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The *Chaîne Opératoire* of Middle Kingdom smelting batteries and the problem of fuel: Excavation, experimental and analytical studies on ancient Egyptian metallurgy

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ABSTRACT

In this paper, a solution is proposed to answer the problematic issue of the type of fuel used in the early Middle Kingdom smelting furnaces at Ayn Soukhna based on a strict experimental protocol. The protocol was repeated 17 times, for a total of approx. 1500 h of experimental work. In correspondence with the archaeological data obtained at Ayn Soukhna, the combined use of fresh acacia and donkey dung as fuel for smelting is proposed; the use of charcoal for smelting can be excluded.

A holistic evaluation of experimental and archaeological evidence has shown that a combination of fresh wood and donkey dung was most likely the standard fuel choice at Middle Kingdom Ayn Soukhna. This is evidenced by the compatibility of results from experimental smelting using wood and donkey dung in terms of furnace lining impact and colorimetry, slag typology and deposition, ash and charcoal distribution, and raw copper shape and size. The use of charcoal systematically results in incompatible smelting results, as does smelting without donkey dung. In addition to green wood, donkey dung is required to regulate the temperature distribution inside the furnaces and to increase the production of copper prills. Green wood creates a favourable atmosphere for the smelting of copper carbonate ore. It heats the lining of the column less, allowing the furnaces to be reused over several campaigns. The dual fuel use of green wood and donkey dung, however, has resulted in the best agreement between experimental smelting results and corresponding archaeological evidence.

This archaeometallurgical research in the Nile Valley is carried out by a team composed of Georges Verly, in charge of archaeology and experimental archaeology, and Frederik Rademakers, in charge of archaeometry, as well as numerous master students and a *Maître d'art*, Hugues Paridans. Dialogue and interdisciplinarity are the assets of this project, dictating equality between its members, representing the sum of ideas and debates, building all research protocols and publications.

1. Introduction

The intermittent harbour and logistical platform of Ayn Soukhna, located on the northern end of the Gulf of Suez (see Fig. 1, from Abd el-Raziq et al., 2011), played a major role in supplying copper to the Nile Valley during the Old and Middle Kingdoms.[1] Dozens of metallurgical workshops, dating to the early Middle Kingdom (from the reign of Mentuhotep IV to the reign of Senwosret I, ca. 2000–1930 BCE)[2] reveal a

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[1] This paper is part of a Special Issue on the Contributions of Experimental Archaeology to Excavation and Material Studies, guest edited by Frederik W. Rademakers, Georges Verly, Florian Téreygeol and Johannes Auenmüller.

[2] Occupation at Ayn Soukhna at the beginning of the Middle Kingdom is attested for at least three state promoted expeditions under Mentuhotep IV, year 1 (Tallet, 2018, expedition 27), Amenemhat I, year 7 (Seyfried, 1981, expedition 1 = Tallet, 2018, expedition 28) and Senwosret I, year 9 (Seyfried, 1981, expedition 2 = Tallet, 2018, expedition 29).

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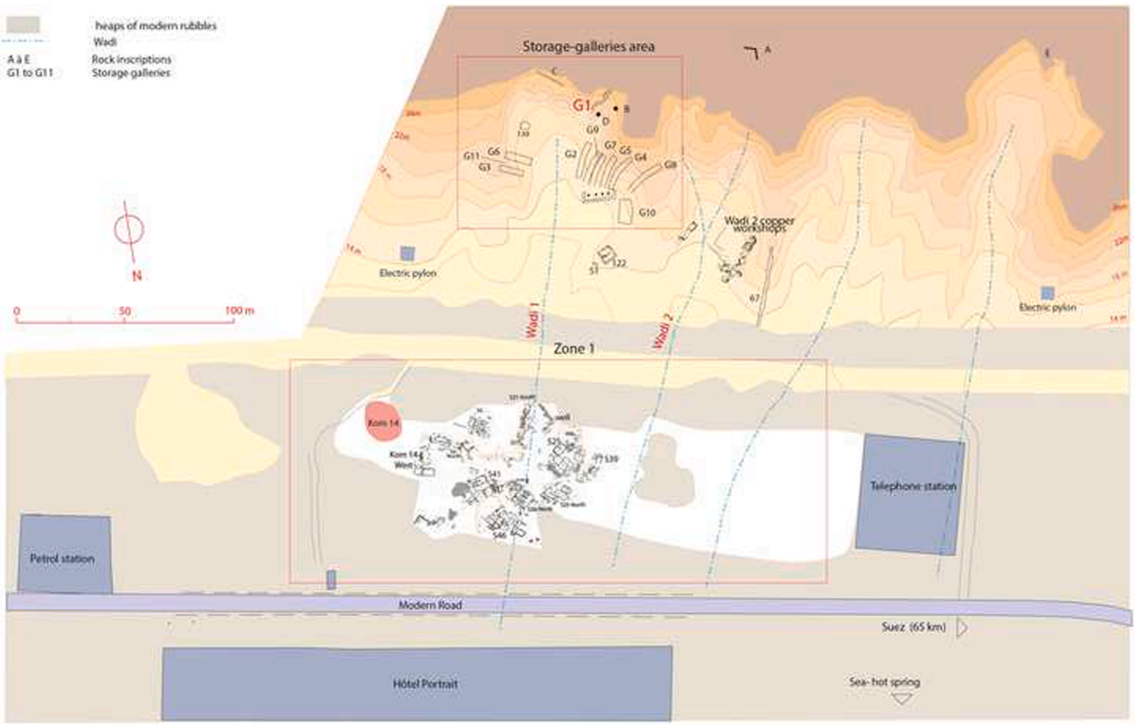

Fig. 1. Map showing the different sectors of Ayn Soukhna © Mission archéologique d'Ayn Soukhna.

highly standardised technological tradition for copper smelting in column-shaped furnaces (Abd el-Raziq et al., 2011; Tallet, 2016–2017; Verly, 2017). As such, they provide the only fully documented example of Egyptian Middle Kingdom primary metallurgy known so far. Detailed study of these furnaces and their broader context offers a unique insight into the organisation of these workshops (Verly, in prep).

One of the main research topics is the identification of the fuel economy at Ayn Soukhna. The high number of smelting furnaces (currently, about 80 furnaces are excavated, with possibly more located in the western sector of the plain and in Wadi 3: Somaglino et al., forthc.) require significant fuel resources. In a marginal desert context, fuel can be a complicated resource to obtain and manage, which has prompted this investigation. What type of fuel was used by the ancient metallurgists in an area with restricted vegetation cover? There are hot springs and an oasis close to the site, but it is unlikely that the regeneration of the local tree and shrub population has allowed local supply during the activity peaks of the individual expeditions.[3] How did the administration manage possible supply problems?[4] The different activities at Ayn Soukhna require different types of fuel. As wood is transformed into charcoal during smelting (cfr. below), its management through different activities is investigated as well. In particular, the recycling of charcoal can be proposed for secondary metallurgy (melting), but also for baking bread (Bats, forthc.).

In the early Middle Kingdom, copper ore was extracted from the South Sinai mining area and brought to Ayn Soukhna by ship for processing, as part of combined mining and smelting expeditions (Abdel-Motelib et al., 2012; Tallet et al., 2011; Tallet and Mahfouz, 2012; Tallet, 2012, 2015, 2016–2017; Tallet, 2018). The present authors' ongoing work leads them to suggest that the management and positioning of smelting centres is partly determined by the availability and type of fuel. The case of Ayn Soukhna could represent a significant example of a new technology, implanted in a new location with respect to the raw materials required for smelting. This induced a change from the usual Old Kingdom *modus operandi* of mining and smelting in metallurgical camps in Sinai (e.g., Petrie and Currelly, 1906), leading the expeditions to carry out smelting on the Egyptian side of the Red Sea. Nonetheless, a substantial ore supply was assured at Ayn Soukhna through these expeditions. Without fuel, however, there can be no metallurgy; and the availability of fuel cannot be assumed in a desert environment.[5] The installation of a large-scale expedition at Ayn Soukhna could not be dependent on a random resource, meaning a careful management of fuel must have been organised as an integral part of the expeditions' organization and smelting technology.

As a comparison, the site of Wadi Gharandel (Sinai) features important remains of primary metallurgy of the Old and Middle Kingdoms. It was not directly associated with a copper mine, but it was a center of concentration that managed ores from various surrounding mines.[6] This characteristic is identical to the Ayn Soukhna site, where

[3] Precise quantitative data on vegetation cover are not available. This discussion will be the subject of a chapter in Verly and Rademakers, forthc.

[4] Since the Protodynastic period, Egyptian expeditions to the South Sinai mining areas were state organised ventures, ordered by the king and managed by the central administration in order to supply the vast building projects of the monarchy (Moreno Garcia, 2013; Tallet, 2018; Tallet et al. 2019). The organisation of such expeditions was not the sole responsibility of the central administration, but also depended on regional resources conscripted in the Nile Valley. One such expedition brought together up to 4000 people to extract turquoise and copper in mineral form.

