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The case of black and green tin glazed pottery from Barcelona between 13th and 14th century: Analysing its production and its decorations

Judith Peix Visiedo *, Marisol Madrid i Fernández, Jaume Buxeda i Garrigós

Cultura Material i Arqueometria UB (ARQUB, GRACPE), Departament d'Història i Arqueologia, Universitat de Barcelona, C/ de Montalegre, 6, 08001 Barcelona, Catalonia, Spain

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ABSTRACT

Black and green tin-glazed pottery is known to be one of the true majolica manufactures at Barcelona during the end of the medieval period. This pottery means the evolution of a production process for achieving opacified decorated tableware, started one century before, with the elaboration of what is known as archaic majolica. Black and green implies a new high variety of decorative motifs made in black, or in black and green, on a white opacified glaze prepared following a recipe adapted by potters to prepare the base glaze and to applicate the pigments for attaining the decorative patterns, characteristics of this period.

Originally thought as imported from Valencia workshops, the development of different archaeometric projects conducted in the core of the ARQUB team, allows verifying an origin in Barcelona. Besides, recent excavations dated back to the 14th century provided a great number of Black and green sherds with new decorative motifs unknown up to now, allowing the specialists to create a new corpus of decorative patterns, which is still in progress. Based on insights obtained from those first archaeometric studies and considering the advances on the classification of decorations based on archaeological arguments, a new sample of Black and green sherds was selected for this study. The aim was to observe whether decoration and technique of manufacture could in some way be related, understanding the technical process as the selection and application of raw materials and the firing temperature at which this pottery was made.

To achieve these objectives, X-ray fluorescence (XRF), X-ray diffraction (XRD), and Scanning Electronic Microscopy (SEM-EDX) have been performed. The results allow verifying the groups previously defined and to identify a new chemical group, probably related to a recipe for the body-paste different from the others. On the other hand, the study enabled us to observe differences also in know-how in firing, which in some cases seem to be connected with differences in the decorative motives, suggesting, perhaps, the discrimination of artisans.

1. Introduction

The origin of black and green vessels is poorly known, and it has been related to the workshops of Raqqah (Syria), from where the vessels were spread into the north of Africa in the 9th century. In the case of the Iberian Peninsula, around the 10th century, vessels with black and green decoration would arrive at Al-Andalus territories in Andalucía (González Milà, 2000). Due to the Christian conquest, this kind of vessels appeared in Christian territories in the 13th century, but with new forms and decorations, adapted to the interests of consumers. From the 13th to the 14th century these products were the main important vessels, until being substituted by blue decorations in the 15th century.

In the case of Barcelona, oriental importations from Syria and Egypt were documented in the 12th and the 13th centuries (Beltrán de Heredia Bercero, 2007; Beltrán de Heredia Bercero and Miró i Alaix, 2008). These vessels appeared with the establishment of consulates in different harbours, in the frame of the Catalan commercial expansion through the Mediterranean. Traders from Genova, Pisa, Greece, Sicily, Alexandria, Egypt or Africa arrived at Barcelona, and with them, maybe the firsts decorated vessels.

What we do not surely know, is how the first majolica arrived in Barcelona, and which vessels could have influenced the decorative motifs of Black and green ones. Some authors (Cirici and Manent, 1977) point out a meridional arrival concerning the conquest of Valencia in 1238. Conversely, other authors (Llubià, 1967: 132) assesses an Aragonese route, rejecting the arrival of the first Black and green vessels

* Corresponding author.
E-mail address: jpeix@ub.edu (J. Peix Visiedo).

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from Valencia. Moreover, López Elum (1984: 85) did not find any references to Black and green pottery in Paterna before the 14th century in the archives.

Even Barrachina and Trallero (1983) suggested a Valencian origin, they also consider majolica from the 13th century as a derivation of the *morisca* ones from Teruel. It is the same for Ainaud (1952), who defends an initial Valencian influence, but only in the first years, since the historical sources tend to indicate a north–south trend, in favour of the hypothesis which defends that Paterna would not be so advanced to produce this pottery.

But, as Beltrán de Heredia observed already more than one decade ago (2007), a Valencian origin has been questioned in the light of some pieces of evidence such as Catalan imports found in Valencia together with the first Valencian products; and, mainly, thanks to the recovery of what is considered to be the precedent of Black and green ceramics in Barcelona, Archaic majolica, for which a local origin has never been questioned, perhaps for the low quality of the glaze showed in some pieces. The finding of several sites with abundant Archaic majolica in contexts dated back at the end of the 12th century (Beltrán de Heredia Bercero, 2007; Beltrán de Heredia Bercero and Lores Otzet, 2005; Huertas Arroyo, 2008) relates such beginning with the setup of the productions of similar pottery in the workshops of Italy and allowed to date back one century earlier the beginnings of tin–lead glazing in the city of Barcelona.

In this sense, it must be highlighted the detailed study carried out by Beltrán de Heredia Bercero (2007) on an important Archaic majolica pottery set recovered from the archaeological site of Sant Honorat which gave rise to the first archaeometric studies which enabled the verification of the local origin for this kind of pottery as well as for coarse glazed pottery (Iñañez and Buxeda, 2007; Iñañez et al., 2009). The study of the shapes and the decorative patterns reveals according to Beltran, an Italian influence, suggesting a north-east diffusion from the Iberian Peninsula to the Levant.

In this sense, the study of the decorative motifs in Black and green vessels is still working progress by archaeologists. The numerous trims and combinations of the central motives have made difficult to systemize the central decorative motifs together with the trims and the shapes of vessels, resulting in a lack of a typology for decorations, using most of the time those based on the history of art criteria. Thus, the typology used up to now is based on two studies (Rivera and Cabestany, 1980 and González Milà, 2000) highlighting the one of González Milà, which proposes a typology to classify vessels appeared in different archaeological sites from Catalonia, taking into consideration the archaeological work. In this sense, González Milà's typology related to decoration is based on 10 trims (named from trim 1 to trim 10), being trim 1 the simplest (with only one black line) and trim 10 the most complicated (formed by 4 lines in manganese combined with zig-zag in green). Trims are combined with different central motifs, classified in geometrical motifs; vegetal motifs (such as branch pine, leaves, starfish); *pseudoheraldic* motifs, and zoomorphic motifs. Regarding the forms, the typology classifies pieces in forms such as plates, bowls, trenchers, salvers and jars.

However, in an attempt of creating a definitive typology according to the abundant findings of the last years, a new one made by

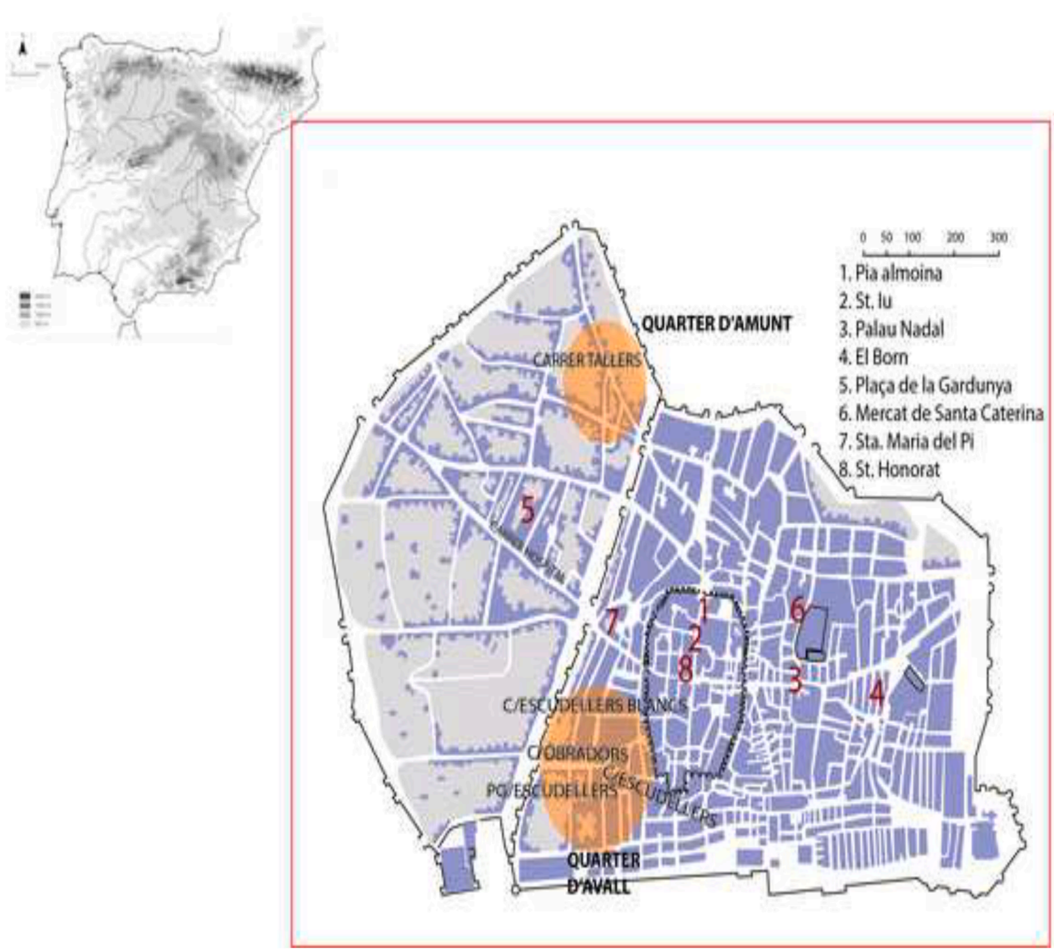

Fig. 1. The situation of Barcelona in the Iberian Peninsula with the main pottery production areas highlighted. On the top "Quarter d'Amunt area" with Hospital st., and Tallers st.; on the bottom "Quarter d'Avall area" with Escudellers st., Escudellers Blancs, st., and Obradors st. Numbers correspond to the archaeological sites where the vessels were gathered.

Table 1
All the individuals analysed related to their chronology, chemical group, type of ceramic (ceramic class and form), and the archaeological site of Barcelona.

