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Jacques Burlot, S.Y. Waksman, Ludovic Bellot-Gurlet, Gulsu Simsek Franci. The glaze production technology of an early Ottoman pottery (mid-14th(?)-16th century): The case of ‘Miletus Ware’. *Journal of Archaeological Science: Reports*, 2020, 29, pp.102073. 10.1016/j.jasrep.2019.102073 . hal-02424450

HAL Id: hal-02424450

<https://hal.science/hal-02424450v1>

Submitted on 7 Mar 2021

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The glaze production technology of an early Ottoman pottery (mid-14th (?) -16th century): The case of “Miletus Ware”

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Abstract

“Miletus Ware”, considered as one of the first wares produced by Ottoman potters in western Anatolia, featured new typological and stylistic characteristics that suggested the introduction of new recipes and the use of new materials in the local repertoire. This study, conducted on archaeological samples of Miletus Ware from eight sites in Turkey and in the Crimea, supports this view. It focuses on the decoration techniques, through the analyses of glazes and underglaze decorations using SEM-EDS and Raman spectroscopy. The results show that, compared to what Byzantine and Beylik potters previously produced in western Anatolia, there were two main innovations in the Miletus Ware glaze production technology. First, its glaze recipe included new sodium-based fluxes. Second, some underglaze decorations as represented by the black and dark-blue-colored ones - obtained respectively through the use of pigments featuring magnesiochromite and cobalt - were produced with materials that had not been used in the region before.

Keywords: Miletus Ware, Ottoman ceramics, Technology, Glaze, Color, SEM-EDS, Raman spectroscopy.

1. Introduction

Ottoman ceramics are mainly known for Iznik Ware, which refers to the pottery produced in Anatolia from the end of the 15th century onward, made with a stonepaste body (mixture of 70-80% quartz, 10-20% clay and 10% frit) and decorated with a varied palette of underglaze colors (Paker, 1964-1965; Tite, 1989). Although bearing the name of Iznik, this pottery was also produced in Kütahya (Şahin, 1981; Carswell, 2012), and in Istanbul during the 18th century, at the imperial workshops located in Tekfur Saray (Yenişehirlioğlu, 2003, 2009). This high-quality pottery was widely exported outside the Empire. Due to their luxurious and ‘high-tech’ nature, many examples from these productions are now exhibited in museums and have been subject to several archaeometric studies (Kiefer, 1956a, 1956b, 1956c, 1985; Henderson, 1989; Henderson and Raby, 1989; Tite, 1989; Henderson, 2001; Geçkinli *et al.*, 2002; Okyar, 2002; Colomban *et al.*, 2004; Paynter *et al.*, 2004; Colomban *et al.*, 2005; Şimşek *et al.*, 2010; Şimşek and Geçkinli, 2011; Constantinescu *et al.*, 2014; Tite *et al.*, 2016; Şimşek *et al.*, 2019a, 2019b). These studies provided in-depth

knowledge on the history of techniques of these luxury Ottoman ceramics.

However, it is important to emphasize that these stonepaste ceramics (also called fritwares, or ceramics with synthetic pastes) hardly reflected productions intended for use in daily life at this period, and do not reflect the whole Ottoman ceramic industry. In the post-medieval contexts of western Anatolian sites, the vast majority of pottery unearthed featured a clayey body, and were notably decorated with a green monochrome glaze applied on a white slip layer (Crane, 1987; François, 2001; Vroom, 2005; Böhlendorf-Arslan, 2008; Findik, 2015; Vroom and Findik, 2015). Another mass-produced ceramic excavated in these contexts corresponds to the so-called Miletus Ware. Despite its relative abundance, no extensive archaeometric studies have been carried out on this ceramic group. The Miletus Ware was considered in some of the studies previously mentioned, which were mainly devoted to Iznik fritwares and used Miletus Ware samples as comparative evidence to attempt to define the origin of the Ottoman stonepaste technology (Henderson and Raby, 1989; Tite, 1989; Okyar, 2002). In these studies, only one or two samples of Miletus Ware were analyzed.



Fig. 1. Examples of Miletus Ware samples, showing the variety of colors in the underglaze decorations (Pictures: S.Y. Waksman, I. Teslenko; CAD: J. Burlot).

Miletus Ware is one of the first ceramic types produced by Ottoman potters in western Anatolia (Lane, 1957; Erdmann, 1963; Aslanapa *et al.*, 1989). It was named by Friedrich Sarre since he found it abundantly at Miletus, a site located on the Aegean coast of Anatolia, and assumed that it was locally manufactured (Sarre, 1930-1931, 1935). This ceramic was generally produced in open forms such as plates and large bowls (Fig. 1). It may be described as reddish earthenware covered with white slip on both sides, though the external side is not fully covered. Its interior surface features painted decoration, usually in dark-blue, turquoise, purple and black, and main motifs include flower rosette or sunburst at the center and radial lines around this central motif. The painted decoration is then covered with a transparent glaze. Such underglaze painted decoration may also be used on the outer surface of Miletus Ware, but the details are less accurate since, according to Paker (1964-1965), customers paid no attention to this less visible surface. In other examples, the exterior is only covered with a monochrome glaze, which is generally green.

Miletus Ware has mainly been excavated in central and western Anatolia, but also on the Greek islands, in the Crimea, and in the Balkans; all regions that were conquered by the Ottomans (Burlot, 2017). Archaeological excavations have provided evidence of the production of Miletus Ware in four centers in Anatolia: Kütahya (Şahin, 1981), Pergamon (Mania, 2006), Akçaalan (Akarca, 1979) and Iznik considered as the major one (Aslanapa, 1969; Aslanapa *et al.*, 1989; Özkul Fındık, 2007). Discoveries in Miletus (Gök Gürhan, 2010, 2011) and in Aphrodisias (François, 2001) also suggest the local production of Miletus Ware, but further investigations are required to confirm these hypotheses.

The start date of Miletus Ware remains unclear. It is commonly acknowledged that Miletus Ware spread widely from the second half of the 14th century onward (Sarre, 1935; Aslanapa, 1965, 1971; Aslanapa *et al.*, 1989), but no secure historical or archaeological evidence have been proposed to confirm this chronology. In fact, very few archaeological sites, for which the excavation results have been published, indicate well stratified contexts enabling us to date the use of Miletus Ware. At Saraçhane in Istanbul (Turkey), as well as in Sofia (Bulgaria), fragments of Miletus Ware were discovered in contexts dated from the mid-15th century to around 1520 (Saraçhane: Hayes, 1992; Sofia: Guionova, 2015); and in the Crimea, they were found in stratified layers mostly dated from the second half of the 15th century, but ranging up to the end

of the 16th century (Teslenko, 2007), which is the latest date known for these ceramics.

2. Questions and corpus

Although the data on the beginnings of Miletus Ware are still fragmentary, the present study can confirm that the appearance of this pottery in western Anatolia coincides with the settlement of Turkish populations in a former Byzantine region. Hence, the production of Miletus Ware marks the introduction of a new ceramic type in western Anatolia, related to new culture and potentially to new technologies. By analyzing the Miletus Ware decorations, we aim, first, to define the glaze recipes and manufacturing techniques used by early Turkish ceramic production centers in western Anatolia. Second, based on these definitions, we examine whether the recipes and techniques reveal new technological features, and how the latter fit into the diachronic ceramic technological landscape of Anatolia. This paper focuses on glazes technology, the analyses of bodies and slips are summarized below and detailed elsewhere (Burlot and Waksman, forthcoming a, forthcoming b).

