# REVISITING THE ORIGINS OF ISLAMIC GLAZED POTTERY: A TECHNOLOGICAL EXAMINATION OF 8TH-10TH CENTURY AD CERAMICS FROM ISLAMIC LANDS

Moujan Matin

A Thesis submitted for the degree of Doctor of Philosophy

Keble College
University of Oxford
2016

# Abstract

The beginnings of Islamic ceramics have long been attributed to the opaque white glazed wares made in Iraq in the 9th century in response to the imported Chinese porcelain and stoneware. These Islamic glazes were known to have been opacified by tin-oxide and much work has been undertaken to characterise their development and spread. However, little has been done to explain the origins of this technology and its invention in Iraq.

This thesis takes a new approach to investigating the beginnings of Islamic glazed pottery. It examines the technical aspects of early Islamic glazed wares from the 7th to 10th century AD from the Eastern Mediterranean to Central Asia. A significant number of sherds from Fustat, Aqaba, Al-Mina, Raqqa, Samarra, Basra, Kish, Susa, Nishapur, Merv and Samarqand were sampled and analysed using a scanning electron microscope equipped with energy dispersive spectroscopy (SEM-EDS). Particular focus was placed on the opaque yellow glazes from the Eastern Mediterranean and Mesopotamia (7th-9th century) and the opaque white glazes from Mesopotamia and Central Asia (9th -10th century). The analyses showed that the opaque yellow glazes and the opaque white glazes were respectively the result of lead-tin-oxide particles in high lead glazes, and tin-oxide particles in lead-alkali and alkali-lead glazes. Using experimental replication, it was found that at temperatures around 900°C, and in the presence of alkalis and alkaline earths, lead-tin-oxide particles convert to tin-oxide and that the colour of the glaze changes from yellow to white. It was therefore argued that the opaque yellow glazed wares of the 7th/8th-century Eastern Mediterranean represent a precursor to the opaque white glazed wares that flourished in Iraq in the 9th century. The results have helped change the way the beginnings of Islamic ceramics are viewed: origins in the 7th/8th century AD rather than the 9th century AD, first developed in Egypt or the Levant rather than Iraq, and indigenous rather than Chinese-inspired. The eastward spread of Islamic glazed wares to Iraq in the 9th century follows the move of the Islamic capital from Damascus, Syria, to Baghdad, Iraq, and the further spread of this technology east and west can now be traced.

*To my parents,*
*Afsoon and Mehran,*
*for their sacrifices.*

# Acknowledgements

My deepest gratitude goes first and foremost to my supervisors, Prof Oliver Watson, Prof Michael Tite and Prof Mark Pollard, for giving me the opportunity to work with them on this research and for their directions. I cannot thank Prof Oliver Watson enough for his trust and confidence and for his boundless help and support. He walked me through the world of Islamic ceramics with much patience and expertise and provided me with unrelenting encouragement. Never will I forget the joy I found in travelling and visiting some of the collections of Islamic art with him, and in hearing his views on art, literature and life. As this thesis journey comes to an end and as he begins his retirement, I wish him the very best. My special thanks also go to Prof Michael Tite who assiduous and tirelessly guided me during the course of my research and the write-up of the thesis. He has been a constant source of enthusiasm, encouragement and good advice. Prof Mark Pollard has been very generous with his time and advice and his support of the thesis is much appreciated.

My sincere thanks also go to Prof Trinitat Pradell and Prof Judit Molera, who gave me access to their laboratory and research facilities at the Polytechnique University of Catalonia (UPC) and the University of Vic, Spain, and who helped me during the replication experiments. Prof Trinitat Pradell also provided me with the opportunity to carry out SR-Micro-XRD analysis as part of her research team at the ALBA Light Facility, Barcelona, for which I am very grateful. I am also thankful to Chris Doherty, Prof James Allan, Prof Ian Freestone, Prof Jeremy Johns and Prof Nigel Wood for their many helpful suggestions.

My sincere gratitude is extended to professors, curators and researchers at various museums and institutions for their permission to access collections and their support throughout the research project. Without their help it would not have been possible to conduct this research. Among them are Dr Mariam Rosser-Owen (Victoria & Albert Museum, London), Dr Julia Gonnella (Museum für Islamische Kunst, Berlin), Dr Andrea Becker (Museum für Islamische Kunst, Berlin), Dr Yannick Lintz (Musée du Louvre), Ms Delphine Miroudot (Musée du Louvre), Prof Donald Whitcomb (OI Museum, Chicago), Dr Jack Green (OI Museum, Chicago), Ms Helen McDonald (OI Museum, Chicago), Dr Shiela Canby (The Metropolitan Museum of Art, New York), Ms Annick Des Roches (The Metropolitan Museum of Art, New York), Dr Jean-Francois Lapérouse (The Metropolitan Museum of Art, New York) and Dr Federico Carò (The Metropolitan Museum of Art, New York).

