Glazes, colorants and decorations in early Islamic glazed ceramics from the Vega of Granada (9th to 12th centuries AD)

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Abstract

This paper presents an analysis of glazes of vessels unearthed in archaeological excavation in the Vega of Granada (south-east Spain) and that can be dated between the 9th and the 12th centuries AD. The material analysed includes the first glazed ceramics produced in Al-Andalus after the Islamic occupation of the Peninsula. From a very early period (mid to late-9th century) there is a coexistence of lead-alkaline monochrome (green and honey) glazed vessels, and of polychrome decorated (green, white and brown) tin-glazed, wares early examples of the so called green and manganese wares. From the 10th century onwards honey lead-alkaline glazed vessels often with manganese-painted decorations are found. The chemical composition of the ceramic pastes, glazes and decorations as well as of the microcrystallites present in the glazes is obtained from polished cross sections of the glazes by SEM-EDS, OM and EPM. Information about the materials and methods of production is discussed and compared to contemporary productions from other areas in Al Andalus and also to later productions.

Keywords: glazes, decorations, optical microscopy, SEM-EDS, Islamization, Iberia

Highlights:

- Early Islamic glazed wares are analysed with OM, SEM-EDX and EPM

- Lead glazes were raw mixtures over biscuit or fired mixtures over raw ceramic
- Tin glazes were applied over biscuit fired bodies
- Pigment and lead oxide mixtures were applied over the tin glazes for the colour decorations
- There is a chronological change in lead glaze composition

1. Introduction

The process of Islamization of Iberia started after the conquest of the peninsula by Muslim forces coming from North-Africa in 711 CE. Although the conquest was finished in few years (711-714 CE, cf. Chalmeta Gendrón 1994), the process of Islamisation was a more complicated and protracted issue that would be developed throughout a longer period ranging from the eighth to the eleventh centuries. This complex process has been abundantly studied from a political point of view. It happened mainly at the same time as the history of the Umayyad dynasty of Cordoba. This was a branch of the caliphal dynasty of Damascus that escaped from the Abbasid caliphs of the East and seized the power in Iberia shortly after the conquest of the peninsula, in 756. The Emirate of Cordoba grew in power and influence in the Western Mediterranean and, at its zenith, it was proclaimed a Caliphate in 929. The Caliphate lasted until 1031, when it was divided in small kingdoms known as *Taifas* (Manzano Moreno 2006). Different aspects of the material culture of this long and eventful period have been analysed regionally (eg Gutiérrez Lloret 1996; Carvajal López 2008), but the process of Islamisation is still a matter of debate (cf. Carvajal López 2013). This paper aims to shed light on this process by contributing to the history of the glazing technologies in al-Andalus.

The introduction of glazed ceramics is closely related to the process of Islamization, as glazes were extremely exceptional in Iberia before the Islamic conquest and they became part of daily life, even after Iberia ceased to be part of the Islamic world. Although the glazed ceramics in the Middle East have a very long history, starting much earlier than Islam, the first Islamic glazed wares are not documented in the Iberian Peninsula until the ninth century CE (Salinas Pleguezuelo and Zozaya 2015), the time period that has been termed Emiral because of the title of the Umayyad ruler of Cordoba before 929: *amīr*. Among the first Islamic glazed ceramics documented there are some imports from the other parts of the Islamic world and even from China (eg. Carvajal López 2008: Heidenreich 2001), but most of the documented vessels seem to be the result of regional production that features a combination of influences and innovations with respect to the glazing traditions of the Middle East. In this context it must be understood that ‘regional’ may potentially include locations in North Africa, as the information on particular workshops of the early Islamic period is extremely scarce and the links between this region and Iberia were close.

The earliest glazes documented in the Peninsula have been located in different archaeological sites of the south of al-Andalus and are always dated in the second half of the ninth century

(Carvajal López 2008; Iñíguez Sánchez and Mayorga Mayorga 1993**;** Salinas Pleguezuelo 2013). These vessels are small and closed, decorated in one or two colours, usually green and honey brown, and it is thought that they were produced locally. From the tenth century onwards the honey brown glazes, usually decorated with lines of dark brown or black, were abundantly used as a coverture of dishes. Another early glazed product in al-Andalus is the Green and Manganese pottery (also known as green and brown) (Rosselló 1992; 1995), initially dated in the tenth century (Barceló 1993), but now documented in ninth century deposits too (Carvajal López 2008; Salinas Pleguezuelo 2013). This glazing technique was applied to closed and open vessels alike. Finally, from the eleventh century, the application of glaze to cooking wares is documented (cf. Carvajal López 2008). Since the work of A. Bazzana (1979) it is assumed that all glazes in al-Andalus were made using a base of silica with lead as flux, and that Green and Manganese glaze contains tin as opacifier. Bazzana did not have actual analysis of the glazes, he was just extrapolating the results from studies of other Western Mediterranean ceramics. In spite of that, his classification is basically right, as most of the glazes from al-Andalus are based on the use of lead as flux (eg Molera et al 2001a; cf Tite et al 1998) and the few analysis done on Green and Manganese wares have demonstrated the presence of tin (Molera et al 2001a).

A lot of work is still to be done to understand the historical development of glaze technologies in al-Andalus. For example, there is no information on how the glazes were produced and distributed at the local level. Were they produced in a few workshops and widely distributed, or were they the result of a widely spread activity? This paper aims to contribute to the understanding of these questions by focusing on the well contextualized glazes found in the Vega of Granada, a region in south-east Spain.

2. Archaeological background and purpose of this study.

The Vega of Granada is a triangular basin defined by the Genil river, which crosses the area from east to west. Located in the south east of Spain, the Vega was conquered by the Muslims probably in 712 CE. Archaeological research on the Vega has defined four archaeological phases for the social and economic changes that define the process of Islamization in this area of al-Andalus, originally defined in Carvajal López 2008 and 2009, and later expanded to the current version in Carvajal López and Day 2013. These phases form the framework of development in which the glazes under study are included:

2.1. Phase I

Ranging between the sixth and the late eight to early ninth centuries CE, this is the phase that covers the historical developments of the years immediately before the Islamic conquest of 711 CE and those of the first century of Islamic rule. This is an important period that includes not only the arrival of the Muslims, but also the foundation of the Umayyad Emirate of Cordoba (756 CE). Phase I does not come into the scope of this work, because no glazed ceramics have been documented in this period.

2.2. Phase II

Phase II goes from the late eighth or early ninth century to circa 925 CE. This period covers the remaining years of the Emirate of Cordoba (which was transformed into a Caliphate in 929 CE). In the Vega of Granada this period is marked by the existence of a number of fortified and unfortified rural sites that exist in parallel to the two towns of the area, Ilbira and Granada. There are glazed vessels in all of them, usually in small numbers but showing a wide range of colours and techniques.

2.3. Phase III

Phase III starts circa 925 CE and ends circa 1011 CE. This interval corresponds to the length of the Caliphate of Cordoba (929-1031 CE), which marks the zenith and fall of the Cordovan government of al-Andalus. During this period the population of the Vega of Granada was concentrated in the town of Ilbira, the provincial capital, after numerous fortified rural sites were abandoned. Ilbira was a prosperous city placed in the land route that linked Cordoba with the main port in al-Andalus, Almería, until most of its population moved to Granada shortly after a Berber attack in 1011 CE.

2.4. Phase IV

Phase IV begins circa 1011 CE and finishes roughly in the mid-twelfth century CE. In this period the political union of al-Andalus vanished and was replaced by the independent Taifa kingdoms all over al-Andalus, with the Zirid dynasty ruling Granada. These kingdoms would be brought together under Almoravid control from 1090 CE, and in 1147 CE the Almohads took over the region.