[5] Currently, no palaeoclimatological data is available for the site. In 2020, the region of Ayn Soukhna appears as desert. Considering other palaeoclimatological data from the Nile Valley and the Western and Eastern Deserts as proxy, we will here speak of a semi-desert region during the Middle Kingdom. A geomorphological study of the site will be published in the future volume Ayn Soukhna V (Peulvast, forthc.). At Seh Nasb in Sinai, a complex of about 3000 furnaces (in battery layout) has been identified by Pierre Tallet's team (Tallet et al., 2011). The authors will propose a hypothesis linking the design of the furnaces and the type of fuel used in a future publication (Tallet and Verly, forthc.).

[6] Other examples of ore concentration and processing sites are present in the Sinai, such as Serabit el-Khadim and Wadi Maghara (Bauerman, 1869; Petrie, 1906; see also Rademakers et al., forthc.).

different ores were gathered for further processing. The choice to set up a center is also made according to another essential characteristic to enable metallurgical activities: the presence of water.[7] According to Bauerman (1869, p. 19) this proves "that the sites for smelting-works were determined chiefly by the presence of springs, where there is usually some quantity of wood […] *Acacia Vera*, which still flourishes round the springs". Another reason to conduct smelting in such locations is the need for water during certain phases of the smelting process: primary sorting, panning and cooling of copper, oxidized ores and slag, and production of technical ceramics (Tallet and Verly, in prep; Verly, in prep).

The most common wood species used as fuel in Pharaonic Egypt are *Acacia nilotica* and tamarisk in the Nile Valley (Boulos, 1999; Fahmy et al., 2008; Gerisch, 2007, 2010; Neumann et al., 2001; Janssen et al., 2003) and *Acacia tortilis/etbaica*, *Acacia ehrenbergiana*, *Acacia seyal* in the desert areas (Bailey and Avinoam, 1981[8]; Bouchaud et al., 2018, 2020; Boulos, 1999; Fagg and Stewart, 1994; Le Floc'h and Grouzis, 2003; Neumann et al., 2001). *Acacia nilotica* does not grow in the desert and its recurrent presence in the form of charcoal in archaeological contexts related to different metallurgical activities indicates the import of fuel from the Nile valley (Van der Veen and Tabinor, 2007, p. 107; Bouchaud et al., 2018). No detailed study is currently available for the copper workshops at Ayn Soukhna, but a charcoal sample was kindly identified on site as acacia (subgenus unknown) by Charlène Bouchaud (CNRS and Museum National d'Histoire Naturelle, UMR 7209). A detailed characterization of wood species at Ayn Soukhna in the future would illuminate the possibility of imports. At Timna (Israel), the "most dominant species in the assemblage were *Acacia* and *Retama raetam*, two main sources of wood that grow locally (…) but rejuvenate slowly" (Ben-Yosef 2018, p. 37).

The 'simplest possible' *chaîne opératoire* is proposed as a working hypothesis here: the use of local resources and the additional supply of fuel by donkey caravans integral to the expeditions. There were two main alternatives in antiquity for primary fuel: either green wood (cfr. Zschoke and Preuschen, 1932; Eibner, 1982, pp. 399-408) or charcoal produced from green wood. The production chain for green wood includes local collection or caravan supply, cutting and calibration for its use as fuel. The production chain for charcoal includes wood collection, cutting, charcoal making and calibration for fuel use – one or more steps could have taken place in the Nile Valley or at Ayn Soukhna. However, charcoal making consumes a lot of green wood, a relatively rare resource. In (semi-)desert conditions, charcoal production may present a resource management problem. A 'general' estimate of charcoal production from green wood is between 17% and 28% of the initial weight of wood and a volume yield of 60% (Corbion, 2016). With charcoal as the main fuel for primary metallurgy (e.g., Ben-Yosef, 2018; Hauptmann, 2007; Merkel, 1990 [although archaeological evidence for charcoal production is usually absent, cfr. Hauptmann, 2007]), this can present a significant bottleneck to large-scale operations (cfr. Burger, 2008, p. 72).[9]

Table 1

Comparison of two representative experiments (XP): XP-45 using charcoal and XP-49 using green wood. A fuel load of either charcoal or green wood corresponds to a volume of 8 l. A load of donkey dung (DD) corresponds to a volume of 3 l. To start the smelting, the furnace column is loaded from the top (1). When the furnace stabilises its consumption, it has reached its thermal inertia (2). It is at this point that it becomes operational. The first charges of ore can be loaded from the top (3). When the last charge is reduced, the fire is smothered (4) to save as much charcoal as possible. The charcoal (whether it comes from green wood or charcoal) is recovered for other operations (5). In terms of volume consumption, the 35 charcoal loads (45°XP) are roughly equivalent to the 28.5 green wood loads (49°XP). The difference is that the charcoal loads (45°XP) were produced by approximately 58 loads of initial wood volume, indicating a net consumption which is twice as high.

Correspondence table between fuel load/donkey dung (DD) load	XP-45 Malachite Maroc, Dunkey Dung, Charcoal	XP-49 Malachite Maroc, Donkey Dung, Green Chestnut Wood
1. Loading of the column	16 charcoal loads/ 0 DD loads	10 wood loads/0 DD loads
2. Inertia	5 charcoal loads/4 DD loads	6 wood loads/3 DD loads
3. Smelting process	16 charcoal loads/9 DD loads	19 wood loads/12 DD loads
4. Fire control	0 charcoal loads/7 DD loads	0 wood loads/6 DD loads
5. Charcoal recovery	− ***2 charcoal loads***	− ***4 charcoal loads*** (eq.: ca. 6.5 wood loads)
Net consumption	**35 charcoal loads**	**ca. 28.5 wood loads**

However, the eleven smelting operations including acacia wood and donkey dung as fuel out of a whole number of 15 experiments undertaken at Melle in 2016–2017 have shown that smelting can be successfully achieved with green wood (cfr. Gerisch, 2007, p. 172, on the use of green wood in glass working contexts; Espelund, 2014, p. 256, Larsen and Rundberget, 2014, p. 232, on the use of wood in the context of iron smelting). The finding of wood bark (Fig. 11) at Ayn Soukhna in sector S30 (US 331) also points to the use of green wood as fuel.[10] The advantages of green wood over charcoal (considering the number of furnaces) are: reduced manipulation (less complex *chaîne opératoire* and less labour intensive), the consumed volumes of green wood and charcoal are quite similar for a similar quantity of copper produced and consumption of fewer trees (charcoal production requires a lot of green wood, resulting in a net consumption that is around twice as high, see Table 1).[11] All this enables a lighter supply chain management when using green wood. It therefore makes sense to propose two experimental protocols: one using green wood and the other using charcoal.

Furthermore, it is important to take into account another fuel resource, which is present at Ayn Soukhna: the dung from donkeys, which can be considered "one of the most valuable resources in arid countries" (Lancelotti and Madella, 2012, p. 953). Indeed, the expeditions were accompanied by donkeys (Janssen et al., 2003; Tallet, 2067),

[7] The place name Ayn Soukhna means "hot spring". Indeed, there is a sulphur spring close to the site. Moreover, in order to collect rainwater from the surrounding mountains, a well was dug into the bedrock in area S21-South probably at the beginning of the Middle Kingdom.

[8] Bailey and Avinoam (1981, p. 145) note that: "Bedouin convention, moreover, prohibits the cutting down of desert trees and bushes, such as the various Acacia, Pistacia and Retarna, whose branches provide shade to the shepherdesses and firewood for the camp, and whose fruits provide fodder for the flocks. A bedouin proverb explains that "the cutting of trees (in a given region) will eliminate the possibility of living there."

[9] Burger (2008, p. 72): "La nature du combustible est très peu discutée dans le contexte de la métallurgie au Chalcolithique/Bronze Ancien. Il est en général admis qu'il s'agit de charbon de bois, mais sans preuve formelle." Zschoke and Preuschen (1932) and Eibner (1982, pp. 399-408) evoke the idea that the fuel could be green wood.

[10] In the context of glass, Gerisch (2007, p. 172) notes that "The question of whether fire wood or prefabricated charcoal served as fuel for the kilns cannot be answered easily. Looking at the recovered fuel material, the few remains of uncharred and incompletely charred wood and bark as well as the large volume of loose bark in feature [8995], Kiln 3 (glass production), could more likely be an argument for the use of wood."

[11] This paper is not the place to describe all 53 experiments in detail. They will be the subject of other chapters in Verly, in prep. The quantity of copper produced during each experiment varies according to the researchers' learning, the state of drying of the smelting furnace and the type of fuel used. The average production of non-oxidized copper prills is 770g from 5000g of ore. The authors insist that this does not represent an ancient reality but the result of learning on the part of the experimenters about how to manage both fuel and the furnace. They authors can thus still affirm that the ancient metallurgists had developed an efficient system with an operational performance that allowed them to maintain their model over time (balancing needs and efforts).

