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Characterization of Tuyères from Lekie Division, Centre Cameroon (West Central Africa): raw materials and ironworking processes[☆]

Zoila Luz Epossi Ntah [a,c,*], Giuseppe Cultrone [b], Thomas Rose [c]

[a] University of Yaounde 1, Department of Arts and Archaeology, Faculty of Arts, Letters and Social Sciences/ Cameroon, P.O Box 755, Yaounde, Cameroon
[b] Department of Mineralogy and Petrology, Faculty of Sciences, University of Granada, Avenida Fuentenueva, s/n18002, Granada, Spain
[c] Forschungsbereich Archäometallurgie, Leibniz-Forschungsmuseum für Georessourcen/Deutsches Bergbau-Museum Bochum, Am Bergbaumuseum 31, 44791 Bochum, Germany

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ABSTRACT

This paper is focused on the characterization of twelve fragments of tuyères collected from Evian and Pongsolo ironworking sites in Lekie, central Cameroon (15–19th century CE) with the aim of understanding the nature of their raw materials and the temperature reached during the ironworking processes. The analytical methods used for the study were X-ray fluorescence, X-ray diffraction, polarized optical microscopy, hydric tests and colorimetry. The studied samples were produced from silica-alumina rich clay materials with variable amounts of iron oxide, lower in Evian and higher in Pongsolo which explain their different colour (greyish in Evian and reddish in Pongsolo). The chemical correlation between the tuyères and the clays from Lekie suggested a local production of the tuyères using the clays from Lekie. Mineralogical and microstructural variations were observed in the samples from the two sites, due to the differences in temperature during ironworking or firing, and the position of the sample within the tuyère. The mineralogy of the fired clays was used to infer the temperatures the samples were exposed to in the furnace, indicating 1100 °C and 1200 °C as the peak temperatures recorded in the tuyères found in Pongsolo and Evian, respectively. The high content of quartz in the tuyères and their low amount of flux oxides indicates their refractoriness during ironworking process. Similarities in terms of typology and the characteristics of the raw materials have been observed in the tuyères from other African countries, indicating the use of a bellows-operated furnace during ironworking processes in Lekie.

1. Introduction and archaeological background

The Lekie Division is located in the forest part of Central Africa, in the south-western part of the Central region of Cameroon (Fig. 1) between 3°34′ and 4°30′ latitude North; 11°10′ to 11°45′ longitude East. Lekie is among the most extensively surveyed archaeological regions in southern Cameroon, with multiple excavations conducted since the 1980s (Essomba, 1983a, 1983b; Elouga 1985).

Metallurgical artefacts such as slag, tuyères and metal objects associated with pottery have been found at many archaeological sites in the Lekie Division of Cameroon: Nkometou/Mfomakap, Pongsolo, Evian, Efok, Elig Zogo, Okok, Nkang, Lekekoua Nkol-Ngok, Mebomo, Oban I (Fig. 1), attesting to an iron production industry in this region since 3000 BP (Essomba 1992; Elouga 2000). These sites have been divided into three groups according to the activities carried out at the sites. Ore mining sites (Okok, Mebomo), iron smelting sites (Mfomakap, Pongsolo, Evian, Nkang, Elig Zogo, Lekekoua), and iron smithing sites (Efok, Mfomakap, Nkol-Ngok, Oban II). Archaeological excavations have been carried out at the sites of Pongsolo (4°07′ N, 11°17′ E), Evian (4°09′ N, 11°23′ E) and Mfomakap (4°01′ N, 11°33′ E) (Essomba 1992; Elouga 2000). The distance between Pongsolo and Evian sites is between 10 and 15 km.

Excavations began at Pongsolo in 1984–1985, where slag and tuyeres were found. Tuyères are pipes used in the furnace to allow pressing air into the furnace during metallurgical processes. Radiocarbon dating carried out by Essomba (1992) on charcoal samples collected in Pongsolo at depths of 40 cm and 60 cm yielded two dates: 100 ± 60 BP (area 1), corresponding to the period 1660 CE (17th century), and 222 ± 70

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[*] Corresponding author at: University of Yaounde 1, Department of Arts and Archaeology, Faculty of Arts, Letters and Social Sciences/ Cameroon, P.O Box 755, Yaounde, Cameroon.

E-mail address: zoila.epossi@univ-yaounde1.cm (Z.L. Epossi Ntah).

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BP (area 2), corresponding to the period 1490 CE (end of the 15th century and beginning of the 16th century). The archaeological artefacts from Pongsolo are mainly slag, many of which are of small size, and tuyères. According to Essomba (1992), twenty-one fragments of tuyères with a length of 10 to 18 cm and an internal diameter of 3 to 5 cm with a wall thickness between 0.5 to 1.0 cm were found in the Ponsgolo area.

Excavations began at Evian in 1984–1985, where the accumulation of smithing residues in mound form, smelting residues, and furnaces in pits covered by a thick layer of clay material were found. The artefacts found in Evian include fragments of tuyères and pottery sherds, slag, lithic tools and metal objects. Excavation of deposits from ironworking activities yielded a total of 501 tuyère pieces, including 491 sherds and 10 fragments of complete or near-complete tuyères (Elouga 2000). A morphological study of the Evian tuyères showed that they varied in size. The internal diameter ranges between 2–3 cm and 5 cm, and walls are 0.7–1.4 cm thick.

According to Elouga (2000), this diversity in size suggests that the production of tuyères at Evian was not standardised. The average of the length was not determined due to the many missing parts of tuyères. Two types of tuyère morphology were deduced from the reconstitution processes: cylindrical fragments and funnel-shaped hyperboloids. The moulding technique and the surface treatment of the tuyères from Evian were also studied by Elouga (2000). Their inner surface is not smooth and shows fairly thin striations or cannelures, while their outer surface is smooth and undecorated. Tuyères from Evian do not show any heat-related changes on their outer surface, but are slagged and vitrified at the tips. Radiocarbon dating of charcoal fragments found at the Evian site revealed that the iron smelting workshop dated back to the end of the 19th century and the beginning of the 20th century CE (Elouga 2000).

According to the oral tradition, tuyères were made from a clay material. A wooden stick was used to give them shape. Their external surface was smoothened by hand or using a ribbing tool. Afterwards, the wooden stick was removed. The tuyères were dried in the air and remained unfired. This moulding technique is mainly used in the centre of Cameroon where the production traditions are preserved (Essomba 1992; Elouga 2000).

Excavations at Mfomakap began in 1983. Mfomakap is the most important archaeological site in the Lekie Division. Many artefacts such as pottery sherds, slag, pieces of tuyères, lithic tools and metal objects have been found in this locality (Elouga, 2000). However, the study of pottery is predominant in this site (Elouga 2000; Epossi Ntah 2012). Although radiocarbon dating attested iron metallurgy in this area since 3000–2000 BP (Essomba 1992; Elouga 2000), little attention has been paid to the tuyères.

The study of tuyère fragments from Evian and Pongsolo has so far been limited to the morphological and moulding aspects, which is very limiting for understanding the ironworking processes in this region. However, at Oliga, one of the most important iron metallurgy sites located in Yaounde in central Cameroon (3000 BP) and close to the Lekie Division, Essomba (1992) reported the chemical analysis of three tuyères to understand their composition and origin. The results showed that they were produced by a material rich in silica, alumina and iron oxide, which is related to the geological setting of central Cameroon.

The present study is the first systematic chemical, mineralogical, petrographic and physical characterization of tuyères from Lekie and

