

# Paul-Louis Cyfflé's (1724–1806) search for porcelain

MARINO MAGGETTI

Department of Geosciences, Mineralogy and Petrography, University of Fribourg, Chemin du Musée 6,  
1700 Fribourg, Switzerland

\*Corresponding author, e-mail: [marino.maggetti@unifr.ch](mailto:marino.maggetti@unifr.ch)

**Abstract:** Seven unglazed figurines from private collections, attributed to Cyfflé's *Terre de Lorraine* manufacture in Lunéville (1766–1780), were subjected to porosity and scanning electron microscopic analyses to determine bulk element compositions and the composition and nature of their constituent phases. One figurine bears the mark CYFFLÉ À. LUNÉVILLE, three the mark TERRE DE LORRAINE. The studied samples pertain to four compositional groupings: (1) Two porous (18–20 % water adsorption W.A.) bodies containing milled quartz-rich frit + anhydrite (former gypsum) + phyllosilicate + Ca-rich matrix; (2) A soft-paste (artificial) porcelain body (10 % W. A.), containing quartz + calcic plagioclase (An<sub>88–95</sub>) + glassy matrix. The latter is inferred to derive from a former frit. A coronitic, amorphous (as revealed by electron backscattered diffraction analysis) reaction rim is visible around the quartzes. The K-rich and Na-poor composition of the frit is best explained as a mixture of potassium nitrate, alum, calcined gypsum, sand, and moderate amounts of salt and soda; (3) A porous (23 % W. A.), hybrid porcelain body with finely milled particles of quartz, mullite-bearing hard-paste porcelain, Na-Ca-siliceous glass and metakaolinite; (4) Three hard-paste porcelain bodies, some with relict quartz, andesine plagioclase (An<sub>37–45</sub>), pseudomorphs of kaolinite and the liquidus phase mullite in a glassy matrix. Well fired figurines have no W. A. due to the pervasive former melt phase, underfired figurines 7 %. These wares can contain small amounts of lead (1.8 wt% PbO) and SO<sub>3</sub> (0.6 wt%), suggesting the use of lead frit and gypsum. The diversity of Cyfflé's production is now better recognized. His trial-and-error experiments made use of a remarkably wide range of paste mixtures, with porcelain bodies in the French (soft-paste) and the German (hard-paste) tradition.

**Key-words:** Terre de Lorraine, Paul-Louis Cyfflé, soft-paste porcelain, hard-paste porcelain, Lunéville, microchemical, microstructural analysis, technology.

## 1. Introduction

### 1.1. Paul-Louis Cyfflé's *Terre de Lorraine* manufacture in Lunéville (1766–1780)

Paul-Louis Cyfflé (1724–1806), a Belgian sculptor, was «modeler, chiseler and sculptor to the King» during the years 1751–1763 at the court of Nancy or of Lunéville of the former Polish king Stanislas Leszczyński (1677–1766), Duke of Lorraine since 1737 (Fig. 1). A detailed account of Cyfflé's "vita" can be found in Noël (1961), Thomaes & Van den Abeele (2008), Calame (2009), and a short one in Maggetti *et al.* (2010). Cyfflé was an excellent sculptor with great interest in creating ceramic figurines. Only few documents describe his activities in the years 1758–1767. He probably worked in Saint-Clément, experimenting with different recipes of white earthenware and producing moulds for several figurines, and in Ottweiler, where the Count of Sarrebruck offered him the direction of the faïence factory which he had founded 1763 (Noël 1961, pp. 48–57). On 5 February 1763, the faïence manufacture of Saint-Clément, founded 1758 by Jaques II Chambrette (*ca.* 1705–1758), was sold to the Loyal + Mique + Cyfflé associates, but Cyfflé quit his parts October 19 of the same year (Calame,

2009, p. 26). The annexion of Lorraine to the kingdom of France, after the death of King Stanislas (23 February 1766), most probably triggered Cyfflé to ask in Paris in December 1766 the permission to found a manufacture "to produce at home in Lunéville earthenware which, without actually being porcelain, would be more beautiful than pipe clay, and called *Terre de Lorraine*" (Houry, 1954; Noël, 1961, p. 87). He also wrote that the gains of the manufacture would enable him to continue his experiments to obtain a porcelain paste. On 3 May 1768, an edict of the Council of State authorized him "to establish at Lunéville a manufacture where he could fire, or let fire during 15 years, tableware which would be superior to pipe clay, without being porcelain, and named *Terre de Lorraine*, as well as common and ordinary faïence using pipe clay". On 1 June 1768, official permission was given for the porcelain research (Calame, 2009, p. 31). But Cyfflé did not wait for these permissions, since he started to work with several artisans already in 1766. He specialized in the production of small unglazed and undecorated white figurines, which he had probably been experimenting with since the middle of 1765 (Noël, 1961, pp. 87–90, 92). It is an open question if he ever produced crockery. His activities and his porcelain experiments had to respect the edict of the Royal Council of 15

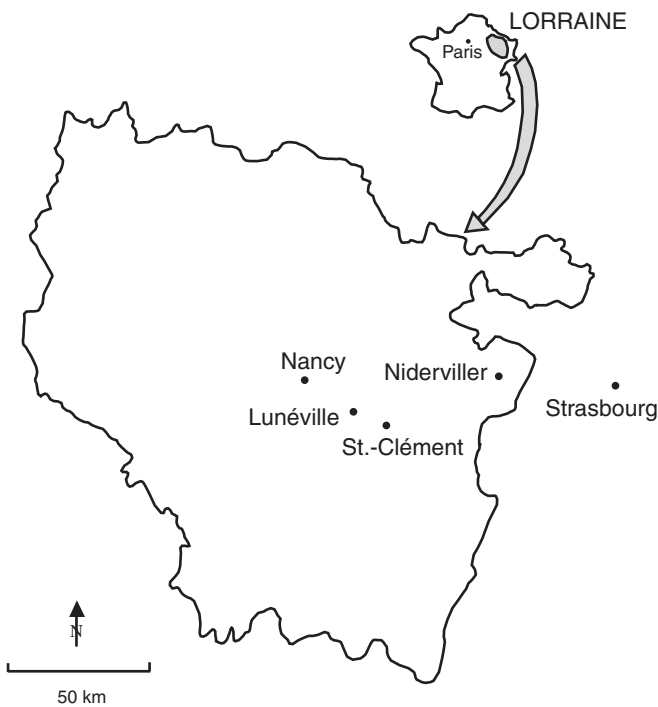


Fig. 1. Map of Lorraine showing location of places mentioned in the text.

February 1766, protecting the Royal porcelain manufacture of Sèvres. Permission was given for the production of porcelain, painted blue or white or *en camaïeu* with another colour, but not for statues, figurines and ornaments in porcelain biscuit, with or without glaze – for a fine of 3000 pounds, the confiscation of the goods and the destruction of the kilns (Gerspach, 1891, p. 103). Therefore, in 1769, having found a washable “marble paste”, Cyfflé asked for another permission to produce biscuit figurines with this “marble” body and to continue his research. With his letter, a small head made of the “marble paste” was sent to Paris and from there to Sèvres. The director of the Royal porcelain manufacture stated that Cyfflé had improved his paste to resemble porcelain and that he should be encouraged to continue his experiments, with the injunction that his objects should not be named other than earthenware (Gerspach, 1891, p. 101). Cyfflé’s manufacture was prosperous – he employed 27 people in 1775 – but closed down in 1780. In the beginnings, the figurines were sometimes marked with CYFFLÉ À LUNÉVILLE, later with TERRE DE LORRAINE or T.D.L.. Cyfflé’s production, mostly classical nudes and sweet rustic figurines, is discussed in Calame (2009).

## 1.2. Short history of soft- and hard-paste porcelain production in France

### 1.2.1. Soft-paste porcelain

French soft-paste (*pâte tendre*), an artificial porcelain, also called frit or glassy porcelain, made without kaolin, was

first produced in Rouen by Edme Poterat, a faïence maker, shortly after 1673 (Soudée-Lacombe, 2006). The Rouen porcelain ceased with the death of Poterat in 1696. In Europe, the industrial production of this ceramic material started around 1690–1693 in Saint-Cloud, a faïence manufacture since 1664, which closed in 1766 (Le Duc & de Plinval de Guillebon, 1991). Many other factories followed such as Vincennes, founded around 1740 (d’Albis, 1984), with the Royal privilege since 1745, granting director Charles Adam the exclusive right to produce figurines of humans and animals. With the intention of imitating the then highly priced German hard-paste porcelain figurines from Meissen, the first soft-paste porcelain biscuit figurines currently produced by Vincennes were sold on 21 November 1752 (d’Albis, 2003, p. 114). The factory was then transferred to Sèvres in 1756 and became exclusive royal property in 1759. Brongniart (1770–1847), director of the porcelain manufacture of Sèvres (1800–1847), distinguishes in his fundamental treatise (1844, II, p. 465) two varieties of artificial porcelain: (1) The *fine artificial soft porcelain*, obtained from a frit made by melting sand from Fontainebleau with five fluxes: sea salt (NaCl), saltpetre (e.g., KNO<sub>3</sub>), calcined alum (e.g., KAl[SO<sub>4</sub>]<sub>2</sub>·12 H<sub>2</sub>O), Alicante soda (Na<sub>2</sub>CO<sub>3</sub>) and calcined gypsum (CaSO<sub>4</sub>·2H<sub>2</sub>O). This frit was crushed and finely ground, then washed with hot water and finally mixed together with white chalk and a marl from the gypsum-bearing strata in Argenteuil, which turns white after firing. Soap and glue were added to improve plasticity. Firing temperatures were around 1200 °C. Sèvres produced this kind of body from 1748 to 1804 (Treppoz & d’Albis, 1987). (2) The *common artificial soft porcelain*, as produced in Tournai (since 1750) or Saint-Amand-les-Eaux (since 1815), had a simpler recipe: a frit (grey sand with Alicante soda) was mixed with white chalk and a white firing marly clay.