Moreover, I acknowledge the various fundings who supported my research and study over the past three years. Namely, The Soudavar Memorial Foundation, The Barakat Trust, The Iran Heritage Foundation, The Collection Research Grant (OI Museum, Chicago), The Oxford School of Archaeology Bursary, The Meyerstein Fund and The Keble Association Fund.

On a personal note, I would like to thank my parents, Afsoon and Mehran, for their constant support and understanding. All I have accomplished and will accomplish are only possible due to their love and sacrifices. This thesis is dedicated to them.

Last but not least, my heartfelt thanks to my husband, Hamid, who continuously encouraged me, assisted me and much cared for me during the process of writing the thesis. His unyielding companionship form early morning laboratory experiment to late night writing-up has been precious to me.

# CONTENTS

# LIST OF FIGURES

# LIST OF TABLES

# GLOSSARY OF TERMS

| | |
|---|---|
| **Abbasid caliphate** | The third of the Islamic caliphates who ruled over the vast Islamic empire from 750 AD until the fall of Baghdad to the Mongols in 1258 AD. |
| **Alkali glaze** | A glaze that contains less than 2 wt% PbO in its composition. |
| **Alkali-lead glaze** | A glaze that contains 2-10 wt% PbO in its composition. |
| **Assemblage** | An archaeologist's grouping of artefacts (such as pottery) from a site by form or function, assumed to represent the material culture of a single occupation or cultural episode. |
| **Buffer layer** | The layer found at the body-glaze interface, with composition and properties intermediate between body and glaze. |
| **Body** | Clay or a mixture of clay and inclusions that is suitable for forming vessels. The term is used as opposed to any additional parts of a vessel such as slips and glazes. |
| **Calcination** | Heat treatment in air or oxygen. Calcination of a metal results in oxidation of the metal. |
| **Calx** | A fine powder formed as the result of calcination of one or more metals. |
| **Ceramic** | Clay products made permanent by heat. |
| **Coarse-grained** | A clay or body whose inclusions are more than 200 µm in size. |
| **Colourant** | Metal oxides that contribute colour to glazes or glasses. |

| | |
|---|---|
| **Coptic glazed ware** | Abbreviated CGW. A group of glazed ceramics found in Eastern Mediterranean during the 7th/8th century AD that occur immediately after the Roman sequence. |
| **Crystal** | A solid whose constituent atoms have a regular repetitive arrangement in space. |
| **Crystallite** | A microscopic crystal. |
| **Fabric** | The texture of a ceramic including clay, inclusions, and pores and excluding surface treatment. Fabric studies entail taking a sample and then grinding one side flat before examination under an optical or an electron microscope. See ***Fine-grained***, ***Medium-grained***, ***Coarse-grained***. |
| **Fine-grained** | A clay or body whose inclusions are less than 50 µm in size. |
| **Glaze** | A layer of glass fused onto the surface of a ceramic body. See ***Alkali-lead glaze***, ***Lead-alkali glaze***, and ***High lead glaze***. |
| **High lead glaze** | A glaze that contains more than 55 wt% PbO in its composition. |
| **Inclusion** | A mineral present in a clay, either naturally or deliberately added by the potter. |
| **Inglaze** | Decoration applied on the surface of an unfired glaze so that it matures simultaneously with the glaze during firing. |
| **Lead-alkali glaze** | A glaze that contains 35-55 wt% PbO in its composition. |
| **Matte** | A matte appearance is dull, without a shine or gloss; normally attributed with ceramic decorations with minimum or no vitreous phase, such as ***Slip***. |
| **Maturity** | 1) A mineralogically mature sediment is one which has undergone prolonged weathering and transport, and is one which consists largely of the stable minerals, particularly quartz, 2) Glazes are said to mature in the final stages of firing by developing qualities of bonding (to the body) and texture. See ***Soaking time.*** |
| **Medium-grained** | A clay or body whose inclusions are between 50-200 µm in size. |

| | |
|---|---|
| **Microstructure** | The fine structure of a ceramic cross section examined with an electron microscope. See ***Scanning electron microscope***. |
| **Opacifier** | A material added to a glaze, glass or enamel to render it opaque. Oxides of tin, antimony, zirconium and titanium are amongst the most common opacifiers in modern ceramic manufacture. |
| **Opaque** | A quality of a glaze or glass which cannot be seen through. See ***Transluscent*** and ***Transparent***. |
| **Overglaze** | Also known as *Onglaze*. Decoration applied to pottery after it has been glazed; the ware is again fired and the decoration fuses into the glaze. An example in Islamic ceramic tradition is lustre decoration. |
| **Scanning electron microscope** | Abbreviated SEM. A microscope in which a finely focused beam of electrons is scanned across a specimen, and the electron intensity variations are used to construct an image of the specimen. This type of microscope can effectively achieve magnifications from 200 to 35,000 times. Over the last decades, scanning electron microscopy has become almost a standard technique in examining archaeological glazes. |
| **Slip** | A fluid suspension of clay and water used for coating clay bodies to give colour or a smooth texture to the surface; often used synonymously with *Engobe*. |
| **Soaking time** | A term used in kiln firing schedules when the top temperature is maintained at the same level for a given period to allow for glaze to mature. |
| **Transluscent** | Not fully ***Transparent***, because of the numerous small air bubbles and undissolved quartz and/or feldspar grains remained in an ***Underfired*** glaze. |
| **Transparent** | Glazes are transparent if they transmit light without scattering or diffusion, so that the underlying body can be seen clearly. |
| **Umayyad caliphate** | The second of the Islamic caliphates who ruled over the Islamic empire from 661 AD until the coming into power of Abbasids in 750 AD (although an Umayyad dynasty continued in Spain, known as the Western Caliphate). Under the Umayyads, the Muslim Empire increased at a rapid rate, stretching from the borders between France and Spain in the west, to the borders of China and India in the east. |