Fig. 2. Experimental furnaces at Aubechies: C for green wood, N for charcoal - Blocks of copper carbonate ore (from Morocco) – Loading the column with green wood blocks, fresh donkey dung and ore blocks © Georges Verly. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

the only pack animal of that time, to maintain a regular supply to the site. Donkey bones are also amongst the faunal material from the site. Donkey dung, the use of which as fuel is also known for other types of technologies (cfr. Sillar, 2000; Janssen, 2005), was thus a locally available combustible with suitable burning properties in comparison to cow dung or wood.[12] Donkey dung is known (in dry form) mainly for cooking food (see, e.g., Albert et al., 2008; Malleson, 2016, forthc.; Miller, 1996; Moens and Wetterstrom, 1988, p. 166; Peet and Woolley, 1923, p. 64; Shahack-Gross and Finkelstein, 2008; Shahack-Gross, 2011; Wilson, 1988, p. 53; Budka et al., 2019). For smelting, it can be used wet. Indeed, the addition of fresh dung to the furnace charge slows down wood/charcoal consumption, thereby increasing the time it takes for ore blocks to descend the column of the furnace (see below) and providing the metallurgist with a tool to control the furnace temperature. This has been shown experimentally to be a valid hypothesis, and confirmed by the latest excavation evidence, as discussed below. The protocol is reinforced by the presence of donkey dung heaps near the smelting centers on the Early Iron Age “Slaves’ Hill Plateau” at Timna, Site 34, Area G (Ben-Yosef, 2018, pp. 35-36). Therefore, the experimental protocols combine donkey dung with green wood on the one hand and charcoal on the other.

2. Experimental materials and methodology

Two experimental protocols are tested: the first uses green wood alternated with fresh donkey dung and the second uses charcoal alternated with fresh donkey dung. This makes it possible to describe the reactions specifically caused by each type of fuel to the furnace lining.

All available archaeological data is integrated in the implementation of the two experimental protocols in order to obtain results that can be validated against the archaeological evidence. Specifically, this implies an exact replica of the furnace architecture. The type of furnace, known only at Ayn Soukhna (Verly, 2017), was designed to meet a specific ancient *chaîne opératoire*, in relation to ore types coming from Sinai. The original Ayn Soukhna structure is composed of three to four L-shaped ovens which form a quadrangular block made of sandstone and sand. A furnace consists of a vertical 1.3 m high column, 0.3 m in diameter and a horizontal outlet of 0.7 m length. The column is protected by a lining (i.e., a thin layer of clay paste) which reacts according to the temperature, the presence of the ore and the oxygen concentrations. In order to be as close as possible to the ancient conditions, also the size of the ore blocks has to be similar to the archaeological specimens. This precaution also had to be followed for the primary sorting system and its working environment (Verly and Rademakers, forthc.). The choice of the experimental ore type also followed this line of reasoning in order to use an ore with characteristics similar to those of the Sinai ores (Rademakers et al., 2020). For the experiments conducted in Europe, it was not possible to obtain the exact same materials available on the Red Sea

[12] Cow dung fire is characterised by the release of much more smoke relative to that of wood fire. However, cow dung and wood fires are both capable of releasing a similar amount of heat (Braadbaart et al. 2012, p. 845). Experimental firing of clay figurines in a small dung-fuel kiln has demonstrated that cow dung fire can reach high temperatures: for example, over 1000 °C in 1h or 1095 °C in 1h and 20 min (Braadbaart et al., 2012; Lancelotti and Madella, 2012; Kenoyer, 1994).

littoral in Egypt to build the furnace and its lining. The furnace was thus built with sandstone and sand of a local variety. The lining was built up using plaques of clay tempered with sand. The observations made during the experiments focused not only on the nature of the lining, but also on its changing behaviour as a result of different temperatures, redox-conditions and smelting reactions. Previous experiments in Egypt (using the same silt deposits that were available at Ayn Soukhna in antiquity) have shown that with the correct fuel, a perfect match between experimental and archaeological linings can be obtained.

Over the last seven years, more than 50 experimental smelts in exact furnace replicas have been conducted in Egypt, France and Belgium. This has provided a detailed record of their operation under different conditions and a deeper understanding of the particular smelting process. The dialogue between the three disciplines of archaeology, experimental archaeology and archaeometry is crucial for the development and understanding of the *chaînes opératoires* (Verly, 2017). The results of the experiments are then validated against the only tangible reality: the archaeological data. The entire methodology has been fully described by Verly and Longelin (2019). Beyond understanding the ancient smelting process, another goal of the experiments is to improve the future excavation of similar structures.

The full protocol of the operating chain is described below in an individual section. In short, it comprises the loading of fuel, donkey dung and ore blocks through the upper furnace opening. In order to allow a uniform path through the furnace column, it is important that the size of the ore blocks and the fuel blocks are the same (Verly, 2017). The only difference between the two protocols is the use of either green wood or charcoal.

The markers to validate which type of fuel was used in antiquity are, in order of importance: the lining, the slag morphology and the resulting metallic copper production. The lining is the primary indicator because this layer is permanently in contact with the fuel at all column levels and directly exposed to the different furnace atmosphere conditions. The main argument to establish a specific *chaîne opératoire* derives from the colorimetry and vitrification zones of the lining. In addition, the (non–) vitrification of the reduced ore blocks provides an indicator for low-temperature smelting (around 900 °C). During the experiments, continuous temperature measurements in the furnace column are conducted. Eight thermocouples are placed at key points in the furnaces. These are defined based on the archaeological lining, which indicates the locations of different atmospheric conditions and areas of fuel or copper concentration through colorimetric variations and different degrees of firing/vitrification. Two identical furnaces were therefore constructed at the Archéosite d'Aubechies, an experimental archaeology platform in Hainaut, Belgium: 'furnace C' dedicated to green wood and 'furnace N' to charcoal (see Fig. 2). In line with the methodology and to ensure a complete reaction of the lining, the 'Wood protocol' was performed fifteen times in Furnace C and the 'Charcoal protocol' was performed fifteen times in Furnace N.

The latest archaeological research at Ayn Soukhna in 2020 has shown that the S39-area furnace linings are in a good state of preservation and only little affected by fire. As the internal stratigraphy of the workshop shows, the furnaces were used many times during two different campaigns. The non-vitrified appearance of the archaeological slag blocks equally indicates a relatively low-temperature reduction process. The first experiments undertaken at Ayn Soukhna in 2013 with local[13] materials procured from the surroundings did use charcoal without any fire control. This demonstrated that these furnaces could very easily produce a temperature well above 1200 °C (Rehder, 2000, p. 7),[14] impacting the lining to the point where it becomes incompatible with the archaeological originals. To arrive at similar thermal impacts in the experimental furnaces, the years of experimentation have shown that column temperatures should on average (a detailed breakdown is discussed below) not exceed 900 °C. Otherwise the vitrification zones are too large, the lining is too damaged and a liquid slag is formed: the experimental data are no longer validated by archaeological evidence.

As far as fuel is concerned, *Acacia nilotica* would be preferable to mimic the ancient process as closely as possible. However, it is impossible to obtain this species in Belgium or in France. Since wood with the exact same characteristics (*Acacia nilotica* has a calorific value [CV] of ca. 6130 kcal/kg and a moisture content [MC] of ca. 5.6% [Chakradhari and Patel, 2016; Telmo and Lousada, 2011; Todaro et al., 2015]) could not be obtained in Belgium, two species of dense wood, easy to obtain in larger quantities, were chosen: chestnut (*Castanea sativa*, CV of ca. 4480 kcal/kg and MC of ca. 11%) and ash (*Fraxinus angustifolia* and *excelsior*, CV of ca. 4500–4600 kcal/kg and MC of ca. 7–11%). It has to be kept in mind, however, that acacia has a higher calorific value and lower moisture content, which could result in somewhat different furnace conditions during combustion. The small number of specific archaeological contexts on the one hand and the small number of charcoal fragments studied in some of these contexts on the other hand call for caution, but nevertheless allow the idea of fuel selection in the context of artisanal activities related to metalworking (acacia) and brick making (mangrove wood), requiring particular control of the intensity and duration of combustion (Bouchaud et al., 2018).

For the experiments, 100 kg of carbonate copper ore was obtained from a single mining gallery (Touissit-Bou Beker district, Jerada Province, Morocco). Obtaining the quantities necessary for this experimental work from Sinai deposits was practically impossible. The ore was selected to mimic the ores smelted at Ayn Soukhna, but cannot be taken as entirely representative. Nonetheless, this provides a valid alternative to test the behavior of ore blocks during the smelting process and a valuable way to assess the geochemical changes taking place from ore to copper metal (Rademakers et al., 2020).

3. Donkey dung as smelting fuel: Experimental indicators

The earliest experimental reconstructions of the Ayn Soukhna process always had the problem of overheating. Switching from charcoal to green wood helped a little to lower the temperature and slowed down the combustion (cfr. Gerisch, 2007, p. 172). This slower burn time increased the reaction time for ore blocks in the column. Green wood[15] alone, however, turned out to be insufficient for stabilizing the archaeologically observed 'low temperatures', so a further thermal regulator was needed. Based on a suggestion by Angelo Bartoli, an Italian metallurgist who maintains the tradition to use wood and donkey dung, and in line with the achaeological data and inscriptions attesting to the presence of donkeys at Ayn Soukhna, the use of their dung in its fresh form was attempted. Compared to wood, fresh dung is a poor fuel: by itself, it does not burn. It produces a lot of ammonia smoke and water vapor. It forms a plug that prevents air from circulating through the column. Combined with wood, however, these characteristics are actually excellent. The layers of fresh donkey dung form airlocks in the column, which are effective thermal regulators.