### 1.2.2. Hard-paste porcelain

In 1751 Joseph Jacob Ringler went from the porcelain manufacture Höchst to Strasbourg, where Paul Hannong (ca. 1700–1760) acquired from him the hard-paste porcelain secret. This production was affected very soon by a Royal council’s edict (19 August 1753) giving Vincennes’s director Eloi Brichard the exclusive privilege to produce porcelain, and Paul Hannong had to destroy the porcelain kilns (Bastian, 2002, p. 73). He quit France with most of his porcelain specialists and established 26 May 1755, patronized by Carl Theodor, Elector of the Palatinate, a new production site at Frankenthal (Bastian, 2002, p. 74). In Lorraine, hard-paste porcelain production started 1759 in Niderviller with the help of craftsmen from Strasbourg and from Saxe using kaolin from Passau in Austria (Soudée-Lacombe, 1984). On 1 September 1763, Pierre Hannong (1739–1793), one of Paul’s sons, sold the secret of making hard-paste porcelain to Sèvres (Bastian, 2002, p. 80). But the production did not seriously start until the discovery of kaolin deposits near Saint-Yrieix-la-Perche (Haute-Vienne, 26 km south of Limoges) in 1768 (d’Albis, 2003, p. 26), after many preliminary work, as

evidenced by Sèvres manufacture's book of experiments for the period 1768–1780 mentioning the testing of more than 200 different recipes, based primarily on the same principle as the Meissen porcelain (Treppoz & d'Albis, 1987; d'Albis, 2003). After the introduction of hard-paste porcelain at Sèvres in the year 1769 (d'Albis, 2003, p. 26) and the publication of Milly's treatise (Milly, 1771), the hard-paste production technique was no longer a secret. The restrictions on other French factories were gradually relaxed and many new blossomed. In Paris alone, 12 small centres made hard-paste porcelain during the years 1773–1784, some under the protection of members of the royal family (De Plinval de Guillebon, 1995, p. 91). According to Brongniart (1844), the early Sèvres hard-paste porcelain was a mixture of 70 wt% kaolinitic clay, 12 wt% coarse sandy kaolin (where the quartz and kaolin are visible with the naked eye), 9.2 wt% kaolinitic clay sand, 5.3 wt% Aumont sand and 3.5 wt% lime (equivalent to 6.3 limestone,  $\text{CaCO}_3$ ). The hard porcelain paste used for sculpture was obtained by mixing 62 wt% coarse kaolin clay, 17 wt% feldspar, 17 wt% Aumont sand and 4 wt% chalk ( $\text{CaCO}_3$ ). The kaolinitic clay is a nearly pure kaolinite obtained by a mechanical treatment of coarse kaolins from Saint-Yrieix. Aumont sand came from the village of Aumont-en-Halatte, situated North of Paris in the department of Oise. Both types of kaolin sand result from the washing of raw kaolin and contain quartz and feldspar.

## 2. Previous research and aim

Cyfflé's recipe(s) for making *Terre de Lorraine* biscuit figurines is not known. Macroscopic examination of his figurines reveals soft and porous as well as extremely hard and dense bodies (Peiffer, 2007; Calame, 2009). The latter are either opaque or translucent, both with signs of the "paste memory", typical for true porcelain, as is translucence (Calame, 2009, p. 57). One gains the evidence that Cyfflé constantly adapted and improved his recipe, from white earthenware to soft-paste porcelain (Maire, 2008, p. 14). The figurine's body could be: (1) Hard-paste porcelain. In his letter to the minister Bertin (7 July 1769), Trudaine wrote that the marble paste head of Cyfflé could only have been made with a porcelain body (Gerspach, 1891, p. 100). Cyfflé himself stated in a letter from 8 April 1780, that he sells "excellent bodies for the manufacture of porcelain as well as terre de pipe" (Grollier, 1906, p. 441). (2) Soft-paste porcelain. According to M. O\*\*\* (Oppenheim, 1807, p. 272), Cyfflé's body is: «a mixture of niter, a little sea salt, Alicante soda, alum, gypsum and a lot of siliceous sand fritted together, and then heated to a state of molten paste. After cooling down, finely ground white marl is added to the mixture». Peiffer (2007, p. 145) compares the *Terre de Lorraine* to Tournai soft-paste porcelain, and the flux could also be a *terre de pipe* (pipe clay). (3) Bone ash phosphatic porcelain (Delorme, 1927, p. 170 with reference to Castara, 1785); (4) White earthenware

(*terre de pipe*) biscuit fired at temperatures of stoneware (Peiffer, 2007, p. 144).

To solve these questions, archaeometric research is needed. However, one understands the reluctance of curators and private collectors to allow sampling of their intact objects. Therefore, Maggetti *et al.* (2010) choose four figurines from the collections of the Castle of Lunéville, destroyed by the blaze of 2 January 2003. Sadly, marks couldn't be found, but meantime it was confirmed that the "Hercules and Omphale" figurine (samples TBL 24, 26–29) had the mark TERRE DE LORRAINE (G. Demat, pers comm 2010). The four figurines are porous and belong to two distinct pastes, a calcareous and an aluminous-siliceous one. The first, a classical Lorraine *terre de pipe* body, consists of a mixture of different proportions of quartz or calcined flint with a refractory clay, ground Pb-glass and calcium carbonate or calcium hydroxide. The second, an invention of Cyfflé, is a mix of different elements such as calcined and ground quartz, ground pure amorphous  $\text{SiO}_2$ , ground porcelain, ground Na-Ca-glass and coarse grained kaolinite with a fine grained kaolinitic clay, giving, after firing, a porous body with a hard-paste porcelain composition.

The aim of the present study was to analyze intact Cyfflé's figurines in order to contribute further to the identification of the compositional nature of Cyfflé's bodies, to the raw materials used and the technology involved.

## 3. Samples and experimental procedures

Seven unglazed figurines from private collections, three with the mark TERRE DE LORRAINE, one with the mark CYFFLÉ À LUNÉVILLE, were sampled (Table 1). Figurines without mark were attributed by Calame (2009) through stylistic analyses to Cyfflé's workshop. The sampling included porous as well as hard, dense and translucent porcelain-like specimens. A small fragment (about  $3 \times 3 \times 3 - 5 \times 2 \times 2$  mm/35–96 mg) was cut from each object using a thin diamond saw. Analytical procedures concerning scanning electron microscopy (SEM), electron backscatter diffraction (EBSD) and open porosity (water adsorption) are outlined in Maggetti *et al.* (2010). Theoretically, a secondary porosity linked to sampling and polishing could increase the pristine porosity. However, the pores of the samples were filled with resin prior to grinding and polishing. It is therefore assumed that the measured porosity is very close to the pristine one. Reported bulk compositions represent the average of 4–10 area ( $1.6 \times 1.2$  mm to  $400 \times 300$   $\mu\text{m}$ ) measurements of the paste. Phase compositions were determined by spot and small-area analyses.

## 4. Results

The SEM examination of the seven fragments led to the identification of five microstructures.



Table 1. List of the analyzed specimens from private collections and their open porosity data. W.A. = water adsorption. Dating according to Calame (2009).

An. No.	Object	Marks and signatures	Photo in Calame (2009)	W.A. (%)
TBL 35	The Naiade	TERRE DE LORRAINE	p. 57	0.23
TBL 36	Leda and the swan, <i>ca.</i> 1770	TERRE DE LORRAINE, C.G. or G.G. <sup>a</sup>	p. 165	22.71
TBL 37	The Gardener with the shovel, <i>ca.</i> 1770		p. 145	19.71
TBL 38	The barefooted Shepherd with his dog, <i>ca.</i> 1770	CYFFLÉ A. LUNÉVILLE, G. <sup>b</sup>	p. 148	17.70
TBL 39	The pleasant lesson, <i>ca.</i> 1770		p. 108	6.64
TBL 40	Venus correcting Amor, <i>ca.</i> 1775	TERRE DE LORRAINE, Jacque	p. 164	0.00
TBL 41	Hercules and Omphale, <i>ca.</i> 1770		p. 167	10.45

Notes: <sup>a</sup>Signature of an unknown artist (Calame, 2009, p. 49).