| | |
|---|---|
| **Underfired** | The state of a glaze being insufficiently fired, normally including air bubbles and undissolved quartz and/or feldspar grains. |
| **Yellow glaze family** | Abbreviated YGF. A group of yellow glazed ceramics that make up the majority of ceramic finds in Tell-Aswad, Raqqa. This group of ceramics represents a further development of**.** ***Coptic glazed wares***. |
| **X-ray diffraction** | Abbreviated XRD. An analytical technique used to identify crystalline substances. The scattering of X-rays by the atoms of a crystal produces a diffraction pattern which gives information on the structure of crystal. |

# Chapter 1

# INTRODUCTION

The abundance of glazed ceramics is a diagnostic feature of early Islamic culture in archaeological records. The range of glazing techniques used during the preceding Parthian and Sassanian periods in Iraq and Iran was limited to transparent monochrome green or turquoise types (Hill 2004; Pace *et al*. 2008). In the Early Byzantine period in the Eastern Mediterranean, transparent green, orange/yellow and colourless glazes were used to cover vessels (Rice 1930; Armstrong 2001; Vroom 2004). The beginning of the Islamic period brought about stylistic and technological revolutions in the production of glazed ceramics; a manifestation of 'Islamic' identity made possible through technical advance.

Archaeological excavations since the early twentieth century have yielded evidence of the first phase of Islamic glazed ceramics in Egypt, Jordan, Palestine and Syria from as early as the 7th-8th centuries, namely Coptic Glazed ware (CGW), Hijazi ware and Cream ware (Whitcomb 1989); and the further developments in the 8th-9th centuries, known as the Yellow Glaze Family (YGF) (Watson 1999). Despite their significance, the mention of these wares hardly goes beyond archaeological reports and classifications and they remain largely unexplored. In fact, these wares were overshadowed by the results of excavations in Samarra, Iraq, during 1912-13. The ceramic findings, published by Sarre (1925), had an enlightening, yet somewhat blinding, impact on research in the field of Islamic ceramics. A diverse range of exceptionally fine ceramics covered by opaque white base glazes and influenced, in shape and decoration, by imported Chinese Tang porcelain and stoneware were found in the Abbasid capital city of Samarra, all securely dated to the 9th century AD. As a result, it became widely assumed that Islamic 'fine' glazed pottery was invented first in Iraq or western Iran in the 9th century, under the influence of imported Chinese wares, and from there it was spread to east and west. Although such an assumption brought new interest into the field, an adverse consequence was that it relegated to a peripheral position other types of early Islamic ceramics.

In a recent paper, Watson (2014) critically discussed the notion of Iraq being the birthplace of fine glazed Islamic ceramics. He suggested that the existence of a market for the production of fine opaque white glazed wares in 9th century Iraq in response to the substantial numbers of imported Chinese ceramics is a testament to a long established market for fine quality wares. This market could not have been developed by pre-Islamic and early Islamic green glazed jars in Mesopotamia as these large functional vessels could have hardly been considered 'fine tableware'. Watson pointed to Egypt, Jordan, Palestine and Syria as the first producers of fine Islamic glazed tablewares since the 7th/8th centuries AD. He argued that the manufacture of fine ceramics in the form of Roman slipped or glossy wares had roots in these Eastern Mediterranean lands. The Roman wares ceased production on the arrival of Islam but demand for fine tablewares continued and was satisfied by the production of Coptic Glazed ware (CGW), Hijazi ware and Cream ware, and later by the Yellow Glaze Family ware. These wares, rather than the Samarra pottery, form the first chapter in the history of Islamic glazed ceramics and demonstrate the development of a pre-9th-century market for fine ceramics in the Near East.

A further aspect of Watson's proposal was that the rapid production of opaque white glazed wares in the 9th century Iraq/western Iran in imitation of Chinese wares required the availability and readiness of the relevant technologies; most important of all, the technical know-how of 'opaque glazing'. Again, this innovation could not have been linked to the pre- and early Islamic monochrome green/turquoise glazes as those were translucent glazes at their best and showed no evidence of 'deliberate' opacification (McCarthy 1996, 51). Attention was once again directed to the early Islamic Eastern Mediterranean wares: Coptic Glazed wares were decorated with matte opaque yellow glazes and the Yellow Glaze Family wares covered by opaque yellow or opaque white glazes.

