace is still visible with the eastern end shaped like an apse while the western end extends slopes down with a central channel that flows into a slag pit (Figure 3). Lack of walls and tuyères indicate that walls were broken in order to remove the bloom at the end of the smelt. This practice explains the considerable quantities of wall and tuyère fragments found on the surface of slag mounds across Phnom Dek. The metallurgical traits of the Angkorian tuyères (11th-13th c) are classified in the medium-gauge category (Internal Diameter ID ranging from 2.3 to 3 cm; External Diameter ED from 4.5 to 6 cm) (Pryce et al., 2014). Unfortunately, the incomplete nature of technological evidence at Tonle Bak (e.g., lack of information about quantity and placement of tuyères, wall dimensions) means that reconstruction of Angkorian smelting practice cannot solely rely on the archaeological record.

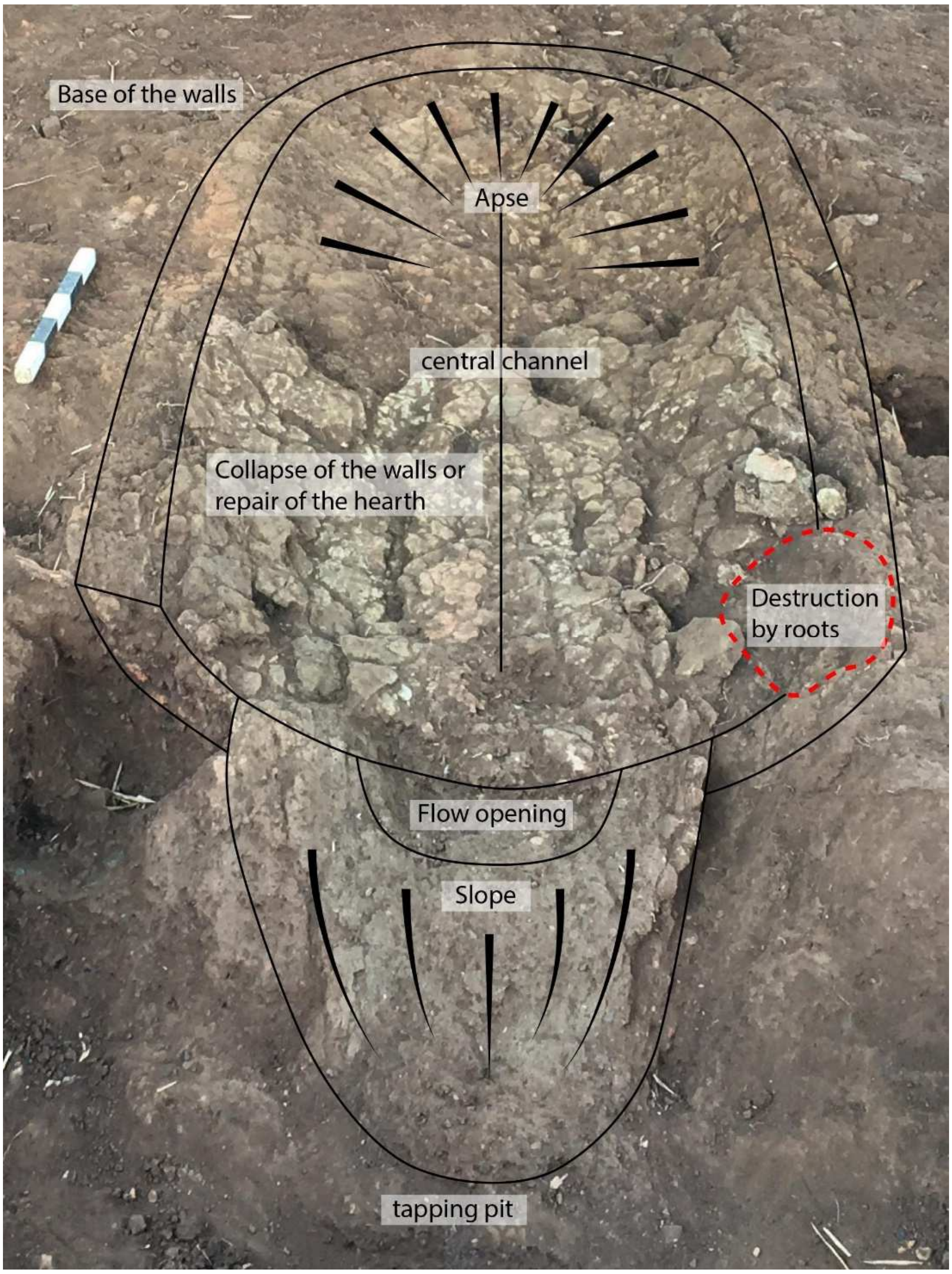


Figure 3: Base of the furnace with tapping pit and key features of the structure.

4. Ethnographic Evidence

The 19th and 20th c. ethnographic accounts describing Kuay iron smelting practice provide another important source of evidence about the construction and operation of furnaces in the Phnom Dek region

(Dufossé, 1934). The long-term Kuay presence combined with similarities in the use of large numbers of tuyères, basic furnace shape and preliminary metallurgical analyses indicating a possible conservatism in late Angkor and Kuay iron smelting parameters (Pryce et al., 2014) suggests continuities in practice suitable for incorporating into the experimental reconstruction.

4.1 Ore and charcoal preparation

The two mineralogical distinctions identified for the Phnom Dek deposit were similarly noted by the Kuay who divided ores based on appearance and weight into the "heavy stones" (*thma thngon)* and the "light stones" (*thma sral*) whose colour is slightly different. The last type seems to be preferred by the Kuays for the smelting (Boulangier, 1888; Harmand, 1876; Moura, 1874). Once procured, the Kuay would roast the ore (Harmand, 1876; Moura, 1874) to transform it into "small grains" (Boulangier, 1881). Charcoal production using the nearby forest was carried out shortly before the smelt as charcoal cannot be stored in advance nor can it be made in the rainy season. According to Dupaigne (Dupaigne, 1987), who interviewed chief smelters nearly 30 years after the last firing, the preferred tree species to produce charcoal are *krokas* (*Caesalpiniaceae sindora cochinchinensis* Baill.), *popil* (*Dipterocarpaceae shorea talura* Roxb.) and *chramas* (*Dipterocarpaceae vatica odorata* Griff.). To make the charcoal, a pile of wood is built on the ground, ignited at the base and covered with soil to prevent oxygen from entering the structure and to thus avoid the complete burning of the wood (Hansen et al., 1876). Descriptions made at the end of the 19th c. all describe the same quantity of raw materials used for the smelting process: between 300 and 420 kg of iron ore is required and the volume of charcoal introduced is twice that of the ore. The expected yield is perhaps 40-50% iron: 85 to 150 kg of iron is thus obtained (Dupaigne, 2016).

4.2 Description of the Kuay furnace

The furnace had a rectangular-shaped furnace chamber with dimensions ranging from 2 to 3 m in length and from 1 to 2 m in width, with walls extending up to 1 m and two holes, at both ends, for the evacuation of the slag during the smelting process (Bouillevaux, 1858; Boulangier, 1888, 1881; Dupaigne, 1987; Fortoul, 1946; Harmand, 1876; Moura, 1883). A 20 to 30 cm wide trench dug along the long axis of the furnace was observed, most likely to facilitate evacuation of slag. Walls were built from a local yellow clay that is abundant in the region (Boulangier, 1881).

4.3 Tuyères and air supply system

The air supply system is one of the unique characteristics of the Kuay smelting process. Bellows are located on the long sides of the furnace and air is passed initially through bamboo tubes and then into clay tuyères. The total number of tuyères per side is between 20 and 30. They are fixed at half the height of the wall in a fanning arrangement above the middle of the base furnace (Dupaigne, 1987; Harmand, 1876). A gap of 2 cm between the bamboo tubes and tuyères allows air to be brought into the bellows before being forced back into the furnace. This ventilation system may cause a significant loss of draught (Boulangier, 1881; Harmand, 1876). Compared to the Angkorian tuyères, the tuyères associated with Kuay sites have smaller standardised bore with an internal diameter ranging from 2 to 2.5 cm and an external diameter between 3 and 4 cm (Pryce et al., 2014). Regardless of size, tuyère manufacture involves wrapping wet clay around a rattan wood about 1 meter long. Once firmed, the clay was removed from the wood, then left to dry 2 hours in the sun before being dried for 24 hours (Dupaigne, 1987).