We analyzed the glazes and their coloring agents applied on the inner surface of 27 Miletus Wares sherds excavated from five Turkish sites (Ephesus, Miletus, Pergamon, Sardis, and Iznik) and three sites located in the Crimea that provide well stratified contexts dating from the second half of the 15th to the third quarter of the 16th century (Table 1).

Samples were selected within three productions previously defined using WD-XRF analyses of the bodies (Burlot and Waksman, forthcoming a, forthcoming b; see e.g. Waksman *et al.*, 2017: 667 for details of the analytical and statistical procedures). They correspond to two production centers located in Iznik and Pergamon, respectively (Table 1: "MW Iznik" and "MW Pergamon Rote Halle"). The third group is related to another production center, whose location has not been identified yet (Table 1: "MW unlocated").

With the exception of two samples, all the slips were of synthetic type (with the main contents as follows: 82-92 wt% SiO₂, 3-9 wt% Al₂O₃, 2-4 wt% Na₂O, and 0.5-3 wt% CaO), featuring the same characteristics as those usually associated to stonepastes (Mason and Tite, 1994; Tite *et al.*, 2011). Only two samples (BZN124 and BZN125, featuring respectively 61.3 and 70.6 wt% SiO₂, 25.1 and 20.1 wt% Al₂O₃, 6.4 and 2.7 wt% Na₂O, and 0.6 and 0.7 wt% CaO) presented a slip of clayey type.

Table 1. List of the Miletus Ware samples, presented by production (*dates based on the archaeological context of discovery, when available; a: Teslenko, 2007, b: Foss, 1976).

Sample ID	Provenance	Period *	Color of the areas analyzed
Production: MW Iznik			
BYZ284	Iznik		Dark Blue; Black
BZN205	Alushta	Late 15th-early 16th century (a)	Dark Blue
BZN206	Alushta		Purple; Dark Blue
BZN209	Alushta	Late 15th-early 16th century (a)	Purple
BZN210	Alushta		Purple; Dark Purple; Dark Blue
BZN221	Funa	Late 15th-early 16th century (a)	Dark Blue
BZN222	Funa	Late 15th-early 16th century (a)	Dark Blue; Black
BZN223	Funa		Dark Blue; Black
BZN228	Balaklava		Dark Blue; Black
BZN229	Balaklava		Black
BZN231	Balaklava		Dark Blue
BZN233	Balaklava		Black
Production: MW unlocated			
BZN 43	Miletus		Dark Blue
BZN 44	Miletus		Purple; Dark Blue
BZN 45	Miletus		Dark Blue
BZN 46	Miletus		Dark Blue
BZN 49	Miletus		Turquoise Blue
BZN 50	Miletus		Dark Blue; Turquoise Blue
BZN118	Sardis	Late 14th-16th century (b)	Dark Blue
BZN119	Sardis	Late 14th-16th century (b)	Dark Blue
BZN285	Sardis	Late 14th-16th century (b)	Dark Blue; Turquoise Blue
BZN286	Sardis	Late 14th-16th century (b)	Dark Blue; Black
BZN287	Sardis	Late 14th-16th century (b)	Purple; Dark Blue
BZN288	Ephesus		Dark Blue; Black
BZN289	Ephesus		Dark Blue
Production: MW Pergamon Rote Halle			
BZN124	Pergamon		Dark Blue
BZN125	Pergamon		Purple

3. Analytical methods

To analyze the ceramic glazes and coloring agents, samples were embedded in a polyester resin, cut in a cross section in order to expose all the layers (Fig. 2), polished up to a 0.25 μm diamond paste and then carbon coated for SEM-EDS analyses. Prior to coating with carbon, the cross-sections were observed under a binocular microscope with magnifications ranging from 10x to 115x.

3.1. Scanning electron microscopy energy dispersive spectrometry

The glazes analyses were carried out with a SEM (FEI Quanta FEG at the Technological Centre for Microstructures (CT μ) in Lyon. The study of the microstructures was mainly performed using backscattered electron (BSE) images which allow the differentiation of the various phases according to the

atomic numbers of their elementary content. Elemental compositions were determined by energy-dispersive X-ray spectrometry (EDS) using a silicon drift detector 30 mm² SAMx. All measurements were operated at 15 kV acceleration voltage in 60 s. Standardless quantification was performed using a PAP correction method of the intensities. IDFix software was used for data acquisition and evaluation, and the reliability of the results was tested by measuring reference glasses and geological standards (Corning Brill B, C and D, DR-N). Concentrations are given as average of three to five measurements on different zones selected within the regions of interest of the vitreous part, avoiding the weathered areas and the slip or body interfaces.

Crystalline inclusions present in the glazes were also analyzed. For some of the smaller ones, analyses were carried out with the lower beam voltage of 10 kV instead of 15 kV, in order to reduce the interaction zone and thus the influence of the surrounding glaze. Analyses by Raman spectroscopy were carried out in order to help to identify the mineralogy of the inclusions, especially the pigments.

3.2. Raman spectroscopy

Analyses by Raman spectroscopy were performed at the laboratory MONARIS (UMR8233) in Paris and at the Koç University Surface Science and Technology Centre (KUYTAM) in Istanbul. They were all carried out on the same cross-sections already analyzed by SEM-EDS. For this reason, a first batch of samples were re-polished to remove the carbon coating, but as it also removed some micro-inclusions of interest, we analyzed the second batch without re-polishing. The very fine carbon coating deposited did not interfere with the identification of the inclusions under the microscope, nor the Raman measurements.

At MONARIS, analyses were performed with a Jobin-Yvon Horiba HR800 spectrometer, using a 514 nm emission of a Coherent (I-90C-6) ionized argon laser, modulated at a power of 600 μW on the sample. Using this power, no modification of the samples was observed, and no evolution of the spectra under the irradiation was noted. Rayleigh rejection is achieved thanks to ultra-narrow band Notch BraggGrate filters. A 600 lines/mm grating was used, which enabled us to obtain a broad spectral window for each acquisition, with a spectral resolution of about 2 cm^{-1} . The scattered light was recorded with a Peltier-cooled CCD detector. The 60 s acquisitions were driven by LabSpec software (Jobin-Yvon Horiba). The spectrometer was coupled to an Olympus microscope equipped with 50x and 100x objectives, which enable us to precisely target the areas to be analyzed. The approximate analysis diameter with these objectives was respectively 2 and 1 μm .

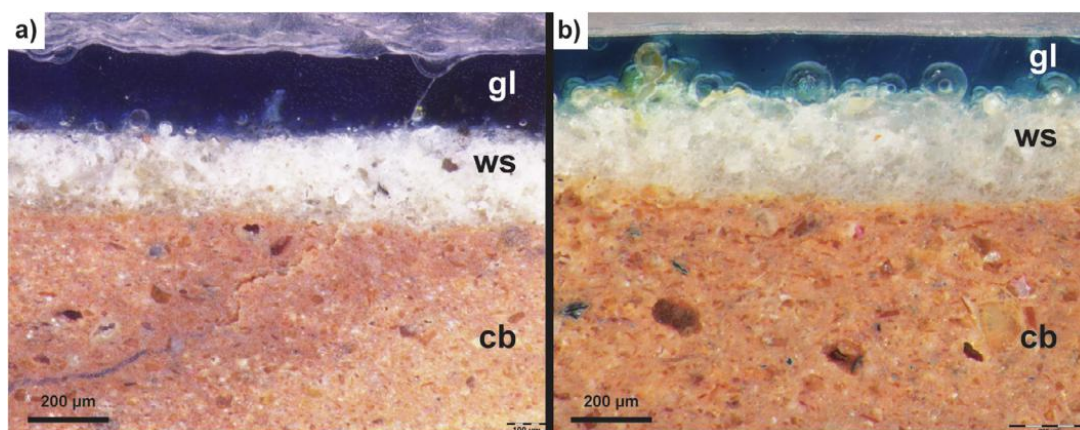


Fig. 2. Pictures of examples of Miletus Ware' cross-sections: a) BZN205 - Dark Blue ("MW Iznik"); b) BZN285 - Turquoise ("MW unlocated") (gl: glaze; ws: white slip; cb: ceramic body) (Pictures: J. Burlot).