<sup>b</sup>Signature of Jean-Baptiste Grandel (Calame, 2009, p. 46).

#### 4.1. Microstructure 1 (TBL 37, 38)

Both pastes contain angular, *i.e.* crushed  $\alpha$ -quartz particles (often rimmed by a small layer of a tin oxide bearing glass), phyllosilicates, calcium sulphates, visible as tiny crystals, and calcium-rich dark particles A (Fig. 2, Table 2). The EBSD identified quartz and proved the amorphous nature of the glass, but failed, due to the high porosity and the microgranular aspect, to solve the crystallographic nature of the Ca-S phase, assumed to be anhydrite ( $\text{CaSO}_4$ ). The phyllosilicates show compositions of illitic biotites.

Both bodies are silica-rich, with around 70–74 wt%  $\text{SiO}_2$  and 10 wt%  $\text{Al}_2\text{O}_3$  (Table 3). CaO is present at 11 wt%, and  $\text{SO}_3$  at 2–5 wt%. The other oxides are below 2 wt%. TBL 37 shows some ZnO (0.3 wt%). The open porosity is high, at around 18–20 %.

#### 4.2. Microstructure 2 (TBL 41)

At lower magnification, one can see dark grey particles (5–15  $\mu\text{m}$  length) embedded in a glassy matrix (Fig. 3a). Porosity (10.4 % open porosity, Table 1) is documented as rounded pores with maximum sizes of about 25  $\mu\text{m}$ . A  $\text{TiO}_2$  phase, most probably rutile, is present in very subordinate amounts. At higher magnification, the dark grey particles show  $\alpha$ -quartz cores, as shown by EBSD, with an outer, amorphous Si-Al-Ca corona (Fig. 3b and c). The matrix is a heterogeneous mixture of tiny subidiomorphic particles and xenomorphic areas, with different greyish to whitish shades under the SEM. Owing to the fine grain size, analysis proved difficult. Microchemical analyses of these phases show varying amounts of three main constituents, namely  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and CaO (Table 2). Both

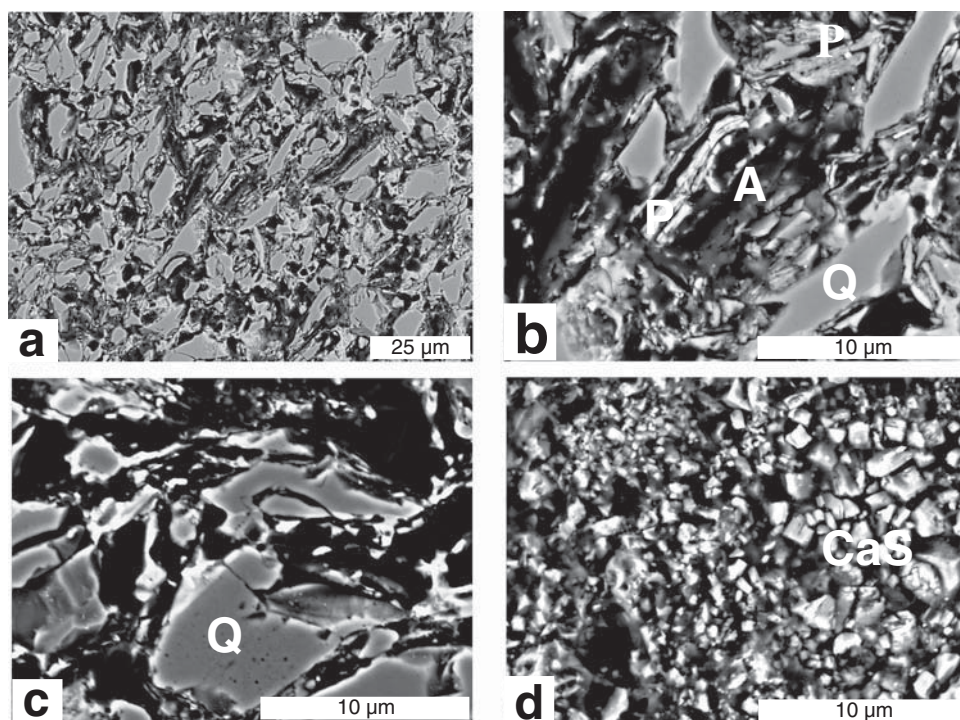


Fig. 2. Backscattered electron images of microstructure 1: (a, b, c) The body contain angular, unreacted quartz (Q) with small rims of a glassy layer (whitish), phyllosilicate (P) and dark particles (A). TBL 38; (d) Tiny clustered Ca-S crystals, probably anhydrite (TBL 37).

Table 2. Compositions of specific phases as revealed by EDS analyses, normalized to 100 %.

An. No. and Phase	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	SO <sub>3</sub>	K <sub>2</sub> O	SnO <sub>2</sub>	CaO	TiO <sub>2</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	Fe <sub>2</sub> O <sub>3</sub>	ZnO	PbO
TBL 36														
Phase A ( <i>n</i> = 11)	0.7	0.1	22.1	73.2		2.9		0.6				0.4		
Phase B ( <i>n</i> = 12)	1.8	0.1	27.1	64.2		1.7		4.7				0.4		
Phase C ( <i>n</i> = 16)	0.5	0.1	41.6	54.2		2.4		0.4				0.8		
TBL 37, 38														
Glassy rim of quartz ( <i>n</i> = 11)	0.3		9.1	77.7	0.3	0.9	2.3	9.0	0.1			0.3		
Phase A ( <i>n</i> = 13)	0.6		12.2	58.0	1.9	1.7		24.7	0.3			0.6		
Phyllosilicate ( <i>n</i> = 1)		4.7	13.5	46.7		7.7		3.9	2.2		1.1	20.2		
TBL 39														
Phase A ( <i>n</i> = 8)	4.4		26.1	61.2	0.2	0.7		6.2				0.1	0.2	0.9
Phase B ( <i>n</i> = 9)	1.6		33.3	59.9	0.2	1.4		1.2				0.3	0.2	1.9
Phase B (Fig. 7c)	1.9		42.3	51.9		1.2		1.1				0.3		1.3
TBL 40														
Phase A1 (Fig. 8c, <i>n</i> = 1)	0.8		37.5	58.3		1.8		1.2	0.1			0.3		
Phase A2 (Fig. 8d, <i>n</i> = 1)	1.1		41.7	53.9		1.6		1.1	0.2			0.4		
Phase B (Fig. 8c, <i>n</i> = 1)	0.8		38.5	57.0		1.8		1.4	0.2			0.3		
TBL 41														
Phase A1, light grey ( <i>n</i> = 4)	0.2	0.2	32.7	49.1		0.6	0.2	16.0		0.5		0.5		
Phase A2, light grey ( <i>n</i> = 4)	0.3		24.9	60.6		0.8		12.5		0.4		0.5		
Phase B, whitish ( <i>n</i> = 9)	0.3		14.5	73.2		0.9	2.0	7.9	0.3	0.4		0.5		
Phase C, grey ( <i>n</i> = 22)	0.1	0.2	14.1	73.4	0.1	3.5		7.5	0.3	0.2		0.6		
Phase D, corona ( <i>n</i> = 8)	0.2	0.1	9.6	82.2	0.1	0.8	1.6	4.8	0.1	0.1		0.4		

Table 3. Bulk compositions of the analyzed specimens by EDS, reported as normalized oxide percentages (wt%) compared with selected analyses of other manufactures.