In this paper, glazes coming from Phases II, III and IV will be analysed. They come from the archaeological sites of Manzanil (Loja), two different sites in Ilbira (Atarfe): Sombrerete and Tejoletes, the Albaycín in Granada and El Castillejo (Nívar) (Fig 1), which have yielded glazes in one or several of the phases described above (Table 1; for information on the sites, see Ruiz Jiménez 2008 for Manzanil; Carvajal López 2008; Jiménez Puertas and Carvajal López 2008; Malpica Cuello et al 2010 for Ilbira; Adroher Auroux and López Marcos 2001; Carvajal López 2008 for the Albaycin; Jiménez Puertas et al 2009; Jiménez Puertas and Carvajal López in press for El Castillejo). It is worth noting that glazes have been located in every site and phase considered in this study, with the exception of Phase I (which is too early). The missing gaps in Table 1 correspond to a lack of evidence in the archaeological record of the sites under discussion. Finally, it must be said that glazed pottery has been found in other sites of the Vega as well, but they were not as numerous or well contextualized as in the sites discussed in this text. This means that glazed pottery had a very powerful presence in the early Islamic Vega of Granada.

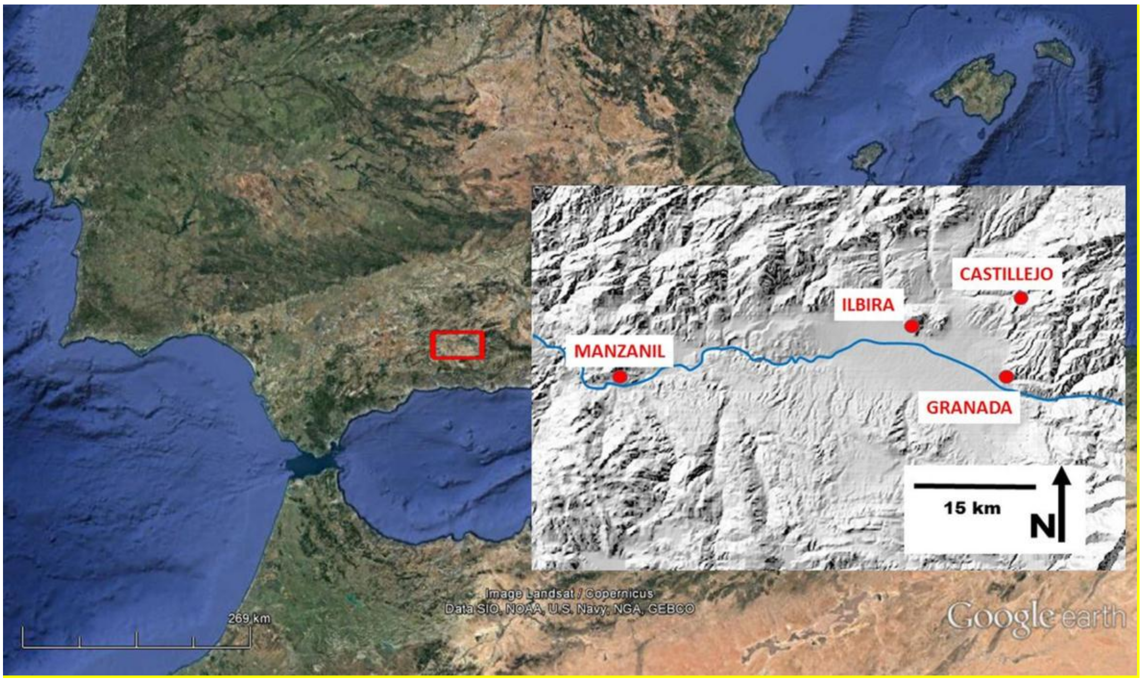


Figure 1. Map showing the location of the area and sites considered in this text.

The glazes found in the Vega of Granada are representative of all the categories described above: green and honey-coloured lead glazed wares, two-side lead glazed wares with one side green and the other honey-coloured, Green and Manganese decorated tin glaze wares and lead glaze cooking pots. Therefore, this study offers the chance to look at the regional development of a technology that was spread all over al-Andalus.

Table 1. A summary of the five assemblages, occupation period and type of wares studied. G = Green lead glazed vessels, H = Honey-coloured lead glazed vessels, 2col = Two-colour lead glazed vessels, GM = Green and Manganese decorated tin glazed vessels, CP = lead glazed cooking pots. n.i.a. = No information available

Chronology / Site	**Emirate Period**		**Caliphate Period**	**Taifa-Almohad Period**
	Phase I	Phase II	Phase III	Phase IV
	711 to circa 800	Circa 800 –925	Circa 925-1011	Circa1011-1150
Manzanil	n.i.a	G, H, GM, 2col	n.i.a	n.i.a
Ilbira Sombrerete	n.i.a	G, H, GM	n.i.a	n.i.a
Ilbira Tejoletes	n.i.a	n.i.a	H	n.i.a
Granada	Early phase without glazed ceramics	n.i.a	n.i.a	H, CP
Castillejo	Early phase without glazed ceramics	n.i.a	n.i.a	G, H, CP

3. Materials and Methods

3.1. Glazed vessels

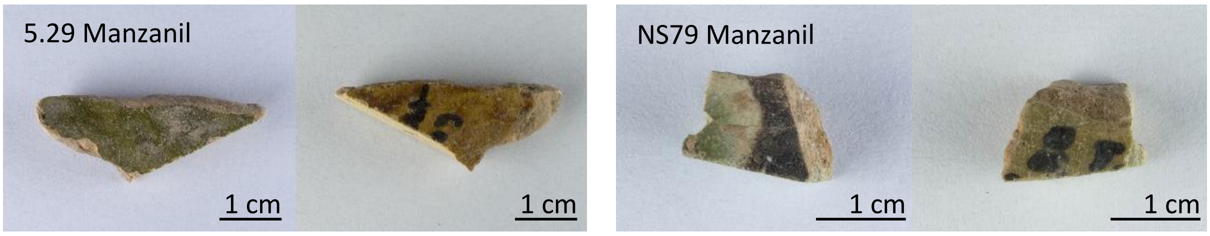
For this study twenty samples were selected from all the sites mentioned above. The samples chosen were representative of the main types of glazed wares described above (Fig.2) and are summarised in Table 2: flasks and dishes with green and honey-coloured glazes from the tenth and eleventh centuries and dishes with Green and Manganese decorations and tin glazes dating from the ninth century. Two glazed cooking pots characteristic of Phase IV are also included to explore the similarities with the other glazed vessels.

Ceramic pastes and glazes of the twenty samples were explored. In the development of the study the results of the analyses of the paste of Sample NS30 (Green and Manganese ware) and of the green glaze of Sample NS27 (Green lead glaze), which were too altered, gave unreliable data and have not been included in the study that follows. As a result, there are nineteen samples considered in for pastes and for glazes, but only eighteen samples can be considered to be completely analysed for paste and glaze together.

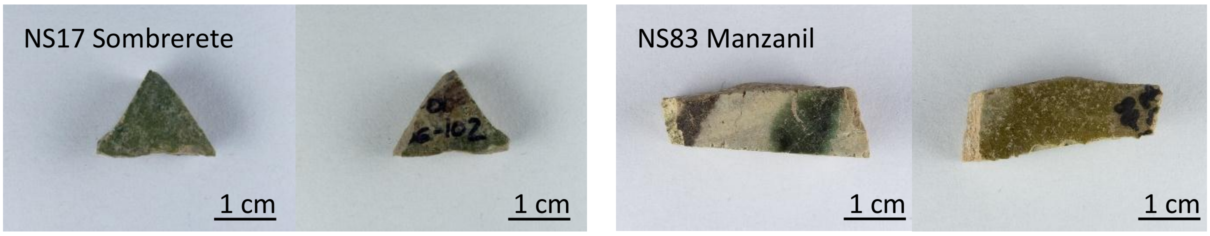
It is also important to mention that the more superficial areas of the glazes appear often broken showing conchoidal fractures and flakes of the glazes are missing. Therefore, the analyses were obtained in many cases from areas relatively close to the ceramic-glaze interface. This may give more variability to the analyses. However, as the areas analysed showed good conservation, we still think that they are valuable.

Table 2. Description of glaze categories.