The present study aims to evaluate this new hypothesis, to explain the beginnings of opacification of glazes and to investigate the development of opaque glazes throughout the Islamic world from the 7th to 10th centuries AD. In the following sections, the CGW and YGF wares are discussed and the relationship between them in design and glazing techniques is addressed. Furthermore, a review of the opaque glazed wares of the Samarra-type, as well as similar types in the Eastern Iranian World and Central Asia is given in order to establish a framework for evaluating the evolution of opaque glazes throughout Islamic lands during the early Islamic period.

### 1.1. Coptic Glazed Wares (CGW): Early Steps

Coptic Glazed Wares (CGW) is a term first used by Rodziewicz (1976a, 63-4; 1976b, 209; 1978; 1983) to describe a certain group of ceramics found in Alexandria, Egypt, that occur immediately after the late Roman levels in the archaeological sequence, in layers containing the most ancient Islamic coinage. Rodziewicz suggested Egypt as the original production centre for the CGW given that their shapes and modes of decoration closely imitate those of the local late Roman wares.

The CGWs are classified into two distinct decorative types: first, a painted type covered with an overall translucent amber glaze, and second, a matte, opaque yellow, green, and/or brown glazed ware. The latter is the focus of Chapter 2. The characteristic of this type is that, rather than an overall decoration, it is covered by glaze in discrete bands with some areas left unglazed (Fig. 1.1.a). Such vessels were recorded also from Fustat (Scanlon 1998 and Gayraud 2009), Abu Mina (Engeman 1990), Tod (Joel 1992), Aqaba (Whitcomb 1989 and Whitcomb 1991, 48-56), Tiberias (Stacy 1995, 164-166, 286), Pella (Walmsley 1995, 664, 667-8, and Walmsley 1997, 2-3), Yoqne'am (Avissar 1996) and Caesarea (Arnon 2008, 35 and 400) (Fig. 1.2).

### 1.2. Yellow Glaze Family Wares (YGF): A Transition

Further development and use of CGW decorative techniques is characterised in ceramics excavated in, amongst other sites, Tell Aswad, near Raqqa (Watson 1999), Al-Mina (Vorderstrasse 2005, 75-8), Qinnasrin (Whitcomb 1999, 81-3), Antioch (Waage 1948), and Tarsus (Bagci 2016) (Fig.1.2). The wares were labelled 'Yellow Glaze Family' (YGF) by Watson (1999, 81) after his classification of the sherds from Tell Aswad. Despite its name, the YGF encompasses not only opaque yellow glazed wares but also opaque white glazed wares with green and/or brown decorations (Fig. 1.1.b). The latter is, however, distinguished from the Samarra type wares discussed below in Section 1.3 (Watson 1999, 82-3). Of all the ceramics found in the aforementioned sites, YGF wares make up one of the main types in the corpus; three-quarters of the excavated glazed sherds in Tell Aswad, for instance (Watson 1999).

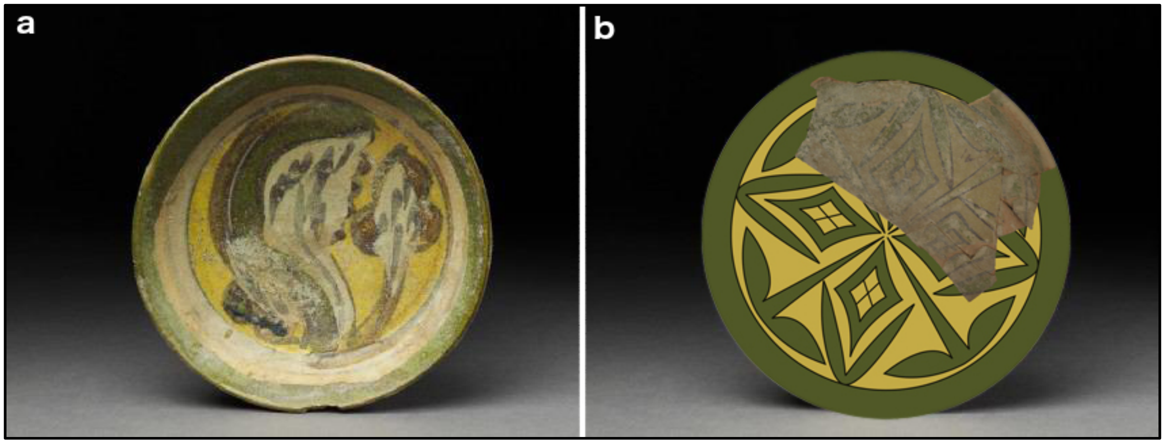


Figure 1.1. *(a) Coptic Glazed Ware from Fustat (Ashmolean Museum, University of Oxford, acc no. EA1974.48); (b) Yellow Glaze Family sherd from Al Mina (Victoria and Albert Museum, acc no. C242A-1937).*