An. no. (TBL)	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>	ZnO	PbO	Total	Reference
35 ( <i>n</i> = 5)	64.3		28.9	0.3		2.1	2.5	1.9				100.0	
36 ( <i>n</i> = 5)	65.3	0.2	29.9	0.8	0.4	0.5	0.4	2.1	0.4			100.0	
37 ( <i>n</i> = 4)	70.3	0.4	10.2	0.4		11.3	0.3	1.3	5.5	0.3		100.0	
38 ( <i>n</i> = 6)	74.1	0.4	10.2	0.5		11.5	0.2	1.4	1.7			100.0	
39 ( <i>n</i> = 9)	58.8		31.1	0.3	0.3	2.4	2.9	1.5		0.9	1.8	100.0	
40 ( <i>n</i> = 10)	66.1		27.1	0.4		2.0	2.3	2.1				100.0	
41 ( <i>n</i> = 5)	81.0	0.2	9.8	0.5	0.3	6.1	0.2	1.3	0.6			100.0	
Previously analyzed Cyfflé's figurines (XRF)													
17	56.44	0.49	21.37	0.84	0.42	16.08	0.88	2.46			1.97	100.95	Maggetti <i>et al.</i> (2010)
24 <sup>a</sup>	63.97	0.11	29.97	0.32	0.22	1.25	2.23	2.31			0.40	100.78	Maggetti <i>et al.</i> (2010)
25	81.55	0.32	9.33	0.34	0.23	6.35	0.94	1.12				100.18	Maggetti <i>et al.</i> (2010)
34	71.45	0.30	24.67	0.55	0.12	0.47	0.92	1.69			0.30	100.47	Maggetti <i>et al.</i> (2010)
Continental, Mg-poor soft-paste porcelain													
Saint-Amand <i>ca.</i> 1835	75.3		8.2			10.0		5.0				98.50	Brogniart (1844)
St. Cloud SC-5	79.59	0.10	4.26	0.49	1.02	7.10	1.96	4.37			0.06	98.95	Kingery & Smith (1985)
Continental hard-paste porcelain													
Meissen 1715	57.4	0.2	35.2	0.1	2.0	3.6	1.2	0.3				100.00	Ullrich & Ballmaier (2002)
Sèvres 1770–1836	58.0		34.5			4.5		3.0				100.00	Brongniart (1844)
Sèvres figurines 1794–1834	64.23		30.05			2.89		2.79				99.96	Brongniart (1844)

Note: <sup>a</sup>Mean of TBL 24, 26–29 (Maggetti *et al.*, 2010).

phases A1 and A2 approach the composition of anorthitic feldspar (Table 2, Fig. 4). The EBSD patterns of these particles were weak, impossible to identify. Phase A1 (An<sub>91–95</sub>) is concentrated in patches, whereas phase A2 (An<sub>88–92</sub>) occurs as irregular grains protruding from these

patches into the K-bearing matrix C or as idiomorphic to subidiomorphic particles in this phase. Particles B, present in minor amounts, differ in their composition from phase C by a significant concentration of tin oxide. The quartz corona has very high SiO<sub>2</sub>, with moderate Al<sub>2</sub>O<sub>3</sub> and

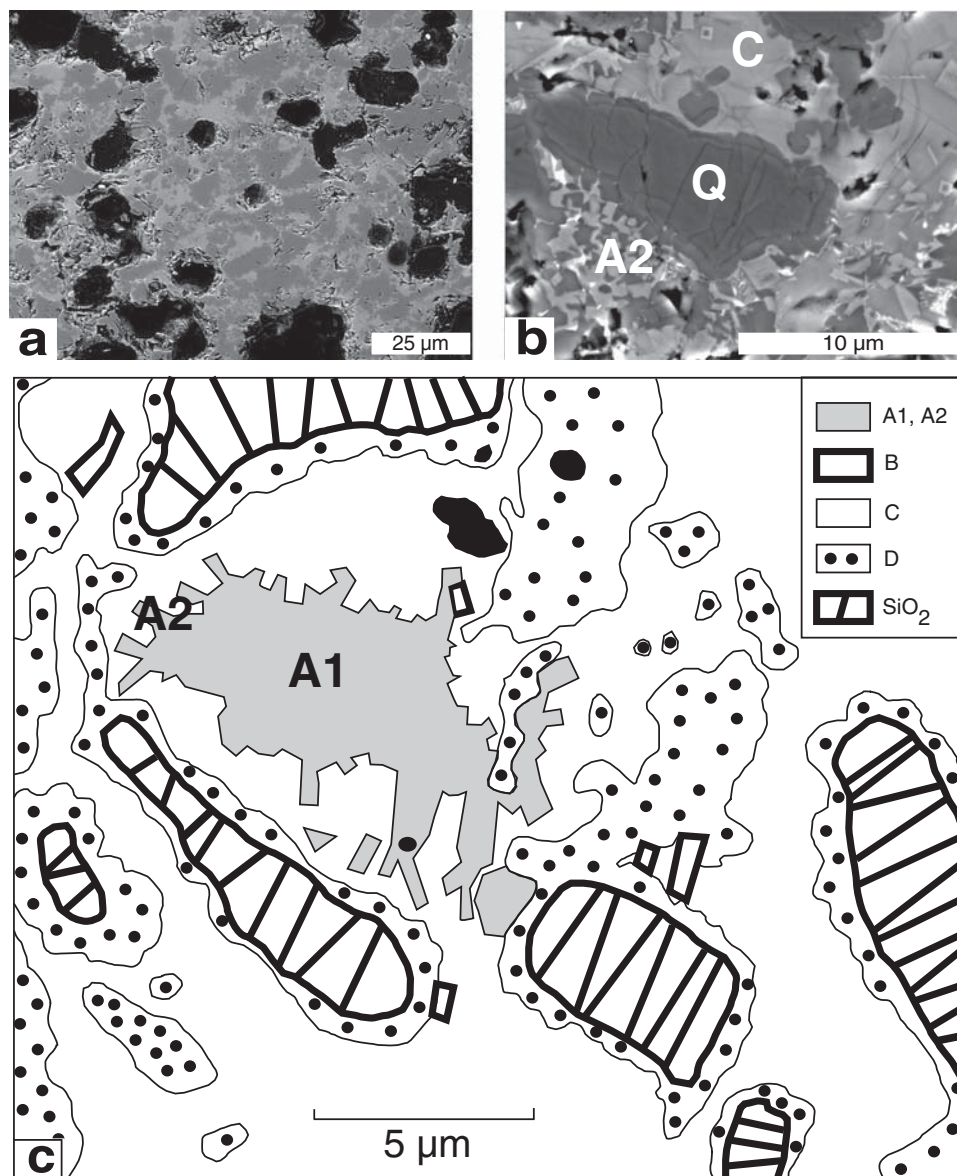


Fig. 3. Section of TBL 41, as seen in the SEM: (a) Grey particles are  $\alpha$ -quartz in a glassy matrix (light grey). Roundish voids appear black; (b)  $\alpha$ -quartz (Q) grains are rimmed with a darker grey, glassy corona. Whitish inclusions in the glassy matrix (C) are crystals of anorthite A2; (c) Sketch of microstructure 2.

CaO, and subordinate  $K_2O$  and  $SnO_2$ . Tin oxide is dissolved in the glass since no cassiterite crystals were observed.

The bulk composition of the body is characterized by elevated silica (81–82 wt%  $SiO_2$ ) contents, moderate lime (6 wt% CaO) and alumina (9–10 wt%  $Al_2O_3$ ) contents, and low alkalis ( $K_2O$  1–1.5,  $Na_2O$  < 1 wt%) contents (Table 3).

#### 4.3. Microstructure 3 (TBL 36)

This microstructure bears four major inclusions, embedded in a small quantity of an aluminous and siliceous matrix made up of small platy grains of former clay minerals: (1) angular grains of  $\alpha$ -quartz (Fig. 5a); (2)

roundish to sub-angular grains of mullite-bearing glassy particles (phase A, Fig. 5b); (3) roundish grains of a Ca-Na aluminous-siliceous glass (phase B, Fig. 5c) and (4) platy grains of an Al-rich siliceous phase C with a phyllosilicate-type cleavage (Fig. 5b, d, and e). Very few rutile ( $TiO_2$ ) grains were also detected. The mean chemical composition of these phases is given in Table 2. In detail, phase A and B show a considerable range of compositions, particularly with regard to their  $SiO_2$ ,  $Al_2O_3$ ,  $K_2O$ ,  $Na_2O$  and CaO contents, contrasting with the more homogeneous composition of phase C (Fig. 6). Phase A contains 67–84 wt%  $SiO_2$  and 12–29 wt%  $Al_2O_3$ ;  $K_2O$  ranges between 1.2 and 4.5 wt%, and  $Na_2O$  concentrations are comparatively low (0.1–1.1 wt%). CaO contents vary between 0.2 and 1.9 wt%. Low amounts of



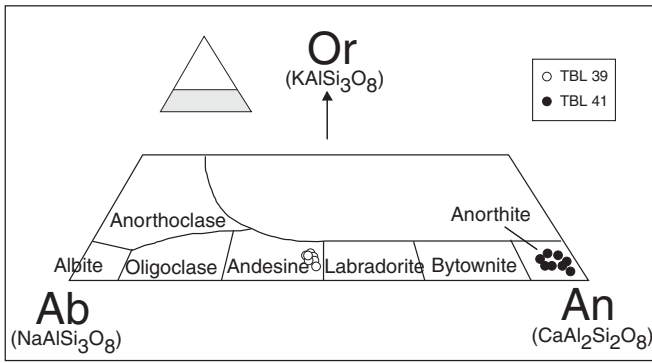


Fig. 4. Classification diagram of feldspars.