In order to test the fresh donkey dung hypothesis, a further series of

[13] The materials used were taken from outside the archaeological area, from the mountain. They are therefore similar to ancient materials.

[14] Rehder (2000, p.7): "In kilns, furnaces, or hearths, which allow an artificial ventilation, i.e., air supply by blow pipes or bellows, the use of wind or the chimney effect, wood fires and charcoal fires could reach temperatures of about 1.400°C and 1.600°C, respectively."

[15] Gerisch (2007, p. 172): "Carbonisation processes occur also as transition stages in the combustion of wood. Because of the remaining water, the temperature of this combustion is connected with lower values."

Table 2
Comparison of smelting results with or without the use of donkey dung (summary).

Features	14-XP Malachite, Green Acacia	15-XP Malachite, Green Acacia, Fresh Donkey Dung
Column temperature	1100 °C	900 °C
Smelting time	5 h45	7 h15 (slower descent of ore blocks)
Atmospheric condition	Upper part: low in oxygen with dense fumes	Upper part: low in oxygen with even denser fumes
Vitrification	More impacted and more vitrified lining	Lining in accordance with archaeological data
Type of slag	Reduced blocks with a substantial part of the blocks not reduced[22]	Increase in reduced blocks and clear decrease in blocks in cuprite stage
Production (at equal ore load)	600 g of copper metal	760 g of copper metal (20% increase)
Type of copper produced	Heterogeneous production: oxidized copper prills, oxidized run-out and copper prills	Homogeneous production in the form of copper prills (oxide-free)

[22]Some blocks seemed denser by hand, so they were checked by breaking. The ore was not yet fully reduced, having progressed only to the formation of cuprite

experiments was conducted under the same conditions with the additional use of donkey dung as the only variable. The results for two experiments are summarized in Table 2, representing fifteen comparative smelts. The results obtained without the use of donkey dung differed systematically from the process traces observed in archaeological remains.

These results highlight the importance of donkey dung as a fuel regulator. To arrive at similar thermal impacts in the experimental furnaces, column temperatures should on average not exceed 900 °C, which is therefore the temperature regime which is voluntarily aimed at. Donkey dung is the only known material present on the site that allows control over the column temperature due to its size and shape (cfr. Matin and Matin, 2016, p. 126, the size and the shape of the pieces of fuel in the fuel bed have an important effect on the rate of combustion) and its slow burning characteristics (cfr. Lancelotti and Madella, 2012, p. 961). Without control, the chimney effect (despite the closed space of the workshop thanks to the walls) can easily reach 1300 °C, a temperature that was even higher than the 1200 °C reached in experiments at Faynan reducing oxidised copper in an artificially ventilated charcoal fire (Kölschbach et al., 2000, pp. 5-22; Burger, 2008, p. 73). The fresh donkey dung further stabilizes the descent of the fuel and increases the process time, as was observed by Matin and Matin (2016, p. 126) concerning the use of cow dung as fuel. This appears important in allowing the blocks to have more time to react during the different stages of reduction.

4. Summary of the smelting process

The Ayn Soukhna copper smelting furnaces are unique in their design and operation (see Fig. 3), differing strongly from Middle Kingdom furnaces at Seh Nasb (Tallet et al., 2011). The workshops are walled to eliminate natural wind variations and roofed to be in the dark in order to visually better control the heat (Verly, 2017). The furnace is loaded from the top with fuel and ignited at the bottom. The particular furnace shape creates a structural chimney effect, resulting in a bottom-to-top draught within the furnace. This enables the fuel to burn without any forced ventilation, thus without requiring *tuyères*, bellows or wind.

Alternating layers of wood and donkey dung are loaded from the top of the furnace and descend into the column as fuel is consumed. This alternating pattern allows for temperature regulation, without which unrealistic furnace conditions are obtained (cfr. above). When conditions have stabilized (i.e., the furnace has reached its thermal inertia), ore blocks can be loaded into the top of the furnace between these alternating layers and descend along with the fuel as it is consumed. This descent is summarized in Fig. 2. In the upper 'Yellow zone', the ore is first 'grilled' (a calcination process whereby malachite alters to cuprite

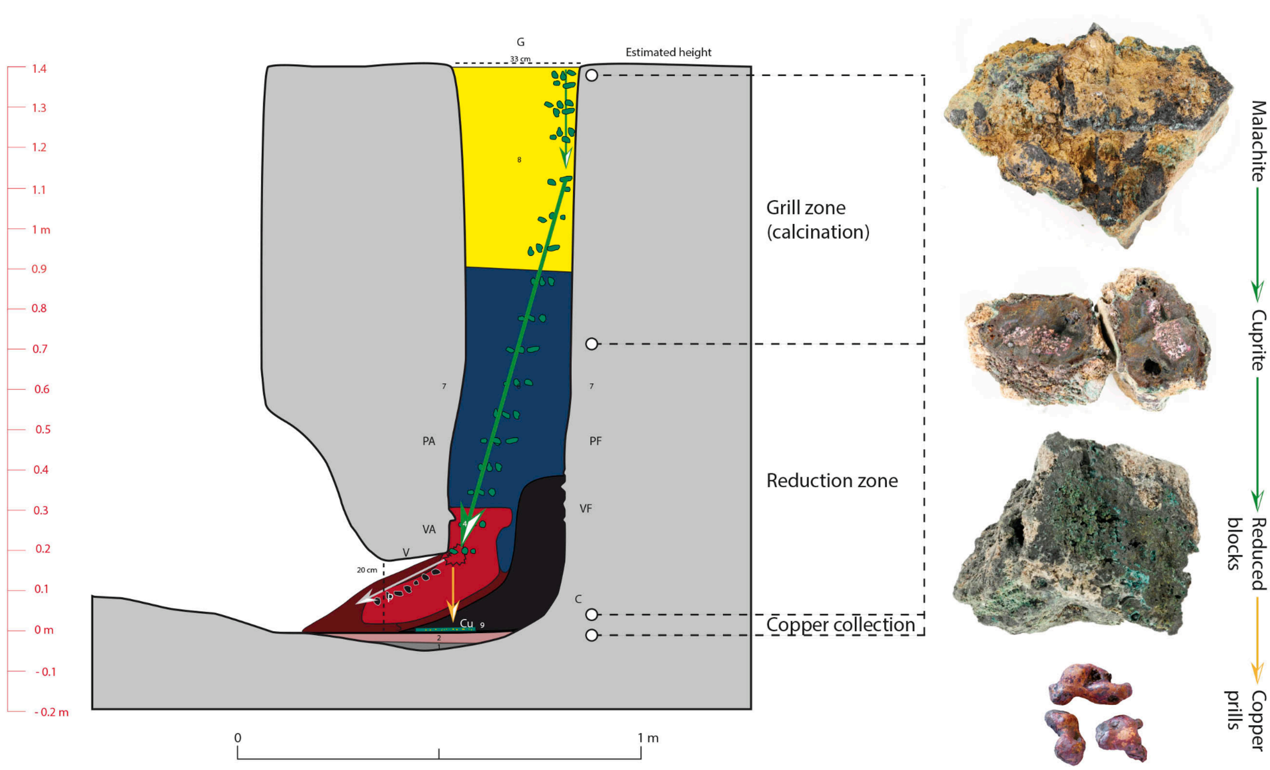

Fig. 3. Ore trajectory in the column. Transformation of ore blocks into reduced blocks and copper prills © Georges Verly and Frederik Rademakers.