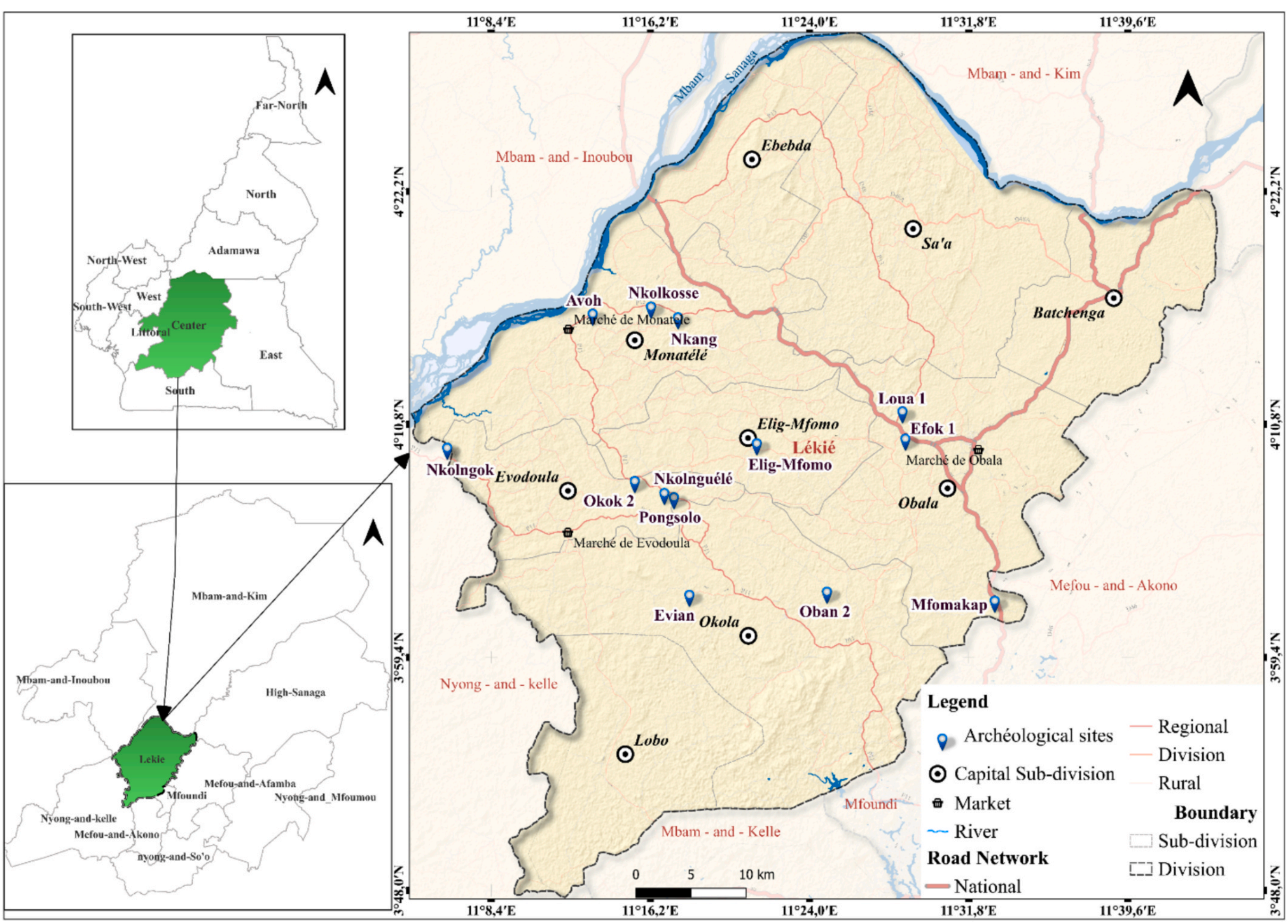

Fig. 1. Geographic location of the Lekie Division and associated archaeological sites of the region mentioned in the study.

more generally in Cameroon. The objective of the study is to determine the temperature to which the tuyères from Evian and Pongsolo sites were exposed during the ironworking processes, as well as the nature of the raw material used to produce them and their treatment. A better understanding of the technology of iron production and the behaviour of the tuyères during the ironworking process will be assessed.

However, it is important to note that studies of African tuyères mostly focus on their morphology (length, diameter, thickness and shape) and the stylistic-morphological approach related to their position, organisation and function in furnaces during the ironworking process in order to reconstruct the *chaîne opératoire*, to determine the type of furnace and to understand the origin of iron technology in west Africa (Killick 1991, 2001; Childs 1991; Childs and Killick 1993; Holl, 2000, 2009; Bocoum 2002; Miller & Van Der Merwe 2009; Chirikure et al. 2009; Zangato and Holl 2010; Chirikure 2015; Tollo 2021). Tuyères are mentioned throughout the metallurgical process, alongside slag, furnaces and bellows, but systematic studies involving an archaeometric approach are sporadic (Lyaya et al., 2012; Mteta and Lindahl, 2017). Furthermore, the study of ironworking technology involving the application of natural sciences methods or archaeometallurgy are more prevalent in southern and eastern than in west Africa (Chirikure and Rehren, 2004, 2006; Chirikure and Bandama, 2014; Chirikure, 2015; De Barros et al., 2020, Bandama et al.2016; Iles, 2020). Although investigations of this type have mostly focused on slag, ores, and metal objects at a few sites in the Central African Republic (Fluzin, 2007a, 2007b) and Nigeria (Whiteman and Okafor, 2003), studies of tuyères are scarce in west central Africa.

Therefore, the results of the present research will be compared with the available data of the tuyères from other parts of Africa in terms of typology, raw materials and production technology with the aim of improving the knowledge of metallurgical technology in west-central Africa.

2. Geology of the region

The soil in the Lekie Division originates from metamorphic rock formations containing significant amounts of gneiss, micaschist, and quartzite (Gazel, 1956). According to the literature, the geology of Lekie belongs to the Southern Cameroon Domain in the Yaounde Group 1 (Fig. 2), which is a *syn*-tectonic basin with deposits younger than 625 Ma (Toteu et al., 2006; Owona et al., 2012; Nkoumbou et al., 2014). The Yaounde Group 1 of the Southern Cameroon Domain is mainly constituted of metasediments and mafic rocks (pyriclasites) to intermediate rocks. These units recorded penetrative deformations and migmatization during tectonic evolution and were successively intersected by pre-, syn- and post-tectonic intrusions (Toteu et al., 2001; Nkoumbou et al., 2014; Tchakounté et al., 2017). According to Tabue Youmbi et al. (2021), at the petrographic level, the Yaoundé series is influenced by the Pan-African orogeny (600 and 500 Ma) and composed mainly of gneisses and migmatites, derived from ancient granitized and metamorphosed sediments (Nzenti et al., 1988). It is subdivided into two units, a *meta*-sedimentary unit and a *meta*-igneous unit. The *meta*-sedimentary unit consists mainly of garnet and kyanite gneisses and garnet and plagioclase gneisses with intercalated layers of marble and calcium silicate rocks, occasionally accompanied by quartzites and orthopyroxenites rich in magnetite, whereas the *meta*-igneous unit is mainly composed of pyriclasites, mafic granulites, pyroxenites and biotite-rich rocks.

3. Materials and methods

3.1. Materials

Twelve samples of tuyères were selected from the INC Laboratory's collection at Elig Essono, University of Yaoundé, according to their stratigraphic context (depth of excavation) and representativeness within the collection: eight are from the Pongsolo site (PO1 to PO8), and four from the Evian site (EV1 to EV4). However, all samples present in the collection are cylindrical. Macroscopically, the samples from the two sites look very different according to the colour of their external surface.

Most of the samples from Pongsolo are red with some black parts possibly due to thermal effects and variation of the atmosphere as in the case of PO1a, PO2, PO3 and PO4 (Fig. 3). Sample PO1b is black and dark brown on the outer surface and red on the inner surface. Samples PO6, PO7 and PO8 are black and partially covered with slagged or vitrified material (with some brown area in the background as in the case of PO8). Therefore, their surfaces appear thicker than the non-vitrified tuyères.

All samples from Evian are characterised by a light grey colour on their external and internal surfaces, and EV4 is covered with slag and gas pores (Fig. 3). This can also be seen in the lower part of EV1. Table 1 presents the general characteristics of the studied tuyères. With a range of 3.5–4 cm, the internal diameter of tuyères from Evian is higher than that of the Pongsolo tuyères with 2–3 cm. Tuyère walls are between 0.5 and 1 cm thick, with an inter-tuyère variation of about 2 mm (Table 1).

3.2. Analytical methods

3.2.1. X-ray fluorescence

The chemical composition of the twelve tuyères was determined by wavelength-dispersive X-ray fluorescence analysis (WD-XRF) using a Panalytical Zetium spectrometer equipped with a Rh tube anode and 4 kV X-ray generator. For major elements, a mixture of 0.6 g of powdered sample and 5.4 g $Li_2B_4O_7$ was melted at 1000 °C. After cooling, the glass disc was analysed. For trace elements, a mixture of 5 g of sample in the same proportion with boric acid was pressed to produce a pellet for analysis. The accuracy of the chemical analyses was assessed by comparison with more than 30 certified geostandards covering a wide range of common silicate rocks (Govindaraju 1994). Typical accuracy is higher than 1.5 % relative to a concentration of 10 %. One chemical analysis per sample type was carried out.

3.2.2. X-ray diffraction

A qualitative identification of the mineral phases for the twelve tuyères was carried out by powder X-ray diffraction (XRD) using a PANalytical X'Pert PRO diffractometer. One analysis per sample was carried out. The working conditions were as follows: Cu Kα radiation ($\lambda = 1.5418$ Å), V = 45 kV, I = 40 mA. XRD patterns were recorded over the 3–70° (2θ) angular range at 0.01 2θ/s goniometer speed. No internal standard was added to the powders. The diffractograms were interpreted using GSAS II software (Toby and Dreele 2013).

3.2.3. Polarized optical microscopy

For the petrographic study, one thin section per tuyère sample was prepared and observed under a Carl Zeiss Jenapol-U polarized optical microscope (POM). Images of the texture and mineralogy in plane- and cross-polarized light were captured with a Nikon D7000 digital camera.