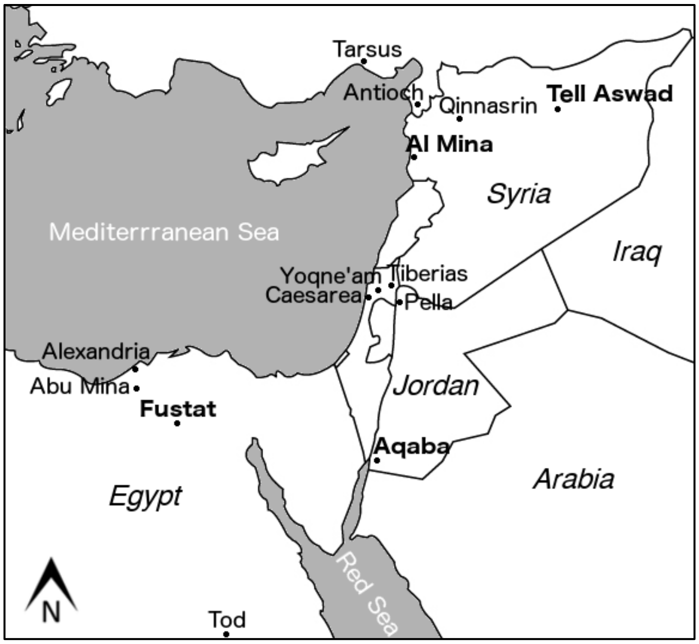


Figure 1.2. *Map showing location of sites with CGW and YGF wares referred to in the text. Those from which ceramic samples were obtained are labelled in bold (see Chapter 2).*

## 1.3. Samarra-Type Wares: The Flourishing Era

The palace city of Samarra, Iraq, located on the east bank of the Tigris (Fig. 1.3), was the second capital of the Abbasid Caliphate after Baghdad. It served as the capital city for a brief period between AD 836 and 892 (AH 221-279). Samarra has remained a key point of reference in Islamic archaeology, primarily for the richness of the material evidence that it represents from a short period of the Abbasid period.

The term ‘the Samarra pottery’ (also 'the Samarra Horizon Pottery’) was first described in detail by Whitehouse (1979) and designates a range of pottery found during the German excavations conducted by Ernst Herzfeld and Friedrich Sarre at Samarra before the First World War. Delicately fine with radically new range of decorations, the corpus represents a turning point in the history of ceramic manufacture. Characteristic wares were covered by a background opaque white glaze, sometimes plain but generally with inglaze or overglaze decoration. Opaque white glazed wares have been widely distributed throughout and beyond the Near East, from Spain in the west to Thailand in the east, and have been found, amongst other sites, in Kish, Hira, Siraf, Sirjan, Rayy and Istakhr (Fig. 1.3).

Samarra pottery has been a subject of various classifications on the basis of the techniques of decoration and styles (Kervran 1977, 88, 152-3, Table II; Northedge and Kennet 1994; 25-34; Northedge 2001, 211-213; Priestman 2001, 90-95). The classification by Northedge (2001) suggests nine different groups of wares: white glaze with cobalt blue decoration (Fig 1.4.a), white glaze with cobalt blue and green or brown decoration (Fig 1.4.b), grey glaze, plain opaque white glaze, splashed glazed (Fig 1.4.c), relief moulded wares (Fig 1.4.d), lustre decoration (Fig 1.4.e & f)) on white glaze, Sgraffiato, and iron-brown glaze. A more detailed sub-division of these wares has been suggested by Priestmann (2011, 90-95) based on the study of a selection of key archaeological assemblages at Jazirat al-Hulaylah, Manda, Siraf and other southern Iranian sites. The proposed categories include monochrome white, turquoise splashed, turquoise and black splashed, monochrome gold lustre, monochrome ruby lustre, polychrome lustre, white on cobalt, cobalt on white, cobalt and green, polychrome splashed, colour painted, white on black, black on white, mononchrome turquoise, and black on turquoise. These sub-groups are used in addition to Northedge (2001) classification to describe the sherds studied in Chapter 3.

Figure 1.3. *Map showing location of the Near Eastern sites with opaque glazed wares referred to in the text. Those from which ceramic samples were obtained are labelled in bold (see Chapter 3).*