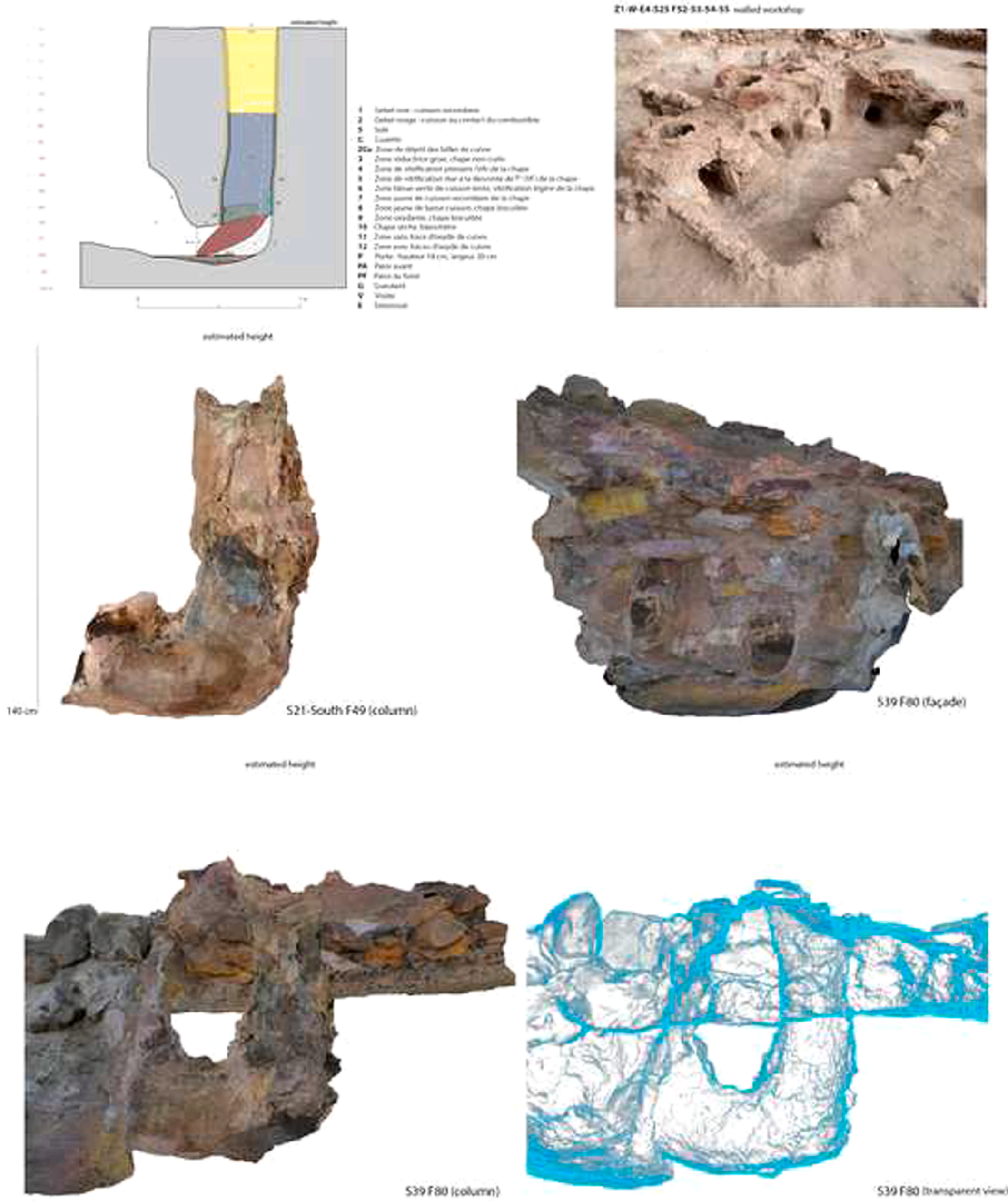

Fig. 4. The archaeological lining is visible thanks to the photogrammetry, e.g., in S21-South F49 (column) - S39 F80 column-façade-photogrammetry (internal view of the column) - S25 F52-55 (workshop view) (for the archaeological description see Verly and Rademakers, forthc.) © Georges Verly and Ayn Soukhna archaeological mission.

[cfr. Rademakers et al., 2020]).[16] This furnace zone is characterized by relatively low temperatures of ca. 100 °C. This is the area where all the fumes are concentrated, creating an anaerobic atmosphere full of ammonia and combustion fumes. It is characterised by a complete absence of flames and high humidity. The lining is lightly to barely fired; it changes from white (the original colour of the clay paste which derives from accumulated deposits formed by erosion and transport through water flowing from the nearby mountains) to yellow. As the ore descends further, it enters a 'Reduction zone', characterized by temperatures of ca. 700 °C. In this 'Blue zone', cuprite is reduced into copper (cfr. Budd, 1991; Coghlan, 1942) under a reducing atmosphere[17], yet the ore blocks remain solid. The lining turns hard and solid and gradually changes from yellow to blue/dark blue. Next, the ore blocks reach the hottest zone of the furnace ('Light Red zone'[18]), situated just above the furnace mouth, where temperatures up to 1000 °C are measured. The 'Light Red zone' has a very oxygen-rich atmosphere, since it is the only

[16] Furthermore, incomplete reduction is often observed in both experimental and archaeological blocks, revealing these intermediate steps from carbonate to metal.

[17] Increasing the air flow rate accelerates the combustion of the fuel according to the reaction: $C+O_2 = CO_2$. This reaction is exothermic (free enthalpy ΔG_0 (1200°C) = −93905 cal) and leads both to an increase in temperature in the 'Light Red zone' and a decrease in the partial pressure of oxygen in the 'Blue zone' (Burger, 2008, p. 72).

[18] It's the only place where the wood/charcoal is combusted in the direct presence of fresh air.

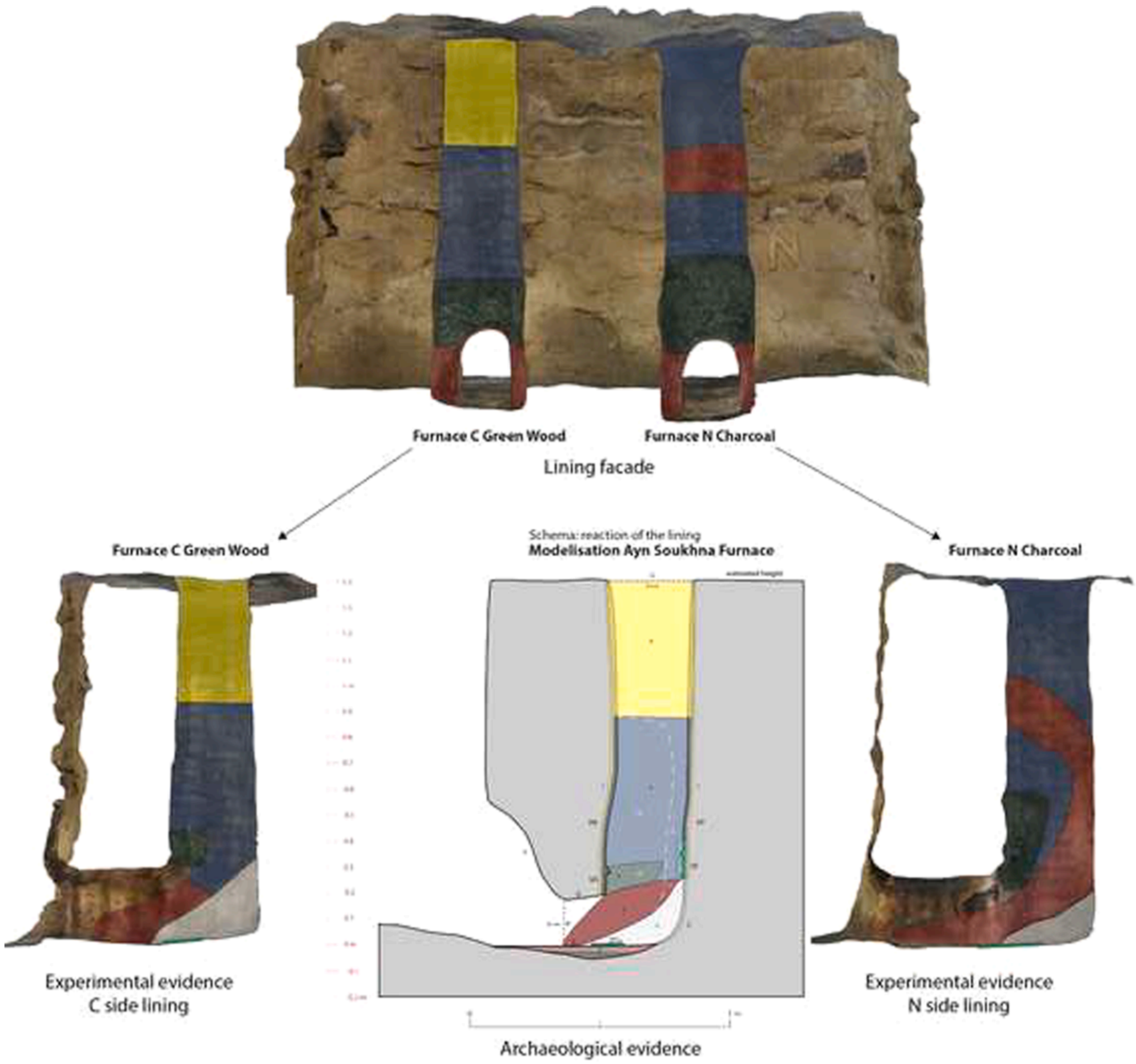

Fig. 5. Colorimetry and vitrification of the experimental furnace lining at Aubechies © Georges Verly.

zone in which wood/charcoal is combusted in the direct presence of fresh air. Here, metallic copper is 'drained' from the ore blocks: the prills drop down vertically through the fuel layer, solidifying as individual prills on the way down to the ash-filled furnace bottom (from 1000 °C to 900 °C just below the 'Light Red zone' to only 70 °C at the bottom 'Grey zone' [see below]). The lining in the 'Light Red zone' becomes gradually vitrified over multiple smelting runs, particularly above the furnace mouth ('VA') and horizontally on both sides ('VL'). The vitrification gets dark blue. Finally, the reduced ore blocks 'roll' over the remaining layer of ashes and charcoal accumulated at the furnace's rear bottom part, the so-called 'Grey zone', where they cool down in the 'Dark Red zone' within an oxidising atmosphere at ca. 250 °C. There, the lining is again physically intact (not vitrified), fired and oxidized red in colour. It can be remarked that, in contrast with many smelting technologies attested in other archaeological contexts, no liquid slag is thus produced in this process: 'reduced ore blocks' (hereafter called 'slag blocks', see Fig. 8) are the primary waste product. In the 'Grey zone', the most anaerobic atmosphere in which not only the straw and twigs used to kindle the fire but also the firewood itself do not burn (it is only slightly carbonised), the newly formed copper prills are collected as they fall to the furnace floor (in a layer of ash at ca. 70 °C throughout the process). The lining is dark as a result of carbon penetration. It is very brittle and often missing from the archaeological remains (see Fig. 4).