Ic	Chronology	Group	Ceramic class	Form	Decoration	Arcaheological Site
BCN120	13th century-14th century	B2	majolica	bowl	BG	Basílica 1967
BCN126	13th century-14th century	B2	majolica	lamp	BG	St. Iu 1949
BCN129	13th century-14th century	B2	majolica	plate	BG	Palau Nadal 1971
BCN134	13th century-14th century	B2	majolica	plate	BG	Pl. Gardunya 2005
BCN446	13th century-14th century	B1e	majolica	plate	BG	Sant Honorat 2000–2003
BCN210	13th century-14th century	B1c	transport	transport jar	green	Mercat de Santa Caterina 2000
BCN440	13th century-14th century	B1e	majolica	plate	BG	Gardunya 2005–2006
BCN121	13th century-14th century	B1c	majolica	bowl	BG	Basílica 1967
BCN122	13th century-14th century	B1c	majolica	plate	BG	St. Iu 1949
BCN123	13th century-14th century	B1c	majolica	plate	BG	St. Iu 1949
BCN124	13th century-14th century	B1c	majolica	plate	BG	St. Iu 1949
BCN125	13th century-14th century	B1c	majolica	jug	BG	St. Iu 1949
BCN127	13th century-14th century	B1c	majolica	bowl	BG	St. Iu 1949
BCN128	13th century-14th century	B1c	majolica	plate	BG	Palau Nadal 1971
BCN130	13th century-14th century	B1a	majolica	plate	BG	Antic mercat del Born 1998
BCN131	13th century-14th century	B1c	majolica	plate	BG	Pl. Gardunya 2005
BCN132	13th century-14th century	B1c	majolica	plate	BG	Pl. Gardunya 2005
MJ0087	14th century	B1a	majolica	plate	BG	Sta. Maria del Pi
MJ0088	14th century	B1a	majolica	plate	BG	Sta. Maria del Pi
MJ0092	14th century	B1a	majolica	plate	BG	Sta. Maria del Pi
MJ0093	14th century	B1a	majolica	plate	BG	Sta. Maria del Pi
MJ0096	14th century	B1a	majolica	jug handle	BG	Sta. Maria del Pi
MJ0098	14th century	B1a	majolica	jug	BG	Sta. Maria del Pi
MJ0099	14th century	B1a	majolica	closed shape	BG	Sta. Maria del Pi
BCN448	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN449	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN450	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN451	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN452	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN453	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN454	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN455	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN456	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN457	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN458	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN459	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN460	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN461	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN462	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN463	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN464	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN465	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN466	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN467	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN468	14th century	B1e	majolica	plate	BG	Antic mercat del Born 2001
BCN137	14th century	B2	majolica	plate	BG	Sta. Maria del Pi
MJ0089	14th century	B2	majolica	plate	BG	Sta. Maria del Pi
MJ0090	14th century	B2	majolica	plate	BG	Sta. Maria del Pi
MJ0091	14th century	B2	majolica	plate	BG	Sta. Maria del Pi
MJ0094	14th century	B2	majolica		BG	Sta. Maria del Pi
MJ0095	14th century	B2	majolica		BG	Sta. Maria del Pi
MJ0097	14th century	B2	majolica		BG	Sta. Maria del Pi
MJ0100	14th century	B2	majolica	closed shape	BG	Sta. Maria del Pi
BCN199	14th-15th century	B1b	transport	transport jar		Santa Caterina 1999
BCN211	14th-15th century	B1c	transport	transport jar	green	Mercat de santa Caterina 2000
BCN212	14th-15th century	B1d	transport	transport jar	green	Mercat de santa Caterina 1999
BCN112	15th century	B1c	transport	transport jar		Pia Almoina 1993
BCN113	15th century	B1b	transport	transport jar		Pia Almoina 1993
BCN115	15th century	B1c	commonware glazed	jug	green	Pia Almoina 1993
BCN116	15th century	B1c	commonware glazed	poal	green	Pia Almoina 1993
BCN117	15th century	B1out	commonware glazed	poal	green	Pia Almoina 1993
BCN118	15th century	B1c	commonware glazed	poal	green	Basílica 1967
BCN119	15th century	B1c	commonware glazed	oil cruet	honey-coloured	Pia Almoina 1993
BCN204	15th century (after 1438)	B1d	transport	transport jar		Pia Almoina 1993
BCN205	15th century (after 1438)	B1d	transport	transport jar		Pia Almoina 1993
BCN206	15th century (after 1438)	B1d	transport	transport jar		Pia Almoina 1993
BCN207	15th century (after 1438)	B1b	transport	transport jar		Pia Almoina 1993
BCN208	15th century (after 1438)	B1d	transport	transport jar		Pia Almoina 1993
BCN209	15th century (before 1474)	B1b	transport	transport jar		Sant Agustí 1994
BCN156	16th century	B1c	commonware glazed	basin	transparent glaze	Pl. Gardunya 2005
BCN157	16th century	B1b	commonware glazed	chamber pot	green	Pl. Gardunya 2005
BCN158	16th century	B1b	commonware glazed	colander	brown	Pl. Gardunya 2005
MJ0189	14th century	B1a	majolica	plate	BG	Sta. Maria del Pi
MJ0191		B1a	majolica	plate	BG	Warehouse of Museu de la Ceràmica

(continued on next page)

Table 1 (*continued*)

Ic	Chronology	Group	Ceramic class	Form	Decoration	Arcaheological Site
MJ0192		B1a	majolica	plate	BG	Warehouse of Museu de la Ceràmica
MJ0194		B1a	majolica		BG	Warehouse of Museu de la Ceràmica
MJ0195		B1a	majolica		BG	Warehouse of Museu de la Ceràmica
MJ0196		B1a	majolica		BG	Warehouse of Museu de la Ceràmica
MJ0190		B2	majolica	plate	BG	Warehouse of Museu de la Ceràmica
MJ0193		B2	majolica		BG	Warehouse of Museu de la Ceràmica
MJ0197		B2	majolica		BG	Warehouse of Museu de la Ceràmica
MJ0198		B2	majolica	plate	BG	Warehouse of Museu de la Ceràmica

archaeologists working in excavations of Barcelona together with the Museu d'Història de Barcelona is being elaborated. This new one is being created considering a high number of Black and green individuals gathered from the excavations of *Rec Comtal* in the archaeological site *El Born* (Barcelona) from which the samples analysed in this study were recovered. The study is attempting to make a typology based on the shape of vessels, adding numerical subgroups to the forms mentioned before (plates, bowls, trenchers…) and to the decorative motifs of the trims and central parts of ceramics, using both publications mentioned before and including new trims and central motifs unknown up to now. The goal is to create an easier typology substituting names by numbers and merging trims and central motifs from González Milà, easing the classification of these vessels. Moreover, in the frame of Archaide project (Anichini et al., 2020l) a typology for these ceramics based only on the decoration was also made. To do that, it had been into consideration the proposal made by archaeologist and the Museu d'Història de Barcelona, with who we work together, reducing the trims to 22 (merging trims from González classification to reduce it with new ones) and changing the descriptive names of central motifs to simple ones (such as *arpia, au, ametlla*, etc). For this reason, in the paper the typology published in Archaide is the one used, waiting for the elaboration of a definitive one.

From the archaeometric point of view, the first attempt to study Barcelona products to establish their provenance and the definition of the Reference Groups (RG) was in the frame of the research of Iñañez (2007). Already in the framework of the Tecnolonial project which started in 2009 a bunch of studies have been realized to provide a wide vision about the pottery production in Barcelona from the 13th to the 18th centuries. In this sense, the studies have been focused on the paste

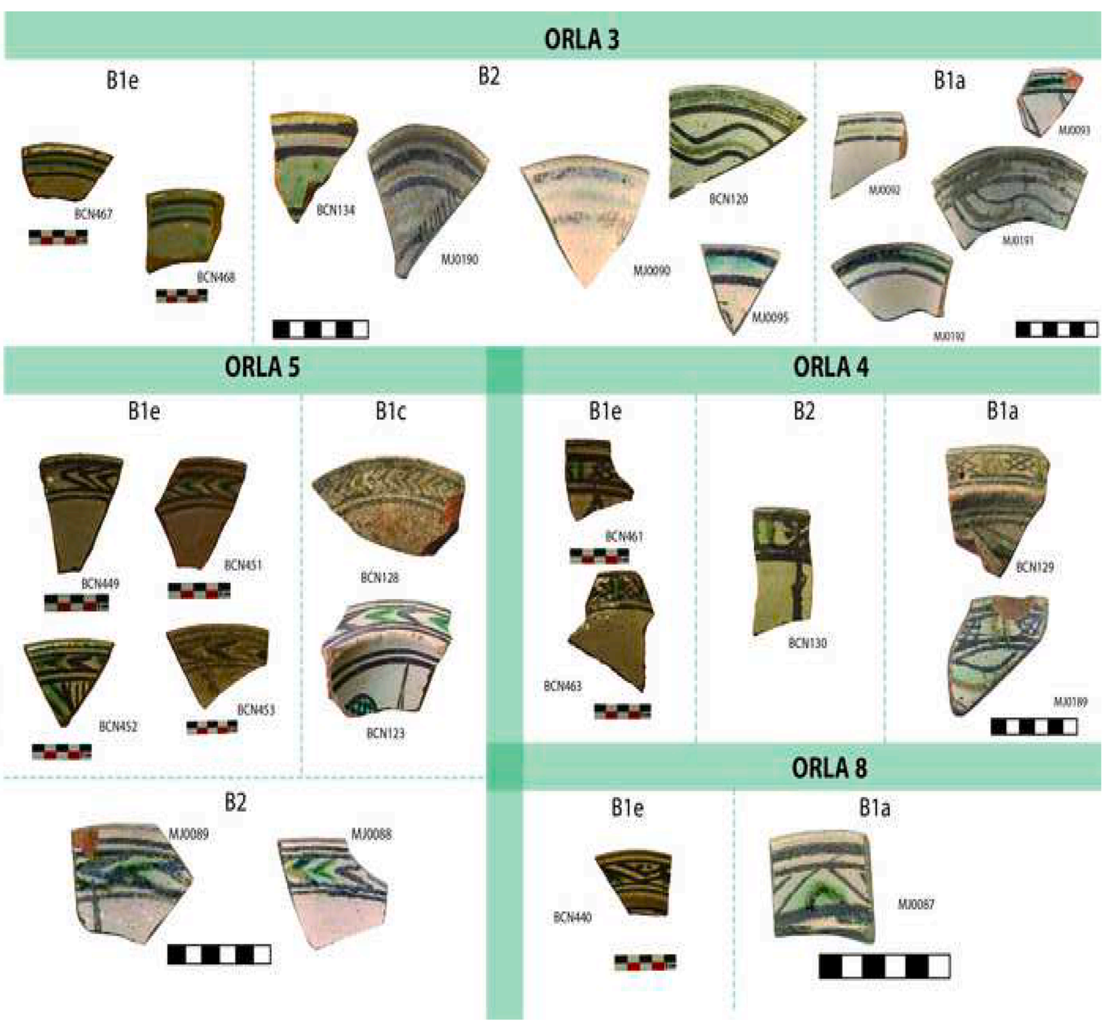

Fig. 2. Some of the black and green individuals analysed in relation to the decorative motifs of the trims, known as "orlas" and their chemical group. Samples studied correspond to "Orla 3", "Orla 4", "Orla 5", and Orla 8". As it can be seen, trims combine geometrical designs made in green and black. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 3. Some of the individuals analysed classified by the chemical group (B1a, B1b, B1c, B1d and B2), corresponding to transport jars, common ware glazed, and black and green vessels. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

clays at chemical and petrographical level (Di Febo, 2015; Di Febo et al., 2012; Iñañez et al., 2009; Madrid i Fernández et al., 2015; Buxeda i Garrigós et al., 2011; Buxeda i Garrigós et al., 2015).

The results of these studies allow us to identify four chemical groups using four different paste clay (A, B, C and E), including vessels of common ware pottery, cooking pottery, transport jars, and majolica, the lasts ones presenting different decorations (from archaic majolica to Black and green, to blue on white, to lustre, and polychrome respectively). The products here studied correspond to vessels dated back from the end of the 13th and the beginning of the15th centuries, with Black and green decorations, known to be the first majolica manufactures in Barcelona, after the first products of archaic majolica in the 12 and the 13th centuries.

Thanks to the previous studies we can include Black and green vessels analysed in this article in group B, which are diversified in turn exhibiting different amounts of calcium in B1 and B2. The B1 group exhibits an increase of the CaO content compared to the previous group A (dated back from the 12th century), and it is the one which the production of majolica starts. The characterization of these vessels showed that the same paste B1 was used to produce majolica, coarse green glazed, honey-coloured glazed and transport jars. This would be related to a non-specialization of the production at this stage, although the advancement from archaic majolica to majolica products took place here.

Continuing with that, in the 15th century, group B2 reached the market. This new group implied a new increment in the CaO content (Table 3) but, from the technical point of view, the most significant change is that this paste used in B2 was restricted only for majolica production, whilst paste B1 was still in use also for glazed coarse ware. This change seems to happen at the same time that the potter's guild was created in 1402 in Barcelona (called *Confraria de Sant Hipòlit*) having a great impact in the organization of the pottery production: the regulation of their activities was an important step in the process of improvement (García-Oses, 2018a, 2018b).

Potters were spread through the city of Barcelona, to guarantee a correct development in the artisanal activity. This means that there was not an only workshop producing vessels in a specific moment. Moreover, the first payslip to potters dated back from 1389, allowing us to identify 15 potters divided into three quarters: on the one hand, two potters were working on *Quarter del Mar* (those who made bowls), four in *Quarter de Framenors* (making jars and pots), and nine in *Quarter del Pi* (six making pots and three tiles) (García-Oses, 2018b). This shows that in Barcelona were two main areas for producing pottery; one related to construction materials, in the north of Raval and the district of el Pi; and another related to cooking and table products, at the end of the Ramblas and in Framenors (Fig. 1).