3.2.4. Hydric tests

Hydric tests were carried out to assess the water behavior of the twelve tuyères. These tests provide valuable information on the pore system of the materials, predicting their greater or lesser susceptibility to degradation, as saline solutions can circulate easily through the pores and fissures of materials. Each tuyère used for hydric tests were previously dried in an oven at 70 °C for 24 h to remove any moisture. Free water absorption (Ab), forced (under vacuum) water absorption (Af), drying index (Di), degree of pore interconnection (Ax) (Cultrone et al., 2004), open porosity (Po), saturation coefficient (S) and apparent (ρa) and real densities (ρr) were calculated according to UNE-EN 13755 (2008), NORMAL 29/88 (1988) and RILEM (1980) standards. Although the standard for hydric tests suggests analysing three samples per type with specific shapes and sizes (RILEM, 1980), this cannot be applied to

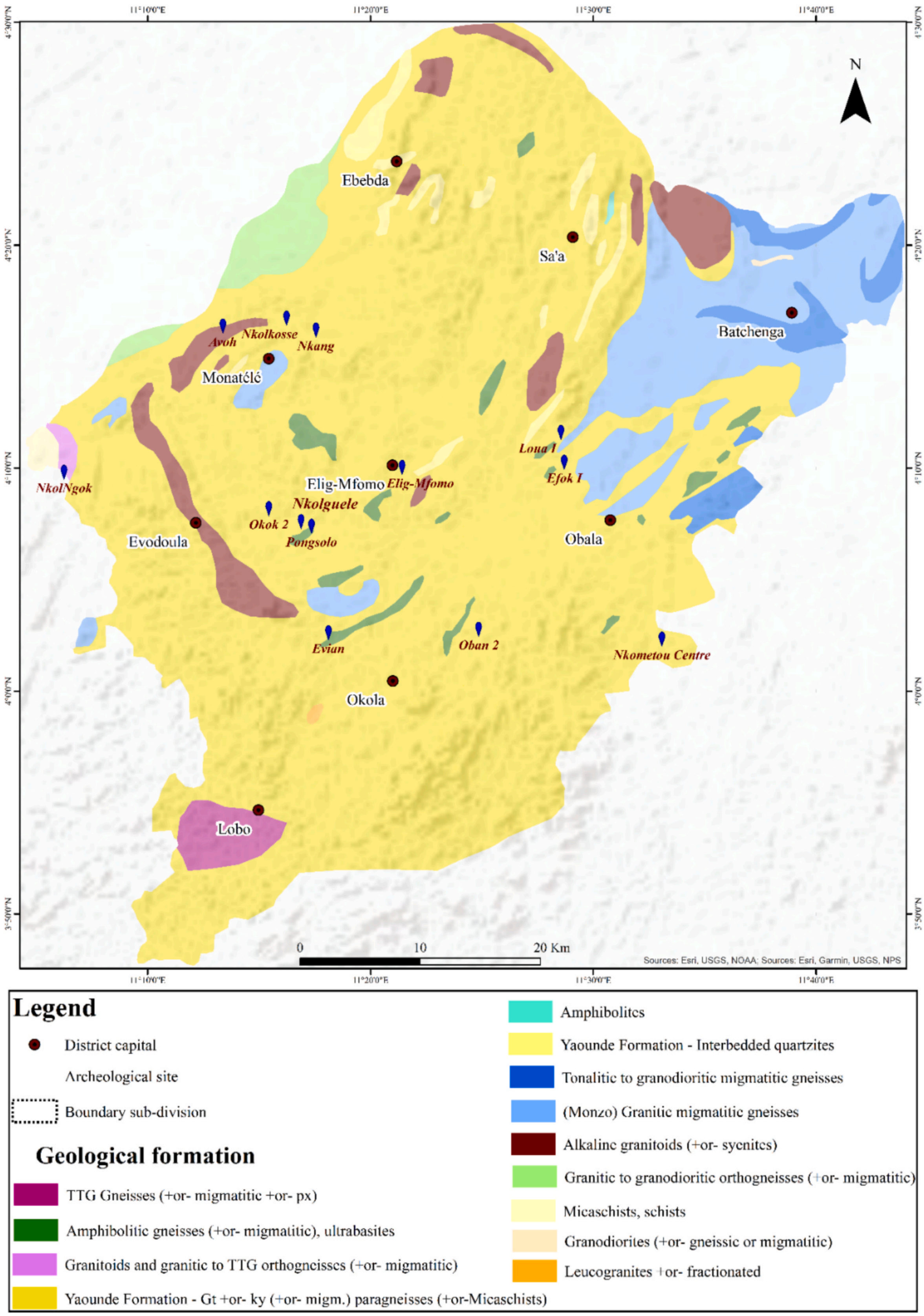

Fig. 2. Geological map of the Lekie region (after Delor et al., 2021).

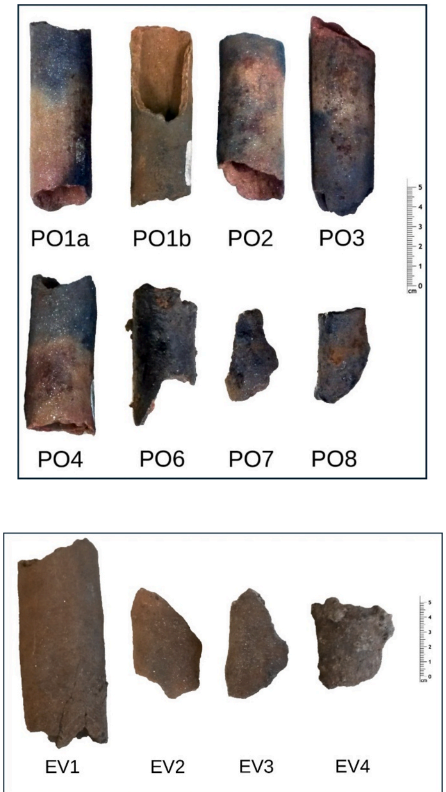

Fig. 3. General view of the tuyère samples from Pongsolo (PO1–8) and Evian (EV1–4).

archaeological materials, as these are unique samples that should not be broken into multiple pieces for testing.

3.2.5. Colorimetry

Colour measurements on the surfaces of each tuyère were performed with a portable Konica Minolta CM-700d spectrophotometer to quantitatively determine their colour and to detect possible differences between samples. Lightness (L*) and chromatic coordinates (a* and b*) were determined in the 400–700 nm wavelength range by selecting the CIE illuminant D65 that simulates daylight with a colour temperature of 6504 K. Circular areas of the samples with a diameter of 3 mm were analysed (10° vision angle). Illumination was provided by a pulsed xenon lamp with a UV cut filter, while a silicon photodiode array detected and measured both incident and reflected light.

Except for the preparation and observation of the thin sections, all analyses were carried out at the Department of Mineralogy and Petrology of the University of Granada (Spain). The thin sections were prepared at the Institute of Petrography and Mineralogy in Hamburg (Germany) and observed at the Department of Arts and Archaeology of the University of Yaoundé 1 (Cameroon) and at the Deutsches Bergbau-Museum Bochum (Germany).

X-ray fluorescence analysis will help answer questions about the nature and origin of the raw material used to produce the tuyères. X-ray diffraction will be used to identify the mineral phases and inform about the temperature the tuyères where exposed to in the furnace, whereas the atmospheric conditions will be deduced from the macroscopic observations of the tuyères, mainly the colour of the fracture surface. Polarized optical microscopy will be used to analyse the fabric of tuyères to determine the nature of the inclusions present in the paste, clay preparation and nature of temper. Finally, colorimetry and hydric tests (water absorption and drying) will complete the characterization of tuyères by providing some physical parameters that will help in defining their refractoriness during ironworking processes. These results will give an idea on the origin and the manufacture techniques of the tuyères, their thermal behaviour and the estimation of the temperature during the ironworking processes. This information will contribute to the knowledge on the iron metallurgical processes in Cameroon and Sub-Saharan Africa.

4. Results and discussions

4.1. X-ray fluorescence (XRF)

Table 2 shows the major elements of the tuyères. The SiO_2 content is between 65 and 70 wt% in all samples from Pongsolo (PO) except for the samples PO1a, PO4 and PO8. Evian samples (EV) have a generally higher amount of SiO_2 compared to PO samples. The SiO_2 content suggests that the raw material is rich in quartz.

Al_2O_3 content varies from 18 to 22 wt% in all samples, which could indicate the presence of phyllosilicates and feldspars.

Evian (EV) samples have a lower amount of Fe_2O_3 (from 1 to 5 wt%) when compared to PO samples, which explains their grey colour. Most of the PO samples have a Fe_2O_3 content varying from 5 to 8 wt% except PO1a and PO8, whose iron content is 10 and 13 wt%, respectively. This

Table 1
General characteristics of the tuyères (in thickness section: max = maximum and min = minimun).