5. Results and discussion

5.1. Validation: Furnace lining

At the end of the experiments, the two experimental furnaces at Aubechies were excavated and recorded as archaeological specimens. The study of the linings, after fifteen experimental smelts in each furnace, made it possible to delimit zones along the furnace column (see above). These are mainly indicated by colorimetry and the degree of vitrification of the lining, as illustrated in Fig. 5. Furnace C (wood + donkey dung) shows the same zones, practically at the same locations. The vitrification zone above the bottom opening (VA) and on both sides (VL) is still in formation. This illustrates how these can be reused many times without the need for repair, if used properly. The side vitrification (VL) extends horizontally, reaching the back side of the column only after continued use (over 20 smelts). The horizontal position of the VL confirms the effect of combustion regulation by the accumulation of fresh layers of donkey dung. Indeed, the heat cannot rise vertically through the fuel and ends up stagnating at its maximum in this area. Furnace N (charcoal + donkey dung), on the other hand, does not exhibit the thermal impact seen in the archaeological remains (Fig. 4). The 'Yellow zone' is totally absent due to the nature of the fuel. Charcoal concentrates more heat. According to Rehder (1999), the nature of the fuel influences both the adiabatic temperature and the

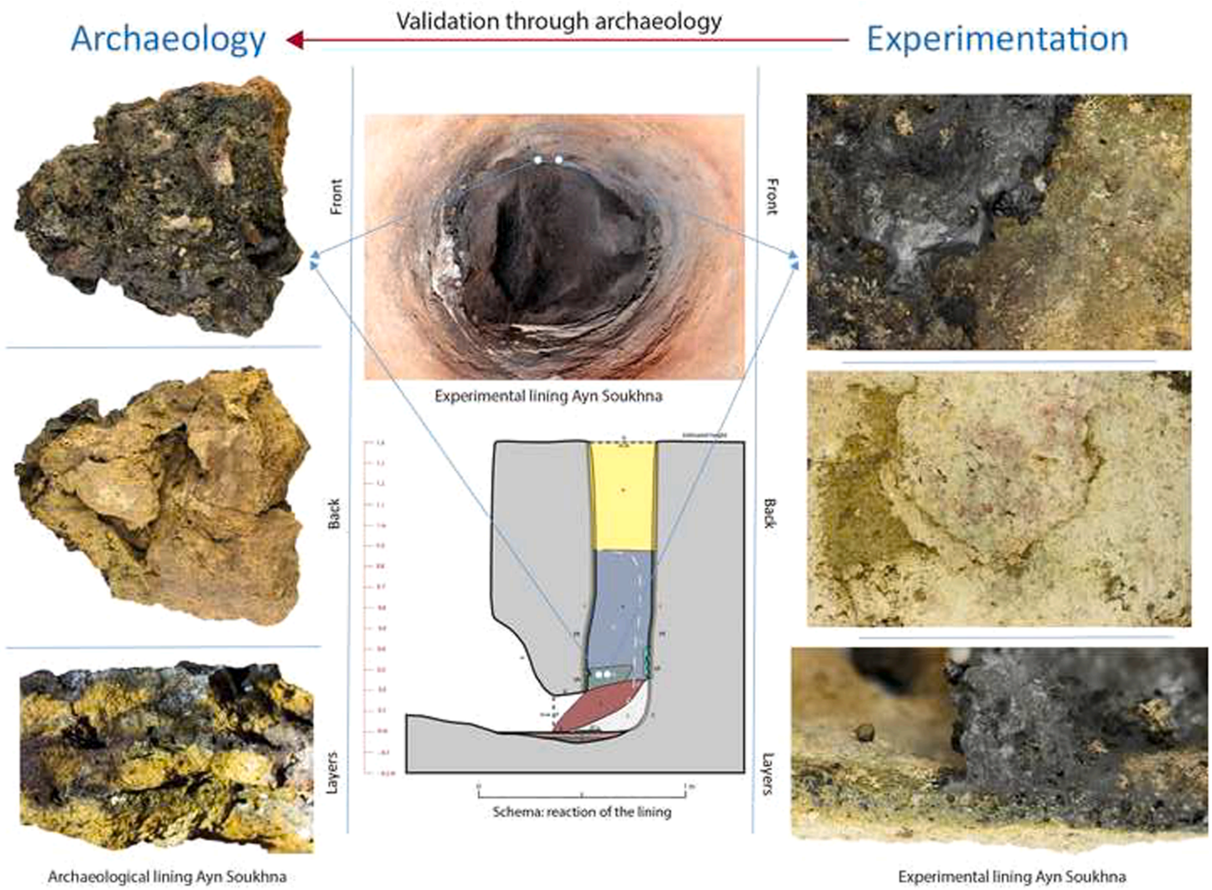

Fig. 6. Validation through archaeology: the experimental lining reacts exactly like the archaeological specimens. The experimental lining materials are made from a clay paste similar to the archaeological lining © Georges Verly.

distribution of the atmospheres. The heat, despite the addition of layers of donkey dung, manages to pass through and to fire the lining at the top of the column ('Yellow zone'). The 'Dark Red oxidation zone' rises completely through the 'Blue reducing zone'. Finally, the VL zone is smaller while VA is much larger and rises up along the column, in line with the heat distribution. Finally, the VA zone is very strongly vitrified.

In 2018, new experiments were carried out at Ayn Soukhna with locally available and thus more similar materials. When the furnaces are used at low temperatures with green wood and donkey dung, the lining reacts exactly as represented by the archaeological specimens (see Fig. 6). The vitrification zone is located in the same place with the same limited area. The colorimetry conforms in all points throughout the entire column. Finally, the type of reduced blocks is also equivalent to the ancient ones. This corresponds with the conclusions obtained from the experiments at Aubechies. It has thus been possible to validate the protocols solely on the basis of the ancient archaeological data, as required by the authors' methodology.

The difference between furnaces C and N can be better understood through the temperature profiles collected by eight thermocouples (see Fig. 7; for a better readability, only the significant curves have been selected). Overall, experiment XP-49 (green chestnut wood) is more stable than experiment XP-45 (charcoal) and the thermal curves of XP-49 are lower than those of XP-45, explaining the differences in reaction between the linings. For experiment XP-45, the 'Blue zone' is almost as hot as the 'Light Red zone' (*Front vitrification VA line*) and the strongly oxidizing environment (deduced from the furnace lining) no longer allows for cuprite reduction. The 'Dark Red zone' (*Back VF line*) exceeds 500 °C,[19] allowing this 'Dark red oxidation zone' to pass through the column. The 'Yellow zone' (*Column line*) disappears, because the temperature exceeds 300 °C. Finally, the *Front vitrification line* is close to 900–1000 °C, explaining the slagging of the ore blocks and the vitrification of the lining.

A note should be made here to explain the 'draining' of copper prills from the slag blocks. The minimum required temperature to melt copper is ca. 1084 °C. This can be lower for copper alloys, but analysis of experimentally produced copper prills (Rademakers et al., 2020) and prills retrieved at Ayn Soukhna (on site pXRF analysis, January 2019 field season) reveals the production of highly pure raw copper. The discrepancy between the theoretical melting temperature and those observed should thus be explained. One possibility is that the temperatures at the core of the furnace column are higher than those measured by the thermocouple, which is located right next to the furnace lining at a distance of ca. 2 cm. The extended duration of the process may play a role as well.

5.2. Validation: Ash deposits

Furnace F63 in sector S38-North is the only archaeological witness that has its reduced blocks (US [Unité Stratigraphique] 102) still *in situ* (see Fig. 8). This exceptional structure, which has been re-lined over time and is positioned on US 106, a man-made protection layer laid out during two expedition campaigns, confirms the experimental chain of operation that has been proposed. Firstly, these experiments produce reduced blocks located in the same place as the confined unit US 102.

[19] It is almost at the same temperature as the *Cuprite line* in Experiment 49.