Figure 4: The smelting process and the Kuay furnace. (Top) Illustration "Fonderie de fer chez les Kouys" (Boulangier, 1888). (Bottom) Photo of the remains of a Kuay furnace in Rumchek (1969) (courtesy B. Dupaigne). Remaining wood walls from the air supply system exhibit the numerous holes for the air supply.

5. The Experimental Smelt

Recognizing the limitations of ethnographic analogy and potential technical differences, we used Kuay accounts on ore and charcoal preparation, furnace operation and air supply to complement the missing archaeological data. For some settings partially documented by both archaeology and ethnographic accounts, other technical choices already experienced by the authors in different contexts have been made while being aware that these choices may interfere the experiment's result. Nevertheless, this provides favorable conditions for a successful smelt, which remains a primary objective of this first experimental attempt.

5.1 The furnace

Archaeological evidence and ethnographic descriptions were combined to reproduce the experimental furnace at the EFEO Siem Reap/Angkor in December 2018. As this was the first attempt to smelt iron and given the wide range of potential parameters to control, we selected to reproduce the structure and materials at a 1:2 scale (Table 1). The experimental furnace had a rectangular internal shape with two apsidal ends and an internal dimension of 0.5x1 m (Figure 5). The total possible load height ranged from 0.7 to 0.8 m depending on the internal 5° slope of the furnace base. Ten holes for ventilation were made on the longest sides of the furnace walls, placed 20 cm above the external base. There is no archaeological evidence for the inclination/position for the Angkorian tuyères. In the Kuay manner they are described as being fixed mid-way in the wall (Dupaigne, 1987). Nevertheless, this position was not embraced into the experimental furnace setting for two main reasons. First, tuyère position and angle relative to the fuel bed are important for controlling the size and shape of the combustion zone of the furnace. We did not master the Kuay configuration sufficiently to meet with this condition. Second, in the lack of additional compositional data on technical ceramics, the risk of clogging the tuyères during smelting was too high with this configuration.The choice for inclination and position was thus dictated by knowledge acquired by the authors in other archaeological contexts, particularly in contexts dating from medieval times in Europe (M. Leroy et al., 2015) p. 156). The angle of inclination of the tuyères was approximately 8°, and they were therefore fixed closer to the external base of the wall. As suggested by the ethnographic data, the tuyères were arranged in a fanning orientation to cover the entire internal surface of the furnace and were separated from each other in the centre by a distance of 10 cm. The tuyères were made from a clay plate rolled around a wooden handle with a diameter of 2.2 cm using a plastic sheet. The tuyères were then dried around the wood until they became rigid enough to allow for its extraction (3 to 4 hours in the sun). A last drying of 24 hours in a ventilated space makes the tuyères solid enough and ready to use. Their final dimensions are ca. 50 cm long, with an internal diameter of ca. 2.2 cm and an external diameter of ca. 5 cm. This resulting morphology is therefore between the archaeological data and the ethnographic ones. The air supply system was an important focus of experimentation due to the difficulty to set up the very delicate bellows and ventilation system as described in the ethnographic accounts. To generate sufficient heat within the furnace, limit the random parameters and avoid issues due to changing temperatures, the ventilation of the furnace was produced by electric fans that created equal air pressure for each tuyère.

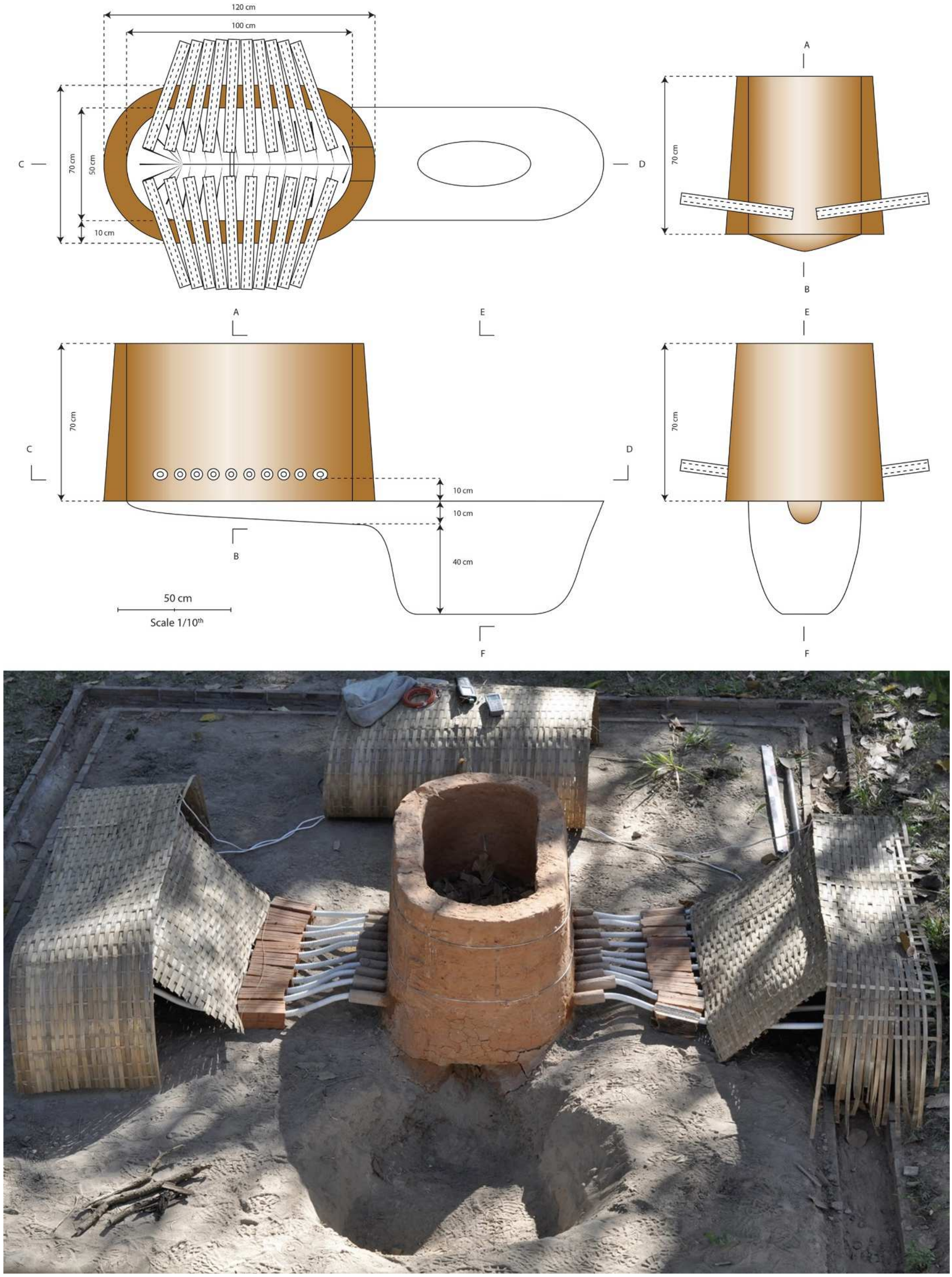


Figure 5: Schematic proposition and final construction of the experimental furnace and air supply system.