At KUYTAM, a Renishaw inVia Raman spectrometer equipped with a 532 nm (green) diode pumped solid state laser was used to identify the coloring agents and inclusions found in the glaze. A Leica objective with a magnification of 50x (NA: 0.75) was used for the spot analyses. The recording times ranged between 10 and 20 accumulations per second. A 2400 lines/mm grating was used for a higher spectral resolution. The laser power was kept 10% (~5 mW). Any blackening on the analyzed area and/or modification of the Raman spectrum was observed during the measurements. All the spectra were recorded with the WIRE 4.0 software and the baseline correction was done with LabSpec Version 5.

4. Results

4.1. Chemical compositions of the glazes

According to their chemical compositions (Table 2), all glazes are of the lead-alkali type, and more precisely of lead-soda type (6.2-33.3 wt% PbO; 5.4-17.3 wt% Na₂O; 0.4-2.3 wt% K₂O). With the exception of four marginal samples, they all match within a main cluster when comparing their PbO contents to their alkali ones (Na₂O+K₂O) (Fig. 3). The main cluster - alkali rich lead-alkali glaze (6.2-21.6 wt% PbO, 10.0-18.5 wt% Na₂O+K₂O) - is only composed of glazes applied on internal surfaces of Miletus Ware belonging to the Iznik and unlocated productions.

Two of the marginal glazes (BZN124 - dark blue and BZN125 - purple) correspond to the ones applied on the two samples belonging to the Pergamon production (Fig. 3: yellow squared symbols). These glazes, with alkali contents similar to those of the glazes of the main cluster, differ by their higher lead content (20.6-26.5 wt% PbO). The third marginal glaze, which features a high lead content (33.3 wt% PbO) and also the highest lime content (2.8 wt% CaO), corresponds to the one applied on the sample BZN 46 from the unlocated production. The last marginal glaze is the dark blue one applied on sample BZN289 from the unlocated production, that differs from the glazes of the main cluster by much lower alkali contents. This may be explained by the heterogeneity of the latter, featuring a lot of inclusions, the composition then being influenced by different microstructures. Among these are the coloring agents whose cumulative contents in this glaze is equivalent to 16.4 wt%.

All the glazes are transparent, neither opacifier such as cassiterite inclusions, nor bubbles were observed. However, other kinds of microstructures are present, especially in purple, black and blue glazes. The majority of these inclusions correspond to the coloring agents (see infra). The colorless glazes and the turquoise ones in which the colorants are totally dissolved, are practically devoid of crystalline inclusions.

Despite two marginal samples (BZN 46 and BZN289) and fairly large ranges of variation, the glazes of the Miletus Wares samples belonging to the group "MW Iznik" do not seem to differ significantly from those of the group "MW unlocated", while the two glazes of the samples from Pergamon differ from the others by higher concentrations of cumulated fluxes (36.4-40.8 wt% PbO + Na₂O + K₂O, compared to a maximum of 34.0 wt% in the other two productions, with the exception of the glaze BZN 46 - dark blue: 41.4 wt%).

4.2. Colors of the underglaze painted decorations

Among the samples of the present study, the chromatic panel of the painted decoration of Miletus Wares is relatively wide. It varies from turquoise to purple, dark blue and black. For three samples (BZN 44, BZN206 and BZN287), the glazes we analyzed correspond to areas where the blue and purple colors are juxtaposed (Table 2: marked with an asterisk). We will discuss these samples both with purple and dark blue underglaze decors because they show features of each of these two colors.

4.2.1. Copper Turquoise Blue

The turquoise areas feature the highest copper oxide contents, all higher than 2.4 wt% CuO (Table 2). This confirms what was already well known, that copper-based minerals in an alkali-based glaze generates a turquoise blue color. The copper-based colorants were fully dissolved in the glaze during the firing since no copper-rich crystalline inclusion was observed, unlike the colorants corresponding to the purple, dark blue and black colors.

4.2.2. Manganese Purple

In the purple areas, manganese-based minerals served as colorants, since the manganese contents quantified in these decors are the highest ones (≥ 1.8 wt% MnO). Manganese-rich inclusions were observed in purple areas, located at the interface with the slips (Fig. 4). Their chemical compositions show very high contents of manganese (≥ 63.2 wt% MnO), as well as significant ones in silicon, iron, lead and sodium; the latter two probably mainly belonging to the surrounding glaze (Table 3). Some of these inclusions were analyzed by Raman spectroscopy, whose results enabled us to identify different crystalline phases.

Fig. 5 shows Raman spectra with bands that seem to be characteristic of a braunite signature (like a Mn²⁺Mn³⁺₆SiO₁₂ based structure), as well as a band characteristic of hausmannite (like a Mn²⁺Mn³⁺₂O₄ based structure) at 659 cm⁻¹ (Coentro *et al.*, 2018, Fig. 11). In some inclusions, these two mineral phases occur simultaneously (Fig. 5, spectrum 1), while in others, braunite is associated with other unidentified crystalline phases (Fig. 5, spectrum 2). Hausmannite can result from firing pyrolusite (MnO₂), a common manganese ore, at ca. 1000 °C (Coentro *et al.*, 2018), while braunite may form during firing by reaction between manganese oxides and the silicon-rich slip matrix at temperatures above 1000 °C (Pradell *et al.*, 2012; Molera *et al.*, 2013). According to Coentro and colleagues (2018: 8), "the coexistence [of braunite] with hausmannite may indicate an incomplete transformation to braunite, which may be due to either the temperature being barely above 1000 °C or to a not long enough firing time." Braunite inclusions have already been documented in manganese brown decorations of 10th-18th century Spanish ceramics (Molera *et al.*, 2013), 15th-16th century Hispano-Moresque tiles (Coentro *et al.*, 2018), and 17th century Portuguese tiles (Coentro *et al.*, 2012). Another type of Mn-rich inclusion identified is jacobsonite (Fig. 5, spectrum 3), a spinel based on a MnFe₂O₄ structure that can be formed by the combination of manganese and iron oxides during firing (Molera *et al.*, 2013).

4.2.3. Cobalt Blue

One of the features of the blue glazes is the presence of cobalt quantified in their chemical composition with contents between 0.4 and 2.2 wt% CoO (Table 2). What also differentiates their compositions are the higher contents in iron and copper oxides, and the detection of nickel oxide. This Co-Ni-Fe-Cu association was also observed in dark blue glazes of a ceramic sample similar to the Miletus Ware discovered in Prilep (Macedonia) (Tanevska *et al.*, 2009), and in the earliest Iznik fritware dating to the late 15th-early 16th centuries (Henderson, 1989).

A large number of inclusions was analyzed in the blue glazes (Fig. 6). The combination of SEM-EDS (chemical compositions and microstructures) and Raman data enabled us to identify at least three categories of inclusions. Two of them correspond to cobalt-iron (Fig. 7, spectrum 1) and nickel-iron (Fig. 7, spectrum 2) based-spinels based respectively on a CoFe₂O₄ and NiFe₂O₄ like structure (Wang *et al.*, 2015), which correlates well with the glaze's chemical compositions.