The present paper has the aim of deepening our knowledge in the production and decoration of Black and green vessels, aiming to observe whether we can discern if the decorative patterns and the ways of producing the vessels and the glazes can be used as a piece of evidence to discriminate different techniques or crafts.

2. Materials and methods

Intending to get a wide overview of Black and green pottery from Barcelona, 23 new Black and green samples have been recently characterised. For the statistical treatment of their chemical data, the results of those individuals have been put together with the individuals of the database of the chemical groups B1 and B2, the ones which Black and green pottery belongs according to the studies mentioned above. This makes up a set of 82 individuals including Black and green vessels, but also green and honey-coloured glazed coarse ware and transport jars, covering the end of the 13th century and the beginning of the 15th century (Table 1). 62 of those individuals correspond to majolica vessels with Black and green decoration, including sherds of bowls, jars, servers, chandelier, and most of all plates. The samples were gathered from archaeological sites of Barcelona, including *El Born, Pl. De la Gardunya, St. Iu, Sta. Maria del Pi and St. Honorat* (Fig. 1). Concretely, 14 individuals were included in the previous established group B1a, 9 correspond to B1c, 16 to B2 and finally, 23 individuals correspond to a new group, B1e (Fig. 2). 21 individuals correspond to transport jars and glaze coarse ware showing different colours from brown, honey or green. Those individuals were recovered from *Pl. de la Gardunya, Mercat de Santa Caterina* and *Pia Almoina* (Fig. 1) from contexts dated back from 14th to 16th century. Those individuals correspond to group B1a, 6 to B1b, 7 to B1c, and 5 to B1d. As can be seen, any of these individuals corresponds to B2 (Fig. 3, Table 1).

Chemical characterization of the individuals was performed by means of Wavelength Dispersive X-ray fluorescence (WDXRF) analysis. The elements determined were: Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, K_2O, CaO, TiO_2, V, Cr, MnO, Fe_2O_3 (as total Fe), Co, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Mo, Sn, Ba, Ce, W, Pb and Th [1]. Loss on ignition (LOI) was determined by firing 0.3 g of dried specimen at 950 °C during 3 h. Mineralogical characterization of all individuals was performed by means of X-ray diffraction (XRD). The previously prepared powder specimens were manually side-loaded and pressed with frosted glass in a cylindrical sample holder of 27 mm diameter and 2.5 mm in height (PW 1811/27). Finally, the study of the glazes and decorations has been undertaken by means of SEM coupled to an energy dispersive X-ray detector and using the backscattered electrons detector (SEM-BS-EDS). The emphasis was given to the following features: the thickness of the glazes, their composition (including the presence of tin oxide particles, bubbles, quartz and alkali feldspar inclusions, responsible all of them for the opacity of glazes), observations of the interface between paste and glaze to infer whether the application of the glaze was made on the ceramic body before firing or after the first firing, in an already biscuit ceramic, and, finally, nature of pigments used for decorations.

SEM observations were performed on polished sections. Bulk specimens were fixed on metal specimen stubs using silicone adhesive, and the non-conductive ceramic specimens were made conductive. Colloidal silver paint was applied on excess silicone adhesive and lateral sides of the ceramic bulk specimen. Microanalyses were carried out with an Energy-Dispersive X-ray spectrometer (EDX) INCA 250 (Oxford Instruments). The observations were performed using an acceleration voltage of 20 kV, a working distance of 15 mm, and the microanalyses are counted for 100 live seconds. Each chemical analysis is the result of the median of 5 microanalyses by SEM-EDX performed at 5 different areas at 3000X. All the analysis to determine the composition of the glazes of this study have been carried out on homogeneous areas, avoiding possible altered parts and also avoiding the ones presenting extreme values. In this sense, the analysis by SEM-EDX belongs to a wide study, including samples from the 13th to the 17th centuries, covering different decorations (archaic majolica, black and green, blue on white, or lustre). For more information, a detailed description of the method, as well as precision and accuracy, see the publication Madrid and Sinner (2019).

3. Results

3.1. Chemical characterisation

As we already said above, statistical treatment of the new 23 individuals chemically characterised have been carried out jointly with the individuals with the same chronology from ARQUB database. Those vessels are included into groups B1 (subdivided into B1a, B1b, B1c and B1d due to slightly compositional differences) and B2 (subdivided into B2a, B2b and B2c) previously studied (Buxeda et al., 2009, 2011; Madrid et al., 2015). B1 (14th century) is the group related to the first true majolica pottery. At this time, the same clay was prepared for the elaboration of tin-glazed or majolica, green and coarse honey-coloured glazed pottery and transport jars, whilst B2 used a specific paste exclusively for tin-glazed products. These vessels have been considered when comparing the chemical results with the new samples, together with black and green ceramics from B1and B2.

The results of the chemical analysis, that is the elemental concentrations determined, corresponds to a projective space $d + 1$ dimensional, the simplex S^{d+1}, where the projective points are represented by homogenous coordinates, having a constant sum k *(k eR +):*

$x = [x_1,\ldots, x_d, x_{d+1}] \mid x_i \geq 0\ (i = 1,\ldots, d, d + 1), x_1 + \ldots + x_d + x_{d+1} = k,$

(in this case, k = 100). The vector space of these projective points is the positive orthant $R^{d+1}{}_{+}$ and this projective points and their projections in the simplex represent distances which suggest a multiplicative model with a logarithmic interval metrics (Barceló-Vidal et al., 2001; Aitchison, 2005; Buxeda, 2008). For that, in the statistical treatment, the chemical data were transformed into log-ratios (Aitchison, 1986; Buxeda, 1999), using the clr transformation in log-ratios centered, based on:

$$\mathbf{x} \in S^{d+1} \rightarrow \mathbf{z} = \log\left(\frac{\mathbf{x}}{g(\mathbf{x})}\right) \in R^{d+1}$$

where S^{d+1} is the d-dimensional simplex, and g(**x**) is the geometrical average of all the d + 1 components of **x** (Aitchison, 1986; Buxeda, 1999).

In the development of the statistical treatment using R (R Core Team, 2017) the concentrations of Mo and Sn were discarded because of analytical imprecision, as were those of Co and W because of the possible contamination from the tungsten carbide cell mill that was used.

Moreover, it has been observed in other studies about glazed ceramics (Buxeda i Garrigós et al., 2001; Iñañez, 2007) that all the individuals show atypical high concentrations of Pb and Sn, due to contamination produced by the diffusion of these components present in glazes, into the matrix during the firing process. For that, those elements have been excluded. Furthermore, the interferences presented in Pb concerning other elements cannot be well corrected. For that reason, elements such as Y, Ga, Th and Rb cannot be taken into consideration, and because of changes in the analytical routines.

Above the total of the individuals analysed, the compositional variation matrix has been calculated, to quantify the total variation (vt) in the data, and to investigate the origin of this variability: which are the components responsible for the exististing differences. Elements mentioned above have not been considered due to contamination problems. Moreover, Cu and P_2O_5 have been eliminated from the statistical treatment, since some individuals exhibit high concentrations that seem to correspond to contaminations. The statistical treatment has been developed with the language and software R (R Core Team, 2014).

[1] Major and minor elements are expressed as concentrations of oxides in percentage by mass (also referred to as wt %). Trace elements are expressed as concentrations of elements in μg g^{-1} (or ppm —parts per million).

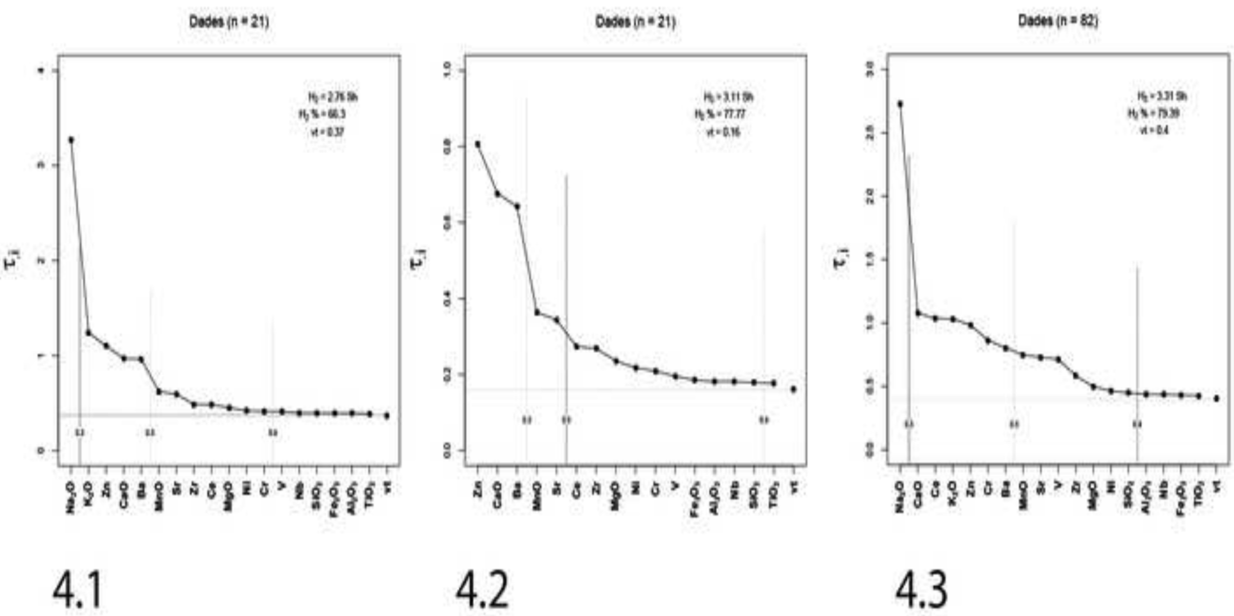

Fig. 4. Compositional evenness plot of the last 23 samples (4.1). Compositional evenness plot without Na2O and K2O (4.2). Compositional evenness plot of all the individuals (82) from B1 and B2 (4.3). H2 = information entropy (in Shannons, symbol Sh, i.e. bits). H2% = percentage of information entropy relative to the possible maximum attainable. Vt total variation. τ.i sum of variances of alr transformed concentrations using element i as denominator.

To study the variability existing in the chemical data, the compositional variation matrix has been calculated, allowing to quantify the total variation (vt) and find out the origin of this variability. The graphic of uniformity (Fig. 4) of the new 23 samples analysed, let us make a visual summary of how the vt is distributed (Buxeda and Kilikoglou, 2003). In the present study, the vt is considered high (vt = 0.37) (Fig. 4.1), suggesting a non-homogeneous aggrupation, with some variability. As it can be observed in Fig. 4.1, most of the chemical variability is associated to Na_2O and K_2O, related to an alteration and contamination process occurred during burial, that will be explained in the technical part. Exploring the composition variation matrix without taking into consideration both elements (Fig. 4.2), it is shown a vt of 0.16, more homogenous, and characteristic from a monogenic group. Comparing the new samples with Black and green vessels from B2 and with all the individuals from B1, a vt of 0.4 is gathered (Fig. 4.3). This implies that the chemical composition has high variability, far from what can be considered as a homogeneous group (Buxeda, and Kilikoglou, 2003), suggesting the use of different clays, or differences in the clay treatment or paste body which can be related to different chemical groups, without implying different geographic location.

The results of the statistical treatment (Tables 2 and 3) are summarized in the dendrogram resulting from the cluster analysis performed on the previous sub compositions, using the squared euclidean distance and the centroid algorithm, performed by Na_2O MgO, Al_2O_3, SiO_2 P_2O_5, K_2O, CaO, TiO_2, V, Cr, MnO, Fe_2O_3, Ni, Zn, Sr, Zr, Nb, Ba (Fig. 5).