Samples	Sites	Excavation depth (cm)	Fracture surface colour	Inner surface colour	Outer surface colour	Thickness (min – max, cm)	Internal Diameter (cm)
PO1a	Pongsolo	0–50	light grey and slice ight line with some coarse grains	dark brown	dark grey	~0.6	3.0
PO1b	Pongsolo	0–50	reddish/black-reddish per area	reddish	red and black	0.6 – 0.8	3.0
PO2	Pongsolo	0–50	light grey and llight red wish some coarse grains	reddish	light red and dark/ black area	0.5 – 0.8	2.5
PO3	Pongsolo	0–50	dark grey with coarse grains / Dark brown in some part variable	reddish	grey	0.7 – 0.9	2.0
PO4	Pongsolo	0–50	black (big margin) and reddish with coarse inclusions (slice margin)	reddish	black with shinig reflections of mica	0.6 – 1.0	3.0
PO6	Pongsolo	0–50	black –light brown with coarse inclusions	mostly reddish but with reddish part as well	black and very slagged	0.7 – 0.8	3.4
PO7	Pongsolo	0–50	bizonal: light grey-light red/brown	dark grey with fine grains	light grey	0.9 – 1.0	3.0
PO8	Pongsolo	0–50	bizonal: black or dark grey with the presence of coarse inclusions	black-dark grey	black-dark grey slagged	0.5 – 0.6	3.0
EV1	Evian	60–80	light grey	light grey	light grey	0.7 – 0.8	4.0
EV2	Evian	40–60	grey- light red fine grains and some coarse grains	reddish	reddish and some black areas	0.5 – 0.8	4.0
EV3	Evian	40–60	light grey with fine grains	light grey	light grey	0.9 – 1.0	4.0
EV4	Evian	40–60	dark grey with coarse grains	dark grey	dark grey and slagged	~0.5	3.5

Table 2
Chemical composition of the tuyeres: major elements (wt.%).

Samples	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	LOI	SUM
PO1a	63.45	20.41	10.00	0.03	0.54	0.33	0.36	1.57	0.85	0.12	1.84	99.50
PO1b	66.42	17.53	8.04	0.04	0.82	0.47	0.42	1.86	0.90	0.14	2.92	99.56
PO2	70.45	18.06	5.52	0.02	0.64	0.49	0.54	1.47	0.89	0.09	1.20	99.37
PO3	66.09	18.54	7.20	0.05	0.73	0.49	0.53	1.89	0.89	0.14	2.83	99.38
PO4	62.08	19.63	7.29	0.04	0.61	0.64	0.45	2.28	0.94	0.08	5.52	99.56
PO6	69.29	19.14	6.48	0.12	0.51	0.37	0.30	2.06	1.01	0.12	0.18	99.58
PO7	69.63	19.75	5.73	0.04	0.52	0.24	0.30	1.98	0.95	0.10	0.37	99.61
PO8	61.67	18.67	13.96	0.45	0.68	0.69	0.44	1.71	0.90	0.23	0.00	99.39
EV1	69.44	20.63	4.77	0.03	0.56	0.26	0.10	1.43	1.56	0.15	0.41	99.34
EV2	67.42	21.19	2.94	0.01	0.66	0.52	0.27	1.86	1.09	0.08	3.60	99.64
EV3	68.20	21.27	4.51	0.03	0.73	0.54	0.19	2.40	1.16	0.13	0.12	99.28
EV4	70.14	19.18	1.77	0.01	0.49	0.53	0.22	1.87	0.95	0.07	4.44	99.67

is consistent with, but not necessarily exclusive to, the reddish colour observed. Moreover, the higher Fe_2O_3 content is probably related to different raw material used for the samples PO1a and PO8.

MnO content is very low in all samples, in the range of 0.01 to 0.04 wt% except for the samples PO6 and PO8 with values of 0.12 and 0.54 wt%, respectively.

MgO, Na_2O and CaO contents are less than 1 wt% in all samples, while the values for K_2O range from 1.4 to 2.5 wt%. This suggests the absence of carbonates and confirms the possible presence of phyllosilicates and K-feldspars.

TiO_2 content varies from 0.80 to 1.56 wt% in all samples with the highest value in EV1.

P_2O_5 content is very low in PO2, PO4, EV2 and EV4 with the values ranging from 0.07 to 0.09 wt%, while it varies in the other samples from 0.10 to 0.22 wt%. with the highest value in PO8.

The loss on ignition (LOI) is very low in samples PO6, PO7, EV1 and EV3 with values below 0.5 wt%. In the other samples, the LOI value is above 1 wt%, with PO4 recording the highest value of 5.5 wt%. In PO8, the LOI was below the detection limit.

The trace elements of the tuyères are presented in Table 3. The results show that zircon is the most abundant element varying from 194 to 220 ppm in most of them, except for the sample PO8 which has a content of 163 ppm and sample EV1 with a content of 593 ppm. These results can suggest the presence of zircon crystals, especially in EV1.

The vanadium content is between 130 and 180 ppm, and can be easily stored within phyllosilicate or iron oxide structures.

The chromium content ranges from 130 to 287 ppm, except for the sample PO8 which has a content of 545 ppm. The zinc content is below 100 ppm in EV samples, and between 100 and 200 ppm in PO samples, except for sample PO8, which reaches 399 ppm.

Sample PO8 also stands out for having the highest Sr and Ce content. The content of other elements is generally low in all samples. The elevated Cr, Zn, Pb, Sr and Ce values observed in PO8 may be due to the contamination from the ore in the vitrified material.

4.2. *X-ray diffraction (XRD)*

X-ray diffraction shows that all tuyères are rich in quartz in accordance with the high SiO_2 content determined by XRF (Table2). The dominance of quartz suggests the use of a high-silica paste, potentially improving thermal resistance but increasing thermal shock resistance. However, there are differences between the samples based on the other identified mineral phases (Fig. 4). Samples PO1a, PO2, EV2, and EV3 contain kaolinite, muscovite and quartz, whereas EV1, PO1b, PO3 and PO4 present muscovite and quartz but no kaolinite. Samples PO6, PO7 and PO8 contain mullite and quartz, whereas EV4 is composed of cristobalite, mullite and quartz. The presence of high-temperature phases such as mullite and cristobalite could be related to the presence of slag or vitrification observed on the surface of the last four samples.

4.3. *Polarized optical microscopy*

The tuyères from both sites show similar features, above all with regard to aplastic inclusions, which are mainly composed of quartz (Fig. 5a, b). Quartz grains have an angular morphology. They appear either as isolated grains or together with phyllosilicates, forming gneiss fragments. They are poorly sorted in all samples. Quartz has undulose extinction, typical of metamorphic rocks. Phyllosilicates are easily distinguishable in low fired tuyeres and are composed exclusively by muscovite. They show second order blue-green interference colour. The phyllosilicates and elongated materials appear well aligned due to the pressure exerted on the raw material during shaping, according to the oral tradition when placed around a wooden stick (see above). The pores, which are elongated and have an irregular morphology, also have the same orientation as the phyllosilicates. The moulding technique can be seen in EV1, which has a subparallel, wedge-shaped orientation of the mica (Fig. 6a). Mica is completely absent in PO6, PO8, and EV4. I In PO3 and PO7 it has lost birefringence and appears isotropic (Fig. 5c). Moreover, PO3 and PO7 show clear signs of heat impact. In these five cases, a high temperature may have been reached. The abundance of

Table 3
Chemical composition of tuyères: trace elements (ppm).

Samples	Zr	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	La	Ce	Nd	Sm	Pb	Th
PO1a	258	23	132	169	12	106	39	199	24	91	56	16	64	105	38	4	32	21
PO1b	196	22	143	154	11	108	46	176	22	88	82	19	75	137	60	8	28	11
PO2	210	23	143	138	12	93	31	134	21	75	93	16	87	136	57	5	25	12
PO3	203	23	149	157	16	166	34	147	21	88	99	22	103	165	67	12	36	11
PO4	194	23	173	160	13	69	31	107	24	89	89	22	80	121	49	14	25	12
PO6	208	23	145	236	13	74	35	125	22	82	90	16	93	167	57	7	23	12
PO7	214	22	140	178	10	61	30	105	22	81	85	14	100	163	66	9	23	13
PO8	163	24	152	545	40	185	59	399	23	80	122	33	102	194	69	11	56	13
EV1	593	21	154	226	9	63	21	86	30	97	45	24	101	160	61	8	22	15
EV2	213	24	166	167	13	74	31	82	25	84	87	26	67	102	48	10	26	12
EV3	212	22	176	287	19	116	25	70	23	93	76	21	67	105	36	5	18	11
EV4	218	23	153	154	3	30	15	49	21	68	82	25	63	113	52	10	21	8