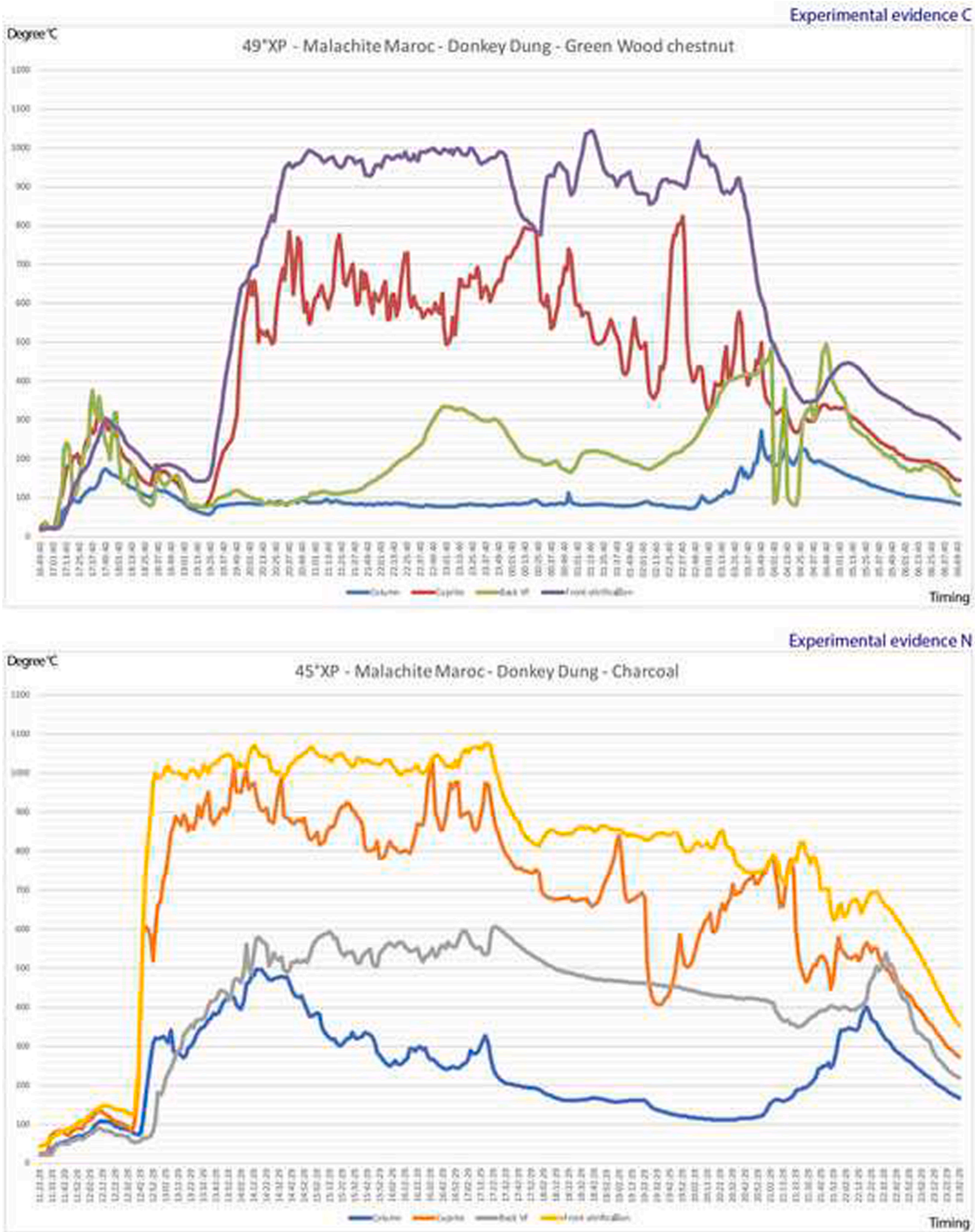

Fig. 7. Temperature diagrams of chestnut furnace C and charcoal furnace N. Experimental data from XP-45 and XP-49 © Georges Verly.

Secondly, no blocks are vitrified or fused together with other blocks. Thirdly, there is no production of liquid slag. Interestingly, this group of reduced blocks exhibits no charcoal residue between them *in situ*, suggesting that the combustion process was not interrupted because the fuel is converted into ashes (cfr. Gerisch, 2007, p. 172). This intriguing observation highlights another possible relation between the type of ancient fuel used and the resulting waste. At the time of excavation, some charcoal elements were still in place at the back of the furnace bottom. The layers under the slag blocks (such as US 104, 109 and 113), in contrast, were very ashy with small charcoal residues, suggesting that green wood was used as fuel and not charcoal (cfr. the observations of Matin and Matin, 2016, p. 127).

Based on this evidence, the experimental smelts were carefully investigated.[20] This yielded some interesting results, summarised in Fig. 9. In the case of smelting with charcoal and donkey dung (furnace N), the products and by-products do not correspond with the

[20] Instead of a hot extraction of the products and by-products from the furnace (which allows the easiest separation between metal and waste, cfr. Verly, in prep.), the fuel column was left to be consumed completely in order to reproduce the situation encountered in the excavated examples (e.g., F63 S38-North).

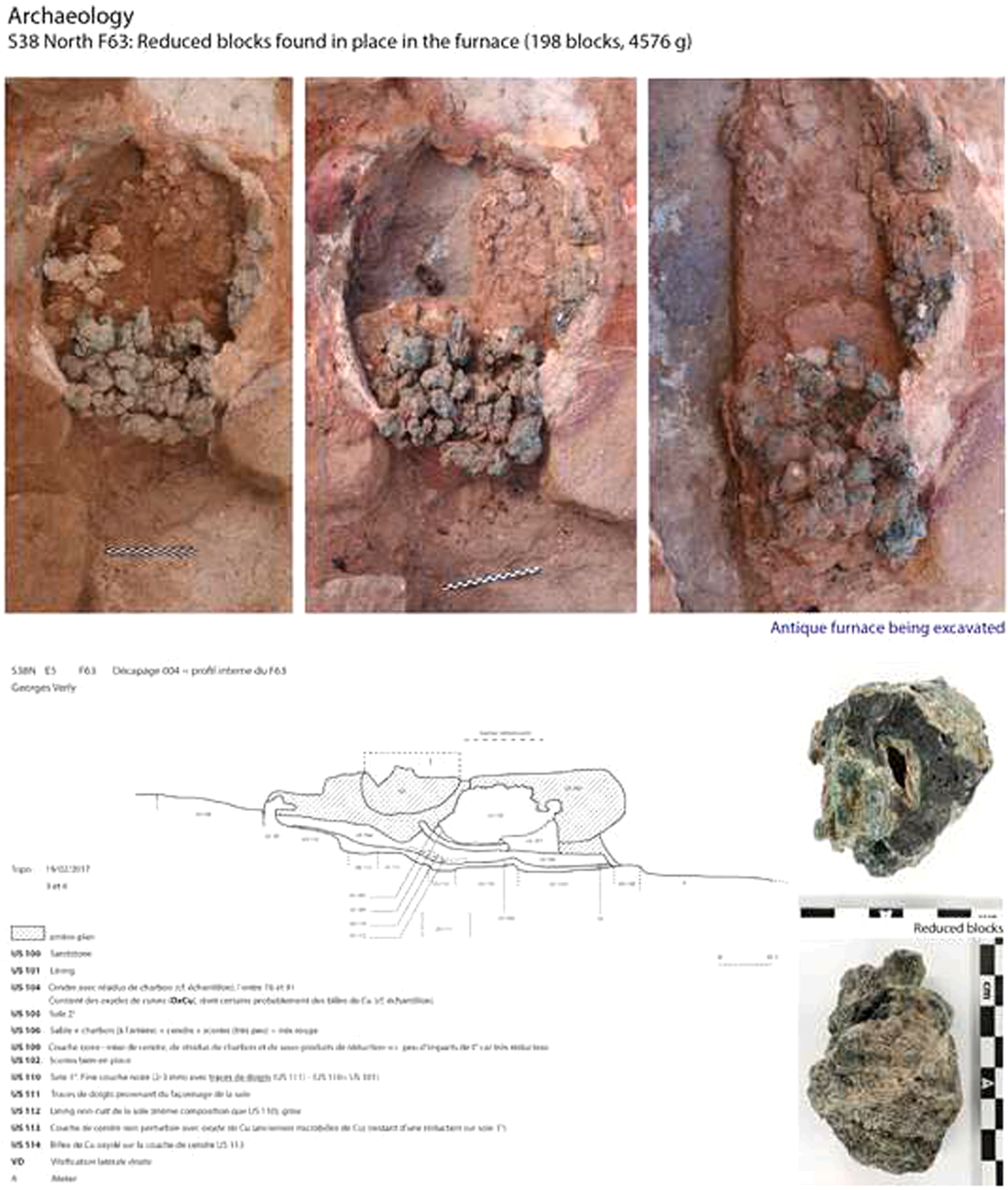

Fig. 8. Reduced blocks found *in situ* in furnace F63 S38-North (for the archaeological description see Verly and Rademakers, forthc.) © Georges Verly and Adeline Bats.

archaeological data. A partially oxidized copper cake is produced instead of only copper prills preserved in the ash. Much of the copper is either oxidized or trapped in liquid slag, which in both cases represents a loss of production, since there is no evidence at Ayn Soukhna that copper prills were mechanically extracted from the slag blocks. A compact block of fused slag completely blocks the lower opening. It is suspended from VA and rests on the furnace bottom. The lining has melted completely, obstructing the lower opening and is mixed with the liquid slag. The lining had to be removed and re-applied. Concerning the fuels, the whole surface of the furnace floor is covered with ashes and large pieces of charcoal. Finally, there are charcoal fragments between the blocks of liquid slag. None of this could be validated with regard to the archaeological data. On the contrary, smelting with green wood and donkey dung (furnace C) results in remains that can be validated by the discovery of F63 (S38-North). Reduced blocks are produced and are deposited in the same place as observed in this furnace. The copper prills are deposited in a bed of reducing ash, protecting them from oxidation. The lower opening is not blocked. The lining is operational for a new (in this case sixteenth) smelting. As far as the fuel is concerned, there is no charcoal between the blocks, but a thick layer of ash at their base. At the back, there are still fragments of charcoal. All the observations made in furnace C are in line with the archaeological evidence.