Table 1: Comparison of archaeological and ethnographic features, and experimental choices

Data sources	Tuyères	Furnace design	Air supply mechanism
Archaeological evidence (13th c.-14th c.)	**Type**: Medium-gauge (cm) (2.3<ID<3 ;4.5<ED<6) **Number on each wall**: unknown **Fixation heigh**t: unknown	**Dimensions:** 2m long, 1m wide, unknown height **Hole (tapped slag**): 1 **Central channel**: a V-trench	Unknown system
Ethnographic evidence	**Type**: Thin (cm) (1.9<ID<2.5 ;3<ED<4) **Number on each wall**: between 20 and 30 **Fixation height**: half-height of walls	**Dimensions:** 2-3 m long, 1-2 m wide, 1 m height **Hole (tapped slag**): 2 **Central channel**: 20 to 30 cm	Forced draught with bellows But 2 cm space between tuyères and bellows (no pressure)
Our experiment (½ scale)	**Type**: *Medium-gauge* (ID: 2.2 cm ED: 5 cm) **Number on each wall**: 10 **Fixation height**: 10 cm above outside floor	**Dimensions:** 1 m long, 0.5 m wide, 0.8 m height **Hole (tapped slag**): 1 **Base Trench**: a V-trench	Forced draught with connection (pressure)

5.2 Selection of Parent Materials

5.2.1 Iron ores

Two tons (2000 kg) of the two varieties of iron ores found at Phnom Dek were collected in 2018 for this experiment. A mix of both varieties (ca. 50%/50%) was used to ensure sufficient quantities of gangue and to create slag. All ores were roasted in an open fire and crushed into 2 to 3 cm pieces prior to smelting.

5.2.2 Clay

The clay for the furnace walls and tuyères came from a local brick factory in Siem Reap. Different proportions of local sand were mixed with the clay for each technical component. For the furnace walls, 25% sand was introduced for the overall assembly and the internal surfaces of the furnace (walls and floor) were then coated with a liquid clay mixed with 50% sand. The furnace was constructed two months before the experimental smelt. The tuyères need to be particularly refractory as they are in contact with the hottest area of the furnace (around 1300 to 1400°C). To prevent clogging, the clay was therefore saturated with sand (around 50 to 60%) to increase resistance during the smelt and to avoid them collapsing on themselves. The clay as well as the technical ceramics from the experiment are planned to be investigated by the ArAr laboratory (Archaeology and Archaeometry) in Lyon (France).

5.2.3 Charcoal

A quantity of 600 kg of charcoal was purchased from a producer located 20 km north of Siem Reap, who obtains wood from forested areas in northern Cambodia. The exact species are not precisely known but the charcoal producers surveyed for this experiment frequently mentioned the names *phchök (Shorea Obtusa)* and *châmbâk'* (*Irvingia Malayana*). These are hardwood species largely used in Cambodia for general construction as well as charcoal production (Francois et al., 2015). Modern charcoal is made in reusable charcoal kilns built with clay, mud or termite soil. The carbonised logs, approximately 60 cm length and between 10 and 50 cm in diameter, were cut into pieces of 5-8 cm at the maximum.

Figure 6: The different stages of preparation for the ore, charcoal and tuyères.

5.3 The Smelting Process

The furnace was lit in the morning of December 5 and the firing was conducted over a period of 12 hours, including 1 hour of heating and 1 hour of final combustion with no ore charge.

Temperatures were recorded each time a new charge of ore and charcoal was added using two thermocouples placed at the rear apse of the furnace (Figure 7). One 'low' probe (T1) was placed 22 cm from ground level and 48 cm from the top and a second 'high' probe (T2) was placed 43 cm from ground level and 27 cm from the top of the wall. T1, which was supposed to record the highest temperature, could not be placed at the level of the air intakes as this was also the level at which the metal and slag formed. For this reason, the temperatures recorded are well below the maximum temperatures reached.

We used ethnographic data for the quantities of raw materials. Thus, a final fuel-to-ore ratio of 2:1 was selected for the smelt, while studies of the known slag compositional data for 13th c. sites appear to favour a 1:1 ratio (see above). Indeed, previous bloomery experiments in other contexts have shown that a ratio close to 2:1 is ideal for producing high-steel metal (Crew et al., 2011; Tylecote et al., 1971). Nevertheless, these conditions remain to be confirmed especially since the observations were made for a different metallurgical process. As a first experiment with a wide range of unknown parameters, we preferred to opt for a 2:1 ratio in order to ensure a suitably reducing atmosphere and meet the conditions to produce iron. Charcoal and ore loads were added every 20 to 25 minutes. The first and second charges incorporated 6 kg of ore each and subsequent additions of 4 kg were made, up to a total of 96 kg. After 4 hours and 30 minutes of reduction, a first and only run of tap slag was evacuated through the front hole of the furnace. Slag formation after this event only accumulated at the bottom of the structure, mixing with metal, slag, charcoal and tuyères. At the end of the process, the furnace was opened by breaking the walls to extract the bloom. The final mass comprised of dense slag, light slag and metal that had collected on the ends of the tuyères. This mixture issue may be due to several parameters including the blowing technique, position of the tuyères as well as the slope of the furnace base. The following morning, the light and bubbly slag was extracted and about 30 kg of dense, metal-rich masses could be separated and individualised into seven blocks. Most of the slag was furnace slag that accumulated around the tuyères, adhering to the furnace wall, or entrapped in the upper part of the bloom.

A systematic sorting and precise inventory of the remains of the experimental furnace, wastes, and raw metal allowed us to select specimens for a preliminary investigation at the Archaeomaterials and Alteration Prediction Laboratory of the Institute of Archaeomaterials Research (LAPA-IRAMAT) and at the Laboratory for the Measurement of Carbon-14 (LMC14-LSCE).

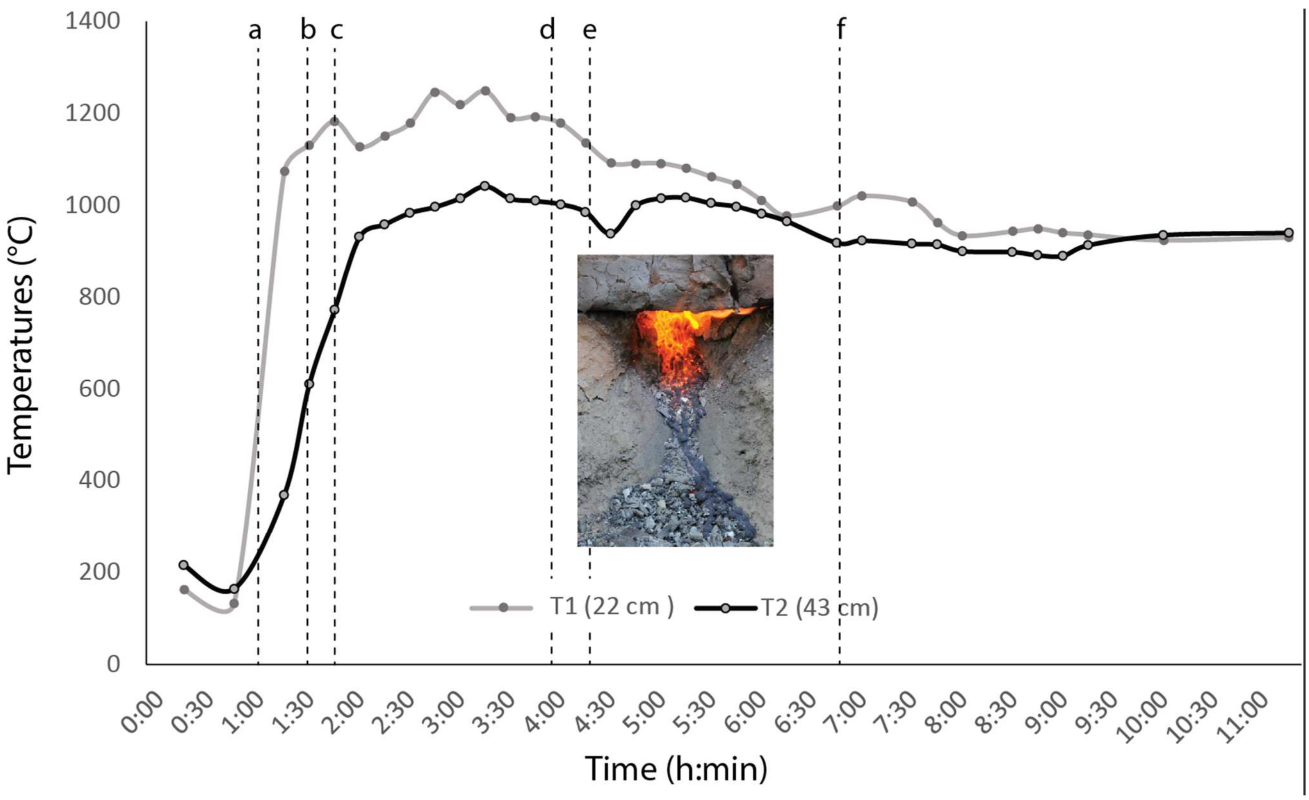


Figure 7: Temperatures reached at the thermocouples (T1, T2) during the smelting. (a) Blower start-up. (b) 1st charge of ore (6 kg). (c) Quantity of ore charge reduced to 4 kg. (d) Quantity of charcoal charge reduced to 6 kg. (e) Temperature decrease: opening of the front hole to flow the slag. (f) Unsuccessful attempt to unclog the tuyère.