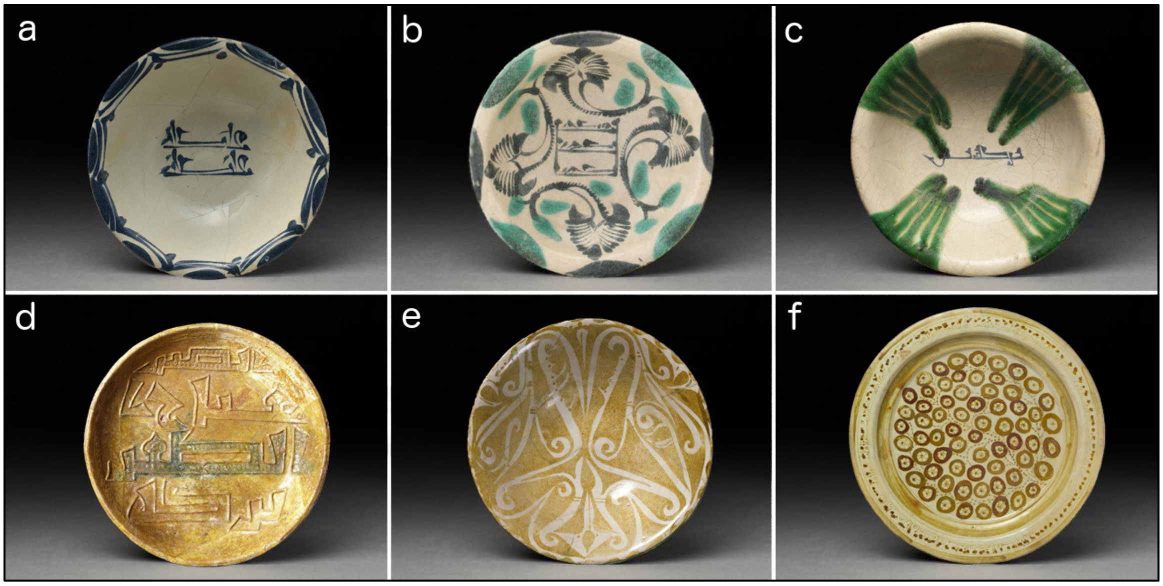


Figure 1.4. *Typical Samarra-type pottery: (a) white glaze with cobalt blue decoration (the Metropolitan Museum of Art, acc. no. 63.159.4); (b) white glaze with cobalt blue and green decoration (Freer and Sackler Galleries, acc. no. F2000.2); (c) splash glazed (the Metropolitan Museum of Art, acc. no. 30.112.46); (d) relief moulded ware (The David Collection, acc. no. 50/1999); (e) monochrome lusterware on opaque white glaze (The David Collection, acc. no. 26/1962); (f) polychrome lusterware on opaque white glaze (the Metropolitan Museum of Art, acc. no. 1976.309).*

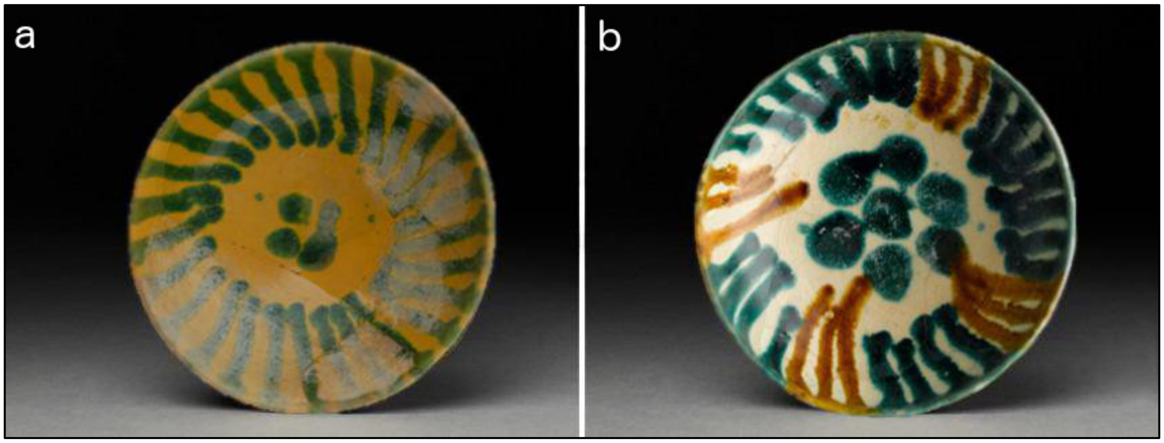


Figure 1.5. *Two bowls from Susa, Iran; reproduced from Watson (2014, 141): (a) green decoration on opaque yellow glaze (Musée du Louvre, acc no. MAO S.576); (b) green on brown decoration on opaque white glaze (Musée du Louvre, acc. no. MAO S.439).*

Another key archaeological assemblage of Samarra-type pottery occurs in significant quantities in Susa, Iran (Koechlin 1928; Rosen-Ayalon 1972; 1974; Kevran 1977; 1979; Guillermina *et al*. 2005). The assemblage also includes local examples of YGF wares with green and/or brown decorations. Characteristic types are decorated by chequer board, hatched or dotted patterns. Moreover, bowls with runs of green and/or brown from the rim are found amongst the opaque yellow and opaque white wares of Susa (Fig. 1.5), which appear similar to the splashed glazed wares from Samarra. The similarities are evident both in shape, essentially with flat bases or low footrings and steep walls, and in decorative patterns. On this ground, Watson (2014) argued for connections between the Eastern Mediterranean opaque yellow glazed wares and the Mesopotamian opaque white glazed wares. The Susa assemblage is therefore important for the study of relationships between these two traditions.