Table 2. Semi-quantitative SEM-EDS analysis of the glazes, in wt%. Samples are presented by production (*both dark blue and purple colors merged on the same sample; **Monochrome Turquoise Glazed Wares belonging to two early Turkish (Beylik) western Anatolian productions (data from Burlot, 2017); -: below detection limits; m: mean; σ : standard deviation).

Sample ID - Color of the area analyzed	SiO ₂	PbO	Na ₂ O	K ₂ O	MgO	CaO	Al ₂ O ₃	Fe ₂ O ₃	MnO	CuO	CoO	NiO	Cr ₂ O ₃	SnO ₂
Production: MW Iznik														
BYZ284 - Dark Blue	62.1	14.5	12.4	1.6	0.6	1.0	2.1	2.7	–	0.5	0.6	0.4	0.3	–
BYZ284 - Colorless	63.4	14.7	13.6	1.5	0.6	1.1	2.1	0.6	–	0.4	–	–	–	0.3
BYZ284 - Black	61.3	13.3	13.0	1.3	1.1	1.0	2.5	1.5	0.5	0.6	–	–	2.6	–
BZN205 - Dark Blue	61.4	15.4	14.8	1.2	0.5	0.8	1.6	2.3	–	0.4	0.4	0.3	–	–
BZN206 - Dark Blue & Purple*	57.6	18.0	10.1	1.5	0.4	0.8	2.4	4.9	2.2	0.5	0.6	0.6	–	–
BZN209 - Purple	57.7	15.3	12.5	2.0	0.7	1.0	5.0	0.9	3.7	–	–	–	–	–
BZN209 - Colorless	62.0	13.8	12.3	2.3	0.8	0.8	6.7	0.3	–	–	–	–	–	–
BZN210 - Purple	62.9	13.3	14.8	1.4	0.5	0.8	2.1	0.6	1.8	0.3	–	–	–	–
BZN210 - Dark Purple	62.8	13.9	14.1	1.6	0.4	0.8	1.7	0.8	2.3	–	–	–	–	0.4
BZN210 - Dark Blue	64.0	12.6	13.5	1.7	0.4	0.6	1.9	3.1	–	0.3	0.5	0.4	–	–
BZN210 - Colorless	63.2	16.1	13.9	1.4	0.5	0.8	1.9	0.5	–	–	–	–	–	–
BZN221 - Dark Blue	65.6	11.5	13.3	1.5	0.4	1.0	1.5	2.0	–	0.7	0.7	0.5	–	–
BZN222 - Black	62.3	12.8	11.5	2.0	0.9	0.8	2.7	4.1	–	0.4	0.4	–	1.5	–
BZN222 - Dark Blue	61.1	16.0	9.4	2.0	0.4	1.1	1.9	4.4	–	1.2	1.2	0.7	–	–
BZN223 - Black	54.4	13.8	10.9	1.5	2.2	1.1	4.0	3.6	0.6	0.8	0.5	0.3	6.0	–
BZN223 - Dark Blue	61.1	17.4	10.8	1.8	0.7	1.1	2.0	2.6	–	0.5	0.6	0.5	–	–
BZN223 - Colorless	61.1	21.6	10.9	1.5	0.6	1.0	1.9	–	–	0.4	–	–	–	–
BZN228 - Dark Blue	61.8	11.0	16.3	1.6	0.7	1.1	2.5	2.2	–	0.5	0.6	0.6	–	–
BZN228 - Black	50.4	7.9	13.3	1.4	3.9	1.5	4.8	5.4	–	0.8	0.8	0.9	7.7	–
BZN229 - Black	63.7	10.7	14.6	1.4	1.0	1.5	2.1	1.8	–	0.4	–	0.3	2.0	–
BZN231 - Dark Blue	58.0	13.5	11.3	1.7	0.7	1.4	2.6	5.1	–	1.1	2.0	1.6	–	–
BZN233 - Black	53.5	11.8	11.6	0.9	2.5	1.5	4.4	4.2	0.6	1.4	0.3	0.3	6.4	–
Production: MW unlocated														
BZN 43 - Dark Blue	66.4	14.1	12.9	1.0	0.5	0.8	1.4	1.2	–	0.5	0.3	0.3	–	–
BZN 44 - Dark Blue & Purple*	55.9	7.6	15.3	1.3	0.8	1.1	3.2	8.6	2.1	0.5	1.1	1.1	–	–
BZN 45 - Dark Blue	59.9	10.8	14.5	1.1	0.5	1.6	1.9	5.3	–	1.4	1.4	1.1	–	–
BZN 46 - Dark Blue	50.1	33.3	7.7	0.4	0.6	2.8	2.0	1.0	–	0.5	0.3	0.4	–	–
BZN 49 - Turquoise Blue	56.9	20.9	10.4	0.9	1.0	1.9	2.5	0.8	–	3.9	–	–	–	–
BZN 50 - Dark Blue	61.1	20.2	11.1	1.0	0.4	1.4	1.4	1.1	0.3	0.7	0.4	0.6	–	–
BZN 50 - Turquoise Blue	60.8	21.1	8.8	1.2	0.5	1.1	2.5	0.4	–	2.4	–	0.4	–	–
BZN118 - Dark Blue	59.8	9.3	17.3	1.3	0.7	0.8	2.0	5.5	–	0.6	0.7	0.8	–	–
BZN119 - Dark Blue	61.2	10.7	15.9	1.4	0.9	0.7	2.1	2.7	–	0.5	0.4	0.4	0.3	–
BZN285 - Dark blue	65.0	12.3	11.3	1.2	–	0.9	1.4	4.1	–	0.8	1.0	0.9	–	–
BZN285 - Turquoise Blue	62.5	15.3	12.3	1.1	0.7	1.6	1.5	0.6	–	3.7	–	–	–	–
BZN286 - Dark Blue	69.4	8.8	13.1	1.2	–	0.7	1.0	3.2	–	0.7	0.7	0.6	–	–
BZN286 - Black	60.2	6.2	13.7	1.2	2.5	0.3	3.9	2.9	0.3	0.4	0.3	0.3	7.8	–
BZN287 - Dark Blue & Purple*	55.8	14.1	14.1	0.9	0.3	0.9	1.6	5.3	1.8	1.2	1.3	2.2	–	–
BZN288 - Black	59.1	8.1	15.2	1.0	1.9	0.7	3.7	3.4	0.6	0.3	0.3	0.3	4.8	–
BZN288 - Colorless	65.1	12.7	16.2	0.9	0.6	1.2	1.7	–	–	0.5	–	–	–	–
BZN288 - Dark Blue	58.9	9.9	16.1	0.9	0.6	1.0	1.8	6.9	–	0.8	1.3	1.0	–	–
BZN289 - Dark Blue	61.1	10.2	5.4	1.4	1.2	2.3	1.9	9.9	–	2.5	2.2	1.8	–	–
Production: MW Pergamon Rote Halle														
BZN124 - Dark Blue	49.8	26.5	13.0	1.2	1.4	2.3	1.8	1.2	–	0.8	0.4	0.6	–	–
BZN125 - Purple	52.3	20.6	14.5	1.2	0.4	0.8	2.2	0.5	6.0	0.3	–	–	–	–
Monochrome Turquoise Glazed Ware**														
Production: Ephesus region (b/2)														
m (n = 6)	43.5	33.2	4.8	1.4	1.1	2.1	2.2	0.6	–	1.5	–	–	–	8.1
σ	4.8	9.6	3.6	0.7	0.4	0.7	0.6	0.3	–	0.4	–	–	–	1.8
Production: "Pergame F"														
m (n = 5)	44.7	32.4	3.4	1.6	1.4	2.9	2.9	0.6	–	1.3	–	–	–	7.4
σ	2.2	3.7	1.5	0.6	0.4	0.3	1.2	0.2	–	0.3	–	–	–	3.0

Similar inclusions were observed in 9th century Samarra faience blue glazes (Kleinmann, 1991), and in 15th-16th century Hispano-Moresque tiles (Coentro *et al.*, 2014, 2018). Hematite inclusions were also identified (Fig. 7, spectrum 3) (Bersani *et al.*, 1999; Colomban *et al.*, 2004).