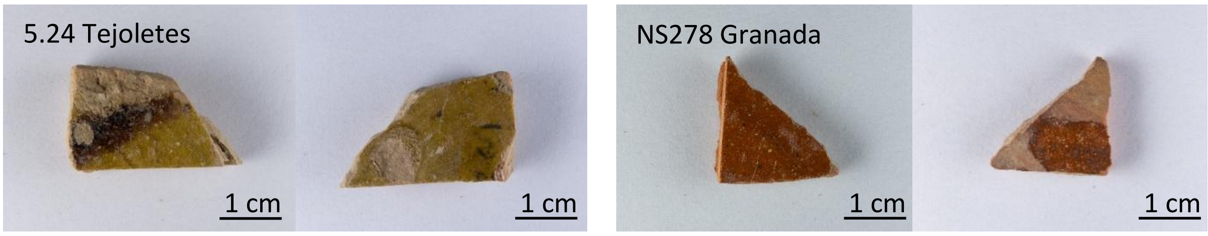
Lead Glazes	Honey-coloured (H)	5 samples	Flasks and dishes covered on one or both sides with a yellowish to honey, sometimes light green glaze. The main side may have dark brown decoration lines
	Green-coloured (G)	2 samples	Flasks covered on one or both sides with deep green glaze
	2 colours (2Col)	2 samples	Flasks. One side has a honey glaze the other side a deep green glaze.
Tin Glazes	Green and Manganese (GM)	9 samples	Dishes. The main side is decorated with dark brown and green motives over a white background glaze. The other side may be covered with a yellow or green glaze.
Cooking pots	Cooking Pots (CP)	2 samples	Red-Honey glaze inside, sometimes outside as well



Two-coloured glaze **Green & Manganese decorated glaze**



Green glaze **Green & Manganese decorated glaze**



Honey-glaze **Cooking pot glaze**

Figure 2. Pictures of the different glaze categories.

3.2. Methods of analysis

Small fragments (a few millimeters) of the samples were embedded in an epoxy resin to obtain polished cross sections for Optical Microscopy (OM) and Scanning Electron Microscopy (SEM) observation and analysis. A Zeiss DSM 960A SEM with Link Isis L200B EDX detector was used (20 kV, 1.5 nA). It was calibrated using mineral and glaze standards. Typical limits of detection are about 0.1% for most of the elements except lead and tin, 0.5%, and calcium and copper, 0.2%. At least two measurements were obtained and average and standard deviations determined from polished cross sections of the ceramic bodies covering each an area of 2x2.5 mm^2 adequate for the analysis of fine pastes and, also from well preserved regions of the glazes covering an area of a few hundreds of micrometres; spot analysis of some of the crystallites present in the ceramic matrix, in the glazes and also in the decorations were also performed to determine their nature. The data have been renormalized for comparison among samples and measurements with low totals were rejected. Backscattered electron (BSE) images of the pastes and glazes were obtained as well.

In order to elucidate the nature of some of the microcrystallites found in the glaze decorations chemical analyses were also obtained by EPM (JEOL JXA-8230). This equipment, with 5 wavelength dispersive X-ray spectrometers (WDS), has a lower limit of detection: for most of the elements of about 0.01%. The operating conditions were 20kV and 15nA with a focused beam (spot analysis). A minimum of six analyses of the crystallites and of the glaze around were acquired and the average and standard deviation obtained.

4. Results

4.1. Ceramic pastes

The chemical analysis obtained by SEM-EDS of the pastes, lead and tin glazed wares are given in Table 3, 4 and 5 respectively. Some characteristic results of the Optical and SEM observation of the polished sections of the pastes, glazes and decorations are shown in Figure 3, Figures 4 and 5 and Figures 6 and 7 respectively.

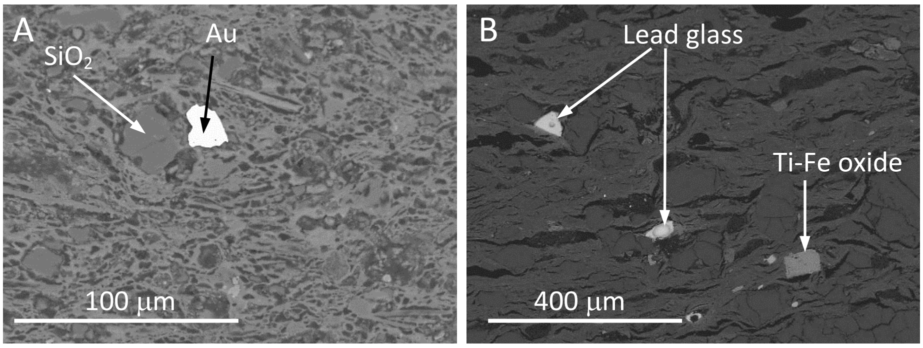


Figure 3. SEM-BSE images of A) gold fragments found at the ceramic body of Sample NS79 and B) lead glass fragments and some tin-iron oxide particles found in the ceramic body of Sample NS291.

Table 3. Chemical composition of the ceramic bodies measured by SEM-EDS. All the amounts represent wt%

Phase	Glazes	decor.	Site	Ref.	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	FeO	PbO
II	Tin	GM	Manzanil	NS83	1.2	3.1	15.6	56.6	1.6	14.8	0.7	6.2	0.3
				NS82	0.1	2.5	19.4	52.1	2.1	13.4	0.8	7.7	1.8
				NS81	0.6	2.5	19.8	52.8	1.1	12.8	0.8	7.4	1.5
				NS79	0.9	3.0	15.4	55.7	1.8	14.6	0.7	6.4	0.9
				NS78	0.4	2.7	19.8	54.5	1.2	13.0	0.8	7.7	0.0
				NS77	0.6	2.6	17.3	53.2	1.8	15.3	0.6	6.8	1.9
			Sombrerete	NS22	1.0	2.6	16.6	54.8	2.8	13.9	0.7	6.1	1.2
				NS27	0.9	3.3	14.6	54.5	2.6	17.4	0.6	5.3	0.9
	Lead	2Col	Manzanil	5.28	0.7	2.5	20.0	52.1	2.0	12.5	0.8	8.0	1.3
				5.29	1.1	2.9	16.6	56.1	1.4	14.2	0.7	5.9	1.1
		G	Sombrerete	NS17	0.7	3.5	15.2	54.0	3.1	16.8	0.7	6.0	0.0
			Castillejo	NS121	0.8	3.1	17.1	53.2	2.7	15.2	0.6	6.5	0.7
III	Lead	H&B	Tejoletes	5.24	0.4	2.6	16.4	52.5	3.0	16.5	0.7	5.9	1.6
				5.20	0.3	1.5	13.8	63.2	3.0	8.7	0.7	5.9	2.8
		H		NS291	1.0	2.9	17.2	51.0	2.0	18.1	0.6	5.9	1.1
IV	Lead	H	Castillejo	NS124	1.8	4.1	19.4	51.1	2.2	14.0	0.6	5.5	1.4
			Granada	NS275	0.6	4.0	17.8	53.9	2.9	13.6	0.6	6.5	0.0
	Cooking Pots	H	Granada	NS278	0.5	0.9	24.7	58.5	3.1	1.2	0.5	10.4	0.2
			Castillejo	NS110	0.7	1.0	19.3	67.5	2.5	1.3	0.5	7.0	0.0

The ceramic pastes are typical fine calcareous pastes (~14.4% CaO, varying between 12.8 and 18.1%) and show a relatively similar composition with some chemical variability, an understandable phenomenon considering the long period and the various sites under study. The exceptions are Sample 5.20 (only 8.7% CaO) and the cooking pots, which are not calcareous (less than 1.5% CaO). The ceramic pastes show also small inclusions of gold particles, in some cases containing also zirconium or palladium (Fig 3A). Those inclusions are very characteristic of these pastes, and appear in almost all the shards, including the cooking pots. Finally, the pastes contain also small lead glass fragments (Fig 3B), which are responsible for the lead content determined in the pastes. Nevertheless, the low reaction of those glass fragments with the ceramic paste indicates relative low firing temperatures. It is not known at this stage if this is an intended addition or a side result of working with ceramics and glazes in the same or close spaces. The presence of similar small glass fragments has also been observed in ceramics from early Islamic Iraq (Mason and Tite 1994: 79-83), where it was suggested that this glass inclusions could have been added intentionally, thus setting a precedent for later fritware recipes. Yet fritwares were never developed in the Western Mediterranean. In principle, this lends support to the idea of accidental inclusion of glass fragments in the ceramic paste.