The study of the dendrogram shows the existence of four different groups (Fig. 5). Na_2O and K_2O have not been taken into account since it causes the separation of each group in several groups due to the alteration and contamination process undergone by part of those samples during burial causing the crystallization of analcime which affects the values of sodium, potassium and sometimes, rubidium (Buxeda i Garrigós, 1999; Schwedt et al., 2006; Buxeda i Garrigós et al., 2002).

Firstly, B1e contains the new Black and green individuals analysed from the excavations in *Rec Comtal*, in the archaeological site of *Antic Mercat del Born*, dated back to the 14th century. Continuing to the right, the next group is formed by individuals from B1a dated back from the end of the 13th century, and from the 14th century. Next, is group B2, formed only by Black and green tin-glazed pottery and can be dated back to the 14th century, and by two samples classified previously (Iñañez, 2007) in B1a (MJ0088 and MJ0191). Last and bigger group is the combination of individuals from groups B1b, c and d, which corresponds to the paste prepared for some transport jars and mainly for glazed coarse ware, being in use for the elaboration of this latter until the 16th century. In this sense, group B1d is formed mainly by transport jars covering a wider period of two centuries, (14th and 15th) when Barcelona stopped manufacturing these large containers, whilst group B1c is the only one which comprises those categories plus Black and green tin-glazed pottery from B2 (marked with a purple square in Fig. 5) and is the oldest one, covering a large period from the end of the 13th century to the 16th century.

The result has been the definition of a new subgroup, B1e that corresponds to the new Black and green tin-glazed individuals analysed. Concerning BCN461 and BCN453 (Fig. 5), they remain ungrouped in the dendrogram. In the case of BCN461, the contents in MnO, Ce and Zn are much higher concerning the other samples. In contrast, the individual BCN453 shows the lowest Al_2O_3, and differences in Ce and Zn that recommend keeping it on ungrouped (Table 2).

3.2. Mineralogical study

Chemical results show that the individuals analysed correspond to ceramics technically considered as calcareous (CaO > 5%-6%). In terms of phase transformations and densification during firing inducing microstructural changes (Maggetti, 1981; Maniatis and Tite, 1981; Tite et al., 1982; (Heimann and Maggetti, 2014)), calcareous ceramics develop more high-temperature phases than low calcareous pottery and a lighter microstructure with a gradual formation of a vitreous phase. As is shown in the triangle ceramic phase diagram CaO($+Fe_2O_3$ + MgO)--SiO_2 + Al_2O_3 (Fig. 6), all the individuals analysed in this study are placed in the quartz-anorthite-wollastonite thermodynamic equilibrium triangle, characteristic of calcareous ceramics.

The study of the XRD diffractograms allowed the identification of five fabrics[2] (F1 to F5), i.e. different categories of association of

[2] Fabric is the final product of a paste after firing. In this respect, a paste can end in more than one different fabric (Buxeda et al., 1995; Buxeda and Madrid 2016, 36)

Table 2
Elemental concentration determined for the 82 individuals in this study. Major and minor elements and loss of ignition (LOI) are expressed in mass percent. Trace elements are expressed in micrograms per gram.

	Na2O	MgO	Al2O3	SiO2	P2O5	K2O	CaO	TiO2	V	Cr	MnO	Fe2O3	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Mo	Sn	Ba	Ce	W	Pb	Th	LOI
BCN440	0.75	1.86	15.45	56.88	0.34	3.73	11.38	0.78	108	86	0.08	5.9	18	39	96	95	8	187	159	41	209	18	1	82	556	112	52	3959	10	3.11
BCN446	1.31	1.95	15.67	57.76	0.25	2.88	10.67	0.8	106	92	0.07	6.02	18	40	105	104	8	182	144	40	207	19	1	65	499	105	51	3759	11	3.01
BCN448	1.04	1.98	15.69	56.69	0.21	3.05	10.62	0.77	105	91	0.08	5.88	20	41	82	88	6	192	170	43	218	18	1	30	548	129	55	5165	9	1.63
BCN449	1.18	1.9	15.41	57	0.23	2.9	10.29	0.77	94	87	0.08	5.84	17	37	33	77	10	188	189	34	201	18	1	33	576	106	41	2260	13	2.4
BCN450	0.84	1.8	15.9	59.48	0.24	3.75	8.34	0.81	114	87	0.09	6.09	18	40	84	75	7	190	177	42	222	19	1	151	728	123	50	4243	10	1.98
BCN451	0.5	1.82	15.63	57.51	0.26	4.01	9.01	0.77	109	82	0.08	6.03	17	38	45	75	8	192	200	41	222	18	1	60	606	108	30	3952	12	2.81
BCN452	1.29	2.01	15.5	56.69	0.23	2.71	10.81	0.78	107	84	0.08	5.95	18	38	204	89	9	177	176	39	217	18	1	40	533	107	47	3213	12	3.39
BCN453	0.52	1.78	13.93	55.48	0.3	4.01	11.38	0.72	95	74	0.08	5.25	16	32	136	164	11	180	166	32	196	17	1	81	613	91	34	1513	13	5.46
BCN454	0.65	1.86	16.41	60.27	0.34	3.92	7.71	0.83	107	94	0.08	6.26	17	40	90	83	12	190	162	38	226	19	1	56	643	108	37	2511	13	1.59
BCN455	0.82	1.86	15.29	56.61	0.23	3.44	11.07	0.78	108	92	0.1	6.04	19	38	107	82	9	183	198	38	212	19	1	67	638	104	97	3113	12	2.46
BCN456	1.21	2.04	15.89	56.42	0.22	2.82	11.23	0.78	112	91	0.08	5.96	21	39	83	93	5	179	154	43	208	18	1	74	530	125	37	5458	9	3.12
BCN457	1.6	2.13	15.74	54.48	0.24	2.19	12.67	0.8	100	89	0.08	5.99	19	41	184	89	10	168	182	38	192	18	0	45	552	103	53	3159	12	3.02
BCN458	0.52	2.01	14.75	53.34	0.3	3.84	13.84	0.73	103	82	0.09	5.58	15	36	102	82	10	191	194	35	174	17	1	67	592	97	55	2599	11	3.47
BCN459	0.5	1.75	14.77	56.22	0.42	3.97	10.61	0.75	104	81	0.08	5.62	16	36	79	80	8	197	168	39	210	18	1	15	1042	112	27	3368	11	4.44
BCN460	0.64	1.88	16.03	57.97	0.31	3.85	9.46	0.8	107	92	0.08	6.02	19	40	62	85	11	194	159	38	226	19	1	21	736	106	74	2505	13	2.46
BCN461	0.9	1.81	15.3	57.73	0.36	3.8	8.82	0.78	124	89	0.12	5.81	18	42	78	155	7	151	157	40	216	18	2	46	928	116	25	3850	11	2.67
BCN462	1.69	2.06	15.81	53.06	0.26	2.07	13.12	0.76	110	86	0.08	5.88	29	39	73	81	11	177	192	34	176	19	1	32	595	99	94	2147	13	4.73
BCN463	1.39	1.92	15.79	56.03	0.22	2.63	10.97	0.78	111	88	0.07	6.1	31	41	90	86	6	175	180	42	208	18	1	29	588	118	133	4762	9	3.16
BCN464	0.36	1.88	14.98	53.14	0.5	4.06	12.16	0.74	99	76	0.1	5.44	13	32	132	76	10	196	199	33	171	18	1	46	840	91	24	1689	14	5.71
BCN465	0.78	1.89	15.7	57.77	0.27	3.73	9.3	0.79	105	87	0.08	5.88	55	40	81	79	8	192	175	40	217	19	1	33	622	110	148	3742	12	2.93
BCN466	0.94	1.95	15.82	57.61	0.22	3.35	9.72	0.78	105	81	0.07	5.97	43	39	152	80	10	191	196	37	218	19	1	43	612	106	185	2607	13	3.2
BCN467	0.78	2.08	15.38	55.35	0.27	3.5	10.81	0.76	102	84	0.08	5.88	30	38	88	84	10	187	199	38	207	18	1	46	615	97	117	2945	12	4.02
BCN468	0.82	2.08	15.07	53.17	0.42	2.47	14.81	0.73	93	75	0.07	5.45	29	35	117	76	8	143	207	37	173	18	1	64	538	100	116	3297	12	5
BCN210	0.54	1.83	16.03	57.32	0.34	3.91	9.55	0.77	103	65	0.08	6.02	19	38	36	105	20	195	173	33	211	17	0	7	569	77	85	346	13	4.37
BCN211	0.57	1.73	15.97	57.82	0.12	3.55	8.67	0.77	111	62	0.08	5.97	18	39	33	102	20	172	132	32	216	17	0	5	639	77	68	166	13	5.34
BCN212	0.47	1.71	14.05	53.66	0.13	3.72	12.89	0.7	96	59	0.07	5.43	19	33	26	93	18	177	140	29	205	16	1	7	632	74	55	108	12	7.9
MJ0087	1.06	1.81	14.74	56.73	0.17	3.23	11.26	0.71	81	75	0.08	5.39	18	35	146	103	45	180	146	39	224	17	4	32	437	58	77	4191	19	4.25
MJ0088	0.63	1.86	15.02	55.16	0.24	3.89	11.84	0.73	84	69	0.06	5.63	16	36	122	111	42	198	151	37	205	17	3	16	432	57	74	3456	18	3.88
MJ0092	1.28	1.88	15.28	57.58	0.18	2.89	10.48	0.74	86	81	0.07	5.67	18	35	101	102	46	173	144	39	232	18	3	63	437	67	65	4193	17	3.04
MJ0093	0.75	2.03	15.57	56.47	0.18	3.8	10.33	0.76	82	74	0.08	5.84	21	37	98	150	46	189	147	39	224	18	3	34	441	70	109	3982	17	2.94
MJ0096	0.76	1.82	14.94	57	0.19	3.64	10.52	0.72	80	79	0.08	5.6	18	34	44	108	34	183	135	34	225	17	3	51	446	57	80	2183	17	3.76
MJ0098	0.64	1.92	14.96	57.51	0.24	3.91	9.72	0.72	72	81	0.08	5.6	17	34	46	111	29	187	145	33	235	18	3	22	531	79	65	1453	18	3.58
MJ0099	0.87	2.09	15.38	58.14	0.21	3.9	8.82	0.75	77	75	0.08	5.77	22	34	56	119	35	187	143	34	232	18	4	57	457	71	127	2371	18	3.1
MJ0189	0.62	1.93	14.86	54.63	0.39	3.86	12.44	0.7	74	71	0.09	5.46	16	35	370	109	42	188	155	38	207	17	3	47	490	57	51	3659	18	3.99
MJ0191	0.63	1.86	14.79	54.64	0.22	4.14	12.93	0.69	77	70	0.07	5.02	15	32	103	104	39	210	158	35	196	17	3	41	462	61	35	3113	17	3.93
MJ0192	1.08	1.84	15.4	57.73	0.76	3.24	9.43	0.76	77	76	0.07	5.64	17	36	102	121	43	212	166	39	227	18	3	27	486	67	60	3599	18	2.73
MJ0194	0.56	1.81	15.5	57.45	0.22	3.99	9.85	0.74	79	75	0.07	5.69	24	32	54	101	28	198	137	31	221	17	3	21	514	73	82	1265	18	3.09
MJ0195	0.67	1.89	15.06	56.14	0.26	3.75	11.15	0.75	75	75	0.07	5.58	16	34	147	108	34	195	165	34	208	18	3	90	458	63	50	2348	17	4.09
MJ0196	0.65	1.83	15.1	56.15	0.44	3.65	10.8	0.7	78	73	0.07	5.46	17	33	187	120	41	190	151	37	225	17	3	31	442	69	58	3387	18	3.61
BCN120	0.52	2.07	15.68	53.49	0.23	4.23	13.45	0.71	81	52	0.07	5.59	18	41	166	115	36	210	161	37	182	18	2	93	539	68	86	2985	15	3.26
BCN126	1.49	1.95	14.9	53.62	0.27	2.38	13.65	0.68	88	50	0.07	5.48	20	38	359	113	34	176	154	37	201	18	2	47	480	73	158	2914	15	4.58
BCN129	0.46	1.87	14.63	54.85	0.47	4.1	11.45	0.68	79	51	0.07	5.37	16	37	54	116	28	202	185	35	199	18	2	86	665	79	55	1809	13	5.25
BCN134	1.35	1.85	14.94	56.72	0.55	2.93	10.33	0.69	80	57	0.08	5.4	20	37	89	105	42	167	166	39	213	18	1	53	593	76	100	3901	14	3.86
BCN137	1.74	1.8	15.84	58.59	0.22	2.4	10.32	0.71	92	55	0.06	5.4	26	41	141	111	75	114	139	47	210	18	2	30	537	81	215	8350	13	1.56
MJ0089	0.63	1.86	15.02	55.16	0.24	3.89	11.84	0.73	84	69	0.06	5.63	16	36	122	111	42	198	151	37	205	17	3	16	432	57	74	3456	18	3.88
MJ0090	0.56	1.88	15.35	49.64	0.22	4.15	16.49	0.68	68	63	0.07	5.25	16	32	102	112	31	211	180	32	165	17	3	8	611	61	38	1784	18	4.6
MJ0091	0.52	1.99	15.09	53.55	0.34	4.29	12.81	0.7	74	68	0.07	5.43	15	34	106	110	28	223	181	31	173	17	3	23	564	64	30	1294	17	4.17
MJ0094	1.14	2.16	16.1	53.78	0.3	3.78	12.13	0.73	88	74	0.07	5.67	20	40	91	112	60	205	160	45	187	19	3	52	512	64	80	5953	19	2.11
MJ0095	1.34	1.98	15.4	54.65	0.32	3.16	12.43	0.72	80	74	0.06	5.92	14	36	145	117	34	221	189	34	183	18	3	48	494	57	48	2008	19	3.26
MJ0097	1.62	1.98	15.84	55.78	0.2	2.56	10.98	0.76	90	80	0.08	5.92	17	38	179	116	44	187	160	38	204	18	3	119	447	72	83	3680	17	2.93
MJ0100	0.8	1.74	15.52	60.51	0.21	4.26	6.95	0.76	81	79	0.08	5.83	34	34	177	105	34	190	133	35	238	18	3	26	622	59	193	2210	19	2.44
MJ0190	0.44	1.99	14.95	51.25	0.32	4.18	14.66	0.67	78	66	0.08	5.21	14	35	80	106	38	210	162	34	167	17	2	39	518	50	46	2749	20	5.18
MJ0193	0.64	1.87	14.61	57.92	0.46	3.23	7.21	0.73	71	67	0.16	5.36	17	35	169	119	41	192	194	36	182	17	2	63	505	61	43	3481	17	4.99
MJ0197	0.5	2.1	14.13	49.31	0.23	3.75	16.24	0.64	69	65	0.06	4.79	15	31	97	106	44	179	167	36	174	15	2	44	439	56	36	4024	17	6.99
MJ0198	1.09	1.96	14.66	52.37	0.16	2.82	14.12	0.69	77	67	0.08	5.35	17	33	75	103	37	174	163	33	181	17	3	57	477	60	49	2816	18	5.32