Because of the rarity of these spinel-like mineral phases in nature, it is more likely that they are the result of the preparation of pigments by potters, including roasting, or that they are formed by interaction with the components of the vitreous matrix during firing. The inclusion presented in Fig. 6a-b seems to illustrate this hypothesis. Several facies are indeed visible in this inclusion, in particular its core appears lighter under optical microscope (Fig. 6b). Raman analyses carried out in this area revealed hematite, while those carried out at the periphery of this same inclusion showed nickel-ferrite based structure signatures.

4.2.4. Chromium Black

In the black decorations, the coloring oxide is that of chromium, quantified at contents higher than 1.5 wt% Cr₂O₃ (Table 2). The magnesium oxide contents are also significant, with percentages higher than 1 wt% MgO. For some of the black glazes, iron, cobalt, nickel and copper oxides are also detected. In these cases, the presence of these elements is probably a contribution from the glaze adjacent to the black decoration which is of a cobalt blue type; a phenomenon already observed and commented with glazes of Iznik fritwares (Tite, 1989; Colomban *et al.*, 2005), and also with glazes of 12th-14th century Islamic ceramics from the province of Nimrôz (Afghanistan) (Pernicka and Malissa, 1980).

With the Miletus Wares, the black motifs are represented by fine straight or curved lines which did not diffuse within the glaze, contrarily to the dissolved colorants used for the blue and purple colors.

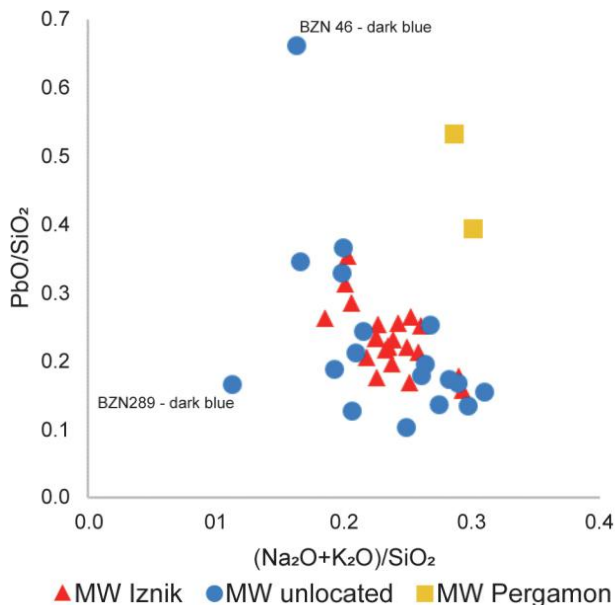


Fig. 3. Binary diagram showing PbO/SiO₂ vs. (Na₂O+K₂O)/SiO₂ ratios in the glazes.

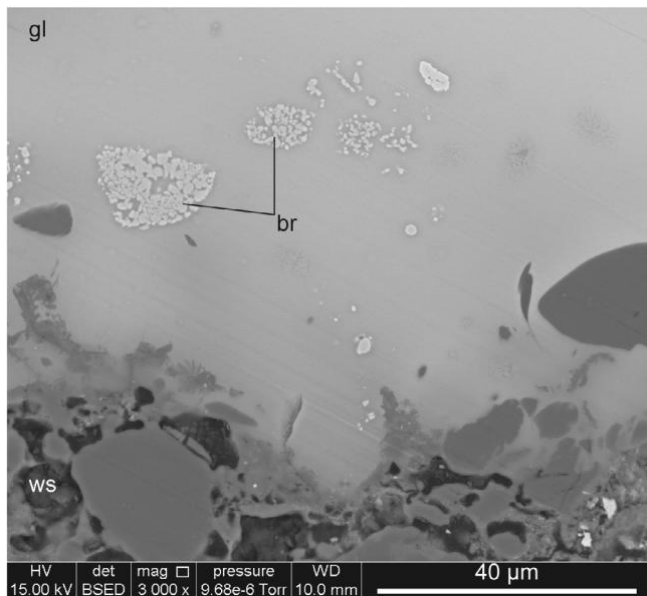


Fig. 4. Braunite inclusions in the purple glaze of sample BZN206 (“MW Iznik”), observed with a SEM in BSE mode (gl: glaze; ws: white slip; br: braunite).

We observed very characteristic inclusions in these black “lines” (Fig. 8). They are located either at the interface between the slip and ceramic body, or in the entire thickness of the glaze. Their very angular shape testifies to a crushing of the pigment used to paint the decoration and the preservation of its integrity during firing.

The chemical compositions of these inclusions indicate chromium contents higher than 48.8 wt% Cr₂O₃, as well as significant aluminum, magnesium and iron contents (approximately 16 wt% Al₂O₃, 13 wt% MgO, and ≥4.5 wt% Fe₂O₃) (Table 4). Although in varying proportions, some inclusions also contain relatively significant amounts of copper, manganese, cobalt and nickel, issued probably from the adjacent painted decorations (Pernicka and Malissa, 1980; Tite, 1989; Degli Agosti and Schweizer, 2001).

The Raman spectroscopic analyses carried out on these inclusions show major bands at 566 and 701 cm⁻¹, with a shoulder on the latter at 666 cm⁻¹ (Fig. 9). These Raman signatures could correspond to magnesiochromite, which

would be consistent with the high chromium and magnesium contents. Thus, to paint the fine black motifs, potters used pigments likely based on crushed magnesiochromite, the grains of which did not dissolve in the vitreous matrix of the glazes. These grains are also visible under the binocular microscope, unlike the pigments used for the other colors.

Chromite inclusions have already been observed in glazes of western Anatolian ceramics, notably in Iznik fritwares (Henderson, 1989; Tite, 1989; Paynter *et al.*, 2004; Şimşek *et al.*, 2010, 2019a). In western Anatolia, mines of magnesiochromite are known in the provinces of Eskişehir, Bursa, Denizli, and Kütahya; the analysis of which could be a starting point for future provenance studies of the pigments.

5. Discussion

5.1. Comparison with other Turkish productions

By comparing the glazes of the Miletus Wares with the previous analyses of other western Anatolian pottery, we observe that the recipes used to produce the Miletus Wares are new in the region. The technical tradition hitherto defined for the glazes of the Late Byzantine and early Turkish (Beylik: 14th-early 15th century) ceramic productions in Western Anatolia was that of transparent high-lead glazes (Burlot, 2017; Burlot *et al.*, 2018). Among the Beylik ceramics in this region, one type was an exception: the Monochrome Turquoise Glazed Ware. Archaeometric studies on this ceramic defined the turquoise monochrome glazes as lead-alkali and tin-opacified (Table 1). The tin opacification and the much higher Pb/(Na₂O + K₂O) ratio (Fig. 10a) still clearly differentiate the recipe of this glaze from the one of the Miletus Wares.