5.3. Validation: Ore blocks

Thanks to the rigid application of the experimental protocol, the teleology (Verly and Longelin, 2019) of each element could be successfully verified. In addition, a further validation of the results above

Fig. 9. Excavation and survey of the products and by-products of the two types of fuel in the experimental furnaces, in comparison with the archaeological furnace F63 S38-North © Georges Verly.

was gained through the study of the ore blocks (see Fig. 10). The first validation was obtained by comparison of the experimental and archaeological blocks, which shows that only the reduced blocks from furnace C are in conformity with the ancient reduced blocks (there is no layer of green neo-mineralization on the surface of the experimental blocks, which has formed over time on the archaeological blocks). The surface is rough with still sharp edges (as evidence of ore extraction and/or calibration). The copper prills have escaped through the natural layers of copper carbonate and gangue, and along cracks formed in the ore blocks during heating. On the other hand, the slag from furnace N is inconsistent with the archaeological evidence: its surface is vitrified, often encapsulating oxidized copper prills. The second validation was obtained by voluntarily letting the temperature rise during smelting experiment XP-29. As such, the experiment was deliberately left to fail to see the difference in the behaviour of the same batch of ores with the right and the wrong temperature, by no longer putting on layers of donkey dung during the smelting process. The first blocks, taken out at a low temperature of 900 °C, are in conformity with the archaeological data (with donkey dung layers), whereas the gangue of the same ore liquefies if the temperature rises without control to 1200 °C. This experiment attests to the use of fresh donkey dung as a temperature regulator. In the current state of research, no other ancient regulator such as, e.g., water (the addition of which was tested, however, the experimenters were not able to maintain a stable temperature curve, while huge amounts of water were necessary to extinguish a column in full smelting) can be suggested for Ayn Soukhna. Another strong argument in favour of fresh donkey dung is that the use of this type of dung increases the percentage of copper prill production by 15–20% and the copper quality, using the same type of copper ore (see Table 2).

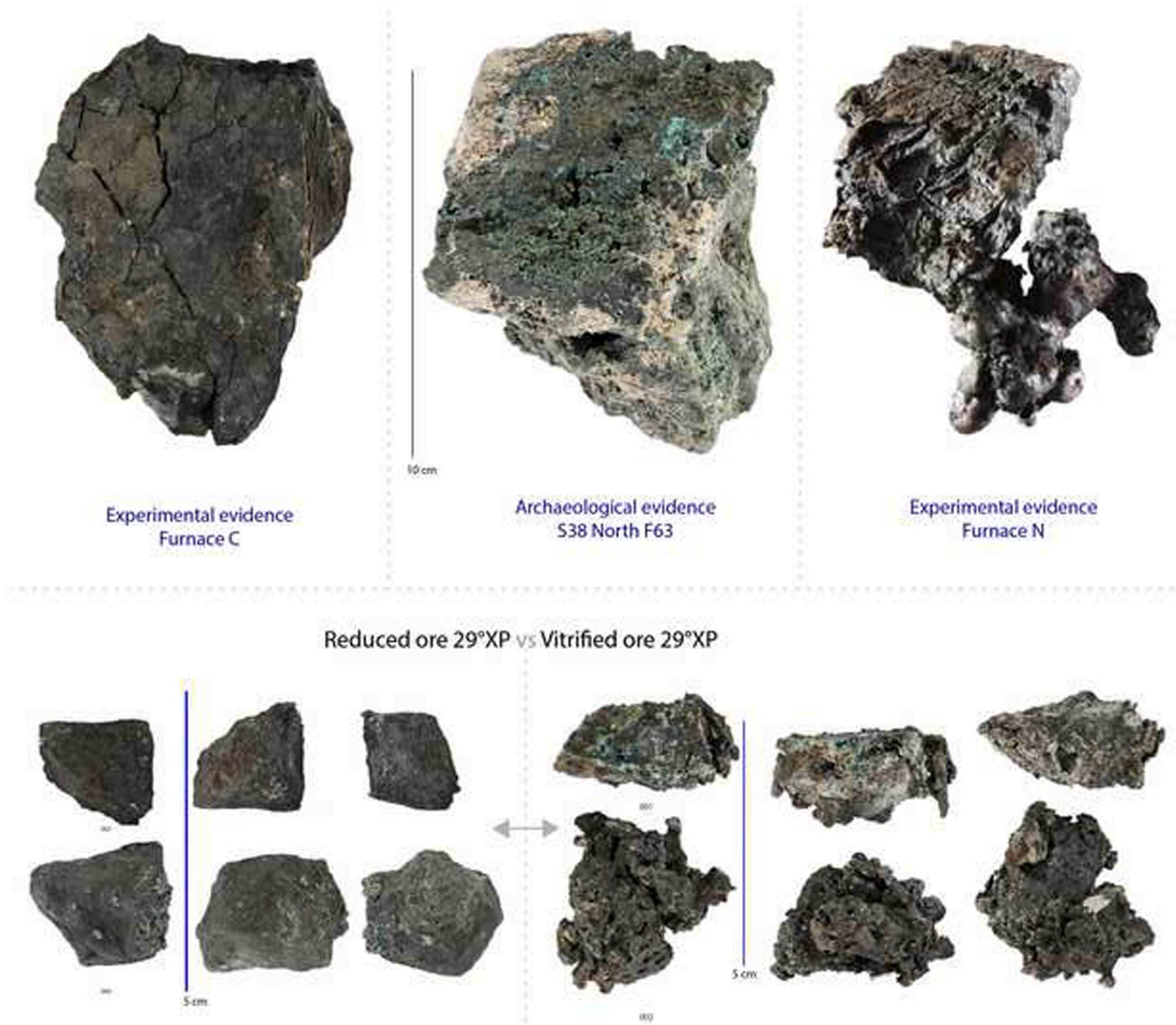

Fig. 10. Experimental data validation process using archaeological artefacts: comparison between types of reduced blocks © Georges Verly.

5.4. *Improved interpretation of excavation data*

The interest in practicing experimental archaeology is to be able to better excavate complex areas such as the workshops of Ayn Soukhna. This results in a dialogue that enriches both the excavation and the experimental protocols. In order to maintain a scientific approach, attempts are made to falsify the working hypothesis, and strengthening it by accumulating experimental and archaeological evidence. The excavations of 2020 present the latest exponent of this methodology. They have confirmed earlier observations and experiments with two further archaeological findings discovered in the waste layers of sector S39, directly related to the smelting activity (see Fig. 11), guided by experience from the experiments. As a first example, small spheres, weighing only a few grams and quite fragile and porous, regularly appear during the experimental primary sorting[21]. They derive from the heating and vitrification of donkey dung. Such spheres were recovered during the excavation of the metallurgical waste from sector S39 (US 306–309) in 2020. Without experimentation, it would have been impossible to identify and understand these spheres as proof of the use of donkey dung in the smelting phase, which is crucial for the understanding and establishment of a specific operating chain at Ayn Soukhna.

As a second example, the hot extraction of the experimental material made it possible to observe an interesting phenomenon during the experiments. The wooden blocks at the back of the column, its most reductive part, can sometimes come out intact and unburned (see above). The primary sorting of smelting products – probably at a high temperature (ca. 800 °C) and thus imposing an uncomfortable body management (Verly, in prep) – carried out on a flat stone with water, allows to save the carbonised wood (charcoal) as well as the intact blocks. A similar finding has been observed in the archaeological metallurgical waste from sector S39 (US 331–309): an antique fuel mixture composed of dry and carbonised wood. Finally, at the bottom of the sector S39 reduction furnaces, fragile balls of fibers were found in a mixture of rubbish. These balls were not burnt and are suspected to be dried donkey dung. These finds again corroborate the working hypothesis, established and repeatedly tested through experimentation: the use of green wood blocks as fuel, along with donkey dung, for smelting copper at Ayn Soukhna.

6. Conclusions

Green wood creates a favourable atmosphere for the smelting of copper carbonate ore. It heats the column at lower temperatures, allowing furnaces to be reused over several campaigns. The dual fuel use

[21] Due to lack of space, the entire smelting *chaîne opératoire* cannot be discussed here. After smelting in the furnace column, two sorting phases take place in order to recover all the copper prills. This hypothesis is based on excavation data (Verly, in prep.).

Fig. 11. Experimental data validation process using archaeological artefacts: thanks to experimental replication, it has been possible to identify, for the first time, vitrified ancient dung (a product in smelting) - presence of a mixture of dry wood and charcoal in the antique smelting waste © Georges Verly and Sara Zaia.

of *green wood and donkey dung* has resulted in the best agreement between experimental smelting results and corresponding archaeological evidence. A holistic evaluation of experimental and archaeological evidence has thus shown that a combination of fresh wood and donkey dung was most likely the standard fuel choice at early Middle Kingdom Ayn Soukhna. This is evidenced by the compatibility of results from experimental smelting using wood and donkey dung in terms of furnace lining impact and colorimetry, slag typology and deposition, ash and charcoal distribution, and raw copper shape and size. The use of charcoal systematically results in incompatible smelting results, as does smelting without donkey dung. In addition to green wood, donkey dung is required to regulate the temperature distribution inside the furnaces and to increase the production of raw copper prills.

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