4.2. Lead glazes

The lead glazes (Table 3) are contain abundant lead (~49%PbO), but also relatively large amounts of calcium and aluminium (~4.4%CaO and ~4.4%Al_2O_3) and low quantities of potassium (~1.7%K_2O), sodium (~0.5%Na_2O) and magnesium (~0.5%MgO). Iron is present in all samples in similar amounts (~2.0%FeO), except in the honey glazes of Samples 5.29, 5.20, 5.24 and NS278, where it is found in even higher quantities (ranging from 3.3 to 6% of FeO). This suggests that in these cases iron was added as a colourant. Other added colourants are copper for the green and manganese for the brown decorations. The dissolution of Cu^{2+} in the glaze gives a dark green colour, Fe^{3+} a yellow colour and Fe^{2+} a green colour to lead glazes. Manganese is mainly present as Mn^{2+} in the lead glazes, giving a violet tinge when the amount of manganese dissolved is low and brown if it is high. The small amount of tin determined in some of the green glazes is probably due to the use of bronze scrap as copper source. The cooking pots are typical high-lead glazes, richer in lead (~57%PbO) and aluminium (~6.6%Al_2O_3), but containing similar amounts of iron (~2.6%FeO) with regards to the other lead glazes.

Green and honey-coloured glazes, including the lead glazed and two-colour glazed ceramics, as well as the cooking pot groups (Fig. 4) are thick (≈200μm), show a limited glaze-ceramic interaction layer full of potassium-lead feldspars, few large bubbles and large cracks perpendicular to the glaze surface. Lead-potassium feldspars nucleate and grow at the interface due to the reaction of the PbO with the clay (Molera et al 1993, Molera et al 2001b, Pradell et al 2010). The cracks show the characteristic pattern developed due to the mismatch between the glaze and the ceramic shrinkage during cooling, although crack patterns can also develop immediately after removal from the kiln or years later (Zamek 1995). Moreover, iron bearing pyroxenes (typical composition: $CaMg_{0.85}Fe_{0.15}Si_2O_6$) floating into the glaze towards the surface are found in the lead glazes but not in the cooking pots; pyroxenes form due to the reaction of Ca- and Mg-rich grains from the paste or dissolved in the glaze; due to the lower density of those crystallites in comparison to the lead glaze, they detach from the interface and float in the glaze (Molera et al 2001b). The pastes of the cooking pots contain very low amounts of calcium (~1%CaO) and consequently pyroxenes are not formed. Quartz particles or any other remnant of the original raw glazes are not found. The limited interaction layer and lack of quartz grains is consistent with a double firing, that is, the glaze raw materials being applied over fired ceramic bodies. However, this could also be consistent with the use of a pre-fritted glaze mixture applied over a raw ceramic body.

Table 4. Chemical composition of the lead glaze wares measured by SEM-EDS, b.d. below detection limits. G: green; B: brown; H: honey. All the amounts represent wt%

Phase	**Dec.**	**Site**	**Ref.**	**colour**	**Na_2O**	**MgO**	**Al_2O_3**	**SiO_2**	**K_2O**	**CaO**	**FeO**	**MnO_2**	**CuO**	**SnO_2**	**PbO**
II	2col	Manzanil	5.28	G	0.3	0.6	2.8	39.3	0.9	3.7	2.0		2.2		48.2
				H	0.4	0.4	3.8	39.1	1.3	3.3	1.6		0.8		49.2
			5.29	H	0.4	0.6	4.1	35.9	1.9	3.6	6.0		0.1		46.7
				G	0.7	0.8	5.9	37.9	2.0	4.6	1.7		0.9	0.7	44.6
	G	Sombrerete	NS17	G	0.2	0.5	2.5	30.6	0.7	3.6	1.0		2.0		58.8
					0.2	0.4	3.6	33.4	1.0	4.1	1.4		1.6		54.2
		Castillejo	NS121	G	1.5	0.5	3.6	37.5	1.7	3.9	1.9		0.8	0.9	47.4
				Red band	1.3	0.5	3.9	38.1	2.0	4.2	1.8		0.9	0.4	46.6
III	H	Tejoletes	NS291	H	0.6	0.7	4.8	35.6	2.1	5.2	2.5				48.5
			5.20	H	0.3	0.4	5.1	37.7	1.6	4.7	4.7				45.2
				H	0.3	0.5	5.7	37.9	2.3	5.3	2.6				44.8
			5.24	H	0.5	0.8	4.9	33.2	1.8	4.6	1.8				52.2
				Brown band	0.5	0.4	4.7	32.9	1.8	4.2	3.8	1.5			50.2
IV	H	Castillejo	NS124	H	0.4	1.0	4.9	35.2	2.0	5.6	2.4				47.7
		Granada	NS275	H	0.4	0.6	4.2	35.5	2.1	5.2	1.9				50.1
					0.4	0.7	5.9	35.9	2.3	4.7	2.3				47.4
	Cooking Pots	Granada	NS278	H	b.d.	b.d.	6.5	26.9	0.5	1.0	2.3				62.8
					b.d.	0.2	6.9	31.9	0.6	0.9	3.3				56.1
		Castillejo	NS110	H	0.3	0.5	6.4	33.1	1.3	3.2	2.2				52.5

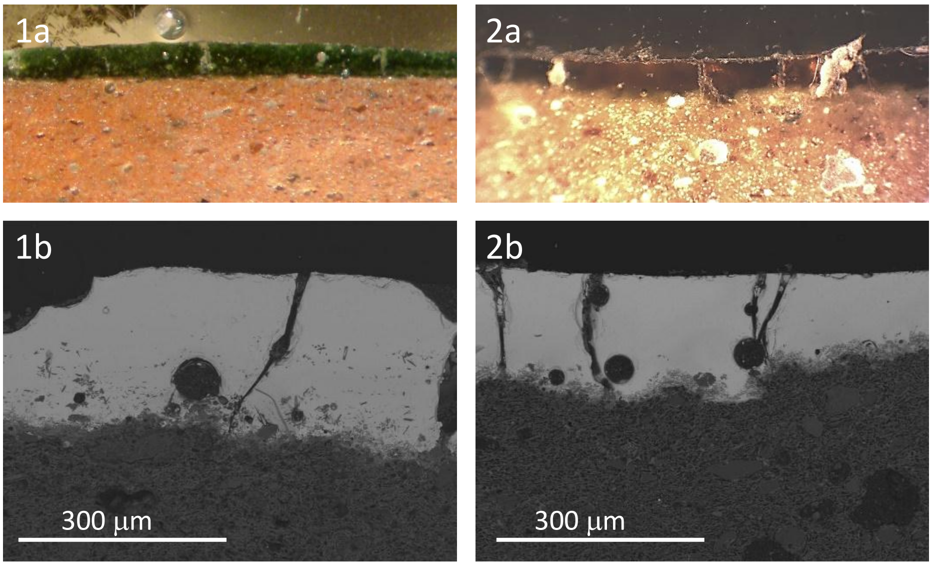


Figure 4. 1a) Optical and 1b) SEM-BSE images of a green glaze from Sample 5.28. 2a) Optical and 2b) SEM-BSE images of a honey glaze from Sample 5.29.

One of the green glazes, Sample NS121 (Fig 5), shows a curious feature, observed by Optical Microscopy: a red band very close to the interaction layer between the glaze and the body (Fig 5B). Micro-XRD analysis of similar bands in other samples (Salinas Pleguezuelo, Pradell, Molera, study in preparation) showed the presence of metallic copper nanoparticles. The small size of the particles (a few nanometres) makes them virtually undetectable by SEM-BSE. Copper nanoparticles scatter light strongly due to the Surface Plasmon Resonance (SPR) at about 560 nm, producing the bright red colour shown. Copper nanoparticles may form if a copper containing glaze is submitted to strong reducing conditions during the firing (Pradell 2016). In fact, the glaze looks very dark as it corresponds to a heavily reduced lead glaze (Fig 5A).