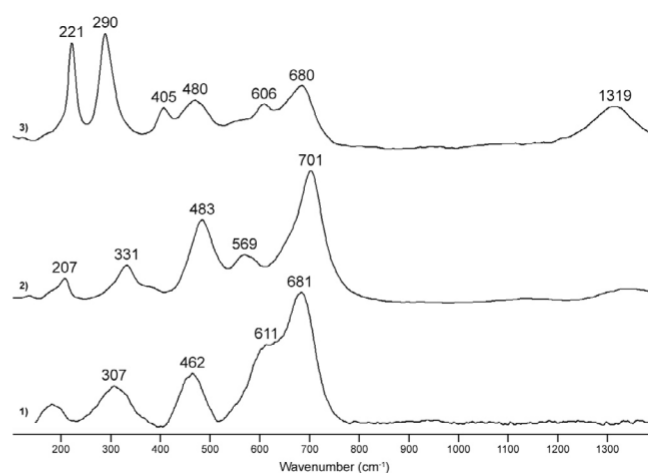
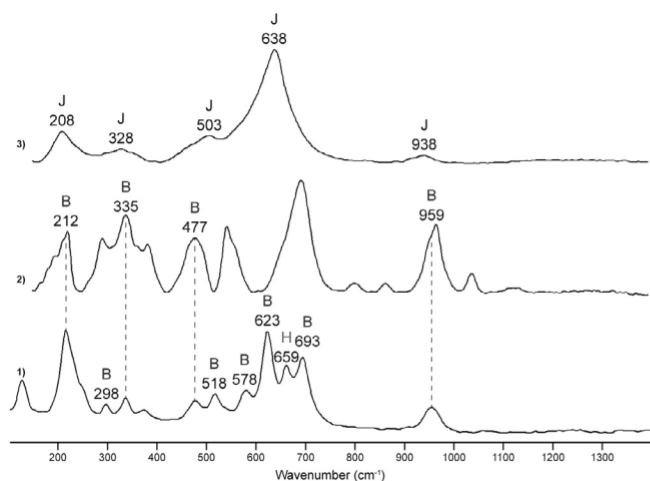
Other earlier Turkish ceramics include the Seljuk productions from central and eastern Anatolia, generally dated to the 12th-13th centuries. The wares that have been more studied are the tiles with synthetic bodies used to decorate monuments; and the best known are those of the Kubad-Âbâd palace located near the Beyşehir Lake, dating from the first half of the 13th century, which corresponds to the date of construction of the palace (Yeğingil and Freestone, 2008; Freestone *et al.*, 2009). Tiles from other Seljuk monuments in Sivas, Tokat and Konya were also studied (Kiefer, 1956a, 1956b, 1956c; Henderson and Raby, 1989; Demirci *et al.*, 2004; Colomban *et al.*, 2006), but the data about their contexts of discovery, their chronology, and their chemical compositions are relatively incomplete, which means it is not possible to propose a “solid” Seljuk referential, nor to directly compare the glaze compositions. Nevertheless, these studies propose that the glaze technologies of the Seljuk pottery - which are characterized by the use of soda-lime and alkali fluxes for transparent glazes, and lead-alkali fluxes and tin-oxide opacifiers for monochrome turquoise glazes - were different from those of Miletus Ware.

A corpus of the so-called “Seljuk” clayey-body ceramics found at Korucutepe has also been studied (Demirci *et al.*, 2004). According to the researchers, these ceramics featured a lead-alkali glaze, but no more detailed information about their chemical composition and no archaeological documentation about this corpus were provided.

Finally, the lead-alkali glazes applied to the internal surfaces of Miletus Ware are more similar to the glaze recipes of the Iznik fritware, since the latter are also of soda-lead type and also feature a low amount of CaO (< 3 wt%). However, there is a major difference between them, namely the presence of tin oxides. No tin oxide has been detected in the glazes of Miletus Ware, while SnO₂ contents of around 4 to 8 wt% were measured in the glazes of the first Iznik fritware (Henderson, 1989; Tite, 1989; Okyar, 2002; Paynter *et al.*, 2004).

Table 3. Chemical compositions of manganese-rich inclusions identified in several purple decors (wt%, SEM-EDS; -: below detection limits). Samples are presented by production.

Sample ID	SiO ₂	PbO	Na ₂ O	K ₂ O	CaO	Al ₂ O ₃	Fe ₂ O ₃	MnO	CuO	CoO	NiO
Production: MW Iznik											
BZN206-01	20.5	2.6	1.3	0.4	0.5	1.1	2.8	67.6	0.8	0.9	0.9
BZN206-03	13.1	1.0	0.4	–	0.5	0.4	3.2	78.5	0.7	1.0	0.6
BZN210-02	23.0	2.8	5.0	0.4	0.5	1.6	2.2	63.2	–	–	0.4
BZN210-03	2.4	0.4	0.7	–	–	1.7	4.0	89.1	–	–	–
Production: MW unlocated											
BZN 44-03	14.9	2.5	3.3	0.4	0.7	1.4	2.4	67.7	2.2	0.8	1.3
BZN 44-04	11.3	2.0	1.0	–	0.3	1.1	1.9	77.1	2.6	0.5	0.6
BZN287-03	1.7	0.4	–	–	–	–	1.5	94.3	1.4	–	–
BZN287-07	22.4	2.2	1.8	–	0.7	2.2	1.7	65.9	1.5	0.3	0.5
BZN287-09	8.4	2.4	0.6	1.2	0.4	0.3	2.3	78.3	2.9	1.6	0.4
Production: MW Pergamon Rote Halle											
BZN125-01	11.4	0.8	0.4	–	0.6	0.6	2.9	81.0	0.4	–	–
BZN125-02	13.9	2.2	1.4	–	0.6	0.8	2.9	76.2	0.4	–	–
BZN125-04	14.2	1.7	2.1	–	0.9	0.4	2.0	76.4	–	–	–



- Fig. 5. Raman spectra of manganese-rich inclusions identified in several purple areas: 1) BZN206-03 (“MW Iznik”; laser: 514 nm; 100x objective); 2) BZN221 (“MW Iznik”; laser: 532 nm; 50x objective) (identification based on Coentro *et al.*, 2018, Fig. 11; B: braunite; H: hausmannite); 3) BZN125 (“MW Pergamon Rote Halle”; laser: 532 nm; 50x objective) (identification based on reference spectrum: RRUFF ID R060780).

- Fig. 7. Raman spectra of Co-Fe-Ni-rich inclusions identified in several dark blue decors: 1) BZN287 (“MW unlocated; laser: 532 nm; 50x objective) (cobalt-ferrite based structure’ identification based on Wang *et al.*, 2015, Fig. 3a); 2) BZN 44 (“MW unlocated”; laser: 514 nm; 100x objective) (nickel-ferrite based structure’ identification based on Wang *et al.*, 2015, Fig. 3b); 3) BZN118 (“MW unlocated”; laser: 514 nm; 100x objective) (hematite’ identification based on Bersani *et al.*, 1999, Table 1).