$\text{Fe}_2\text{O}_3$  and  $\text{MgO}$  were detected in many measurements. The siliceous (62–67 wt%  $\text{SiO}_2$ ) and aluminous (25–31 wt%  $\text{Al}_2\text{O}_3$ ) phase B is characterized by comparatively high amounts of  $\text{CaO}$  (2.9–7.6 wt%) and  $\text{Na}_2\text{O}$  (0.3–2.9 wt%), and subordinate amounts of  $\text{K}_2\text{O}$  (1.1–2.3 wt%). The patchy aspect in Fig. 5c is due to slight variations of  $\text{CaO}$  (1–2 wt%). Trace amounts (<0.6 wt%) of  $\text{Fe}_2\text{O}_3$  and  $\text{MgO}$  were detected in about half of the EDS analyses of this phase. Both phases A and B are amorphous, as revealed by EBSD, and have non stoichiometric composition, *i.e.* they cannot be related to a known mineral. The former is interpreted as milled hard-paste porcelain, as evidenced by both chemical composition (Table 2) and microstructure. Phase B is most probably a milled

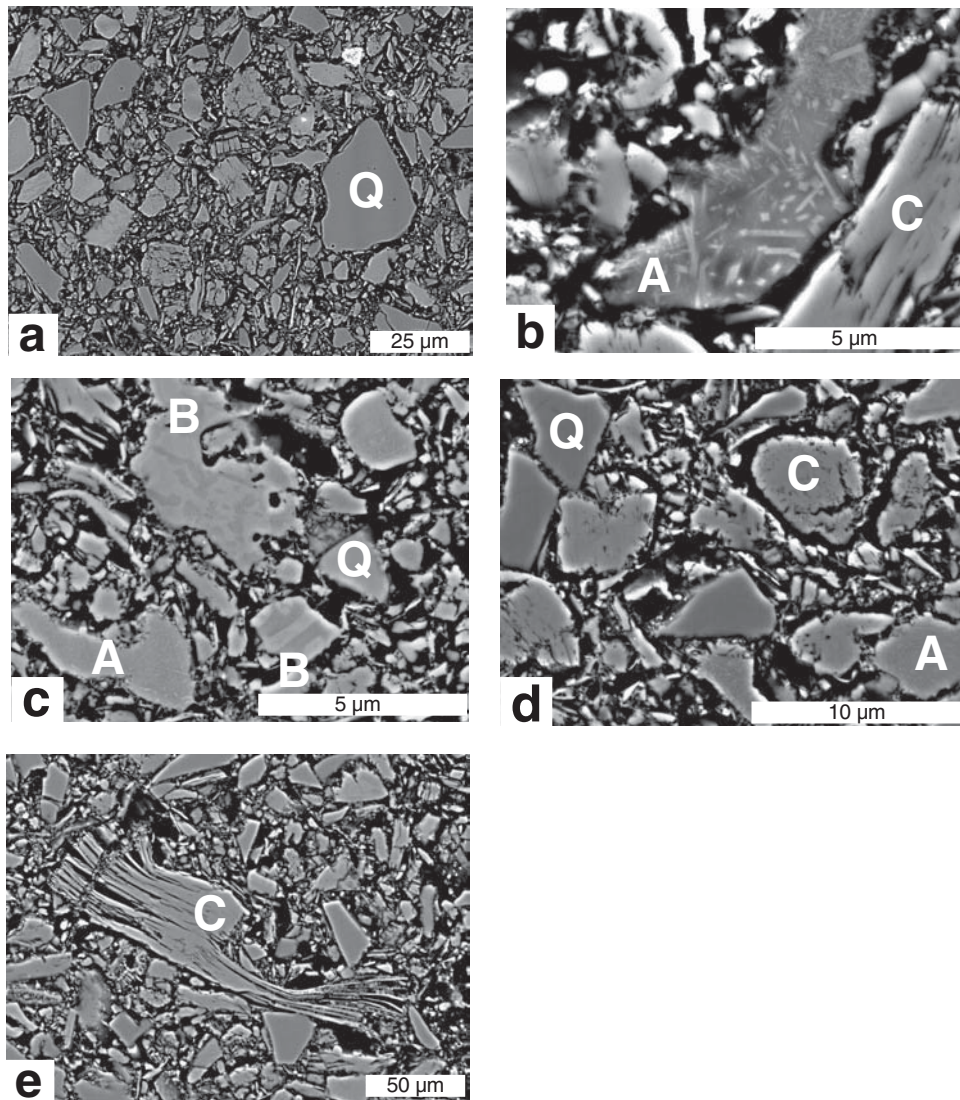


Fig. 5. Different aspects of TBL 36 through SEM observation. The body contains 4 major inclusion types: angular quartz grains (Q), angular mullite-bearing porcelains (A), angular glasses (B) with internal patchy structure and meta-kaolinite (C), with typical cleavage morphology.

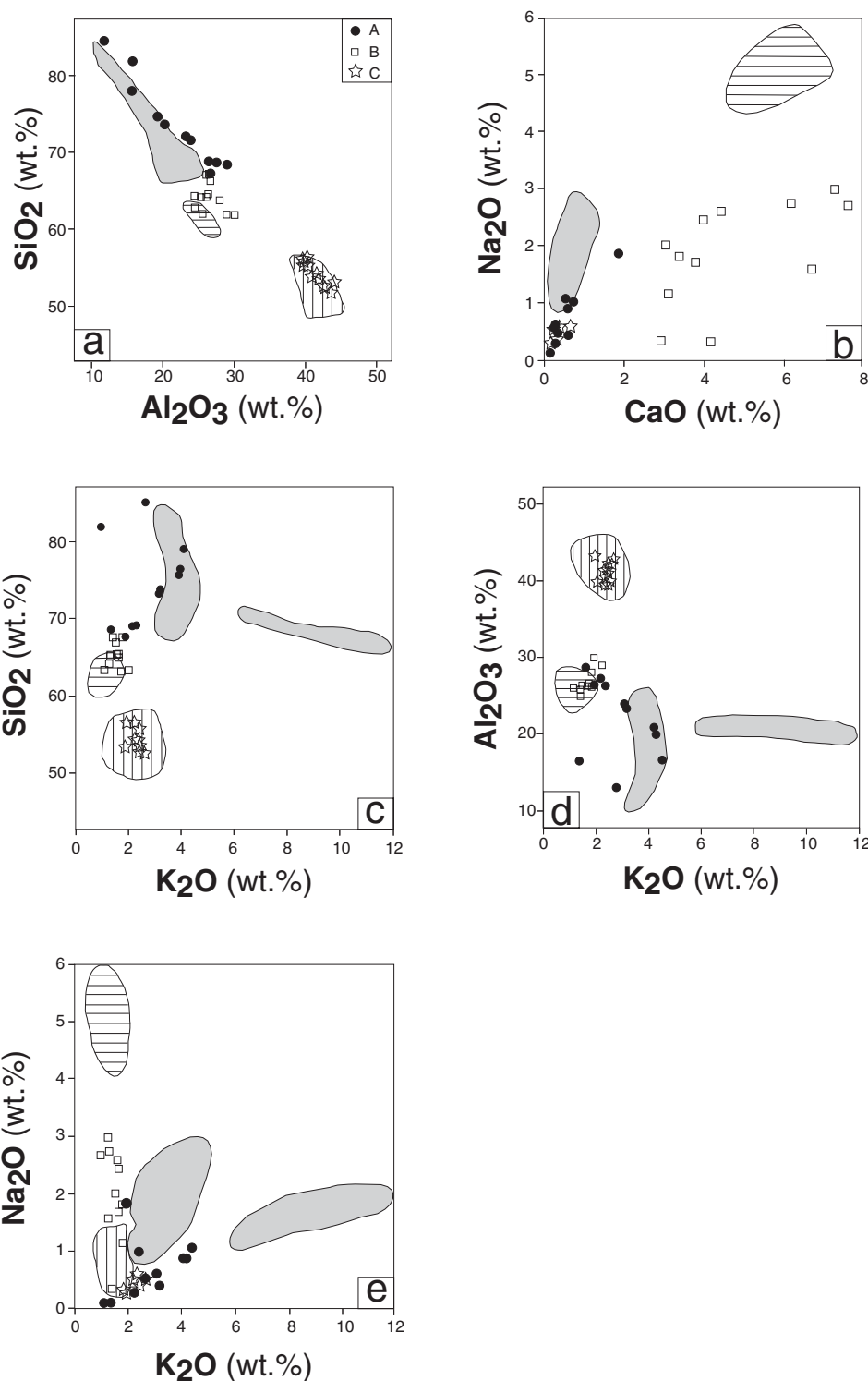


Fig. 6. Bivariate plots showing the composition of the porcelain particles A, the glass B and the meta-kaolinites C of TBL 36, compared to the compositional areas of the porcelain grains (grey) A1 in TBL 24\* and A2 in TBL 34, frit B in TBL 24\* (horizontal ruling) and meta-kaolinites (vertical ruling) C1 in TBL 24\* and C2 in TBL 34. TBL 24\* and TBL 34 from Maggetti *et al.* (2010).