## 1.4. Eastern Iranian and Central Asian Wares: A Continuation

Ceramics from the eastern Iranian world and Central Asia are most known by a class of pottery referred to as slipwares. Decorated by one or more coloured slips over a white background slip, and then covered by a transparent glaze, slipwares are amongst the most elegant, sophisticated wares of the early Islamic World (Fehérvári 2000, 50-63; Watson 2004, 205-245). Less popular were the opaque yellow or white glazed wares, generally covered by a base opaque yellow or white glaze with coloured over-glaze decorations. These opaque glazed wares represent a manifestation of the advances in glaze techniques in the Near East, which spread to Eastern Iran and Central Asia by the 9th/10th century AD. Attested by the pottery kilns found in Nishapur (Wilkinson 1959), Samarqand/Afrasiab (Shishkina and Pavchinskaja 1993, 35-44) and Tashkent/Chach (Burjakov and Filanovich 1999), a new ceramic industry was established in the eastern Islamic World, which was able to produce opaque glazed ceramics of various local decorative styles (Fig 1.6).

Figure 1.6. *Map showing location of the sites in Eastern Iran and Central Asia with opaque glazed wares referred to in the text. Those from which ceramic samples were obtained are labelled in bold (see Chapter 4).*

A problematic issue regarding the discussions on the Eastern Iranian and Central Asian wares has been the lack of a comprehensive typological investigation. Amongst the most thoroughly studied of the early Islamic period in Central Asia have been ceramics from Nishapur, Iran. Wilkinson (1973) identified twelve ceramic types in Nishapur, amongst which the local 'Opaque Yellow Ware' is rather similar in the glazing and decorative techniques to the YGF wares of Eastern Mediterranean and Mesopotamia (Fig. 1.7.a). Moreover, yellow decorations were applied on 'Buff Ware', 'Polychrome on White Ware' and 'Ware with Yellow-Staining Black'. However, their matte appearance, low thickness and poor quality seem to indicate direct application of the yellow pigment on the body, rather than formation of a glaze layer. Similar matte yellow decorated wares have been found in Merv (Herrmann *et al.* 1997; 1999; 2000; 2001) but their occurrence is less significant in the Uzbek sites of Samarqand (Shishkina 1979), Tashkent (Brusenko 1986) and Akhsiket (Henshaw 2010, 64-88). Here, glazed ceramics tend to be of the opaque white type with decorations in green, blue or lilac colours, referred to by the local term as ‘Ishkor’ wares (Rakhimov 2000, 133-4; Henshaw 2010, 84-6) (Fig. 1.7.b).

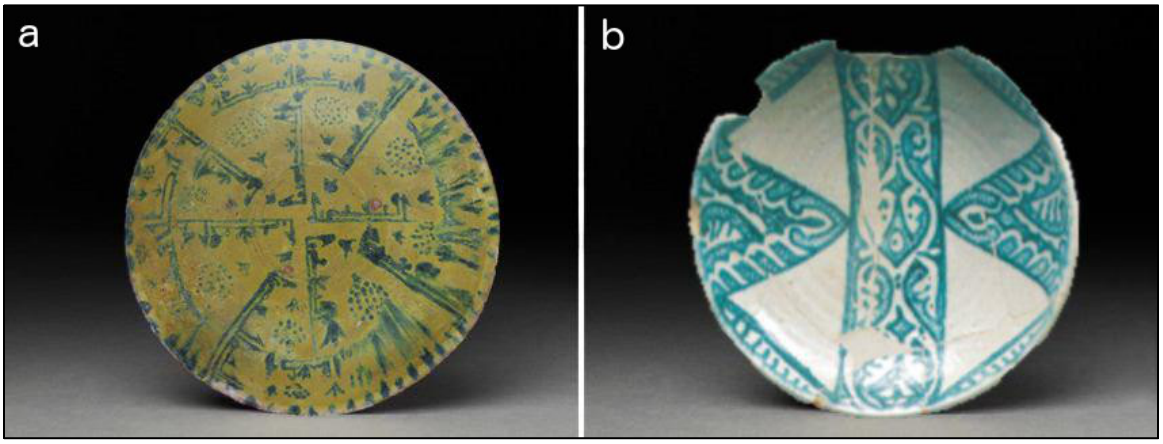


Figure 1.7. *Opaque glazed wares from Eastern Iran and Central Asia: (a) green decoration on opaque yellow glaze, Nishapur, Iran (the al-Sabah collection, acc. no. LNS 548 C [reproduced from Watson (2004, 178)]; (b) turquoise decoration on opaque white glaze, Samarqand, Uzbekistan [reproduced from Shishkina and Pavchinsakaja 1993, 51].*

## 1.5. Scope and structure of the thesis

This thesis examines the origins of Islamic glazed wares through scientific and technical study of early Islamic glazed pottery from the Eastern Mediterranean, Mesopotamia, and Central Asia from the 7th to 10th century AD. The following three chapters discuss ceramics from these three regions, respectively (Fig. 1.8). Each chapter represents results of examination of archaeological sherds. Relevant information on the production of these glazed wares that were described in historical treatises are discussed in Chapters 2 and 3. Chapter 2 also contains a detailed section on experimental replication of Eastern Mediterranean glazed ceramics.