Table 3
Mean and standard deviation (sd) of the different chemical groups characterized by XRF from Barcelona.

Components	Chemical group							
	B1bcd (n = 29)		B1e (n = 21)		B1a (n = 15)		B2 (n = 11)	
	mean	sd	mean	sd	mean	sd	mean	sd
Na2O (%)	0.62	0.18	0.97	0.39	0.86	0.32	0.95	0.49
MgO (%)	1.88	0.13	2.02	0.12	1.99	0.09	2.07	0.13
Al2O3 (%)	16.26	0.54	16.18	0.32	15.87	0.29	15.95	0.42
SiO2 (%)	59.30	1.10	58.61	1.57	58.99	1.16	56.18	2.14
K2O (%)	3.94	0.26	3.41	0.66	3.80	0.49	3.75	0.78
CaO (%)	10.75	1.40	11.34	1.90	11.34	1.23	14.20	2.17
TiO2 (%)	0.78	0.04	0.81	0.02	0.76	0.02	0.73	0.02
V (ppm)	102.62	14.71	109.95	5.55	82.73	4.93	84	7.11
Cr (ppm)	62.45	8.16	89.43	4.76	78.40	4.03	66	9.60
MnO (%)	0.08	0.01	0.08	0.01	0.08	0.01	0.07	0.01
Fe2O3 (%)	6.03	0.28	6.13	0.17	5.88	0.14	5.66	0.27
Ni (ppm)	38.45	2.60	40	2.14	36.40	1.72	37.91	3.48
Zn (ppm)	113.48	8.14	87.10	7.39	118.40	12.28	116.45	4.66
Sr (ppm)	145.17	16.79	187.33	19.93	159	12.94	174.55	16.29
Zr (ppm)	224.21	16.25	213.67	17.02	227.33	16.15	195.91	13.73
Nb (ppm)	18.52	1.24	19.14	5.73	18.47	5.16	18.45	1.04
Ba (ppm)	585.72	57.23	653.95	132.79	489.73	42.68	549.73	69.57
Ce (ppm)	80.83	5.14	112.19	9.36	68.73	7.05	68	9.84

crystalline phases for the new group B1e enabling us to estimate four different Equivalent Firing Temperature (EFT) (Table 4, Fig. 7). F1 (BCN451) exhibits alkali feldspar, plagioclase, quartz, hematite, calcite, pyroxene, gehlenite and illite-muscovite (Fig. 7 F1). Diffractograms of fabric F2 (BCN460 BCN453, BCN461, BCN454, BCN459, BCN454, BCN451, BCN464) and F3 (BCN440, BCN467, BCN455) exhibit the same mineral phases except for illite-muscovite, which is highly reduced for F2 and practically decomposed for F3 (Fig. 7, F2 and F3). The two mineral phases that can provide evidence about the EFT are pyroxene and gehlenite, showing both an initial growth for F1 and an important development for F2 and especially for F3. Finally, F4 (BCN457, BCN462, BCN446, BCN448, BCN456, BCN449, BCN452, BCN463) and F5 (BCN466 and 468), show the total decomposition of illite together with an important reduction of calcite and gehlenite for F4, and the total decomposition also of these phases in F5; and showing the two fabrics the presence of intense peaks of analcime (Fig. 7, F4. Those observations enable us to estimate an EFT in the range of 850–950/1000 °C for F1, slightly higher for F2, in the range of 900–950/1000 °C, and about 950/1000 ⁰C for F3. In the case of F4 and F5, the EFT can be estimated in excess of 950/1000 ⁰C. This wide range of fabrics and the differences in the EFT observed for group B1e agrees with the observations made for subgroups B1a and B1c and could be related to limited technical know-

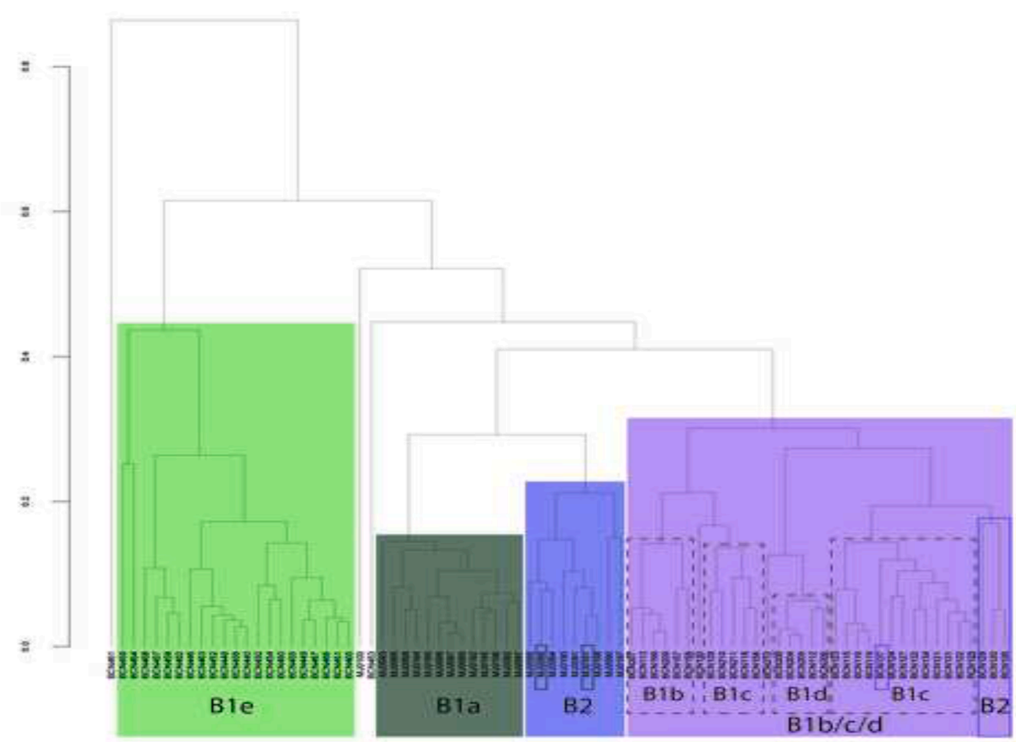

Fig. 5. Dendrogram performed by using the squared Euclidian distance and the centroid agglomerative algorithm on the clr transformed subcomposition Na2O, MgO, Al2O3, SiO2, K2O, CaO, TiO2, V, Cr, MnO, Fe2O3 (as total Fe), Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Ba, Ce and Th. Firstly, in green new group B1e, in brown B1a, in purple B2, finally in pink B1/b/c/d. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

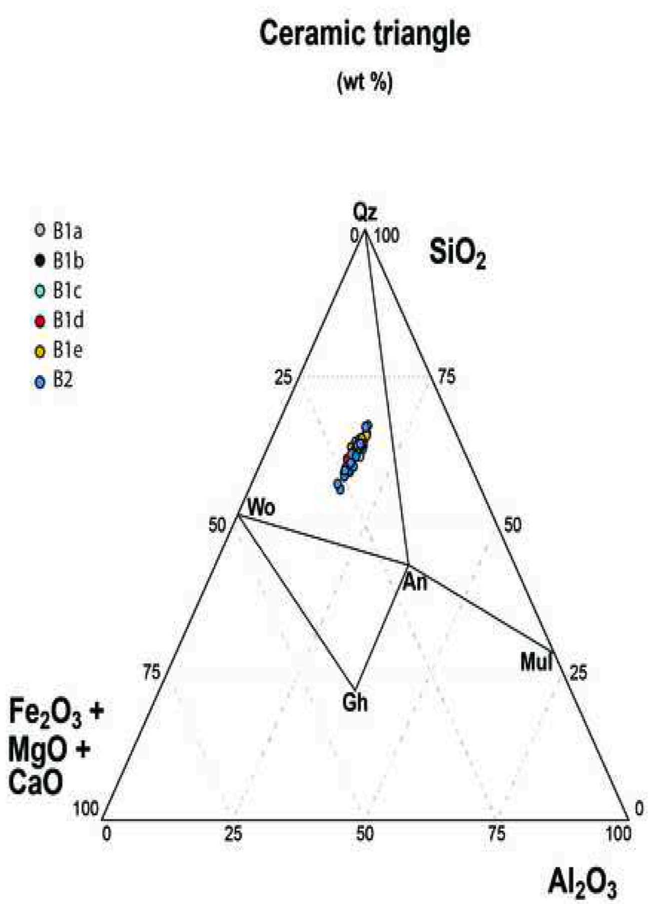

Fig. 6. Triangle ceramic phase diagram CaO(+Fe2O3 + MgO)-SiO2 + Al2O3.

how. In contrast, for B2 only one range of EFT was estimated: 900–950/1000 °C. Indeed, ceramics from B1a shows high know-how than in B1c, with more fabrics and different range of temperatures, allowing us to identify an evolution in group B1. The use of a specific base clay in B2 for majolica and the control of the EFT could be explained by the achievement of deep knowledge and control in the technique of producing black and green vessels.