(cullet?) glass. Phase C has a relatively narrow range of SiO<sub>2</sub> (52–56 wt%) and Al<sub>2</sub>O<sub>3</sub> (40–44 wt%). K<sub>2</sub>O (1.9–2.6 wt%), Na<sub>2</sub>O (0.3–0.6 wt%) and CaO (0.2–0.7 wt%) show a wider scatter. Low amounts of Fe<sub>2</sub>O<sub>3</sub> and MgO were

detected in almost all analyses. These compositions are very similar to those of the mineral kaolinite. The higher K<sub>2</sub>O and Na<sub>2</sub>O are interpreted to pertain to relict mica, converting to kaolinite (Tite *et al.*, 1984).



The body bulk composition shows high SiO<sub>2</sub> (65 wt%) and Al<sub>2</sub>O<sub>3</sub> (30 wt%), low CaO (0.5 wt%), Na<sub>2</sub>O (0.4 wt%) and K<sub>2</sub>O (2 wt%), and negligible TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and SO<sub>3</sub> (Table 3). The open porosity is high (~23 %, Table 1).

#### 4.4. Microstructure 4 (TBL 39)

Sample TBL 39 contains few sub-angular to rounded quartz grains immersed in a speckled matrix (Fig. 7). Some zircon and Fe-oxides were also detected (whitish grains in Fig. 7a). Phase A, visible as rounded, light grey particles in Fig. 7b has the composition of a lead- and zinc-bearing andesine feldspar (An<sub>37-45</sub>, Fig. 4). The EBSD showed the crystalline nature of this phase, but the exact crystallographic identification failed due to the very weak signals obtained. Phase B is chemically similar to a lead-bearing glass; EBSD confirmed its amorphous nature. This inferred melt is moderately siliceous (52–67 wt% SiO<sub>2</sub>), with highly variable alumina (26–42 wt% Al<sub>2</sub>O<sub>3</sub>), and subordinate lime (1.1–1.6 wt% CaO), potash (1.2–1.6 wt% K<sub>2</sub>O), soda (1–2.3 wt% Na<sub>2</sub>O) and lead (1.5–2.2 wt% PbO), and traces (<1 wt%) of SO<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and ZnO (Table 2). The andesine grains were evidently resorbed by melt B as they occur as corroded and embayed grains in the latter. Pseudomorphic meta-kaolinites (Fig. 7c, Table 2) and some tiny mullite crystals in phase B (Fig. 7d) could also be detected.

The bulk composition of the body is given in Table 3. It contains 58.9 wt% SiO<sub>2</sub> and 31.1 wt% Al<sub>2</sub>O<sub>3</sub>. Na<sub>2</sub>O (2.8 wt%), CaO (2.4 wt%), PbO (1.8 wt%), K<sub>2</sub>O (1.5 wt%) and ZnO (0.9 wt%) are minor components. Water adsorption is high (6.64 %, Table 1).

#### 4.5. Microstructure 5 (TBL 35, 40)

Sample TBL 35 shows a patchy aspect with few resorbed relict quartz, some feldspar crystals of probably andesine composition (with needles on the border protruding into the glassy matrix), many patches of roughly kaolinitic composition and some interstitial glassy matrix (Fig. 8a). A reliable quantitative chemical analysis of the phases was however not possible due to the lack of marked chemical contrasts. This figurine shows many rounded pores (10–15 μm diameter) pertaining to a closed porosity, contrasting with the very low open porosity (0.23 %, Table 1). The body is siliceous (64 % SiO<sub>2</sub>) and aluminous (29 % Al<sub>2</sub>O<sub>3</sub>), with subordinate amounts of CaO, Na<sub>2</sub>O and K<sub>2</sub>O (Table 3).

Backscattered electron images of bisque TBL 40 have a mottled aspect, due to the presence of four glassy (former melt) phases (Fig. 8b–d). The clear one contains in some cases relict cores of partly dissolved quartz particles with cracks protruding into the surrounding glassy phase. Quartz increases its volume abruptly at the α-β transition (573 °C) or when transforming into another SiO<sub>2</sub> polymorph such as

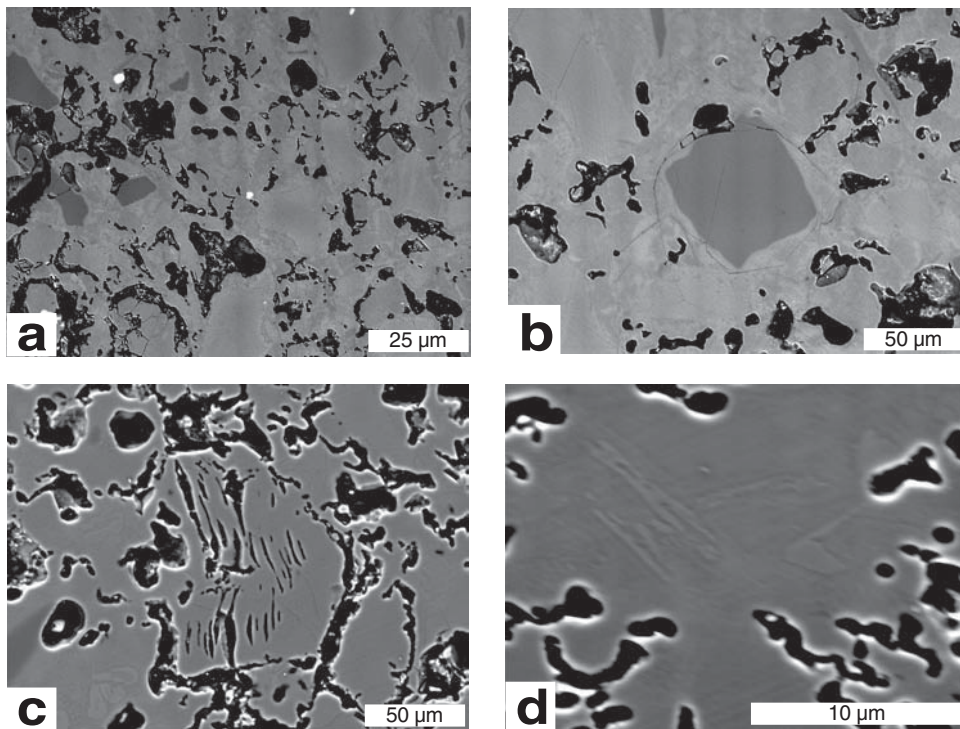


Fig. 7. Backscattered electron images of TBL 39. (a) Irregular shaped pores (black), quartz grains (dark grey), zircon and Fe-oxides (white) in a speckled matrix. (b) Residual quartz particle (dark grey) surrounded by a typical cooling crack. The whitish xenomorphic particles are interpreted to be resorbed andesine in a glassy matrix (light grey). (c) Meta-kaolinite grain with relict (001) cleavage planes, cut perpendicular to the *c*-axis. (d) Mullite needles in phase B.

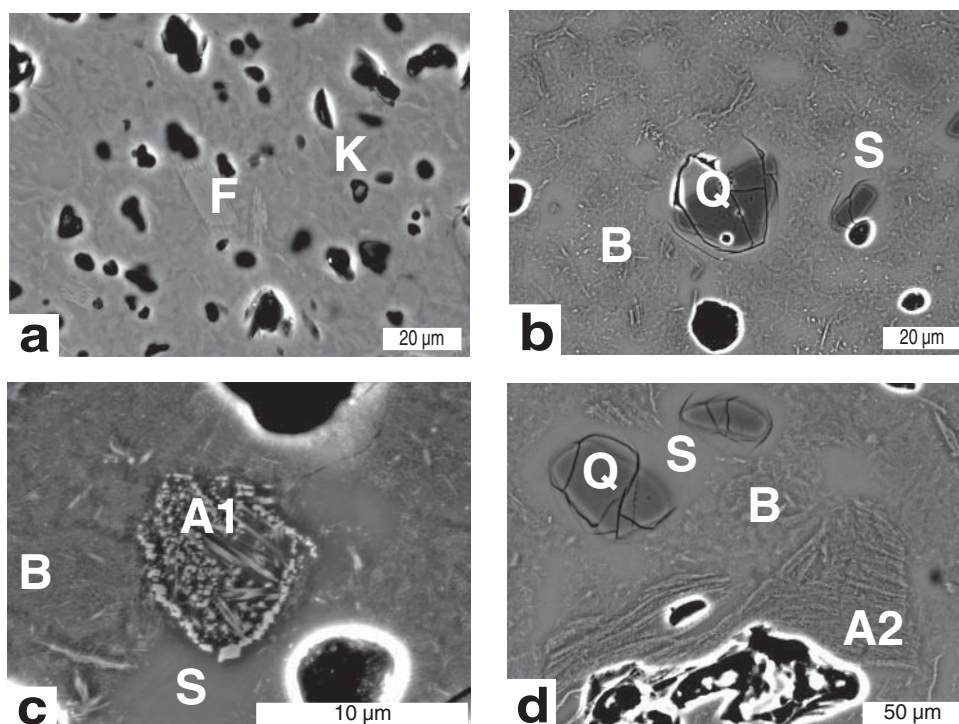


Fig. 8. Backscattered electron images showing: (a) the microstructure of TBL 35 with feldspar (F) and “kaolinitic” patches in subordinate glassy matrix; (b) resorption of quartz (dark grey) by a silica-rich, clear melt phase (S), mullite-bearing melt (now glass) B and black roundish pores (TBL 40); (c) coexisting glassy phases (former melts) A1 with coarse mullites, B with fine mullite needles and mullite-free S in TBL 40; A1 and B show preserved original grain shapes; (d) coarse mullite crystals pseudomorphing the cleavage planes of a former kaolinite in glassy phase A2 (TBL 40).