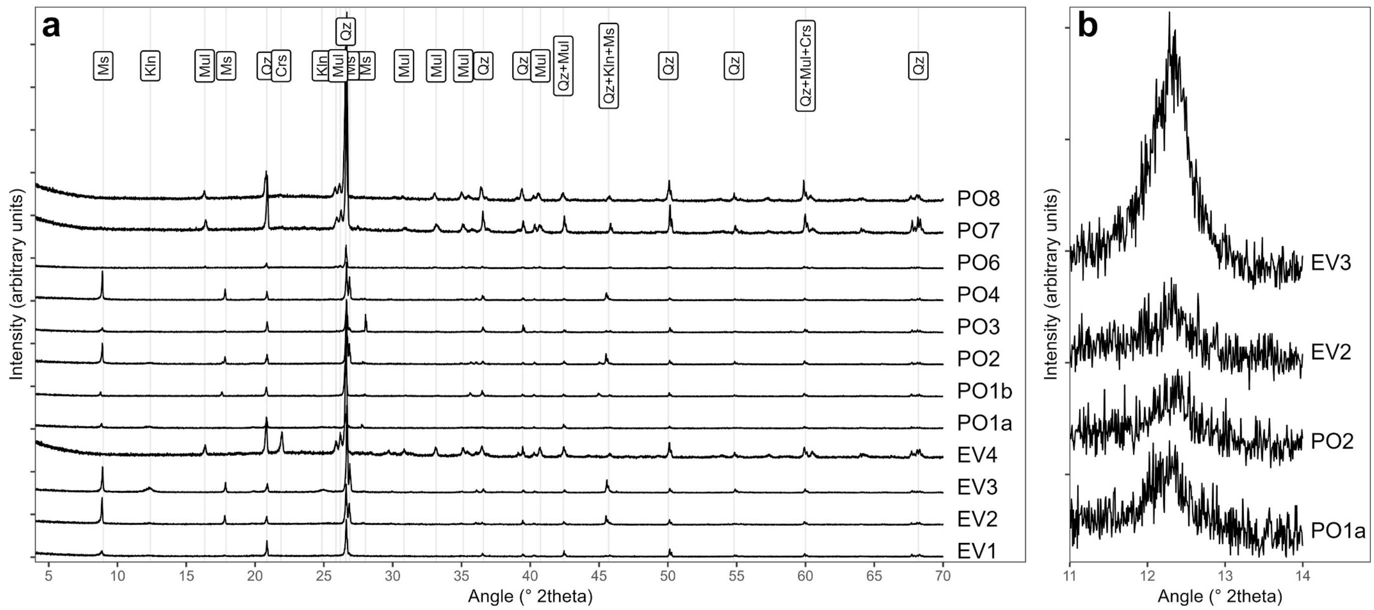

Fig. 4. (a) X-ray diffraction patterns of the tuyères with major peaks labelled: Ms: Muscovite, Kln: Kaolinite, Mul: Mullite, Qz: Quartz, Crs: Cristobalite (minerals acronym after Warr 2021) and (b) The main kaolinite peak for all samples with kaolinite.

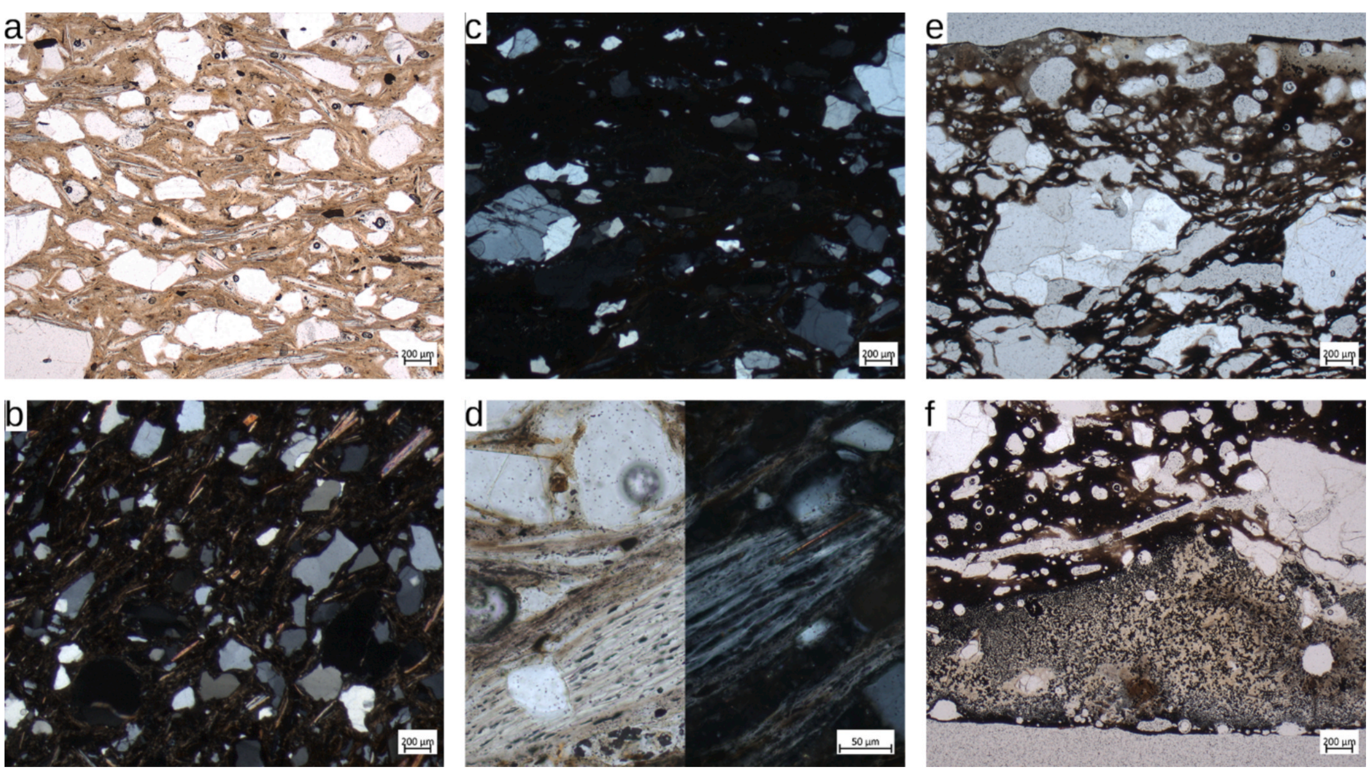

Fig. 5. Microstructures of the tuyeres under polarized optical microscopy. Images are taken in plane polarized light (PPL) and cross polarized light (XPL). (a) clay paste without indicators for heating in EV1 (PPL); (b) clay paste with indicators for low heat impact in PO1a (XPL); (c) clay paste with lost optical activity in PO6 (XPL); (d) bloated mica with lost optical activity in PO7 (PPL, left, and XPL, right); (e) vitrification layer in PO8 (PPL); (f) vitrification with needle-shaped translucent minerals and clusters of opaque minerals in EV4 (PPL).

mica in the paste correlates with the paste colour and sometimes varies within the section (Fig. 6b). Moreover, bloating of mica with the development of a secondary porosity is observed in samples PO3 and PO7 (Fig. 5d), while the tuyères PO6, PO8 and EV4 show the presence of rounded pores in a totally vitrified matrix or slagged areas, generally along an edge of the tuyeres (Fig. 5e). These microstructural features, such as secondary porosity, rounded pores and a vitrified matrix, suggest that these samples were fired more intensely than the other tuyères studied. Generally, red pastes are richer in mica than brown and dark pastes that show a yellowish colour under crossed nicols. Red pastes present small hematite crystals scattered in the matrix. Other mineral phases that have been identified but are only present occasionally include: K-feldspar with incipient argillification on the crystal surface; twinned albite plagioclase; kyanite, whose crystals, rarely twinned,

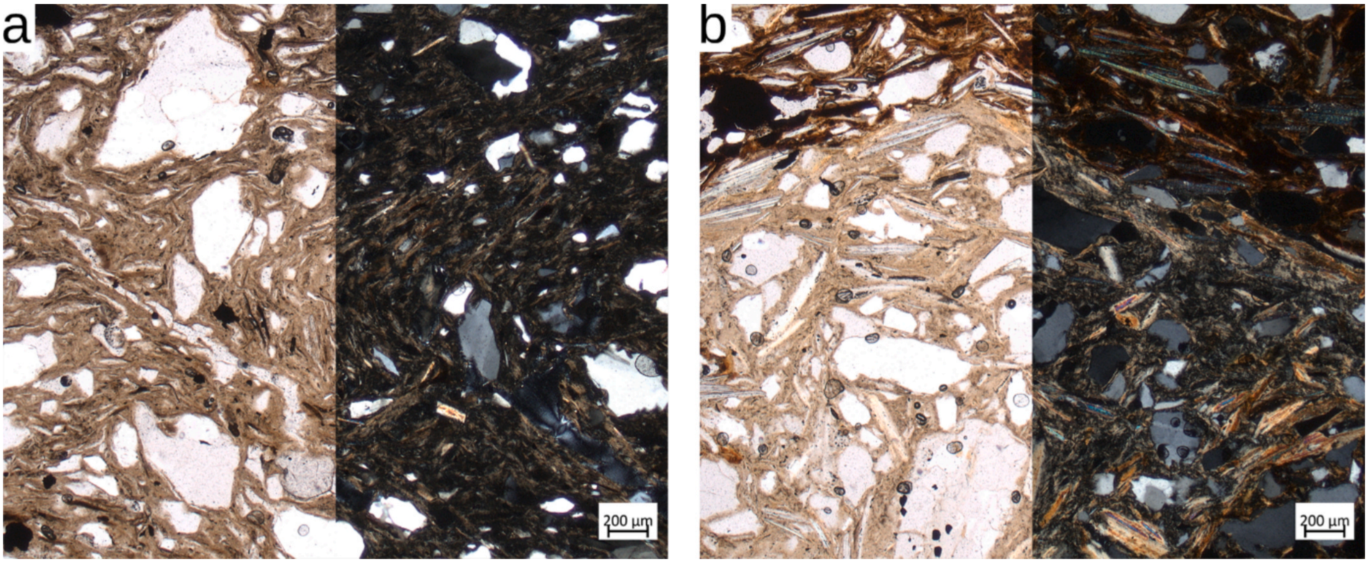

Fig. 6. Indicators for the shaping technique (a) wedge-shaped joint, EV1; (b) Abundance of mica in the paste, PO1b. Left half of each figure in plane polarized light, right half in cross polarized light.