Table 2: Quantity of parent materials (charcoal, ore), iron and slag produced.

Parent materials (kg)		**Products and slag (kg)**	
Charcoal (heating)	66	Raw iron mass	29.8 (ore/bloom=3.35)
Total Charcoal	242	Tap slag	2
Ore	100	Furnace slag	18.8
		Slag accumulated around the tuyères	16

6. Methods

The results of the experimental smelt were then examined within the framework of the technological and chronological analyses used for archaeological remains. By doing this, we provide a direct test of the analyses and shed light on the impact of smelting techniques on the final metal and by-products from the experimental smelt.

6.1 Macroscopic and microscopic analyses

Morphological characteristics were described first to inform on the nature of the waste products and metal formation. The petrographic and metallographic structures were then investigated by microscopic observations after cutting, embedding and polishing of the selected samples to a grain size of 1µm. For iron, after etching with a suitable reagent (Nital - 5% nitric acid diluted in ethanol), the nature of the alloy of the raw metal was determined. A systematic protocol developed at LAPA-IRAMAT acquires mosaics at X5 magnification at each step of the analysis to generate a holistic view of the entire sample and to calculate elements proportions. Optical microscopy of iron and slag samples was done using a Zeiss AxioImager A2m Vario microscope and Zen2 Core software.

6.2 Chemical analyses

Major chemical analyses of ore, slag and slag incorporated in the bloom were conducted to define their respective chemical signatures and explore the relationships between them. Iron ore fragments were analysed by ICP-OES and ICP-MS in the Service d'Analyse des Roches et des Minéraux (SARM-CRPG) (France) (Carignan et al., 2001). Chemical investigation of major elements (> 0.5 wt.%) of representative areas of slag samples and slag incorporated in the bloom pieces was completed by energy dispersive X-ray spectrometry (EDX with silicon drift detector [SDD] large window allowing counting rates about 90,000 c/s) coupled to a scanning electron microscope (SEM-FEG JEOL 7001-F). Each slag sample was subjected to scans of 1 mm^2. Analyses of the slag from the bloom involved selecting areas of 300x300 µm, in which all the mineral phases type are present. The raw NRC data were log-ratio transformed to obtain sub-compositional values (namely XE) to compare the element ratios and perform multivariate analysis (Aitchison et al., 2002; Leroy et al., 2012). Principal component analysis (PCA with Pearson correlation coefficients) was conducted to evaluate the dispersion of the NRC ratios and highlight the transformation of the chemical signatures from ores to bloom pieces. Since MnO is an ore-related compound, the comparison focused on those that may be subject to compositional variations within the smelting system: Al_2O_3, CaO, K_2O, MgO, SiO_2.

6.3 Radiocarbon analyses

The charcoal samples were pretreated using the Acid (HCl)-Alkali (NaOH)-Acid (HCl) protocol to remove carbonates and organic components from the soil (Dumoulin et al., 2017). For the radiocarbon dating of ancient iron alloys, the methodology requires detailed metallographic and SI investigation (S. Leroy et al., 2015b). Iron particles were collected in the carburised areas with CoB coated drills. Both iron and charcoal samples were then combusted in sealed tubes with CuO and Ag wire at 850°C for 5 hours to produce CO_2. CO_2 gas was reduced into graphite to form targets for ^{14}C measurements in the Artemis AMS facility of the LMC14-LSCE (Moreau et al., 2013). The ^{14}C contents measured for the samples were converted into calendar years thanks to the post-bomb atmospheric curve of our region of interest (Hua et al., 2013) complemented by the Hammer and Levin dataset (Levin et al., 2013; Hammer and Levin, 2017) and by LMC14 measurements on 2017 and 2018 nuts. As suggested by Hua (personal communication), a $F^{14}C$ regional offset of + 0.0025 was added to the calibration data for the 2005-2018. These data were implemented in the OxCal v4.3 calibration program (Ramsey, 2009).

7. Measurements and discussion

7.1 Quality of raw iron and slag

Of the 29.8 kg crude reduction mass, two morphological types were obtained and a block of each was analysed (Figure 8). Mass 1 consists of dense, smooth-looking flows and corresponds to a metal that has passed into a liquid state. Mass 2, by contrast is more similar to products from bloomery furnaces that have a more or less spherical or calotte-like morphology and irregular, jagged exterior. Both masses were cut transversely to examine their internal structure (see Figure 8).