Furthermore, it must be highlighted that individuals from B1e and B2 showing an EFT > 950/1000 °C, can be considered overfired, favouring the lixiviation of the crystalline phase, absorption of sodium from the environment and the crystallisation of analcime, which is the cause of the separation from the others in the chemical analysis.

3.3. Glaze study

The study of decorations by SEM-EDX has been focused on eight individuals from B1e (BCN440, BCN446, BCN449, BCN451, BCN452, BCN461, BCN463, BCN467 and BCN468), one outlier from the same group (BCN461), one sample without being included in any chemical group (BCN453), two samples from B1a (MJ0191, MJ0096), and one sample from B2 (MJ0090). The results show a high complexity in the production of glazes, especially in the application of black decoration -even though some problems related to alteration have been detected.

The elaboration of the glazes of most Black and green vessels presented in this article consisted of a double firing process, which means that the application of the glaze on the ceramic body was made after a first firing, in an already biscuit ceramic. This practice avoids problems to the glaze, as bubbling, due to the gas formation during firing, as well as other difficulties related to retractions and porosities although a small part seems to be the result of a single firing (Molera et al., 2001). The

Table 4
Mineralogical results from XRD. Afs: alkali feldspar; Anl: analcime; Cal: calcite; Gh: gehlenite; Hem: hematite; Ilt: illite-muscovite; Ill*: illite-muscovite without the 10 Å, peak; Pl: plagioclase; Px: pyroxene; Qz: quartz. Abbreviations according to Whitney and Evans (2010).

Chemical group	Fabrics	Mineral phases	Samples	Equivalent Firing Temperature (EFT)
B1e	1	Ilt, Qz, Afs, Hem	BCN450	850–950/1000 °C
	2	Ilt, Qz, Afs, Pl, Cal, Gh, Di, Hem	BCN453, BCN461, BCN454, BCN459, BCN454, BCN451, BCN464	900–950 °C
	3	Anl, Ilt*,Qz, Afs, Pl, Cal, Gh, Di, Hem	BCN440, BCN467, BCN455	950/1000 °C
	4	Anl, Qz, Afs, Pl, Cal, Gh, Di, Hem	BCN457, BCN462, BCN446, BCN448, BCN456, BCN449, BCN452, BCN463	>950/1000 °C
	5	Anl, Qz, Afs, Hem, Px	BCN466, BCN468	>950–1000 °C
B1c/d	1	Ilt, Qz, Afs, Cal, Gh, Hem	BCN121, BCN124, BCN132, BCN131, BCN122, BCN125, BCN128	850–950/1000 °C
	2	Ilt*, Qz, Afs, Cal, Gh, Px, Hem	BCN127, BCN122	900–950/1000 °C
	3	Anl, Qz, Afs, Cal, Di, Px, Hem	BCN130	>950/1000 °C
B1a	1	Ilt, Afs, Pl, Cal, Gh, Di, Px, Hem	MJ0098, MJ0195, MJ0191, MJ089, MJ0189	900–950/1000 °C
	2	Anl, Ilt*, Afs, Pl, Cal, Gh, Di, Px, Hem	MJ0093, MJ0099, MJ0194, MJ096, MJ088	900–950/1000°C
B2	1	Ilt*, Afs, Pl, Cal, Gh, Di, Px, Hem	MJ0190, MJ0090, MJ0197, MJ0091, BCN129, BCN121	900–950/1000 °C
	2	Anl, Ilt*, Afs, Pl, Cal, Gh, Di, Px, Hem	MJ0097, MJ0192, MJ0087, MJ0198, MJ0094, MJ0095	900–950/1000 °C

base of the glaze contains a significant amount of tin oxide (Table 5[3]), and few inclusions of quartz and feldspar, possibly accidental. In all the cases the decoration consisted of copper for the green decoration, and manganese oxide for the black motifs, sometimes dissolved into the glaze and sometimes related to crystals of kentrolite $(Pb_2Mn^{3}+_2(Si_2O_7)O_2)$ a lead silicate, and bustamite ($CaMnSiO_6$), a calcium manganese pyroxene, as observed in other similar cases (Molera et al., 2013, 2018).

3.3.1. Double firing and manganese dissolved into the glaze

BCN440 and BCN468 from B1e were the result of a double firing process, as it can be observed at the interface of ceramics, where no interaction between the matrix and the glaze could have been detected. The black decoration was dissolved into the glaze, but in some areas of both vessels, a non-well-distributed glaze was detected, forming concentrations in some parts of the surface where brightly areas correspond to high concentrations of lead without silica, and black areas to silica without lead (Fig. 8.1). These parts underwent the leaching of lead due to an alteration process. For this reason, the analysis of black decoration has been carried out avoiding these altered areas (Fig. 8.3, .5). The black decoration is identified not only because of the accumulation on the upper part of the glaze (Fig. 8.2) but also dissolved into the glaze. The accumulation of manganese in the upper part of the glaze in this sample

[3] SEM-EDX is a semi-quantitative technique, so the values provided cannot be as accurate as in a quantitative technique.

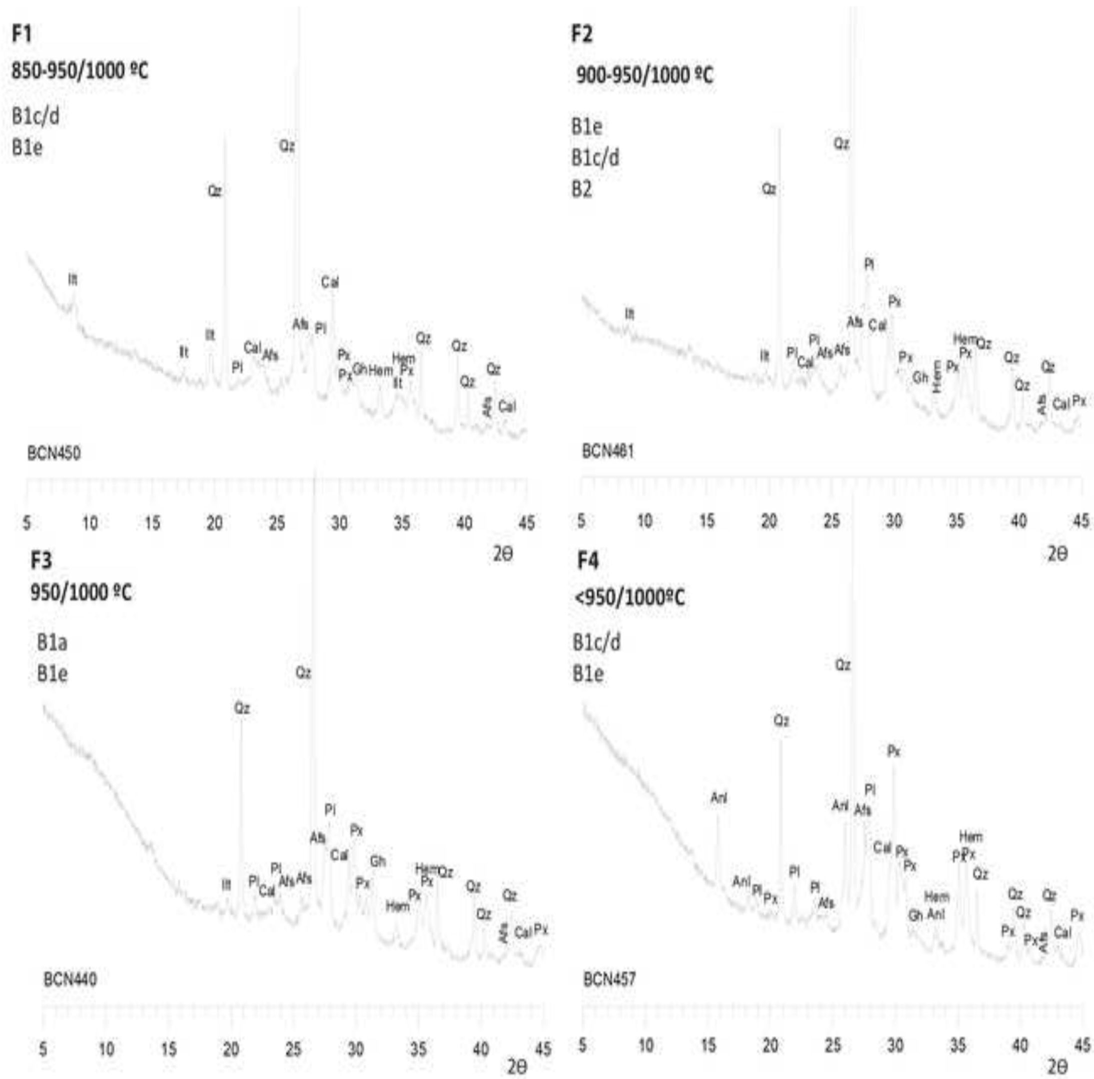

Fig. 7. XRD patterns for the categories of association of crystalline phases as detected by XRD, with all the Estimated Firing Temperature (EFT) determined. F1: FTE 850–950/1000 °C, F2: FTE 900–950/1000 °C, F3: FTE: 950/1000 °C, F4: <950/1000 °C. Afs: alkali feldspar; Anl: analcime; Cal: calcite; Gh: gehlenite; Hem: hematite; Ilt: illite-muscovite; Pl: plagioclase; Px: pyroxene; Qz: quartz. Abbreviations according to Whitney and Evans (2010).

is only observed in vessels with some alteration in the glaze. Nevertheless, in other samples (such as BCN451) this accumulation is present in a non-altered area.

BCN467 (B1e) and BCN461 (outlier) are also the result of a two-firing process, without any interaction between the matrix and the glaze detected (Fig. 8.4). In this case, areas corresponding to black decoration correspond to manganese and iron oxide dissolved into the glaze, whilst green corresponds to copper and iron, also dissolved.

In all cases, black areas show a high amount of tin oxide (Table 5). The amounts of lead, silicon and tin oxide are slightly the same in BCN440, BCN467 and BCN468. Notwithstanding, some differences in the composition of glaze have been identified in BCN461 (the outlier one), due to higher content of lead (about 10%), and a lower value of silicon oxide. Moreover, the use of tin oxide also shows higher contents, from 13.39% to 15.66%.

3.3.2. One firing and manganese dissolved into the glaze

In contrast, BCN452 and BCN463 (from B1e) were the results of a single firing process, as it can be observed a developed area with newly formed feldspars crystals between the matrix and the glaze (Fig. 8.6). In both cases, as in the previous samples, manganese oxide for the black decoration, and copper for the green, were dissolved into the glaze. The amounts of lead and silicon are slightly the same than in the previous one. Concretely, BCN452 shows a great similarity with BCN468 in all the components (Table 5). In the case of BCN463 higher amount of tin oxide (16.98%) has been observed.

3.3.3. One firing, manganese and green dissolved into the glaze, and crystals in green areas

BCN446 (Fig. 9.1) and BCN449 (Fig. 9.2) from B1e were the results of a single firing process, as we are capable to observe a great layer of interaction in the interface where many newly formed feldspar and lead crystals grew up (Fig. 9.2). Black (made with manganese oxide) and green (made with copper) decoration are dissolved into the glaze (Fig. 9.5). The glaze of both individuals is plenty of black crystals only in those areas where green decoration is applied corresponding to pyroxenes which have precipitated in this area (Fig. 9.3 and 4). It should be noted that in BCN446 some differences in the composition of the glaze have been identified, higher silicon oxide and a lower amount of lead (Table 5). These values are similar to the ones observed for MJ0091 from group B2.