show high relief and marked exfoliation; rare tourmaline, slightly pleochroic. Vitrification is usually characterised by a translucent needle-shaped phase and clusters or dendrites of an opaque phase in the vitreous material (Fig. 5f).

4.4. Hydric tests

Free water absorption (Ab) varies between 13 and 20 % in most of the samples (Table 4), whereas PO6, PO8, and EV4 show notably lower values (5–6 %), indicating the lowest water absorption, probably due to their high degree of vitrification.

A similar behaviour is observed with the forced water absorption (Af). While it ranges for most of the samples between 16 and 23 %, PO6, PO8 and EV4 show lower values (less than 10 %). The interconnection between the pores (Ax) is lower in samples EV1 and EV3 (3.92 and 6.62 %), which indicates an easier water flow in the pore network of these two. Samples PO6, PO8 and EV4, those with the lowest water absorption, have the highest values of Ax at or above 30 %. These three samples show the lowest pore interconnectivity, likely due to vitrification and high thermal exposure.The drying index (Di) is similar in all the samples with the values ranging from 0.91 to 0.95 (Table4). These results indicate that the samples have a similar drying behaviour (Fig. 7). However, again, PO6, PO8 and EV4 are the slowest drying samples. The higher degree of vitrification reduces the interconnection between the pores and hinders the movement of water between the pores and capillaries of these samples.

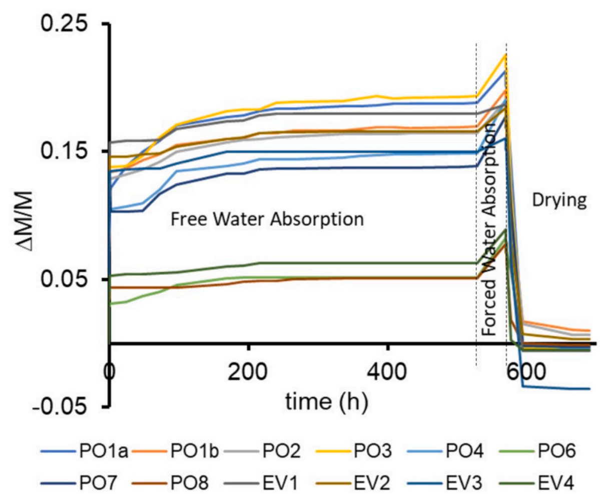

Fig. 7. Free water absorption, forced water absorption and drying curves of the studied samples. Weight variation (ΔM/M) versus time (in hours).

The values of open porosity are between 27 and 37 % for all the samples except again for PO6, PO8 and EV4 with values below 20 %.

Table 4
Hydric parameters of the samples.

Samples	Ab (%)	Af (%)	Ax (%)	S (%)	Po (%)	ρa (g cm^{-3})	ρr (g cm^{-3})	Di
PO1a	18.81	21.33	11.82	70.20	35.34	1.66	2.56	0.921
PO1b	16.94	19.81	14.49	72.45	31.87	1.61	2.36	0.926
PO2	16.47	19.04	13.48	71.63	32.12	1.69	2.49	0.927
PO3	19.37	22.59	14.25	64.96	36.99	1.64	2.60	0.915
PO4	14.89	19.11	22.09	57.21	31.97	1.67	2.46	0.921
PO6	5.14	8.26	37.79	45.15	15.83	1.92	2.28	0.946
PO7	13.9	17.77	21.78	58.42	30.28	1.70	2.45	0.926
PO8	5.1	7.79	34.59	56.02	14.99	1.86	2.18	0.943
EV1	17.96	18.69	3.92	84.94	31.74	1.70	2.49	0.916
EV2	16.59	18.43	9.98	80.79	30.76	1.67	2.41	0.921
EV3	14.96	16.03	6.62	85.17	27.45	1.71	2.36	0.920
EV4	6.25	8.89	29.67	60.98	17.07	1.92	2.31	0.937

Legend: Ab = free water absorption; Af = forced (under vacuum) water absorption; Ax = degree of pore interconnection; S = saturation coefficient; Po = open porosity; ρa = apparent density; ρr = real density; Di = drying index.

This result is consistent with the water absorption values and the texture observations determined previously.

The real density (ρr, Table4) varies from 2.2 to 2.6 g/cm^3 and is linked to those of quartz and other silicate minerals in the tuyères. The values of the apparent density (ρa, Table 4) are lower than those of the real density because they also take into account the empty spaces. The highest ρa values were measured in PO6, PO8 and EV4 because of the densification induced by vitrification, which was already observed under optical microscopy.

The saturation coefficient (S, Table4) varies between 45 and 86 %. Samples with the highest S values have the lowest Ax. This is remarkable for the samples EV1, EV2 and EV3. Sample PO6 also stands out for its highest Ax (37.79 %) and lowest S (45.15 %). These results are in agreement with the literature (samples with a very tortuous porous system and high Ax values attain low S values; Cultrone and Madkour 2013).

4.5. Colorimetry

The values of lightness (L*) indicate that the samples range from medium to dark tones. Only two samples have L* between 51 and 52 (PO2 and PO3) while the darkest samples are PO6, PO8 and PO7 with L* values of 29.87, 31.54 and 34.1, respectively. Samples PO6, PO8 and PO7 are also the ones with the lowest a*, b* and C* in relation to their dark colour. Sample PO6 with the lowest lightness and chromatic values is covered by a layer of slag on its surface, suggesting a high firing temperature.

The samples with the highest chromatic coordinates (a* and b*) and C* are PO1b, PO2, PO3 and EV1 (Table5), among which PO1b and EV1 have a uniform colour in their external surface (light grey for EV1 and dark brown for PO1b), whereas the others show two colours (red and black) due to the thermal-effect.

4.6. Discussion

4.6.1. Raw materials and pre-treatment

According to the chemical results, the tuyères from the two Lekie sites are silica-alumina rich and very low calcareous (Table 2), with a SiO_2 content between 65 and 70 wt% and an Al_2O_3 content between 18 and 22 wt%. They contain a variable amount of iron oxide, lowest in the Evian samples (1–5 wt%) and relatively high in the Pongsolo samples (six samples with an Fe_2O_3 content between 5 and 8 wt% and two with a content of 10 and 13 wt%). The variation of iron oxide influences the colour of the tuyères, reddish for the Pongsolo samples and greyish for the Evian samples. The SiO_2/Al_2O_3 ratio varies between 3.1 and 4 in both sites, indicating a predominance of quartz over clay minerals (Epossi Ntah and Cultrone 2024; Ndjigui et al., 2021; Kılıç et al., 2017). The abundance of quartz could be related to the use of quartz sand as temper during the production of the tuyères, as reported by Essomba (Essomba 1992). However, the angular shape of the quartz grains observed under polarized optical microscopy could suggests that they were naturally present in the raw materials. Therefore, it seems that no quartz temper was added during the manufacture of the studied tuyères. The sum of alkali or flux oxides ($CaO + K_2O + Na_2O$) varies between 2 and 3 wt% in all samples except for EV1 (1.74 wt%). These values below 5 wt% indicate that all tuyères are refractory and withstand high temperatures (Martinon-Torres and Rehren 2014; Quinn 2022). However, it is important to mention that refractoriness also depends on firing process and particle bonding.