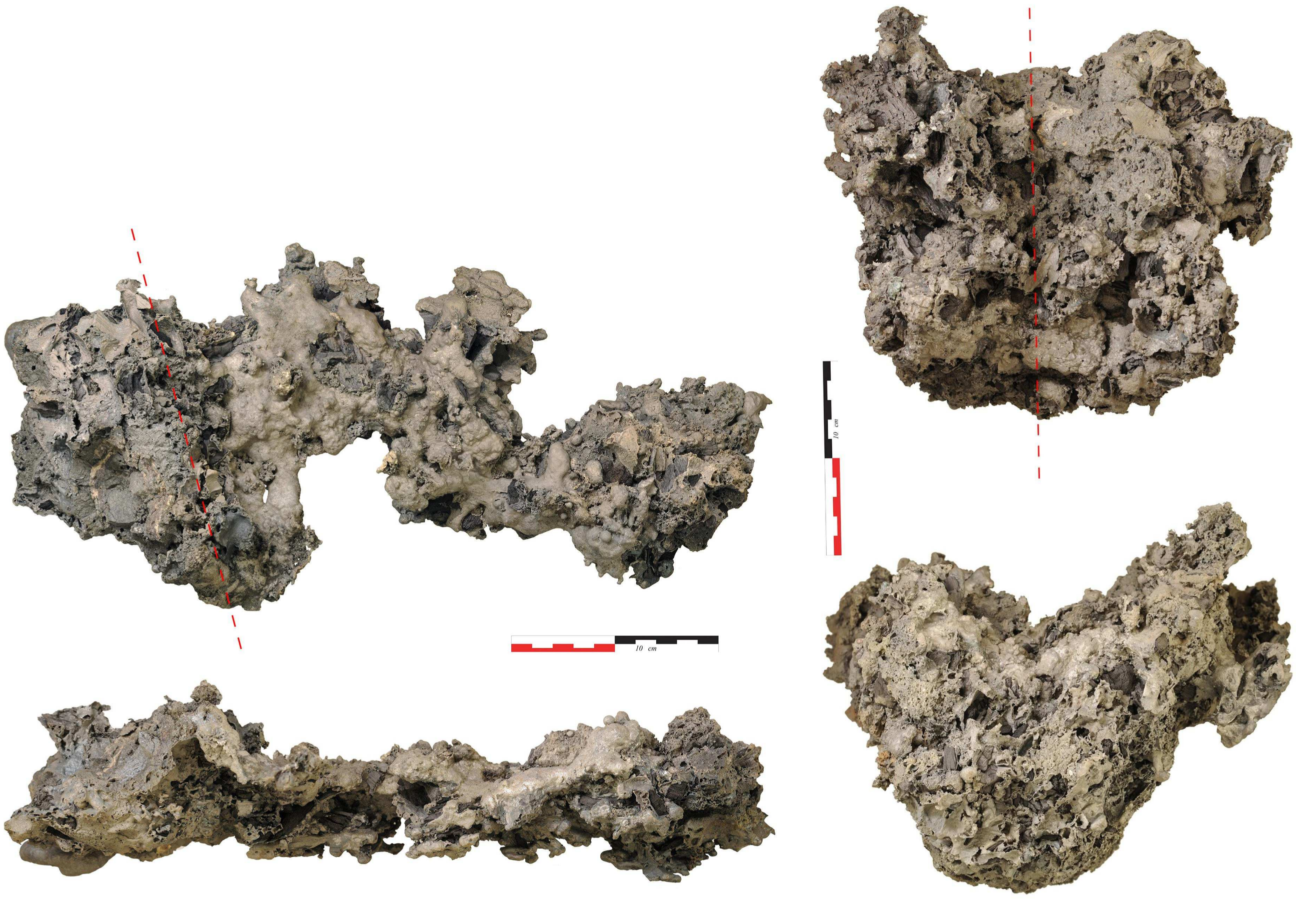
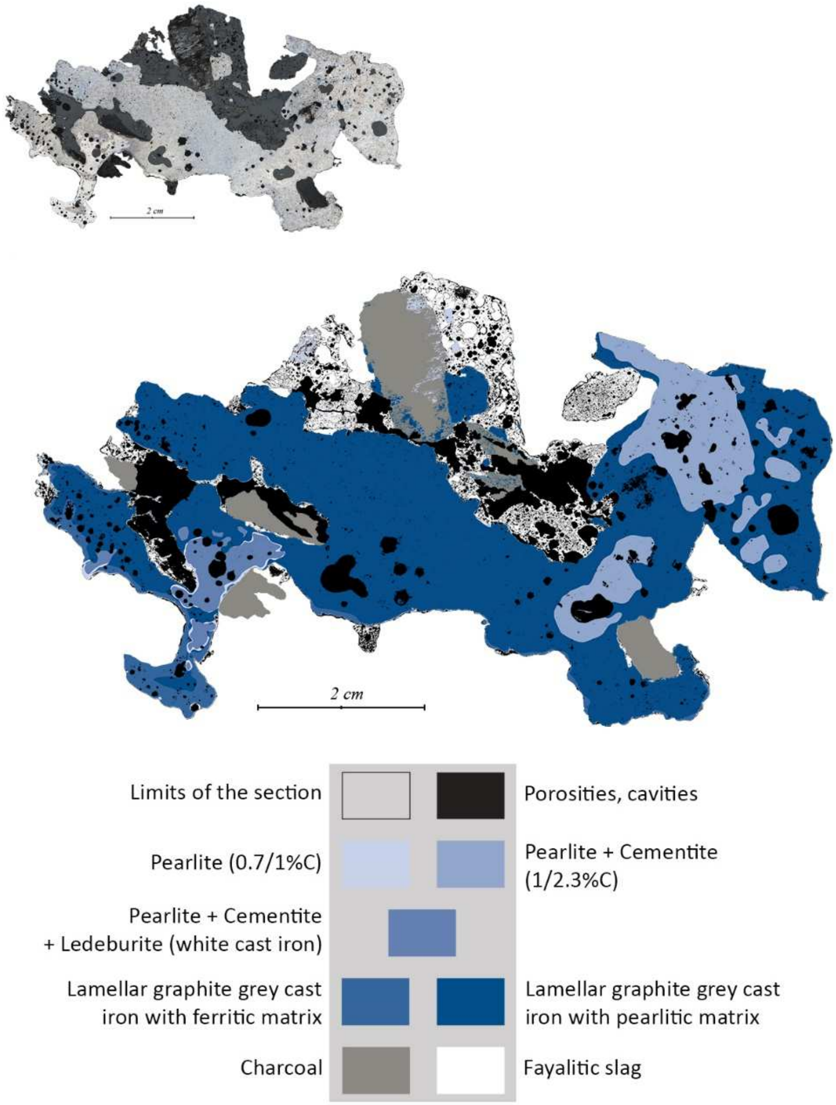


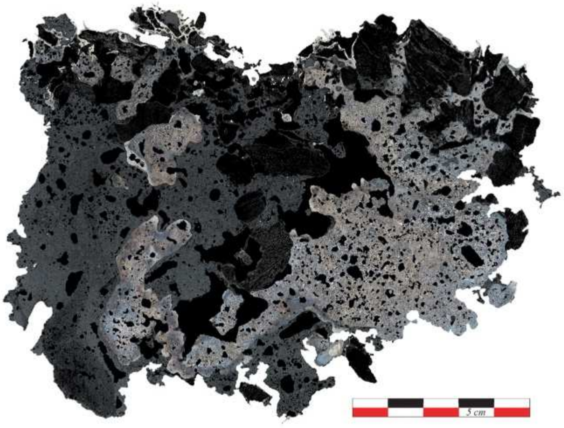
Figure 8: Morphological aspects of Mass 1 (left) and Mass 2 (right). Mass 1 weighs 3980 g and is elongated in shape, 37.7 cm long, 17.5 cm wide and 7.6 cm thick. Mass 2 weighs 5990 g with a length of 24.3 cm, a width of 20.9 cm and a thickness of 14.4 cm. The red dashed lines show the cutting sections.

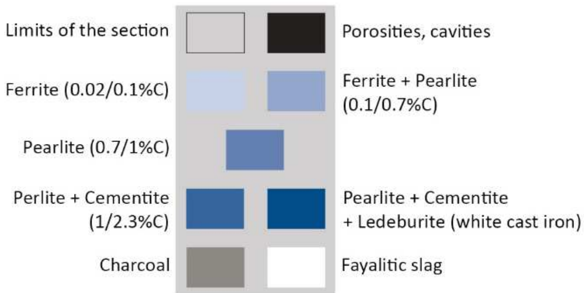
The polished section of Mass 1 contains a majority of metallic areas (69%) covered with large areas of slag (4%) and charcoal (9%). The outline of the metal zones is very regular and appears to be composed of several superimposed metal "castings". The metal itself is mainly composed of cast iron and hypereutectoid steel (Figure 9). Mass 2 is rather hemispherical or bole-shaped with very irregular external surfaces and contains numerous charcoal fragments. The metal parts, slag fragments and porosities within the mass are irregularly shaped and numerous (equal proportions of metal 31% and slag 25%). The metal appears to be very homogeneous and composed of 27% hypereutectoid steel and of 0.8%C eutectoid steel (Figure 10). These indices suggest that the metal has not reached its melting point and, consequently, is less carburised than Mass 1.



Zone		%
Metal	Pearlite + Cementite	0.90
	Ferrite + Pearlite	10.90
	Pearlite + Cementite + Ledeburite	2.56
	Grey cast iron with ferritic matrix	3.16
	Grey cast iron with pearlitic matrix	51.57
Slag		3.90
Porosities, cavities		18.01
Charcoal		9.00
Total		100.00

Figure 9: Metallographic observations and image analysis of Mass 1 section after etching with nital reagent.





Zone		%
Metal	Ferrite	1.09
	Ferrite + Pearlite	1.77
	Pearlite	5.02
	Pearlite + Cementite	22.16
	Pearlite + Cementite + Ledeburite	1.27
Slag		24.93
Porosities, cavities		26.29
Charcoal		17.48
Total		100.00