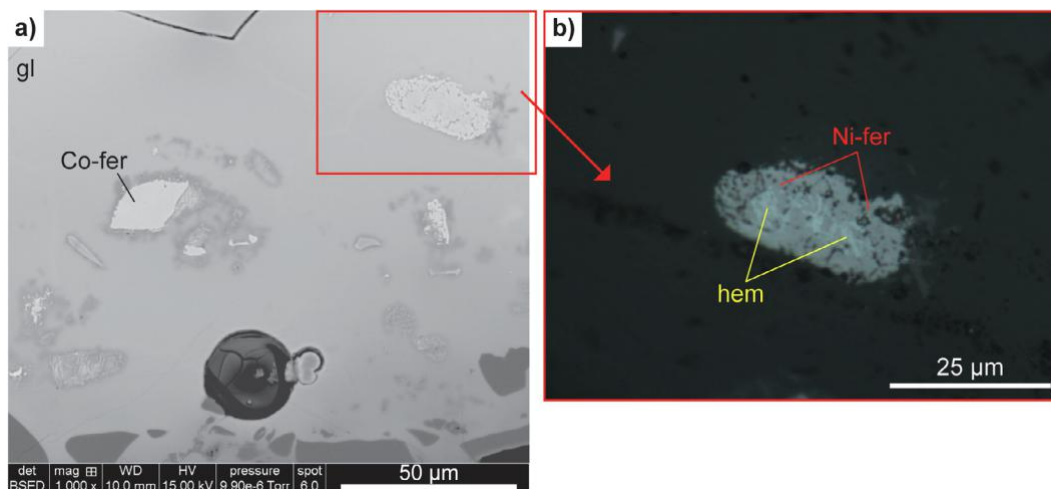


Fig. 6. Co-Fe-Ni-rich inclusions observed in a dark blue decor of sample BZN231 (“MW Iznik”): a) BSE image; b) optical image recorded during a Raman analysis (gl: glaze; hem: hematite; Co-fer: cobalt-ferrite like structure; Ni-fer: nickel-ferrite like structure).

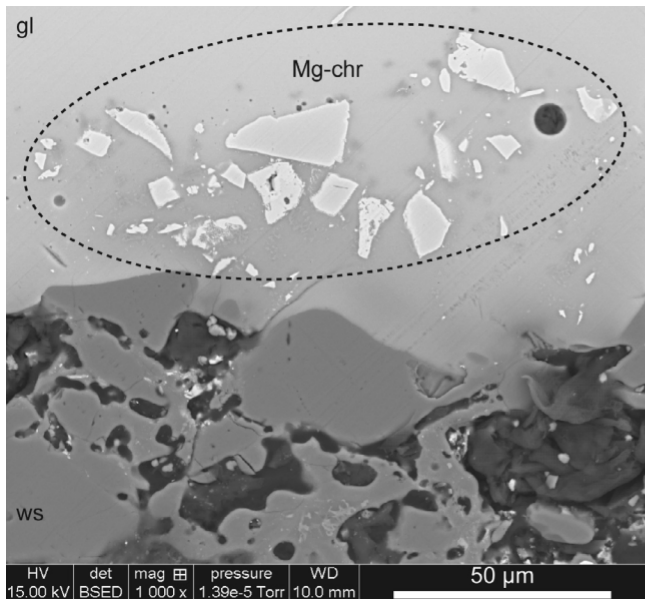


Fig. 8. Chromium-rich inclusions in the black decoration of sample BZN222 (“MW Iznik”), observed with a SEM in BSE mode (gl: glaze; ws: white slip; Mg-chr: magnesiochromite).

Table 4. Chemical compositions of chromium-rich inclusions identified in several black decorations (wt%., SEM-EDS; -: below detection limits). Samples are presented by production.

Sample ID	MgO	Al ₂ O ₃	Fe ₂ O ₃	MnO	CuO	CoO	NiO	Cr ₂ O ₃
Production: MW Iznik								
BYZ284-01	12.0	12.4	5.9	3.6	3.6	2.6	0.7	55.7
BYZ284-02	9.9	14.5	5.5	5.5	4.0	4.2	1.8	50.7
BZN205-01	9.8	15.7	4.5	3.4	2.2	9.7	3.8	49.4
BZN205-03	10.6	13.7	4.5	3.3	1.7	9.5	2.7	52.6
BZN222-12	10.1	16.7	6.1	0.6	5.1	6.1	2.7	50.7
BZN222-16	10.0	16.3	6.4	0.8	4.2	7.8	2.5	49.6
BZN228-01	20.0	17.5	5.0	-	4.1	0.7	1.7	51.1
BZN228-02	19.2	15.9	6.0	-	4.5	0.9	1.6	50.4
BZN228-04	13.0	17.9	4.9	-	3.4	5.9	3.0	50.7
BZN229-01	9.0	19.0	7.5	-	4.7	1.6	2.2	54.8
BZN231-14	16.5	17.5	5.5	-	6.8	2.3	0.5	48.8
BZN231-15	17.3	17.2	6.4	-	5.5	1.8	0.4	49.8
Production: MW unlocated								
BZN286-12	14.9	17.5	5.8	2.5	3.0	2.0	2.5	51.1
BZN286-13	21.1	19.5	7.0	2.0	0.7	0.4	-	48.8
BZN286-14	16.2	17.9	7.0	2.0	2.2	1.1	0.3	51.6

Based on these data, it seems difficult to propose a clear origin for the Miletus Ware’s glaze technology, as it differs from previous productions in western Anatolia, whether Byzantine or Beylik, as well as from central and eastern Anatolian ones (Seljuks). However, we might observe a similarity with the Seljuk ceramics from Korucutepe through their lead-alkali glaze, but more information about the latter and more analyses are needed to compare rightly both technological traditions. Furthermore, to produce the glazes of Miletus Ware, potters introduced soda fluxes that were not used in the previous western Anatolian production centers. The use of alkali fluxes to produce a glaze might also reflect an eastern technical tradition, such as the Seljuk one, for instance.

5.2. Nature of the soda fluxes

The main alkali source in the Miletus Ware glazes is soda (Table 2). By referring to the work carried out by Tite and collaborators on glazes of the Iznik fritwares as well as on Byzantine glasses from western Turkey, analyzed by SEM-EDS and by LA-ICP-MS, we thus tried to determine the nature and the source of the soda fluxes. In their work, the chemical

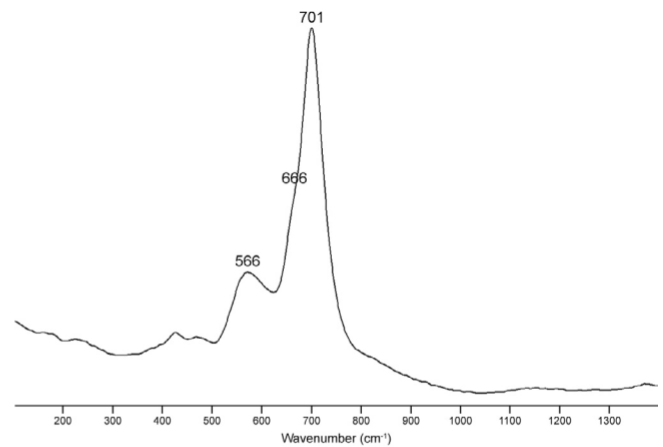


Fig. 9. Raman spectrum of a magnesiochromite inclusion identified in the black decor of sample BYZ284 (“MW Iznik”) (laser: 514 nm; 100x objective; identification based on reference spectrum: RRUFF ID R060796).

compositions obtained showed high Na/K, Na/Mg and Na/Ca ratios (Fig. 10b), as well as small amounts of boron and lithium. Such data enabled the researchers to propose a soda-rich evaporite from western Anatolian hot spring waters as the source of the soda flux (Tite *et al.*, 2016).

After calculating these ratios for the Miletus Ware glazes of the two main productions (“MW Iznik” and “MW unlocated”), we compared them with those of Iznik glazes (Fig. 10b). The results attest also to high ratios for the Miletus Ware glazes, whose ranges are close to the ratios obtained for the Iznik fritware glazes, with the exception of the Na/Mg ratios of the Miletus Ware glazes which are generally lower.