Except for Zr, Cr, Zn, Sr and Ce in samples EV1 and PO8, the trace elements show similarities in almost all samples and suggest a local production of the tuyères. Correlation diagrams of Al_2O_3/SiO_2 versus ($CaO + K_2O + Na_2O$) and V/Cr confirm this hypothesis (Fig. 8 a, b). A comparison of the samples with three local raw materials collected in Mfomakap (30–35 km away from Evian and Pongsolo sites, see Fig. 1a) and the mixture in the proportion 1:1 of the raw material samples (Epossi Ntah 2012) confirm the local production of the samples (Fig. 8 c, d). This is in agreement with Essomba (1992) who reported the chemical composition of three tuyeres from Oliga near Yaounde (Central Cameroon). The tuyères from Oliga are rich in silica and alumina. The SiO_2 content was 60.50 wt%, 47.50 wt% and 51.17 wt%, whereas Al_2O_3 content was 22.58 wt%, 19.46 wt% and 19.38 wt%. The content of iron oxide was higher in these three samples (12.3 wt%, 22.78 wt% and 10.83 wt%) than in our samples. The high content of iron oxide observed in some samples of Pongsolo and in the raw material samples from Mfomakap can be explained by the geological setting of central Cameroon, which is characterized by ferrallitic soils. From the mineralogical point of view, the local clay materials studied by Epossi Ntah (2012) present K- and Na-feldspars with variable amount in each clay (in agreement with the metamorphic origin of the region's rock). However, petrographic analysis shows only occasional feldspars in the tuyères. This result could be explained by the alteration of feldspars during the evolution of the sediment in a fluvial context, which is supported by the observed incipient argillification on the crystal surfaces of some K-feldspars.

However, the quartz grains, which were naturally present in the original clays, made the tuyères resistant to high temperatures by improving their thermal stability during the smelting process (Martinon-Torres and Rehren 2014; Quinn 2022). These findings support the argument for local clay sourcing, with the kyanite as potential indicator for the *meta*-sedimentary unit of the Yaounde series as source.

4.6.2. Thermal behaviour and estimation of the temperature during the ironworking processes

X-ray diffraction analysis and optical microscopy show a similar mineralogical composition of the tuyères from both sites. This mineralogical variation is correlated with the microstructural variation of the samples. According to the oral tradition, the tuyères were not fired before use but only in the furnace during the ironworking process (Essomba 1992; Elouga 2000). The mineralogical and microstructural variation of the tuyères at the two sites is therefore related to the temperatures to which they were exposed during the ironworking process and to the position of the sampled aliquot in the tuyere. Following the suggestion that the studied tuyères were probably produced from the Lekie local materials, previous firing experiments of the clay materials from Mfomakap from 400 to 1200 °C likely provides good analogous materials, even though the experiments were carried out in an oxidising atmosphere in a laboratory kiln (Epossi Ntah, 2012). The mineralogical composition of these fired clay samples obtained by X-ray diffraction analysis showed the decomposition of kaolinite between 400–500 °C, the formation of mullite between 1000–1100 °C and the formation of cristobalite between 1100–1200 °C (Epossi Ntah 2012). The correlation of these results with the mineralogy of the tuyères suggests that the samples containing kaolinite, according to X-ray diffraction results, EV2, EV3, PO1a and PO2, were exposed to temperatures below 500 °C. According to the literature, kaolinite decomposes in a CaO-poor clay

Table 5
Lightness (L*), chromatic coordinates (a*, b*), Chroma (C*) and hue angle (h°) of the tuyères samples by using illuminant D65.

Samples	L*	a*	b*	C*	h°
PO1a	45.60	7.06	16.46	17.91	66.79
PO1b	47.36	10.69	18.41	21.29	59.85
PO2	51.83	10.70	21.98	24.44	64.04
PO3	51.25	11.69	20.96	24.00	60.84
PO4	45.21	7.35	14.33	16.16	65.47
PO6	29.87	2.03	3.11	3.72	56.89
PO7	34.10	4.66	7.54	8.86	58.25
PO8	31.54	4.20	7.555	8.65	61.61
EV1	46.95	10.26	17.53	20.31	59.66
EV2	46.87	6.36	12.50	14.02	63.03
EV3	47.71	4.77	11.98	12.89	68.29
EV4	46.41	6.70	15.59	16.97	66.75

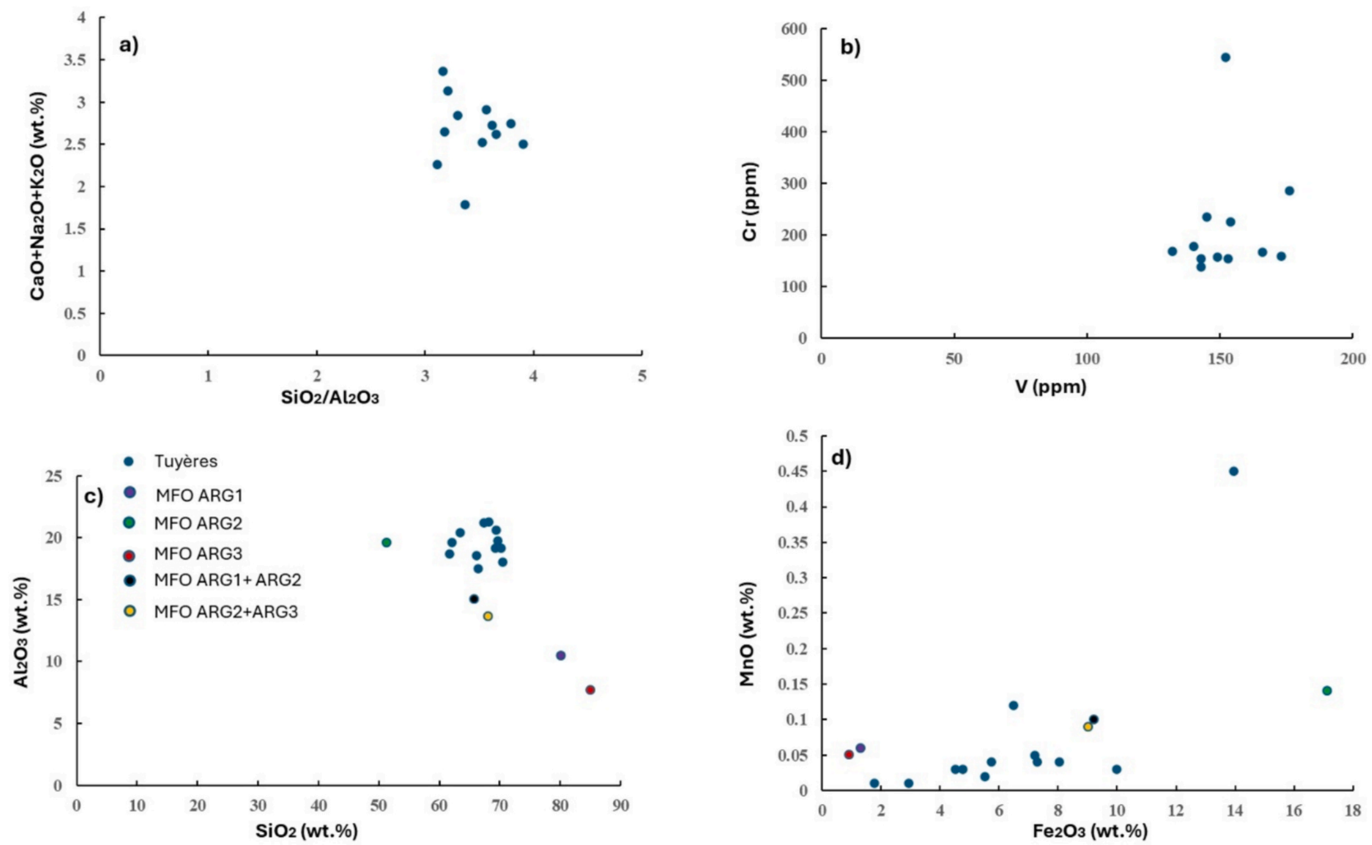

Fig. 8. (a, b) Chemical binary correlation diagrams of the tuyères a. Al_2O_3/SiO_2 versus ($CaO + K_2O + Na_2O$), b. V/Cr; (c, d) Correlation diagrams between local clays (MFO ARG, from Epossi Ntah 2012) and tuyères c. SiO_2/Al_2O_3, d. Fe_2O_3/MnO.

between 350 and 500 °C (Murad and Wagner, 1989) and disappears before 500 °C (Murad and Wagner, 1989), or around 550 °C, but never resists temperatures above 600° C (Mercader et al. 2000). Therefore, the tuyères containing kaolinite were exposed to temperatures below 600 °C. However, they experienced at least 400 °C, when sintering reactions begin (Artioli, 2010), preventing their disintegration after deposition. Kaolinite reacts to metakaolinite between 500 to 600 °C (Epossi Ntah et al. 2017; Cultrone et al. 2001; Cultrone and Carrillo Rosua 2020; Moon et al. 2021). Therefore, samples PO1b, PO3, PO4 and EV1, containing mica but not kaolinite, were exposed to temperatures ranging from 500 to 900 °C because mica completely decomposes between 800 and 1000 °C (Maggetti 1982; Epossi Ntah et al. 2017; Cultrone et al. 2001; Cultrone and Carrillo Rosua 2020; Quinn 2022; Singh et al. 2025). This variation is also related to the hydric behaviour of the tuyères. The samples that absorbed more water are related to the higher porosity (more than 19 %) and low firing temperature exposure around 700 °C for example (Daghmehchi et al. 2023) except for the vitrified tuyères. Tuyères PO6, PO7, PO8 have a vitrified microstructure, they are covered by slag layers and contain mullite, indicating exposure to temperature of at least 1000 to 1100 °C (Lee et al. 2008). EV4, containing cristobalite and having the most vitrified microstructure, was likely exposed to a temperature of 1100–1200 °C.