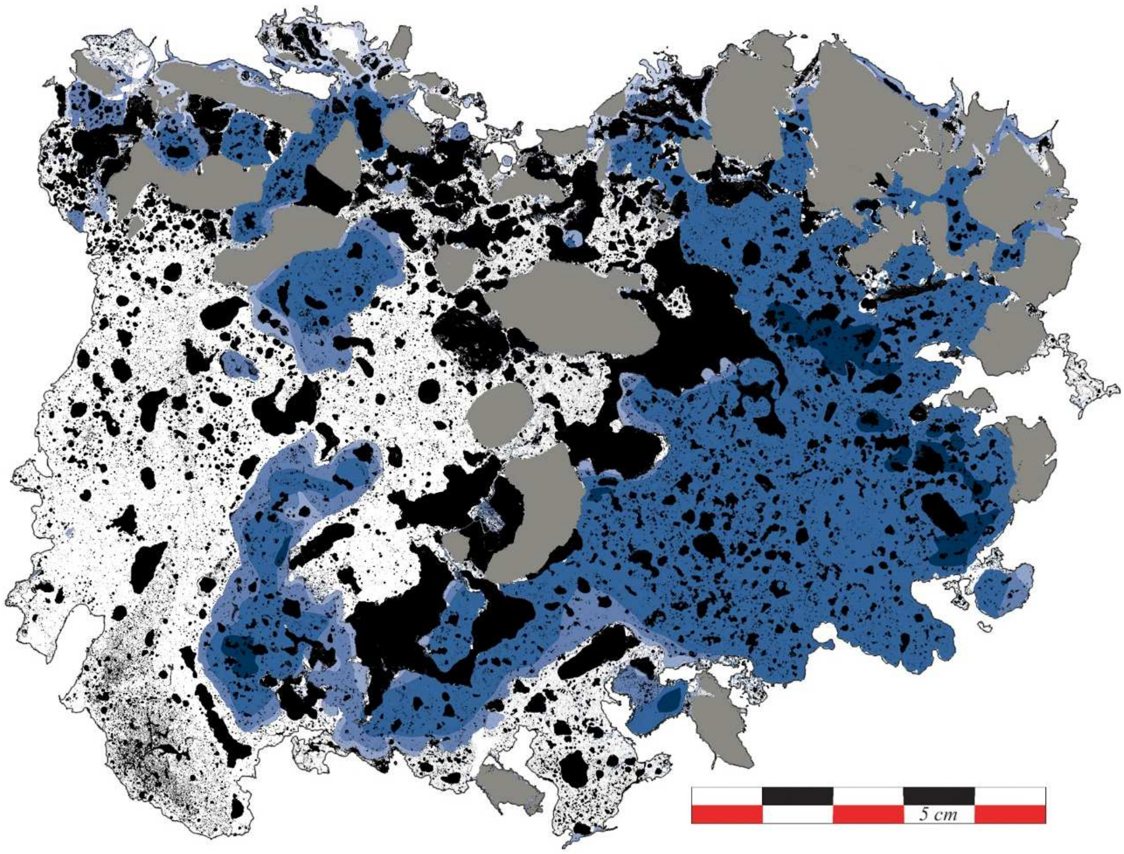


Figure 10: Metallographic observations and image analysis of Mass 2 section after etching with nital reagent.

Overall, Mass 1 contains too much carbon and would be considered unsuitable for forging and therefore rejected by both modern and 13th c. Angkorian smiths. The hypereutectoid metal of Mass 2 could be forged by highly skilled and specialised craftsmen but this bloom would be generally unsuitable as a basis for a final product for everyday use.

Microscopic analyses of ten slag pieces (tap and furnace slags) reveal a clean fayalitic microstructure depleted in iron oxide with nearly no wüstite (Figure 11). This is coherent with high reduction conditions in the furnace and a tendency toward production of a high carbon metal.

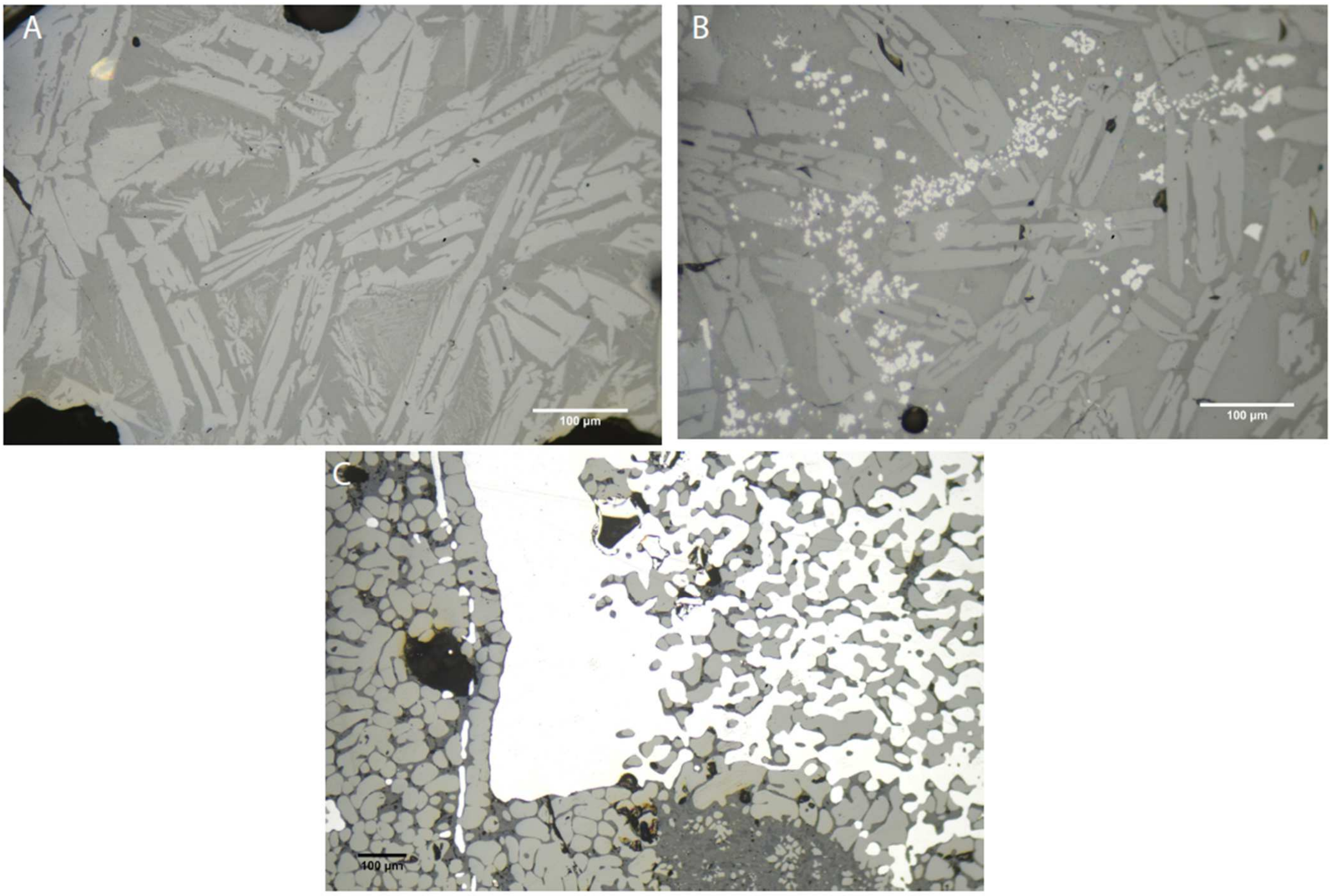


Figure 11: Optical microscopy for experimental tap slag (A and B) and furnace slag (C). (A): Well-developed fayalitic laths. (B) Fayalitic laths and fine wüstite dendrites. (C) Example of partially reduced ore fragment.

Overall, the thermochemical profile of the furnace, slag characteristics, and bloom-slag separation clearly show that the experiment is not yet representative of the Angkorian smelting process. The technical choices made by the authors unsurprisingly hindered the final outcome but also provide clues about where changes need to be effected. Most obvious is that several aspects of the air delivery system were inadequate. Tuyères that failed to melt during the operation quickly became partially blocked by the accumulation of slag at the bottom of the furnace. This points to both an incorrect tuyère inclination and clay composition used to make them. Following some ethnographic accounts, the inclination and placement of the tuyères should arrive at the mid-height of the walls relative to the outside of the furnace. A tuyère and wall fragment recovered from Sanlong Jaya, a 20th c. Kuay smelting site near Phnom Dek (Pryce et al., 2014) may indicate the use of a 45° angle for the tuyères. This new positioning combined with a decreased ratio of sand in the clay, or a different nature of the clay, would allow the tuyères to melt without collapsing on themselves and reduce their size as the metal mass grows inside the furnace. This differences in composition between tuyères and furnace fragments seems to be in line with observations made by Pryce et al. (Pryce et al., 2014) who noted that the tuyère clays contain higher alumina content relative to furnace clays. Nevertheless, it seems that this “alumina enrichment” would not increase their heat resistance during the smelt, it could rather reflect the absence of adding sand for the tuyères compared to the furnace clay. In the light of these new observations, it is fundamental to complete the compositional data on the clay used for both the walls and the tuyères from archaeological sites to provide new data on the technical choices followed by the smelters. A more complete study (DRX, XRF) of the clay used in the manufacture of technical ceramics is planned for later this year and will be carried out in collaboration with the ceramologists of the ArAr laboratory.