Thus, although the ratios calculated for the glazes of the Miletus Wares are also high, it is difficult to confirm that we are dealing with the same type of alkali source because the magnesium contents are slightly higher for the glazes of Miletus Wares. In order to further our research on the nature and origin of the alkali fluxes used in Miletus Wares glazes, we plan to carry out LA-ICP-MS analyses as well, in order to determine the presence of boron and lithium, whose content are characteristic signatures of western Anatolian hot spring waters.

5.3. Painted decoration

For the painted decoration of the Miletus Wares, the potters used copper- (turquoise blue), manganese- (purple), cobalt- (dark blue), and chromium-based (black) pigments. The latter two seem to have been used here for the first time in the ceramic productions of western Anatolia. Studies of the decorations of Byzantine and Beylik Wares from this region have indeed not attested the presence of these two types of pigments in the glazes earlier (Scott and Kamilli, 1981; Demirci *et al.*, 1996, 2002; Okyar, 2010; Okyar *et al.*, 2011; Kırmızı, 2012; Budak-Ünaler, 2013; Budak-Ünaler *et al.*, 2013; Özçatal *et al.*, 2014; Kırmızı *et al.*, 2015; Burlot, 2017; Burlot *et al.*, 2018).

The use of cobalt- and chromium-based pigments echoes Seljuk productions, as well as the later Ottoman ones. Cobalt-blue and crushed-chromite-based black decors were indeed attested in Seljuk tiles from the Kubad-Âbâd Palace (Yeğingil and Freestone, 2008; Freestone *et al.*, 2009) and from Konya (Kiefer, 1956a, 1956b, 1956c), and on Ottoman fritware (Henderson, 1989; Tite, 1989; Paynter *et al.*, 2004; Şimşek *et al.*, 2010, 2019a).

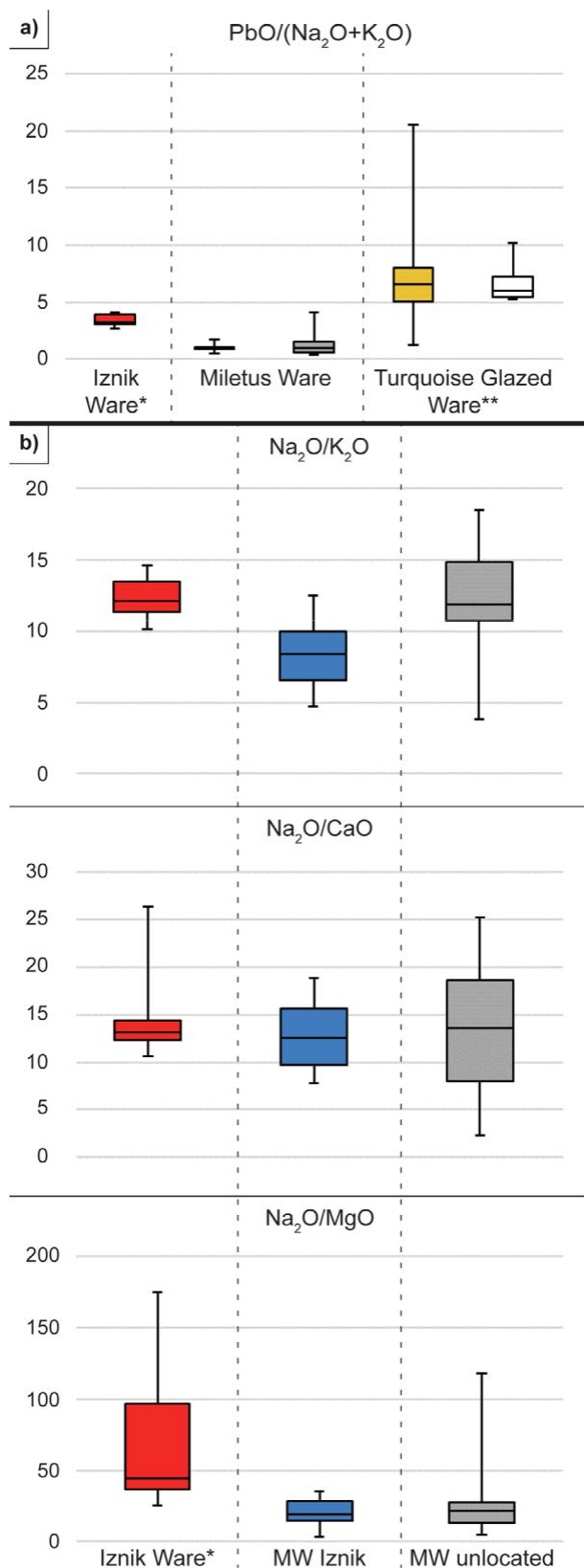


Fig. 10. Boxplots (mean, standard deviation and minimum/maximum values) that compare the ratios between: a) $PbO/(Na_2O + K_2O)$; b) Na_2O/K_2O , Na_2O/CaO and Na_2O/MgO (*refers to Iznik Fritware, data from Tite *et al.*, 2016; ** refers to Monochrome Turquoise Glazed Wares belonging to two early Turkish (Beylik) western Anatolian productions (yellow box: “Ephesus region (b/2)” production; white box: “Pergame F” production), data from Burlot, 2017).

6. Conclusion

Miletus Ware was a widespread ceramic within the Ottoman Empire during the 15th and 16th centuries, whose production bears witness to the introduction of radically different decorations in the western Anatolian ceramic repertoire, as well as new glaze manufacturing technologies. The analyses of the Miletus Wares decoration with SEM-EDS and Raman spectroscopy showed the use of transparent lead-soda glazes applied on the inner surface, which differed from the Byzantine and Beylik ones previously produced in this region. Although the origin of this glaze production technology is not clearly defined, it still seems to have displayed greater influence from the eastern traditions, especially those of the earlier Turkish (Seljuk) productions as suggested by the use of alkali fluxes. The fluxes of the Miletus Ware are sodium-based, which could have been extracted from the soda-rich evaporites from western Anatolian hot spring waters. Deriving the technique from eastern traditions, potters also applied, for the first time in western Anatolia, black and dark blue underglaze decoration, respectively based on magnesiochromite and on minerals characterized by a Co-Fe-Ni-Cu association. These productions, therefore, involved the use of new resources, suggesting new commercial relations in the pottery industry of western Anatolia, likely favored by the expansion of the Ottoman Empire. From a chronological point of view, the Crimean contexts show that the Miletus Ware produced in Iznik was still in use in the second half of the 16th century. It proves that the manufacture of this ceramic was contemporary with that of the so-called Iznik Ware, which may explain their similarities in glaze technology.

Acknowledgements

This study was funded by the French National Research Agency (ANR) through the POMEDOR project, and we acknowledge the support of the ANR with the reference ANR-12-CULT-0008; and by the Koç University Research Centre for Anatolian Civilizations for the Raman spectroscopic analyses at KUYTAM. We would like to thank: the Turkish Ministry of Culture and Tourism, the Directors of the archaeological excavations and the staff of the Museums at Ephesus, Miletus, Sardis, and Pergamon for giving us permission to study the samples; I. Teslenko, B. Böhlendorf-Arslan and J. Vroom for their ceramic studies of the present corpus; the staff of the analytical facilities in Lyon (CNRS UMR 5138 and CTμ), in Paris (UMR 8233) and in Istanbul (KUYTAM).

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