The surface colour/ appearance of the tuyères is also affected by the variation of temperature. Among the samples with the highest chromatic coordinates (a* and b*) and C* (Table 5), the colours of samples PO1b and EV1 do not appear altered by heating; the colours of the original clay (dark brown and light grey) are visible. These samples were exposed to lower temperatures than PO1a, PO2, PO3 and PO4, which exhibit two colours (black and red) as a result of heating. The red colour is due to the formation of hematite, which starts above 700 °C in CaO poor samples and indicates an oxidising atmosphere, whereas a black colour indicates the presence of magnetite due to reducing conditions (De Bonis et al., 2017). Samples PO6, PO7 and PO8 are characterised by their dark colour and the low values of chromatic coordinates (a* and b*) and C*. In contrast to the other samples, the colour of these samples is not from the clay but the slag or vitrified material that covers their surface. Their colour can therefore not be compared to the colour of the other samples.

The reconstructed temperatures suggest a maximum beyond 1000 °C, probably even beyond 1200 °C. This is in agreement with previous research of Essomba (1992) who mentioned that smelting temperature was not higher than 1200 °C in Oliga, and David et al. (1989) who in their ethnographic study of a smelting operation in Northern Cameroon indicated temperatures of at least 1100 °C at the tip of the tuyere, when the tip started to melt. This is also in agreement with Freestone and Tite (1986) and Hein et al. (2007) who estimated the temperature by the degree of vitrification of ancient furnace refractories to the range of 1100 to 1250 °C with a suggested maximum temperature of 1400 °C in early ironworking processes (Freestone and Tite, 1986). Oral sources from Central Cameroon reported that tuyères penetrated the furnace up to the level of combustion (Essomba 1992), which is well reflected in the observed variation of the temperature. Since the shape and operational mode of the furnaces is yet to be reconstructed, and the hottest zone of the furnace is in front of the tuyères, additional research is needed to reconstruct the maximum operating temperature of the furnaces in Pongsolo and Evian. Nevertheless, it can be assumed that PO6, PO7, PO8 and especially EV4 were likely placed closer to the hottest zone, as inferred from vitrification levels and slag presence. This is also confirmed by the low values of the absorption water porosity of these samples (5 % against 18 % for the other samples). The other samples came from parts of the tuyères located further away from the hottest zone with EV2, EV3, PO1a and PO2 maybe placed furthest from the centre of the furnace, i.e. close to the furnace walls. This reconstruction admittedly depends on the sampling location within the tuyère fragment, but the features presented in the samples provide a good coverage over the temperature gradients captured in the different fragments. For example, vitrification and slagging of a fragment is also present in the respective thin section.

4.6.3. Comparative study of the tuyères from other sides of Africa

Regarding the internal diameter of both tuyères, their values of 2–4 cm are small according to Sutton (1976), who described large and small types of tuyères in Nigeria with values of 5–12 cm and 3–5 cm, respectively. The tuyères from Lekie are comparable in their size to some of those found in Nyango (Zimbabwe), with values between 3 and 4 cm (Chirikure and Rehren, 2004). The small internal diameter of the Evian and Pongsolo tuyères (2–4 cm) suggests that they were used with a bellows, as Martinon-Torres and Rehren (2014) stated that internal diameters of 1–2 cm are generally associated with bellows. Moreover, Rehder (1994) confirmed that tuyères with an internal diameter of around 3 cm are characteristic of bellows-operated tuyères. These internal diameter values could also indicate the use of a forced draught shaft in Lekie, as described by Chirikure (2015). This is consistent with the single-piece arrangement, as opposed to the multiple or fused tuyères found in Burkina Faso, Zimbabwe, and Tanzania, which suggest a natural draught furnace (Mtetwa and Lindahl, 2017; Chirikure et al., 2009; Lyaya, 2009) or a natural draft installation (Holl, 2009). Regarding length, the tuyères from Pongsolo belong to the short category (10–12 cm), in contrast to the longest ones (40–50 cm) found in Tanzania (Mapunda and Lyaya, 2014) and Nigeria (Sutton, 1976). Due to missing parts of the Evian tuyères, their length cannot be determined. In terms of wall thickness, the tuyères of Pongsolo and Evian have values between 0.5 and 1.5 cm. This is in good agreement with the values found in Zimbabwe (Nyango) (Chirikure and Rehren, 2004), but they are circular in shape, in contrast to the cylindrical shape identified in most tuyères. However, funnel-shaped tuyères have been found in the Ivory Coast (Seernels, 1998), as well as in Evian (Elouga, 2000).

Regarding the raw materials, chemical analyses show that tuyères from Lekie were produced using a silica-rich, alumina-poor and alkali-poor raw material. This is consistent with the results obtained by Chirikure and Rehren (2004) for some tuyères collected in Zimbabwe, which were also rich in SiO_2 (70 wt%), poor in Al_2O_3 (15–20 %) and alkalis (3–4 %), although the alkali oxide content was slightly higher than that of the Lekie tuyères. Similar results were obtained by Iles (2020) for the chemical composition of tuyères from Tanzania: 60–66 wt% SiO_2, 18–22 % Al_2O_3 and 1–4 % alkalis. These results demonstrate that ironworkers in Cameroon, Zimbabwe and Tanzania used raw materials rich in quartz or quartz sand in order to achieve refractory properties during the ironworking process. Generally, quartz is a natural inclusion in the raw material and is not tempered (Chirikure and Rehren, 2004). Thus, the high quartz content and low flux oxide content of the tuyères indicates their refractoriness during the ironworking process, showing that the ironworkers had the necessary skills to select the appropriate raw materials for the tuyères in West, Central and Southern Africa. Although variation in the typology of tuyères is observed in Africa due to the variety of furnaces, those from Lekie show similarities in typology and raw material characteristics with those of other African countries. These results are consistent with archaeological and ethnographic studies that identified bellows-operated furnaces and/or three-step production processes (smelting, refining, and smithing) in southern and central Africa/ sub-Saharan Africa (Lyaya et al., 2012; Chirikure et al., 2009).

5. Conclusions

The archaeometallurgical study of twelve tuyères fragments from Lekie (eight from Pongsolo and four from Evian) was done by chemical, mineralogical, petrographic and physical analyses with the aim to understand the nature of raw materials and the firing temperatures reached in the furnace. Macroscopically, samples from Pongsolo are reddish with some thermal-effects on their surface except for one sample, whereas three of the four samples from Evian are greyish without evidence of thermal effects on their surfaces. From a typological point of view, these tuyeres are small (Evian and Pongsolo) and short (case of Pongsolo and indetermined for Evian) according the description of the internal diameter and length of the tuyères found in other regions of Africa. They are single pieces and their internal diameter of 2–4 cm could indicate a bellow-driven forced draught shaft furnace. All samples are made from silica-alumina rich clay materials with a variable amount of iron oxide, higher in Pongsolo with respect to Evian samples. The high content of silica in all samples and the morphology of the quartz grains suggest its presence in the natural clays rather than the addition of quartz sand as temper. Chemical correlation between the tuyères and local clay samples indicates the use of local clay as raw materials for the tuyères from Pongsolo and Evian. The mineralogical and petrographic analyses reveal three mineralogical and microstructural groups within the samples, which are the result of different firing temperatures. Based on kaolinite retention, some samples experienced temperatures $<$ 600 °C. This probably represents a part of the tuyère located close to the furnace walls. The presence of mica and the absence of kaolinite in other samples indicate exposure to temperatures of 600–900 °C. Four samples, one from Evian and three from Pongsolo, showed the presence of mullite or cristobalite, indicating firing temperatures $\geq$ 1000 °C. These results are consistent with the low open porosity values of these samples, which suggest a high level of vitrification of these tuyères due to melting close to their tips. The original composition of the local clay very rich in quartz and poor in flux oxides made the tuyères resistant to high temperatures by improving their thermal stability during the ironworking processes. This result shows that the ironworkers have a good understanding of the properties of the raw materials needed to produce tuyères. The observed similarities in terms of the typology and characteristics of the raw material with those of the tuyères from other regions of Africa are consistent with archaeological and ethnographic studies that identified the use of bellows-operated furnaces during ironworking in sub-Saharan Africa.

CRediT authorship contribution statement

Zoila Luz Epossi Ntah: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Conceptualization. **Giuseppe Cultrone:** Writing – review & editing, Methodology, Investigation, Formal analysis, Visualization, Validation. **Thomas Rose:** Writing – review & editing, Methodology, Investigation, Visualization, Validation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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