The ethnographic description of the 2cm space between the bellows tubes and the tuyères could also be a key feature for controlling the rate of air intake to regulate temperature and reaction speed inside the furnace (Rehder, 2006). Inside the furnace, the most immediate change is to increase the slope at the base of the furnace to facilitate evacuation of the slag. The fuel-to-ore ratio (2:1) suggested from the ethnographic data is undoubtedly one of the main reasons for the high-carbon content alloy produced in this experiment. This ratio is therefore extremely important when reducing iron-rich ores, and would be critical when attempting to smelt the eastern iron reserves from Phnom Dek. Again, the cast iron produced in our furnace is linked to our inability to control the rate of air intake (i.e., creating very high temperatures) which was exacerbated by the large number of tuyères. These factors all need to be considered in order to produce the low-carbon blooms typical of the late Angkor period.

7.2 Chemical relationships between ore, slag and bloom for provenance attempts

The NRC ratios measured in ore, smelting slag and slag into pieces of bloom were compared to identify patterns within the experimental smelting system (Figure 12). As expected, the ratios for the slag (tap, furnace, bloom) are different from the ores. These results are in accordance with the fact that the chemical signatures in the slag is strongly constrained by the furnace material and the fuel ash composition. The signatures based on major elements are therefore related to the smelting process itself (Buchwald and Wivel, 1998; Paynter, 2006; Dillmann and L’Héritier, 2007; Blakelock et al., 2009; Charlton et al., 2012) rather than a provenance to a geological ore. Differences are also evident between the two types of slag with the elemental ratios for tap slag being relatively constant in comparison to the more heterogeneous furnace slags. The diversity within the latter type relates to their formation relative to their placement within the furnace and the varying contributions of proximal parent materials. This result is a key reminder of the importance of using tap slag in attempts to identify the provenance of iron products.

Ratios for the large data set collected from the bloom are also quite scattered. Slight compositional differences are discernible between the main body of data from the bloom and the tap slag, especially those including CaO, Al_2O_3 and MgO. The compositional heterogeneity seen here could be a consequence of bloom mass formation at different places within the furnace structure. Such variability complicates direct correlations between tap slag and bloom and may considerably limit the use of major elements in assessing provenance to a smelting process. As already suggested for other contexts (Blakelock et al., 2009), it is fundamental to consider that variable furnace material contributions to the slag could result in chemical overlaps that dilute any specific signature even for different production provenances. Ongoing analyses of these samples will permit to compare and test the consistency of trace elements for defining less-scattered signatures and test relationships throughout this experimental assemblage for provenancing attempts (Coustures et al., 2003; Desaulty et al., 2009; Leroy et al., 2012).

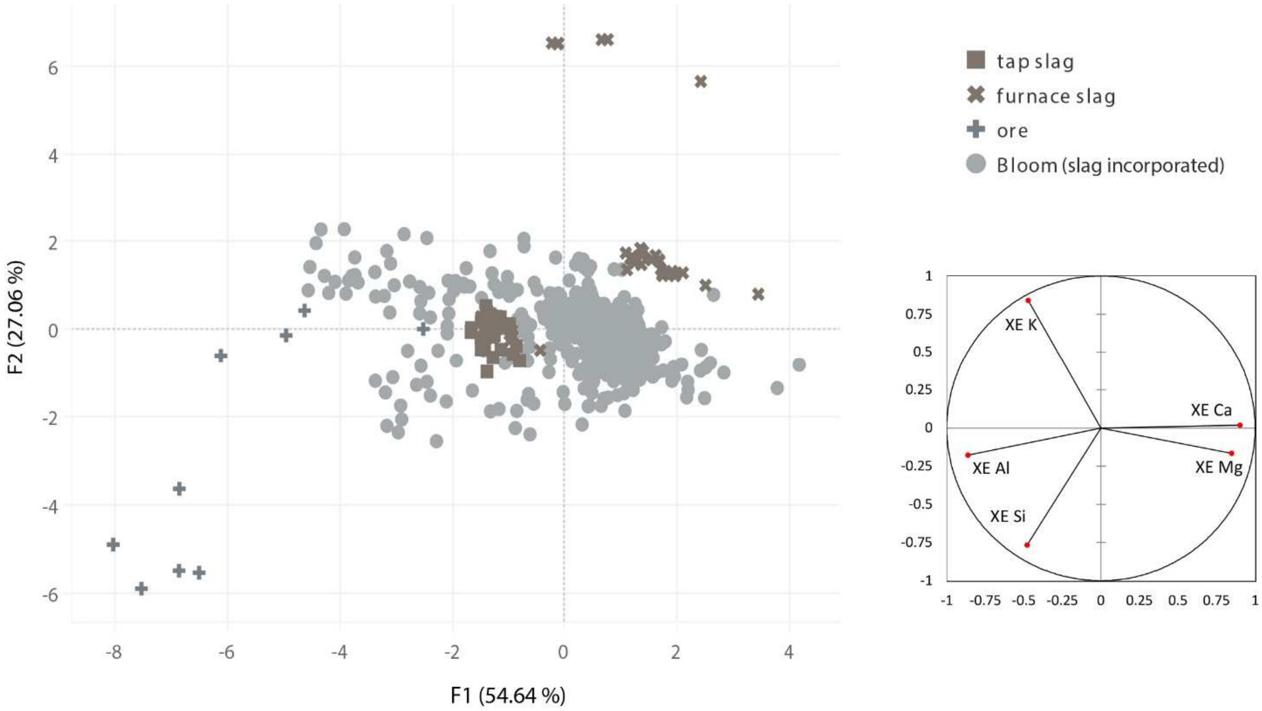


Figure 12: Multivariate plot of the sub-composition values of the non-reduced compounds (NRC ratios XE) from major elemental analyses in ore, furnace slag, tap slag and slag incorporated to the bloom. The comparison is done using the element composition.

7.3 Radiocarbon dating analyses

Six charcoal samples were selected for ^{14}C measurements including two from the internal and external part of a carbonised log (6 cm diameter), two samples from furnace slags, one adhering to the metallic Mass 1 and one from inside the furnace after the smelt. Three samples of metal were extracted from Mass 1 and from a smaller metal piece.

All the results show two dating intervals (Table 3). The first range is between 1957 and 1962 and correspond to the fast rising of the ^{14}C content in the atmosphere caused by atmospheric nuclear detonations. The second range conform to the decreased ^{14}C content from 1965 onwards (Figure 13). Considering the large diameter of some logs used for the experimental smelt, the oldest interval cannot be systematically discarded for the dates on charcoal. Nevertheless, we can reasonably assume that the majority of charcoal pieces introduced in the furnace are posterior to this interval, with some of them ranging from the eighties (SacA 59795: 1981-1983 AD) until the present (SacA 59798: 2009-2013 AD). Unlike the charcoals that all give different radiocarbon dates, the three iron samples show close ^{14}C contents corresponding to an interval of calibrated dates from 1999 to 2003 AD. The strong similarity of the ^{14}C results of the iron samples reflects a homogeneous ^{14}C content in the reducing CO when diffusing into the metal being formed. It demonstrates that iron alloy records the global ^{14}C activity of the wood charcoal used as fuel. Considering the old dates obtained for some charcoal fragments, a reasonable age offset of 15 to 19 years relative to the date of the experiment is obtained for the metal. This result shows that the old wood effect often attributed to iron ^{14}C dating must not be overemphasised and is probably even lower in most cases where the fuel is selected from small trunks and young branches. Furthermore, our experiment shows that iron alloys can be a more reliable dating artefact compared to small unselected charcoals possibly coming from the oldest parts of the wood fuel loaded in the smelting furnace.

Table 3: Radiocarbon dating results for the charcoal pieces and the iron samples. Calibration was performed with OxCal 4.3.2 (Ramsey, 2017); r:0.02; Post-bomb atmospheric NH3 curve (Hua et al., 2013); Post-bomb atmospheric curve (Levin et al., 2013; Hammer and Levin, 2017).