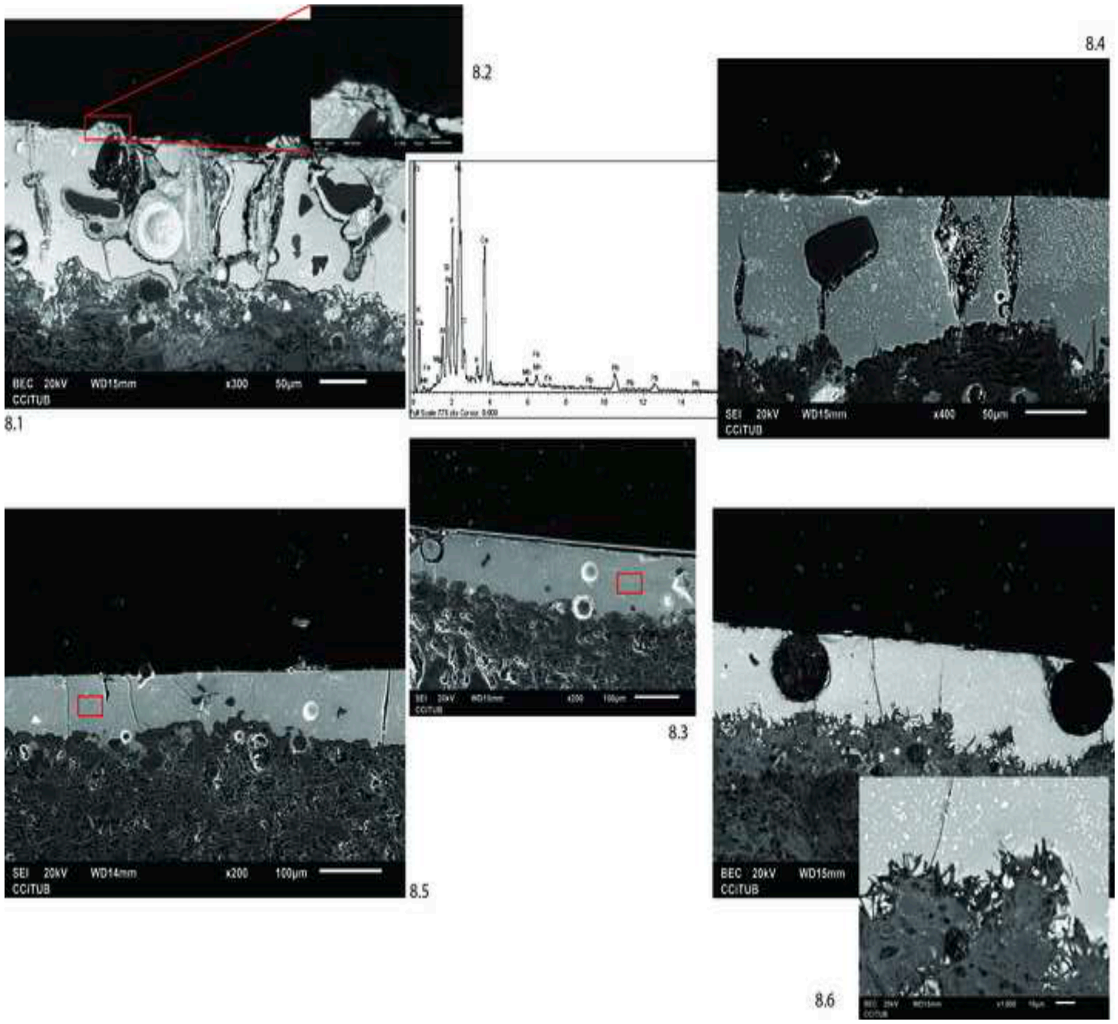

Fig. 8. 1. BCN 440, altered glaze in the black decoration, brightly areas correspond to high concentrations of lead without silica and black areas to silica without lead. 2. Accumulation of manganese in the upper part of the glaze with its microanalysis. 3. Black decoration without any alteration, the red square corresponds to the analysed area. 4. BCN468, black area with manganese dissolved into the glaze. The white crystals correspond to accumulations of tin oxide. 5. Green area, corresponding to copper dissolved into the glaze, in red, the analysed area. 6. BCN463, interface resulting from a two-firing process. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 5
Composition of glazes. 5 microanalyses were done in each sample at 3000X in non-altered areas. The median of the 5 microanalyses has been calculated to estimate the concentrations of the elements.

Sample	Chemical Group	%									
		Na2O	Al2O3	SiO2	K2O	PbO	CaO	CuO	SnO2	MnO	FeO
BCN440	B1e	0.55	2.36	36.34	3.09	39.73	2.36	0.83	13.91	0.82	
BCN446			4.31	43.03	2.35	28.27	4.43		14.67	0.56	1.85
BCN449		0.61	3.98	35.84	2.13	38.78	2.99	0.63	11.55	1.60	1.76
BCN451		0.38	2.43	29.05	1.51	48.76	1.46	0.40	14.18	1.09	0.75
BCN452		0.57	4.06	40.42	3.27	32.34	2.23	1.25	13.00	1.25	1.62
BCN461		0.72	1.82	32.83	1.28	41.86	1.70	2.74	16.99	0.36	0.52
BCN463		0.66	3.12	34.23	2.40	40.50	0.40	0.84	15.66	0.75	1.36
BCN467		0.66	3.04	33.73	1.60	38.68	2.29	0.87	16.98	0.56	1.35
BCN468		0.68	3.43	37.72	3.21	33.24	3.62	0.60	14.88	1.28	1.47
BCN453	Out	1.08	2.34	39.84	6.31	34.70	2.40		12.20	0.63	0.21
MJ0091	B2	1.83	2.68	46.74	6.45	26.60	0.52		15.18		
MJ0191	B1a	0.56	2.50	29.64	1.87	37.81	2.48		18.46	0.43	
MJ0196	B1a	0.72	3.35	36.92	2.09	35.02	1.21	3.32	15.01	0.67	1.68

3.3.4. *Double firing, manganese dissolve into the glaze, with traces of crystallization*

BCN451 (Fig. 10), from B1e, was the result of a double firing process. The black decoration was made with manganese oxide dissolved into the glaze, but also presenting accumulations of manganese on the upper part of the glaze, without being altered. Crystals of kentrolite ($Pb_2Mn^{3}+_2(Si_2O_7)$ O_2) (Fig. 10.2), a lead silicate, are developed in the interface of these areas. Bustamite ($CaMnSiO_6$), a calcium manganese

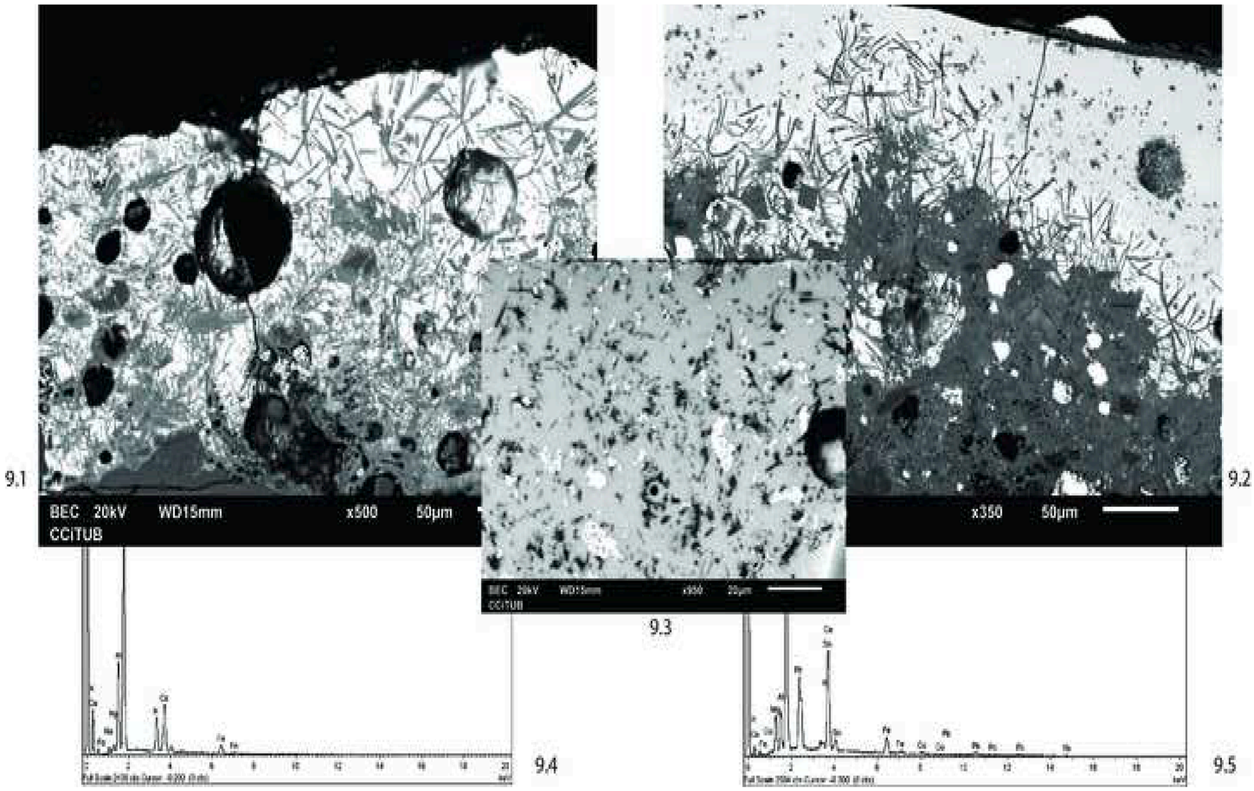

Fig. 9. 1 BCN446. 2. BCN449. Both correspond to an interface resulting from a single firing process with newly formed crystals. 3. Black points correspond to pyroxenes, which have precipitated in the green area. 4. Microanalysis of the pyroxenes. 6. Microanalysis of green decoration. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

pyroxene, is also identified (Fig. 10.3). This is also observed in MJ0191 from B1a. Even the most common way to identify black decorations in medieval and postmedieval tin-glazed pottery is as a dissolution into the glaze (Iñañez, 2007; Pérez-Arantegui et al., 2005), crystals of kentrolite and bustamite have been observed for brown and for black and green products of the Iberian Peninsula, such as Andalusia (Molera et al., 2013, 2018).

Kentrolite crystals are formed due to the reduction of Mn^{4+} first to Mn^{3+} and then to Mn^{2+}, and the reaction with the lead (Molera et al., 2013). This transformation is observed to happen at the low temperature of 650 °C, remaining stable at least at 840 °C. As Molera and collaborators observed, the high contents of lead in these productions may have helped the formation of kentrolite. Bustamite is formed when large amounts of manganese are applied on the glaze facilitating the grown of acicular crystals into the glaze (Molera et al., 2013).

In the case of the individual BCN453, the one ungrouped, a white slip between the glaze and the matrix is detected (Fig. 11.3), which cannot be identified in any of the other individuals. The slip is only present where the green decoration is applied and can even be identified macroscopically. By SEM-EDX, this layer corresponds to Mg, Si, Ca and Fe (Fig. 11.4). Moreover, the green area which is a grey line on the upper part of the glaze by SEM-EDX, corresponds to Pb, Fe, and Cu, indicating that the application of the pigment was realized in the upper part of the glaze (Fig. 11.5).

In the individual BCN453, also with the same crystals of kentrolite and bustamite than in the previous samples detected (Fig. 11.1), crystals in the interface) an accumulation of crystals of manganese was detected (Fig. 11.2). These crystals were also observed in black and green ceramics from B1c (Di Febo, 2015), where the formation of acicular crystals in the interface was interpreted as the application of the pigment straight on the ceramic body. Also, a high presence of crystals of tin oxide showing the content of 16.98% is observed, being the most elevated together with BCN463.

Although the decorative motif is the same than in all the Black and green vessels produced in Barcelona, chemically BCN453 does not fit with any local group. It is interesting to highlight that it is the first case that a slip is identified in majolica from this century. However, it must be emphasised that Italian vessels from 13th century present a fine white layer, a slip, between the clay body and the glaze, probably to save tin oxide for opacifying and obtain a white surface for applying the decoration (Capelli, 1996, 1999a, 1999b; Capelli and Di Gangi, 2001).

Finally, it should be noted that BCN453 and BCN451, from group B1e, exhibit the highest content of lead and the lowest content of silicon oxide (Table 5). In both samples, the high amount of lead compared to the rest of the individuals could be responsible for the line formed by the development of crystals of kentrolite and bustamine (Fig. 10.4).