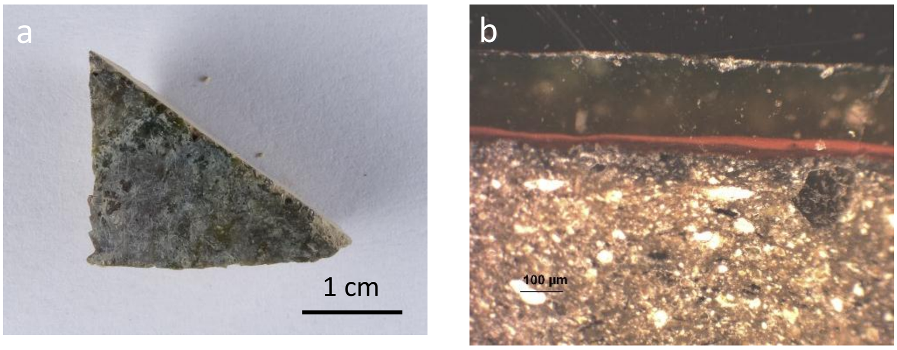


Figure 5. a) Standard and b) Dark Field Microscopy image of NS121 green glaze (Phase II) showing a red band closer to the glaze ceramics interaction layer.

4.2. Tin Glazes

Table 5 shows the analysis of the white and coloured areas corresponding to the Green and Manganese decorated tin glazed wares. Moreover, some of the wares show also a green- or honey-coloured transparent glaze in the other and the corresponding analysis are also included in Table 5. One of the samples (NS27) had both glazes so altered that they were not considered for analyses. Moreover, some of the white glazes appeared very weathered and a consistent analysis was not possible on them. For these reason they are not shown in Table 5 either.

Table 5. Analysis of the glazes corresponding to the tin glaze wares; b.d.: below detection limits; G: green; B: brown; W: white and H: honey. All the amounts represent wt%

Phase	Site	Ref.	Colour	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO	FeO	MnO_2	CuO	PbO	SnO_2
II	Manzanil	NS77	G	2.4	0.4	1.2	38.5	0.9	3.0	0.1	0.7		1.2	43.0	8.7
			H	0.4	0.4	3.5	32.6	1.1	4.4	0.2	0.9			56.2	b.d.
		NS78	B	2.1	0.5	1.7	41.5	1.5	4.1	b.d.	1.1	3.5		39.5	4.3
		NS79	B	1.2	0.5	4.8	47.6	3.4	2.7	1.5	1.4	7.3		28.0	1.5
			G	1.2	0.8	4.5	43.2	4.0	0.2	1.4	1.3		1.2	28.1	14.3
			H	0.6	0.9	4.2	46.1	2.7	5.6	b.d.	1.9			37.7	b.d.
		NS81	B	2.2	0.5	2.9	44.0	1.7	3.6	0.1	1.0	2.3		38.3	3.3
			W	2.1	0.8	2.6	42.8	2.5	7.4	b.d.	1.2			35.3	5.3
		NS82	H	0.3	0.3	4.3	31.2	1.1	3.3	b.d.	1.0			58.6	b.d.
		NS83	G	1.1	0.9	3.8	42.8	2.1	5.8	0.1	1.5	0.2	2.9	36.0	2.9
			W	0.8	0.9	1.7	46.3	2.1	5.6	b.d.	1.1	0.1	0.4	29.1	11.8
			H	0.3	1.0	4.9	45.3	2.5	5.8	0.1	2.1			37.6	b.d.
	Sombrerete	NS22	G	1.0	1.1	2.2	45.6	1.8	5.8	0.1	1.4		1.3	38.4	4.3
			W	0.9	1.0	3.6	45.2	2.6	6.3	0.2	1.2			33.2	6.0
		NS30	G	0.5	0.7	1.8	41.8	1.3	3.4	b.d.	1.0		8.0	36.7	4.5
			W	1.6	0.7	2.6	42.6	2.0	4.4	b.d.	1.3		1.2	40.8	4.4

In the white glaze areas, SnO_2 is found in variable amounts, ranging from 1.5 to 12% (Table 5). Tin is mainly present in the form of small cassiterite (SnO_2) microcrystallites (Figure 6, 1a and 6, 1b), which appear heterogeneously distributed in the glazes, often showing accumulations of crystallites where the original tin-rich particles were present; as a consequence the distribution of the SnO_2 content shows a large variability between samples and even within different areas of the same glaze. Cassiterite crystallites are present in high amounts, about 12 wt% SnO_2 in the white glaze of Sample NS83 (Fig 6, 1b), one of the best preserved. Contrariwise, they are barely seen in the green decoration of the same sample, which is transparent with very few cassiterite crystallites (Fig 6, 1c); still an amount of 2.9 % SnO_2 is determined by EDS. The green decoration is richer in lead than the white glaze, suggesting that the green pigment was applied on the top of the tin glaze mixed with some PbO. All the green decorations contain copper in variable amounts (Table 5), sometimes in very high amounts, as it is the case in the green decoration of Sample NS30, 8%CuO, which is very well preserved and of an intense colour. In this case, the microscopic examination shows without doubt that the copper pigment was applied also on top of the tin glaze.

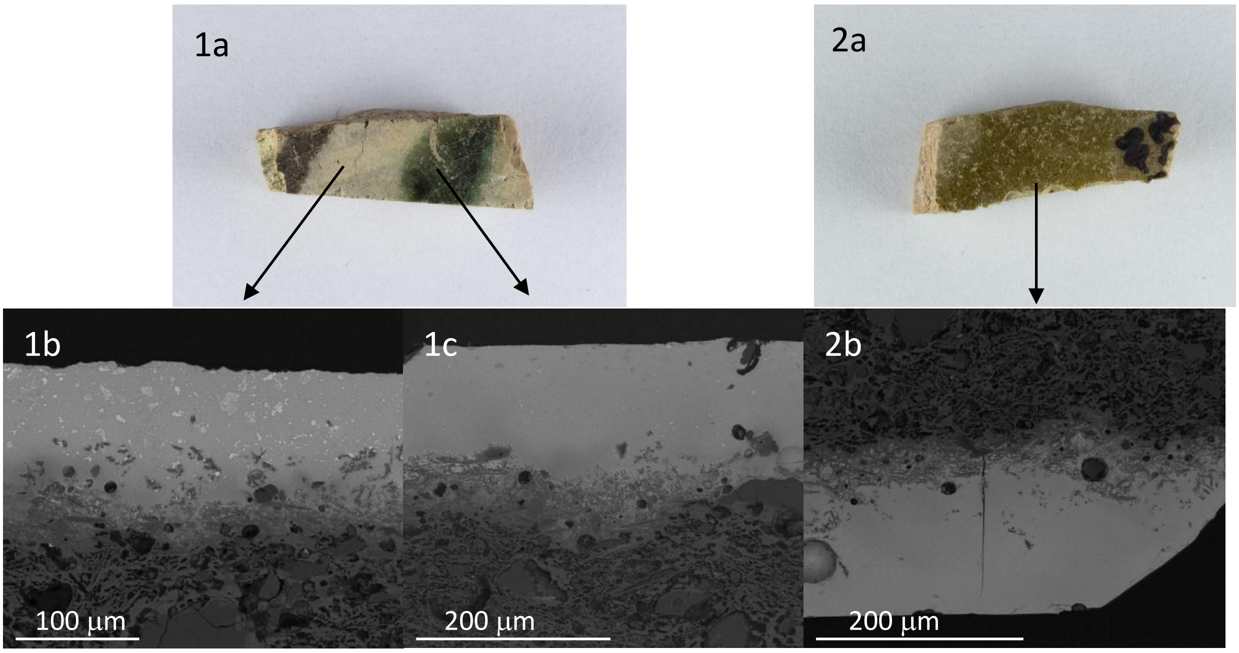


Figure 6. 1a) Standard picture of tin glaze with decoration in brown and green in Sample NS83. 1b and 1c) SEM-BSE images of the white and green glazes of the sample. 2a) Standard picture of the reverse glaze of Sample NS83. 2b) SEM-BSE image of the reverse glaze of the same sample.