tridymite or cristobalite. During cooling, the crystallographic inversions lead to contracting with subsequent cracking. The clear glassy patches are silica-rich (71–72 wt% SiO<sub>2</sub>) and contain on average 22–23 wt% Al<sub>2</sub>O<sub>3</sub>, 2 wt% K<sub>2</sub>O, 2 wt% CaO and 1 wt% Na<sub>2</sub>O, with lesser concentrations of Fe<sub>2</sub>O<sub>3</sub>. The second glassy phase appears as small but very distinct areas containing coarse mullite with quench morphological traits such as central hollows or swallow-tail terminations (phase A1 in Fig. 8c). The aspect of these areas recalls former feldspars in hard-paste porcelain (Salmang & Scholze, 1968, p. 268), but the chemical composition is far away from any known feldspar (Table 2). The third glassy phase A2 has the chemical composition of a micaceous kaolinite (Table 2) with mullite inclusions pseudomorphing the former clay mineral cleavage (Fig. 8d). The fourth glass is visible as small patches containing a felt of very tiny (max. 10 μm) mullite needles (phase B, Fig. 8c). The shapes and the chemical composition (Table 2) suggest a derivation from former fine-grained illitic kaolinites with mullite crystallizing during cooling from the melt. Phase A1 can therefore also be considered a former micaceous kaolinite, as its composition matches the one of B. Sample TBL 40 has no open porosity (Table 1). Some closed porosity is still visible as few rounded pores (Fig. 8b).

The body of TBL 40 is compositionally very similar to TBL 35, as reported in Table 3. Both have intermediate silica (64–66 wt%) and 27–29 wt% Al<sub>2</sub>O<sub>3</sub>. Lime (CaO) is present at 2, soda (Na<sub>2</sub>O) at 2.3–2.5 and potash (K<sub>2</sub>O) at

around 2 wt%. Iron, titanium and magnesium oxides are typically less than 1 wt%.

## 5. Discussion

The diversity of Cyfflé’s bisque bodies, produced in his TERRE DE LORRAINE workshop during the years 1766–1780, is astonishing. However, one needs to consider that Cyfflé developed his recipes in the context of a rapidly evolving political, economical and technological situation. In fact, the authorities could prevail less and less upon the manufactures to respect the Royal privilege of Sèvres, as the secrets of the French *pâte tendre* and the German hard-paste porcelain became well known.

### 5.1. Recipes

As no contemporary recipe for Cyfflé’s figurines were noted, one must rely on the bulk analysis as well as the microstructural and the microchemical evidence to gain some information about the recipes. But this is not an easy doing, since chemically differing primary ingredients contribute to the same chemical constituent in the bulk analysis. Aluminium, for instance, can derive from K-feldspar, from alum, from clay minerals such as illite and kaolinite, and from glass. The exact nature and the proportions of the

primary ingredients in the bodies are therefore difficult to constrain.

### 5.1.1. Microstructure 1 (TBL 37, 38)

The significant amount of sulphate indicates that gypsum was added to the paste, which converted to anhydrite during firing. On the basis of the microstructures and the chemical compositions it is inferred that these bodies were made from a mixture of crushed flint or quartz sand, clay, lime and/or gypsum and alkali (soda, potash). ZnO (in TBL 37) was probably added as whitener (Majewski & O'Brien, 1987). Part of the lime, clay and alkali were pre-fired with quartz sand or milled flint, as shown by the glassy rims around the silica particles. The aspect of the phase A particles of the matrix suggests an organic nature. Theoretically, this question could be solved with infrared spectroscopy, but this needs more sampling (if allowed).

### 5.1.2. Microstructure 2 (TBL 41)

Sample TBL 41 is, as shown by Table 3 and Fig. 9, chemically very similar to TBL 25 analysed by Maggetti *et al.* (2010). The latter was not studied by SEM – the very small sample allowed only X-ray fluorescence and X-ray diffraction (XRD) studies. TBL 25 gives the distinctive XRD patterns of quartz, anorthitic plagioclase and cristobalite, corroborating the microstructural observations. The latter phase is interpreted to occur as minute (sub- $\mu\text{m}$  scale) crystallites incorporated within quartz or in a glassy phase (Miels & Zografu, 1967), as it was not detected by BSE imaging.

The microstructure of TBL 41 is visibly a disequilibrium assemblage of coronitic quartz + islands or individual crystals of anorthitic feldspar +  $\text{SnO}_2$  rich glass fragments in a glassy matrix, very similar to the microstructure of 18th century French soft-paste porcelains (Kingery &

Smith, 1985; Kingery, 1986; Kingery & Vandiver, 1986). But these objects contain wollastonite and no anorthite. Admittedly, the former is very difficult to detect by XRD in the presence of plagioclase. However, wollastonite was not identified with SEM-EDS analyses in TBL 41. The explanation lies in the CaO-rich composition of the samples analyzed by Kingery & Smith (1985). In fact, their bulk compositions plot close to the Alkemade  $\text{SiO}_2$ -phase/wollastonite in the  $\text{SiO}_2$ -CaO- $\text{Al}_2\text{O}_3$  phase diagram (Osborn & Muan, 1960), whereas both figurines TBL 25 and 41 lie on the Alkemade  $\text{SiO}_2$ -phase/anorthite. Similar anorthite particles have been observed in other 18th century porcelains and were interpreted as that they were originally clay (kaolinite), which has reacted with the soda and lime of the matrix to form feldspar (Radkowa, 1973; Freestone, 1993, 1996; Owen & Barkla, 1997). The body composition of TBL 25 and 41 has some characteristics of artificial glassy-type French porcelain, but its content in  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  is higher. CaO and  $\text{K}_2\text{O}$  are significantly lower (Table 3, Fig. 9). The best fit is with sample SC-5 from St. Cloud, dated *ca.* 1750. Higher alumina may reflect higher clay content or the use of alum. Gypsum or alum is the likely source of the small amount of sulphur in the body. All these features show that Cyfflé used a soft-paste (frit) porcelain recipe in the French tradition, based upon fluxes, sand and clay, as documented in the notebooks of Jean Hellot (d'Albis, 1983, 1984, 1985; Brongniart, 1844; Treppoz & d'Albis, 1987). During the frit reaction, a corona developed around the quartz grains. A similar corona in a 1752 figurine from Vincennes was interpreted to be pure cristobalite (Kingery, 1986; Kingery & Vandiver, 1986). In contrast, the corona of TBL 41 is amorphous and has a Si-Al-Ca composition, suggesting strong interactions of quartz particles with the neighbouring ingredients. The tin oxide content of the glassy corona and the presence of tin-rich glassy particles suggest that

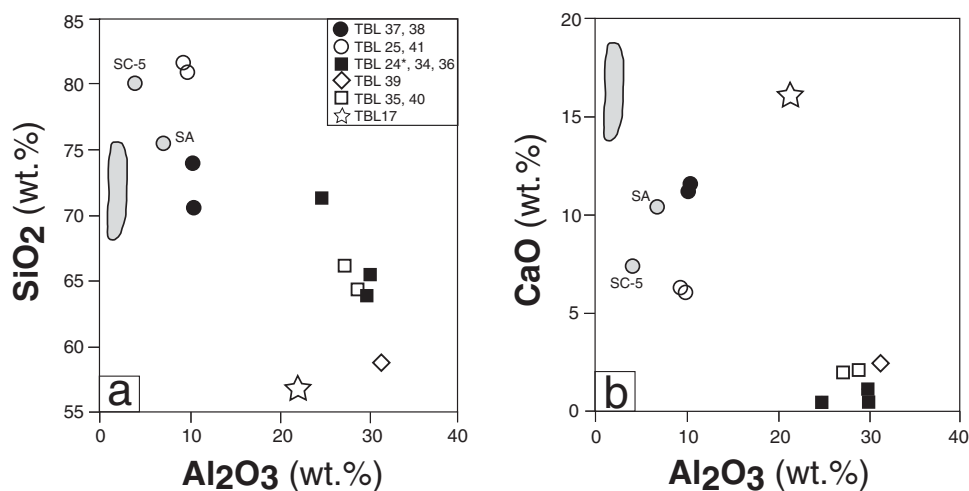


Fig. 9. Body (bulk) compositions for the analysed samples displayed on bivariate plots of selected oxides. Previously analysed figurines TBL 17, 24\*, 25 and 34 (Maggetti *et al.* 2010) are also shown. Grey field = MgO-poor soft-paste (frit) porcelain analyses from Brongniart (1844); d'Albis (1983); Kingery & Smith (1985) and Treppoz & d'Albis (1987). SA = St. Amand (Brongniart, 1844), SC-5 = St. Cloud (Kingery & Smith, 1985).



some (or all?) of the silica was fritted with Sn-bearing cullet glass. The frit was then milled and mixed with an illitic clay and lime and probably more fluxes such as saltpetre, alum, gypsum, and moderate amounts of sea salt and Alicante soda. It is however questionable whether the measured oxide concentrations of the glassy matrix C reflect their original, pre-firing composition. In fact, during the post-frit firing strong interactions between the frit fragments must have occurred, as revealed by microscopic observations.