4. Conclusions

The results of the chemical, mineralogical and decorative study of black and green tin-glazed pottery, together with the archaeological context, suggest an evolution related to the development and improvement of the process of production. Chemically, the new samples analysed are included in group B1 but creating a new subgroup, B1e. The technological study of all the samples (from group B1 and B2) allows us to identify some differences between two groups and intragroup: samples from B1 (including subgroups B1a, B1c and B1e) shows a wide variability related to the technique of firing and glazing compared to samples from B2.

It cannot be possible to link a specific trim with a specific chemical subgroup or with a specific technique in producing the glaze. The study by SEM of B1e individuals allowed us to identify different ways of producing the decoration, a non-homogeneous process of firing (some individuals present a double firing, whilst others only a single firing), and the presence of a slip, undocumented up to now in Barcelona products, which could be related to an Italian tradition.

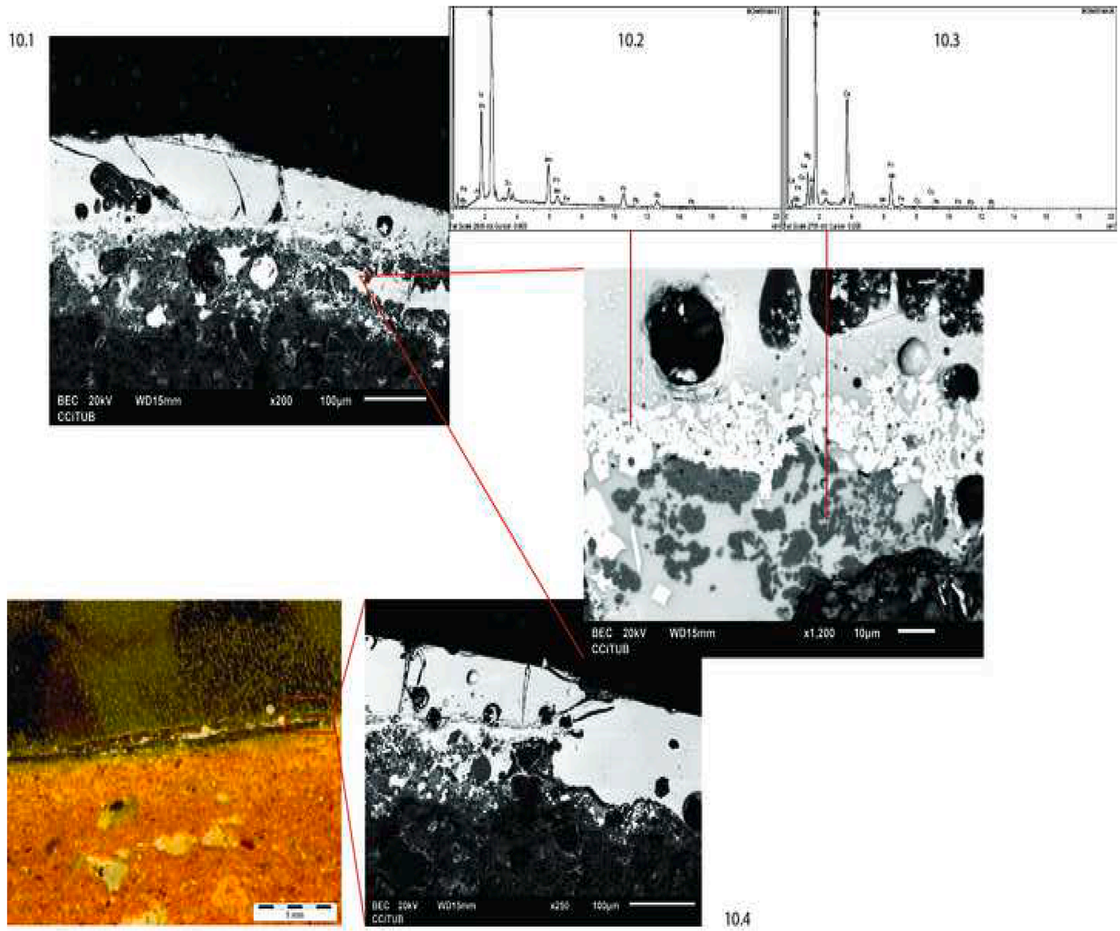

Fig. 10. 1. Crystals ok kentrolite and bustamite observed in the glaze. 2. Microanalysis of kentrolite crystals (Pb2Mn3 + 2(Si2O7)O2). 3. Microanalysis of Bustamite crystals (CaMnSiO6). 4. In the square selected area, crystals can be seen even macroscopically.

Despite these differences, the decorations and the way of applying them are the same. The decorative motifs were made with copper and manganese oxide dissolved into the glaze for all the cases observed in this study. This suggests a standardized technique in adding the pigment to the glazes. Nevertheless, the presence of crystallisation in black parts of some individuals could be related to more content of lead into the glaze, perhaps indicating a different receipt. What is clear is that through the study of decorative motifs it is impossible to assign the samples to centuries or chemical groups. All the trims studied are present in all the chemical groups, and in vessels from the 13th to the 14th century. For that reason, the results of this study show that the chemical and the mineralogical study are more relevant and suggest some interesting things.

First of all, even if group B1 was thought as a production that did not use a specific paste clay for a specific ceramic product, it has been found that B1 can be subdivided into subgroups (B1a, B1b, B1c, B1d and the new B1e) related to the use or types of vessels. In that sense, B1c would be the oldest producing black and green tin-glazed pottery, transport jars and glazed coarse ware, during a long period which covers from the end of the13th century until the end of the 14th century.

In contrast to B1c, B1a and B1e were restricted only to black and green tin-glazed pottery. In that sense, taking into consideration the mineralogical study, it must be highlighted that vessels from B1e exhibit a wide variety of range of temperatures, whilst B1a only shows one range of temperature, indicating a good knowledge and standardization of the firing process. Both groups can be dated back to the 14th century, although the better know-how of the firing technique observed for B1a suggests that would be posterior than B1e. Comparing B1a with B2 (the most modern in terms of archaeological chronology), both can be situated in the same moment and related to good knowledge in the production of these vessels.

With that, new group B1e, with a high variety in firing temperature and some differences in the production of the glaze (presenting some individuals with a single firing and other with a double firing process) and decorations, seems to suggest different potters working at the same time using the same paste but firing at different temperatures, or simply a lack of standardization and not good control of the firing process.

It is important to point out that all the vessels from group B1b are transport jars or glazed coarse ware exhibiting chronologies from the 14th / 15th centuries until the 16th century. So, even that B1 use the same clay (as it is the same chemical group), potters would use different receipts: a difference in B1a, B1b, B1c and B1d have been identified, where B1b, would be almost exclusive for glazed coarse ware and B1d would be almost exclusive for transport jars. From the 16th century onwards, a new clay would be used, restricted only to majolica: B2. Their study suggests a great control of the firing technique, being the beginning of a total specialization using specific clays for specific ceramics.

To sum up, this study allows us to identify specific pastes within group B1 related to the production of black and green tin-glazed pottery. The oldest one would be group B1c, paste with which black and green, but also transport jars and glazed coarse ware were made. Next would be the paste corresponding to subgroup B1e, which would be the starting of using a specific receipt for the exclusive preparation of tin-glazed pottery. Moreover, an increase in the standardization is observed in B1a restricted also to black and green tin-glazed products and showing good

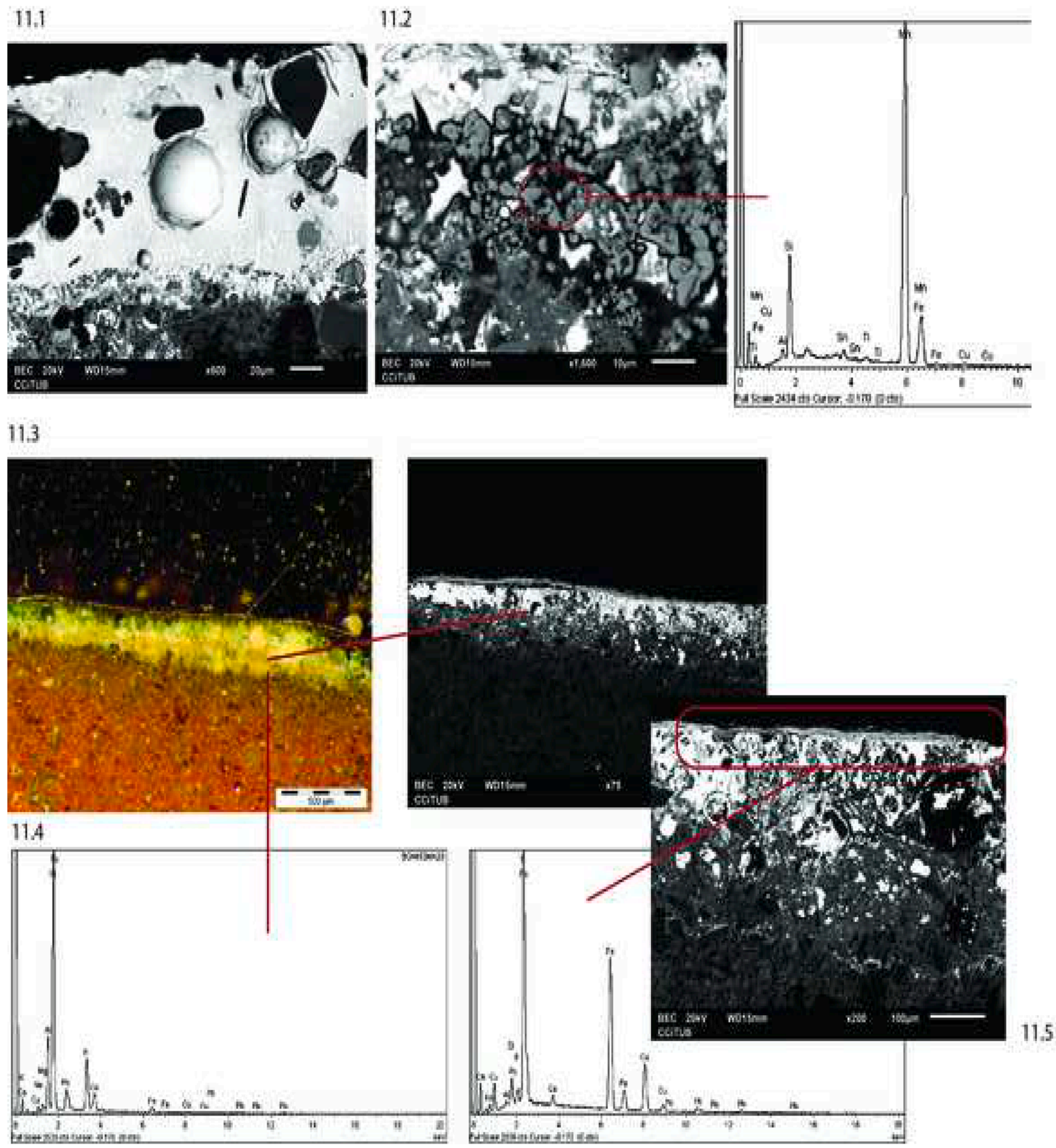

Fig. 11. 1. Accumulation of crystals of kentrolite and bustamite in the interface. 2. Grey crystals correspond to manganese (as it can be seen in the microanalysis). 3. White slip saw macroscopically and by SEM-EDX. 4. Microanalysis of the slip. 5. Grey line on the upper part of the glaze micro analysed, corresponding to the green decoration. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

control of the firing process. It should be remembered that the paste of B1b and B1d was used for producing only transport jars and glazed coarse ware. This could suggest a workshop preparing different pastes to produce different types of vessels; or different workshops using all of them the same clay, but each workshop specialized in a type of production, preparing a specific paste appropriate for each production. So, before changing the clay from B1 to B2 a standardization in B1 is shyly observed.

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