With regard to the honey-green coloured glazes found in the reverse of the shards, only four were measured (the other were too poorly preserved). They are lead glazes (Fig 6, 2a and 6, 2b) and classify in two different groups; NS77 and NS82 have a high lead content

(~57%PbO) and correspondingly, a lower aluminium, potassium, calcium and iron content while NS79 and NS83 a very low lead content (~37%PbO) and higher aluminium, potassium, calcium and iron. The discrepancy in the lead amount can be due to the conditions of the analysis. As some of the glazes are quite weathered, the measurements were performed in areas very close to the ceramic body, and this could result in lower lead contents. However, it cannot be discarded that these discrepancies reflect different glaze recipes. More samples should be analysed to conclude anything about these glazes in the reverse of the tin glaze products.

Coming back to the main decorated side of the tin glazes, the brown motives contain manganese in relatively high amounts (between 2 and 7.3%MnO_2). Standard and SEM images of the brown decoration from Sample NS79 are shown in Figure 7. This sample appears relatively well preserved, although some of the decoration has been lost. The manganese decoration is applied on top of the tin glaze, as can be seen in Figure 7b, and shows light grey needle-like manganese-containing microcrystallites on the surface growing towards the ceramic (Figures 7c and 7d). The large needle-like crystallites have been analysed with the Microprobe, giving the composition: $Ca_{0.4}Mn_{1.6}Si_2O_6$, consistent with a manganese-containing pyroxene, in particular a manganese-rich bustamite. Bustamite and in general manganese pyroxenes have been found in other historical brown decorations (Molera et al 2013). Pyroxens form at a firing temperature above 900ºC in calcium-rich areas of the glaze. Moreover, very small and scarce cassiterite (SnO_2) crystallites are seen in the brown glaze, appearing concentrated in the ceramic-glaze interface; this confirms that the brown paint did not contain cassiterite and was applied on top of the tin glaze.

Figure 7e shows also the presence of some light crystallites in the SEM-BSE images. Their composition was measured by EDS and also by Microprobe. The data were not very conclusive, because of low totals, probably due to poor conservation of the decoration. However, the data are consistent with a lead manganese silicate containing some calcium and chlorine. The formation of a manganese-lead silicate may indicate a low firing temperature; for instance kentrolite ($Pb_2MnSi_2O_9$), a very common lead manganese silicate formed in manganese-rich decorations, decomposes at a temperature of about 950ºC (ongoing analyses by Molera et al). The presence of a lead-manganese compound might be related to the application of a manganese oxide pigment together with some lead oxide on top of the glaze. This oxide did not dissolve with the tin glaze mixture below. Consequently, we conclude that the dark brown colour was obtained firing the manganese-lead pigment on the top of the white at temperatures of about 900ºC, which gave rise to the formation of manganese and lead manganese silicates.

Consequently, to obtain the green and brown decorations, copper and manganese mixed with lead oxide pigments were applied on top of the tin glaze and fired at a relatively low temperature (900ºC).

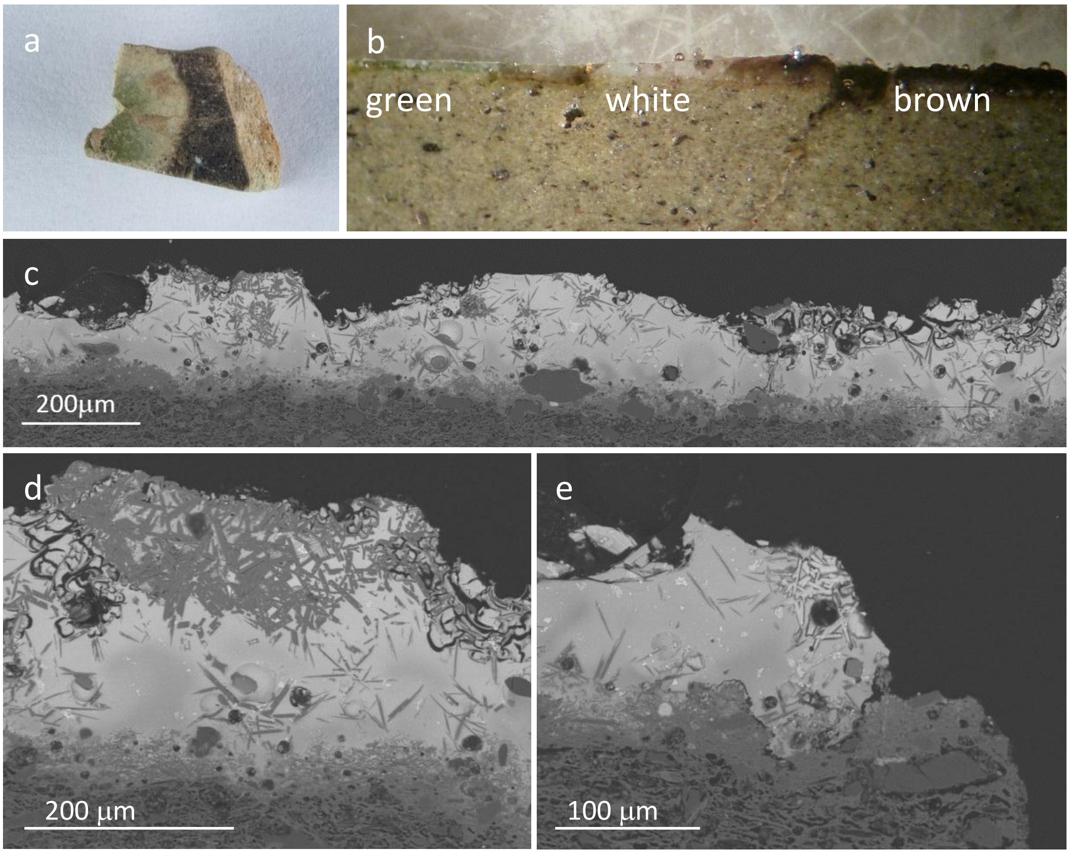


Figure 7. A) Standard and B) Optical and C) D) and E) SEM-BSE images of the brown decoration from NS79

5. Discussion

5.1. Characteristics of ceramics and glazes

In Figure 8 the composition of the ceramic pastes Na_2O+K_2O+FeO versus $CaO+MgO$ is plotted. These two sets of oxides represent two components of the ceramic pastes: the former relates to the composition of the clay and the latter to its content in Ca and Mg, which are highly variable depending on the geological origin of the raw materials. The plot shows that the composition of the ceramic bodies is similar in all the examples analysed and that there are no important changes across the groups or across the archaeological phases under consideration. Nevertheless the group shows a considerable chemical variability, which is understandable considering the long period and diversity of sites studied. The only exceptions to the general homogeneity are the two samples of the cooking pot group, that otherwise do not show much affinity between themselves, and Sample 5.20, which has lower calcium content than the other samples. The case of the cooking pots is clearly due to their different clay recipe, which was designed for a completely different function (cooking) than the rest of the ceramics under consideration.

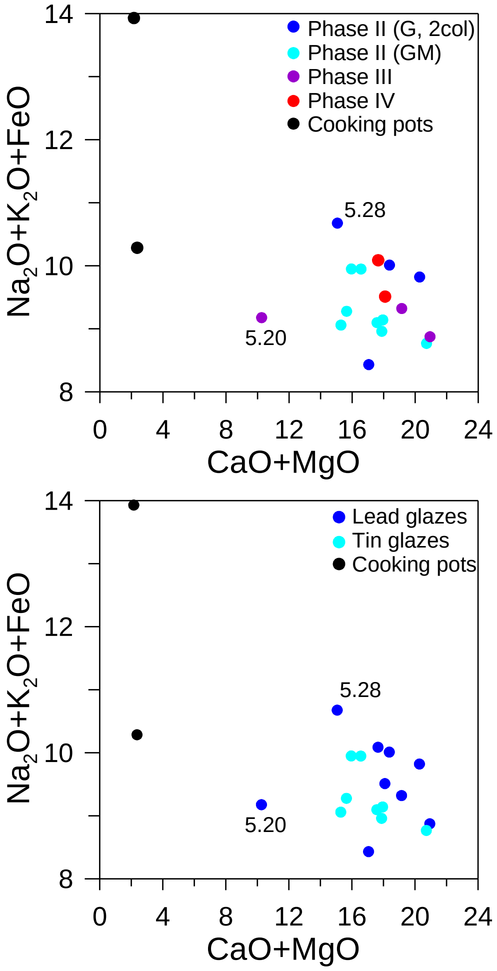


Figure 8. Na_2O+K_2O+FeO versus CaO+MgO plot of the ceramics bodies composition showing their relation by phases and by groups.