Sample	**Sub sample**	**Nature**	**Lab ID.**	**$F^{14}C \pm 1\sigma$**	**Calibrated Age (2σ, 95,4 %)**
Charcoal on slag	XP-18-ChScor	Charcoal	SacA 59794	1.0787 ± 0.0025	1957AD (2.6%) 1958AD 2001AD (92.8%) 2004AD
Charcoal on slag	XP-18-ChScorCo	Charcoal	SacA 59903	1.1686 ± 0.0026	1958AD (17.4%) 1959AD 1988AD (78.1%) 1990AD
Charcoal on metallic mass	XP-18-ChMet	Charcoal	SacA 59795	1.2426 ± 0.0028	1959AD (20.0%) 1962AD 1981AD (75.4%) 1984AD
Charcoal in the furnace	XP-18-ChFos	Charcoal	SacA 59796	1.0817 ± 0.0024	1958AD (3.4%) 1958AD 2001AD (92.1%) 2003AD
Carbonised log	Internal ring	Charcoal	SacA 59797	1.1266 ± 0.0025	1958AD (7.4%) 1958AD 1993AD (88.0%) 1995AD
	External ring	Charcoal	SacA 59798	1.0375 ± 0.0024	1955AD (15.1%) 1957AD 2009AD (80.3%) 2014AD
Dense metal piece	XP-18-f1a	Iron	SacA 59792	1.0841 ± 0.0024	1958AD (4.9%) 1958AD 2001AD (90.5%) 2002AD
	XP-18-f1b	Iron	SacA 59793	1.0822 ± 0.0024	1957AD (4.7%) 1958AD 2001AD (90.7%) 2003AD
Metallic mass 1	XP18-Met1	Iron	SacA 60033	1.0882 ± 0.0027	1958AD (7.3%) 1958AD 2000AD (88.1%) 2002AD

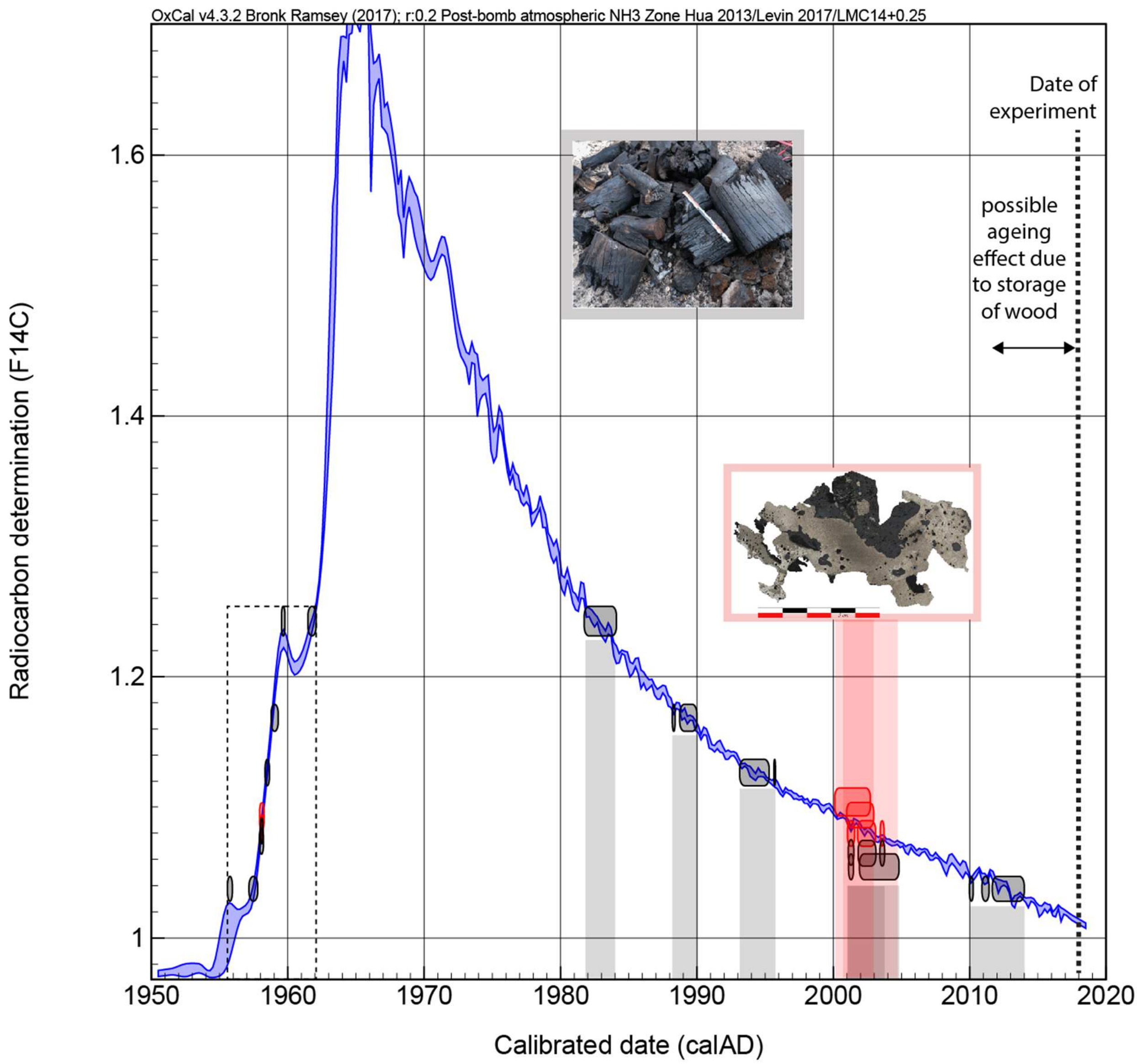


Figure 13: Calibrated ^{14}C dates obtained for charcoal fragments (grey) and iron alloys (red).

8. Conclusions and Perspectives

This initial experimental smelt was designed to address some technical questions relative to the construction of furnaces and the metallurgical process employed during the late Angkor period. The end result, perhaps unsurprisingly, failed to produce comparable quantities and qualities of slag and iron as seen in the archaeological systems around Phnom Dek. However, valuable information was obtained for future tests including changes to the composition, placement and inclination of the tuyères, the ratios of raw materials, limit of fuel size and a reorganization of the air supply system to allow better temperature management necessary to produce a low-carbon bloom. Our analysis of the waste and products of the smelt also provided useful insights into chemical variability and iron dating of an unprocessed iron bloom. The results confirm that direct correlation between iron product and slag using only major elements may be much more complex and should be confirmed with a larger data set and complemented with other scales of analysis. On-going study of trace elements from this experiment will continue to refine our understanding of this smelting system and, more broadly, to confirm the chemical relationship between ore, slag and object for provenance studies. Our results also demonstrate that iron alloys are viable artifacts for direct dating and confirm the reliability of previous dates obtained from crampons recovered from Angkor period temples (Leroy et al., 2017; S. Leroy et al., 2015a). In the near future, the charcoal species identification from archaeological sites would help significantly to select the adequate fuel in order to better approximate the 14C content in the furnace.

Finally, we hope that the experimental work conducted at the EFEO in Siem Reap will encourage local and international scholars to continue the reconstruction and preservation of Angkorian heritage beyond its elaborate temple complexes. This experiment has provided a public display and publication in Khmer (Tauch, 2019). Future attempts to smelt iron – and fire ceramics – will benefit from this dissemination of results and the integration of new technological details recovered from excavation and survey. The results of these collaborative activities in turn provide essential information for archaeological and archaeometric researches and, more specifically, the role of pyrotechnologies within the historical trajectory of premodern Cambodia.

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CRediT authorship contribution statement

Leroy Stéphanie: Conceptualization; Investigation; Project administration; Resources; Supervision, Writing -original draft. **Sylvain Bauvais**: Conceptualization; Investigation; Project administration; Resources; Writing -original draft. **Emmanuelle Delqué-Kolic**: Conceptualization; Investigation; Project administration; Resources; Writing -original draft. **Mitch Hendrickson**: Resources, Supervision, Writing - review & editing. **Nicolas Josso**: Project administration; Resources. **Jean Pascal Dumoulin**: Resources, Writing - review & editing. **Dominique Soutif**: Project administration; Resources, Writing - review & editing.

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