### 5.1.3. Microstructure 3 (TBL 36)

The microstructure of this figurine is similar to the fine-grained aluminous-siliceous bisque TBL 24\* (mean of TBL 24, 26–29) described by Maggetti *et al.* (2010). Both bulk compositions show reasonable chemical consistency, with high SiO<sub>2</sub> (64–65 wt%) and Al<sub>2</sub>O<sub>3</sub> (~ 30 wt%), low CaO (0.5–1.2 wt%), Na<sub>2</sub>O (0.4–2.2 wt%) and K<sub>2</sub>O (~ 2 wt%), and negligible TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and SO<sub>3</sub>. They belong chemically to the family of hard-paste porcelain (Fig. 9), but their porosity is extremely high. In comparing the compositions of these inclusions with the fine (TBL 24\*) and coarse grained (TBL 34) bodies previously analysed by Maggetti *et al.* (2010), considerable differences appear (Fig. 6). The porcelain particles A are richer in Al<sub>2</sub>O<sub>3</sub>, but lower in Na<sub>2</sub>O and K<sub>2</sub>O than the A1 porcelain grains of TBL 24\*. Phase B in TBL 36 is significantly poorer in Na<sub>2</sub>O and has a much wider scattering of CaO than the corresponding phase B of TBL 24\*. Clearly, these bodies were made with milled porcelain and glass of heterogeneous chemical composition, together with milled, calcined flint or sand and coarse meta-kaolinite, mixed with a fine-grained kaolinitic clay used as matrix bonding.

### 5.1.4. Microstructure 4 (TBL 39)

The body has the high alumina typical for hard-paste porcelain (Fig. 9). The bulk composition resembles early Meissen hard-paste porcelain or Sèvres hard-paste porcelain (1770–1836), but with lower CaO (Table 3). The presence of small amounts of lead in the paste as well as in the inferred melt phase B suggests that traces of flint glass were included in the recipe for this sample. It is therefore inferred that this figurine was made from a mixture of quartz sand, kaolin or kaolinitic clay, plagioclase (andesine), lime and some flint glass. ZnO-contents of about 1 % are indicative of an addition of this oxide as whitener (Majewski & O'Brien, 1987). The heterogeneous microstructure and the high porosity of TBL 39 indicate severe underfiring of this object.

### 5.1.5. Microstructure 5 (TBL 35, 40)

Samples TBL 35 and 40 are chemically very similar and plot in Fig. 9 in the area of hard-paste porcelain. TBL 35 has interstitial glass, relict andesine plagioclase with recrystallization features, few resorbed quartz grains and many patches close to kaolinitic compositions. In contrast, TBL 40 consists of an inhomogeneous glassy matrix with

much mullite, a characteristic mineral in hard-paste porcelains, and some unreacted quartz. The CaO content is typically lower than that of early Meissen hard-paste porcelain, Sèvres hard-paste porcelain tableware and Sèvres hard-paste porcelain figurines 1794–1834 (Table 3). Microstructural aspects, body and phase composition, and contemporary recipes suggest that both figurines were made from a mixture of quartz sand, kaolinite, lime (or gypsum) and alkali phases (K, potash or K-feldspar or alum; Na, sea salt or Alicante soda). The high proportion of melt phases in both figurines TBL 35 and 40, the degree of resorption of quartz, the absence of open porosity and the low closed porosity indicate thorough vitrification. The presence of plagioclase particles, the minor amount of glassy matrix and the dominant “kaolinitic” patches in bisque TBL 35 suggests a lower firing temperature or a shorter firing time if compared with bisque TBL 40.

## 5.2. Classification

According to Salmang & Scholze (1968), white clay-based ceramic objects with water adsorptions (W.A.) > 2 % classify as white earthenwares, those with W.A. < 2 % as porcelains. TBL 36, 37, 38, 39 and 41 would therefore pertain to the former, TBL 35 and 40 to the latter ceramic type. But underfired porcelain bodies show significant open porosities, classifying them as white earthenware and not as porcelain. Therefore, one has to consider together the bulk compositions and the firing conditions. On these assumptions, it would be reasonable to classify the four figurines of Maggetti *et al.* (2010) and the seven bisques of this study into five paste types (Table 4). Type 1 and 2 are white earthenwares, type 3 is a soft-paste (artificial glassy) porcelain and type 4 a hard-paste porcelain. For type 5, Cyfflé combined ingredients of (artificial) soft- and hard-paste porcelain. These bodies can therefore be classified as hybrid porcelains.

Table 4. The five paste types of Cyfflé's figurine manufacture in Lunéville.

Paste type	An. no. (TBL)	Marks and signatures
1) Gypsum paste	37	CYFFLÉ A. LUNÉVILLE, G.
	38	
2) Terre de pipe	17	
3) Soft-paste porcelain	25	
4) Hard-paste porcelain	41	TERRE DE LORRAINE
	35	
5) Hybrid porcelain	39	TERRE DE LORRAINE
	40	
	24*	TERRE DE LORRAINE <sup>a</sup>
	34	
	36	TERRE DE LORRAINE, C.G. or G.G.

Note: <sup>a</sup>G. Demat (pers. comm.).

### 5.3. Cyfflé's "marble paste"

Low fired ceramic is very porous. Higher fired ceramic develops a melt phase which fills the pores and reduces the opacity of the body. The unglazed, unpainted "marble paste" invented by Cyfflé to emulate Classical marble figurines was, according to him, washable, *i.e.* its open porosity (or water adsorption) must have been  $\sim$  nil. Further, to obtain the "shine through" effect of a marble, closed porosity must have been very low too. Consequently, all pastes with a high open porosity (gypsum paste, *terre de pipe* paste, soft-paste porcelain, under-fired hard-paste porcelain and hybrid porcelain) cannot be Cyfflé's "marble paste", but are probably experimental mixtures. Only pastes with  $\sim$  nil open porosity and a pervasive melt phase, such as TBL 40 having a high translucence (this object with the mark TERRE DE LORRAINE), is likely to match Cyfflé's "marble paste". Obviously, his statement in the request from December 1766 ". . . would be superior to pipe clay, without being porcelain, and named *Terre de Lorraine*, . . ." did not hinder him to make true porcelain. In effect, he was perfectly right when he wrote, in a letter from 8 April 1780, that he sells "excellent bodies for the manufacture of porcelain as well as terre de pipe" (Grollier, 1906, p. 441).

### 6. Conclusions

The study of seven figurines from three private collections has revealed new types of Cyfflé's bisque bodies, including true porcelain pastes. It is evident that many recipes, perhaps of an experimental nature, were used in his manufacture in Lunéville. Between 1766 and 1780, at least five major body types were produced: *gypsum* paste, *terre de pipe* paste, soft-paste (artificial) porcelain, hard-paste porcelain and hybrid porcelain, mostly all with intra-group variants. Theoretically, the *terre de pipe* and the soft-paste porcelain figurines could have been produced elsewhere, since they bear no marks, but stylistic analyses (Calame, 2009) assign them reliably to Cyfflé's manufacture. The *gypsum* paste figurines, containing unreacted calcium sulphate particles, and the *terre de pipe* paste figurines belong most probably to the very earliest bodies used (1766–1768), since the former bears the mark CYFFLÉ A. LUNÉVILLE and not TERRE DE LORRAINE as prescribed by the State's edict of 3 May 1768. The true hard-paste and the hybrid porcelain bodies – all with the mark TERRE DE LORRAINE – could well have been produced in 1768, just after the issue of the edict. This is a reasonable date considering that Cyfflé sent his "marble paste" to Paris in 1769, and that many preliminary trial-and-error experiments were necessary before. But were all recipes introduced simultaneously or is an evolution of the porcelain pastes (soft-, hybrid-, hard-paste) more likely? Were all five bodies in production until the closure of the manufacture? These questions can probably never be solved

due to the lack of archival information. Notwithstanding, the compositional variation of the TERRE DE LORRAINE bodies has now been firmly established. Next analyses should focus on figurines with the mark T.D.L., in order to fully characterize the range of bodies from Cyfflé's manufacture.

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