The situation is quite different when comparing the chemical composition of the lead glazes. In Figure 9 the Al_2O_3 vs CaO content of the lead gazes is plotted. Two distinctive groups directly related to the archaeological phases are observed. Phase II lead glazes (including two-coloured samples) contain less Al_2O_3 and CaO than those from Phase III and Phase IV, which tend to cluster together (with the exception of the glazes of the cooking pots, which have been discussed above). In fact we can see in Table 4 that Phase III and Phase IV lead glazes are also poorer in silica, containing not only more calcium but also more potassium, magnesium and iron than Phase II glazes. Moreover, when plotting in the same plot the lead glazes from the reverse of the Green and Manganese decorated tin glazed vessels, two of the samples (NS77 and NS82, yellow-coloured) group with the Phase II glazes while the other two (NS79 and NS83, green-coloured) with the Phase III and IV glazes. However, as indicated above, the PbO content of those glazes is also very different, and only four lead glazes have been analysed. Therefore more shards should be analysed to conclude anything regarding these glazes.

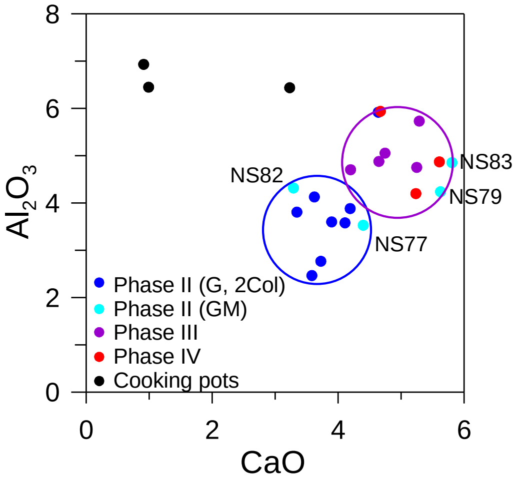


Figure 9.Al_2O_3versus CaO plot for the lead glazes. We can see that the Phase IIG and 2Col lead glazed wares contain less Al_2O_3 and CaO than those of Phase III and IV. Among the lead glazes present in the undecorated side of the tin glaze GM decorated wares, two samples, NS83 and NS79 group with the Phase II group while NS77 abs NS82 with the Phase III and IV group.

With the aim of exploring further the relation between the chemical composition of the ceramic bodies and glazes, the composition of the glazes was renormalized after removing the PbO and the colourants CuO, SnO_2 and MnO_2. The corresponding SiO_2*, K_2O*, CaO* and FeO* values are plotted versus Al_2O_3* (solid dots) in Figure 10 together with the original ceramic bodies composition (open dots). Removing colourants and renormalizing the composition is often performed in order to compare the glazes to which the colourants are added; moreover, glazes react with the clay bodies, which are in turn dissolved to some extend in the glazes. Consequently comparing the composition of the glazes after the removal of colourants and PbO gives information about the addition of sand and other compounds in the glaze applied over the ceramic and also of the amount of clay paste dissolved in the glazes. The SiO_2* vs. Al_2O_3* plot shows that the SiO_2* content of the glazes is higher than expected by simple dissolution of the body or addition of clay into the glaze (the renormalized glaze and ceramic data should coincide only if PbO and clay were used in the glazing mixture) (Walton and Tite 2010). This demonstrates the use of a mixture of SiO_2-rich sand and PbO for the glaze. This can also be seen in the comparison of the data with the correlation line drawn for SiO_2* vs. Al_2O_3* from the glazes. The figure also shows that the amount of SiO_2 originally added to the glaze mixture is larger for Phase II than for Phase III and Phase IV glazes. Finally, the mismatch between the SiO_2* vs Al_2O_3* line with respect to the ceramic body contents shows (see the arrow marked in the figure) that the amount of Al_2O_3 added by the dissolution of the clay paste is relatively small.

A similar behaviour is observed for the potassium content in the glazes. In the glaze mixture, potassium is present in larger amounts than expected by simple dissolution of the clay body or addition of clay. Therefore we can conclude that the sand added contained potassium feldspars (this explains the excess of aluminium and potassium in the glazes than expected by dissolution of some clay paste), probably as impurities. In fact the glazes of Phase II show lower potassium in the glaze formulation than the other phases. This could indicate that the sand added to the glaze mixture during Phase II contained more quartz, while in Phases III and IV contained more potassium feldspars.

The glazes also contain some calcium (and magnesium), which is explained simply by the dissolution of clay paste in the glaze. The same is observed for the iron although we cannot rule out the addition of iron in form of clay. Three samples show clear intentional iron addition not related to the dissolution of the clay paste: the brown glaze 5.24 B (Tejoletes, Phase III) and the honey glazes from Tejoletes (5.20 H, Phase III) and from the Two-colour glaze 5.29 H (Manzanil, Phase II).

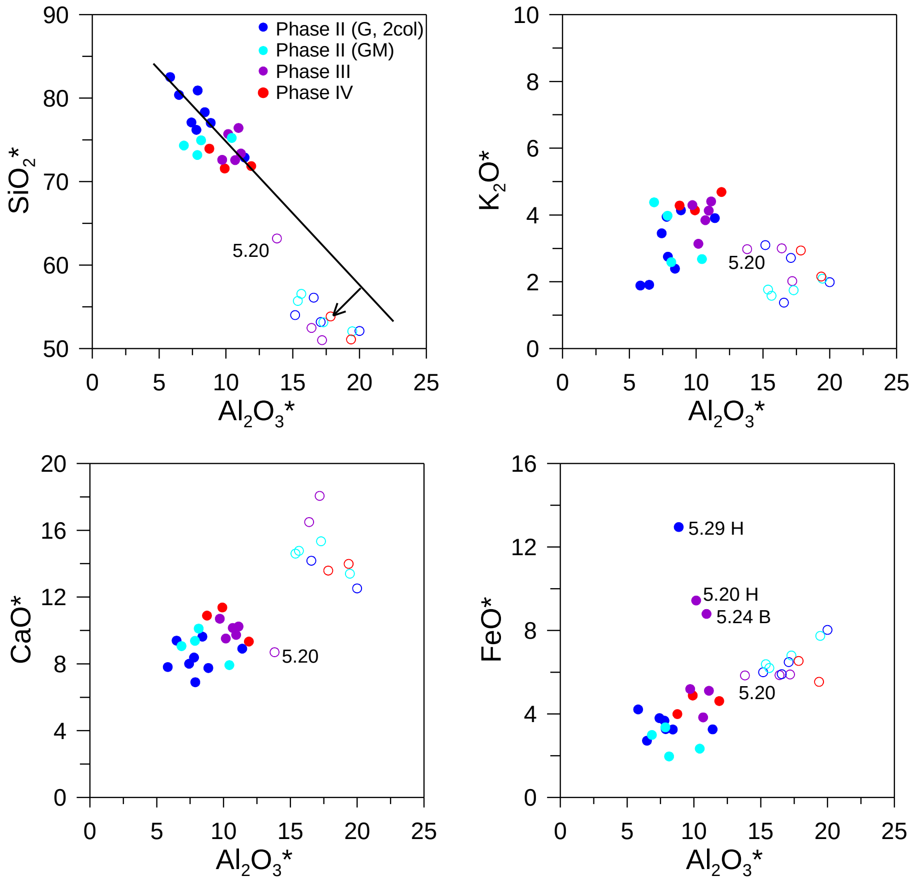


Figure 10.Comparison between the chemical composition of the ceramic pastes (open dots) to those of the lead glazes renormalized after removing PbO and the colourants CuO and MnO$_2$ (solid dots).

5.2. Some archaeological considerations

The analysis presented in this study offers interesting information about the technology of production of the earliest glazes of al-Andalus in the particular context of the Vega of Granada. With the exception of the cooking pots and Sample 5.20, which shows a clearly different features, all the vessels analysed have a similar composition of the ceramic body across all the groups and archaeological phases. This may indicate that the raw clay used for the ceramic body comes from the same area or at least very similar areas from the geological point of view. Since the Vega of Granada shares a common geological history with extensive regions of south-east Spain and the North of Africa, this observation may encompass potentially separated spots. However, the detection of gold particles in the ceramic body certainly makes the composition of the clay recipe of these ceramic vessels quite different from what has been documented so far from the analysis of contemporary productions: the 10th century green, honey and brown lead glazed wares and green and manganese decorated tin glazed wares from the workshop of San Nicolás in Murcia (Molera et al. 2001a). The ceramic pastes from the San Nicolás workshop are also calcareous, but containing lower aluminium and higher calcium and magnesium (10.9%Al_2O_3, 18.3%CaO and 3.6%MgO) than those of the Vega of Granada, and not gold particles. The latter constitutes a good argument to suggest the manufacture of these glazed vessels in the area of Granada. An Arab scholar, Ahmad al-Rāzī, wrote in the 9th century CE that one of the rivers of Granada, Darro, was a source of gold fine grains (Catalán and De Andrés 1974: 26). In fact, gold mining in the river Darro of Granada is attested between Antiquity and the Modern period, and it is related to the name of the river itself (Martín 2000). Consequently, if the clays were extracted from deposits in the river bank, this could explain the presence of gold particles. However, more extensive analyses of the source areas should be conducted in order to clarify this.

Glazes are not so homogeneous. Although in all the cases the lead glazes were obtained by mixing lead oxide with sand, there is a distinctive composition of the lead glazes of Phase II in comparison with those glazes of Phases III and IV, which are in turn similar between themselves. It has been noted above that there is comparatively a higher silicon content and lower potassium in the lead glazes of Phase II in comparison with those of Phases III and IV, which indicate the use of more quartzitic sand in the glazes of Phase II and more feldspatic sand in those of Phases III and IV.

The lead glazes appear calcium and aluminium richer than those found in the 10th century workshop of San Nicolás in Murcia, which are poorer in calcium and aluminium (between 3% and 3.5% CaO, 2.8% and 3.3 % Al_2O_3 depending on the colour of the glazes) (Molera et al 2009) and the green colour of the glazes were produced using Fe^{2+} instead of copper Cu^{2+}. The analysis of San Nicolás glazes showed the use of a mixture of PbO and sand and some iron oxide for the production of the lead glazes. Moreover, the study of San Nicolás demonstrated that the glazing mixture was actually a pre-fired ground lead glass which was applied over the biscuit fired ceramic (Molera et al 2001a). In the case of the Vega of Granada, there are no archaeological evidences to support the use of pre-fired glass as glazing mixture, but it cannot be ruled out, although the presence of calcium and other elements associated to the dissolution of ceramic into the glaze and the limited glaze-ceramic interaction suggest the use of a raw glaze mixture applied over biscuit fired bodies.

The tin glazed wares show specific characteristics. They are often glazed on both sides, but the reverse is glazed with a transparent yellow or greenish lead glaze. The tin glazes are not very well preserved, showing conchoidal cracks with falling flakes. Those that could be analysed show large amounts of cassiterite (SnO_2) in the white glaze. Green and brown decorations were applied over the tin glaze, using a mixture of colourant in large amounts (MnO_2 or CuO) with PbO, but without SnO_2. Copper appears completely dissolved in the glazes but manganese gave rise to the formation of manganese-bearing pyroxene and lead manganese silicate needle-like crystallites on the glaze surface growing towards the ceramic interface. The compounds identified suggest a firing temperature of about 900ºC. The presence of those compounds is explained by the relatively high calcium content of the glazes.

Comparison with the caliphal San Nicolás workshop shows also a similar method of application. In San Nicolás (Molera et al. 2009) a pre-fired tin glaze, containing very characteristic lower amounts of potassium, aluminium and calcium (0.5% Al_2O_3, 1.1% K_2O, 1.5% CaO) and higher quantities of lead (53.8% PbO) than those observed in the present study (Molera et al 2001a, Molera et al 2009), was applied over biscuit fired ceramic bodies. The use of a pre-fired tin mixture applied over the biscuit fired ceramic is also observed in the Vega of Granada wares, but the composition is very different. One characteristic of the San Nicolás tin glazes was the extremely small size and the very homogeneous distribution shown by the cassiterite crystallites in the white glaze, indicative of a very conscious fritting of the glaze mixture (Molera et al 2001a). Moreover, in San Nicolás the colour decorations were pre-fritted mixtures of the pigment with lead oxide and were also applied on the top of the tin glaze, very much like what has been found about the Vega of Granada wares. The presence of manganese crystalline compounds was also determined in San Nicolás, but their nature was different (braunite, Mn_7SiO_{12} haussmanite Mn_3O_4 and kentrolite $Pb_2MnSi_2O_9$) (Molera et al 2013) than that of those found in the Vega of Granada (bustamite, $Ca_{0.4}Mn_{1.6}Si_2O_6$). This is a consequence mainly of the different composition of the glazes, in particular of the high calcium content of the Vega of Granada glazes compared to those of San Nicolás. Both contemporary Green and Manganese decorated tin glaze production are very different both their chemistry and microstructure. But otherwise, the method of application of the glazes and decorations is similar.

6. Conclusions

The results of this study serve to characterize an assemblage of glazed wares from the Vega of Granada. This assemblage shows homogeneity in the composition of the clay body, with the exception of the cooking pots and one of the samples analysed, and in the way in which the glazes are applied. Either a pre-fritted mixture over the raw fabric, or a fritted glass over a biscuit fired fabric were applied to obtain the lead glazes (including cooking pots) whereas pre-fritted mixtures of the tin glazes were applied over the biscuit bodies. Brown and green pigments mixed with some lead oxide were painted on top of the tin glazes, indicating a common technology. The presence of gold particles in the ceramic bodies analysed reinforces this suggestion, and in combination with the historical documentation of the presence of gold fine grains in the Darro river of Granada, pinpoints a possible area of origin of the raw materials used for the ceramic.

Another result that is relevant is the change observed in the silicon and potassium contents of the glazes between Phase II and Phases III and IV. The explanation of this change is outside of the scope of this exploratory first study, and the authors will only remark the need of further studies to understand it.

Nevertheless, the change occurs between Phase II and Phase III, which is when other technological developments are determined in the archaeological record. The transition between Phase II and III has been already noted as a key turning point in the production of ceramics in the Vega of Granada. Changes are also in the production of other types of ceramics, although they are not so well defined yet (Carvajal López 2012). For the cooking pots, it seems to be the moment when a model of production based on dispersed rural workshops changed into another urban-based centralized model (see Carvajal López and Day 2013; 2015). The Vega of Granada was set initially as a network of loosely connected individual systems attached to a rural settlement; during the transition between Phase II and Phase III they start to coalesce in several areas and to form interconnected and complex systems that will define the agricultural area of cities like Granada itself (Jimenez Puertas 2007; Jimenez Puertas and Carvajal López 2011). In the field of political relations, Phase III marks the beginning of a closer relation of the Vega of Granada with the power of Cordoba and with the growth of towns like Ilbira and Granada (Carvajal López 2008; 2013). Therefore the changes observed in the glazes through time are part of a clear archaeological and historical pattern of reorganization of the landscape around towns. As artisans tended to concentrate in towns, all these transformation may have had an impact in their access to different resources and raw materials, and therefore there is a chance that this is one of the reasons for the technological changes noted in glazed ceramics. However, the particular reasons for the change in the glaze composition are not clear yet, and more research is required to understand how the lead glazes of Phase III and IV fit in the overall picture of transformation.

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