Sherds from Fustat, Aqaba, Al-Mina, Raqqa, Samarra, Basra, Kish, Susa, Nishapur, Merv and Samarqand (Appendices C and D) were sampled and analysed using a scanning electron microscope equipped with energy dispersive spectroscopy. The microstructure of sherds consisted of three different layers around which the discussions are centred: The first, external layer was the glaze. The second was the glaze-body interface, referred to as the interaction layer or the buffer layer. The third layer was the body itself, containing various amounts of mineral inclusions embedded in a clay matrix. Particular emphasis was placed on investigating the chemical composition and microstructure of glazes and the various technical aspects of their production. Different glaze types were classified based on their general compositional types: high lead glazes (>55 wt% PbO), lead-alkali glazes (35-55 wt% PbO), alkali-lead glazes (2-10 wt% PbO) and alkali glazes (<2 wt% PbO). Bulk compositions of bodies have been also included in order to enable a rough association of various types of glazes to body fabrics. General descriptions of bodies were given according to the properties of the body matrix and dominant inclusions, e.g. 'fine-grained+microfossils'. In this context, fine refers to inclusion sizes less than 50 µm, medium to sizes between 50 to 200 µm and coarse to more than 200 µm.

Archaeometric work on the beginnings of Islamic glaze opacification had already been conducted prior to this thesis. These studies suggested a progressive development in the production technology of opaque glazes: beginning in the pre-Islamic period by under-firing of glazes, to gradually adding more opacifying particles during the 8th to 10th century AD (Kleinmann 1986; Mason and Tite 1997). According to this theory, the beginnings of Islamic opaque glazes can be explained by the deliberate application of a slip (i.e. a suspension of clay in water applied thinly over ceramic bodies) containing opacifying particles in early Islamic glazes and therefore the observation of opacifying particles at the interface between the body and the glaze. Subsequently, according to this theory, opacifying particles were further distributed in the glaze thickness. However, replication experiments conducted by Paynter *et al.* (2004) established that the concentration of opacifying particles in the interface layer is most probably the result of the settling of the particles in glazes rather than deliberate application of a slip. As a result, this theory has been largely abandoned and the question of the beginnings of Islamic opaque glazes is once again raised. The replication experiments conducted in this thesis offer a new perspective to explain the production of first Islamic opaque glazed wares. Building on the archaeological and historical data, the results provide new evidence on the beginnings and transfer of early Islamic ceramic technologies.

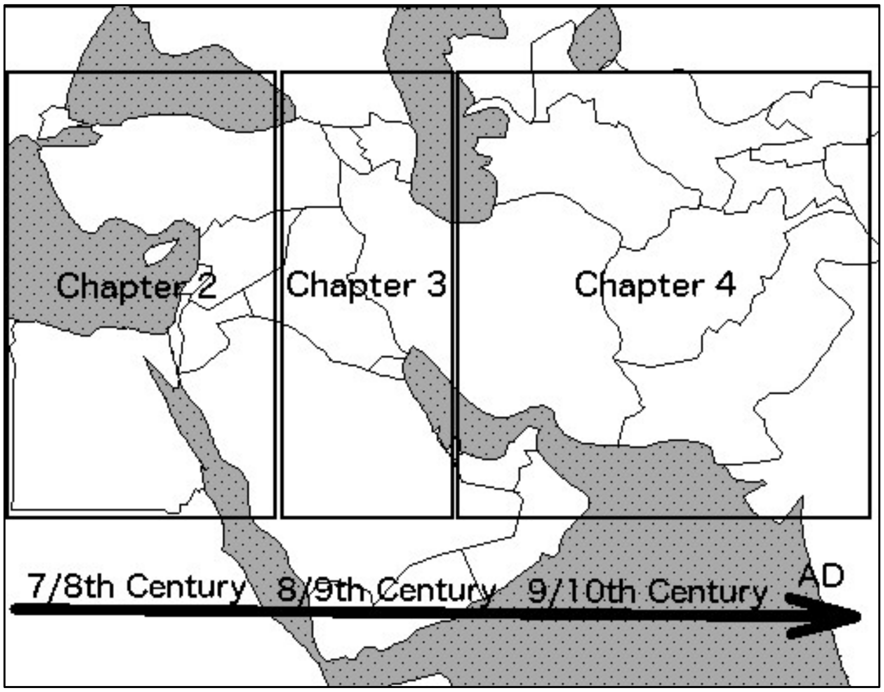


Figure 1.8. *Map outlining ceramics discussed in Chapters 2,3, and 4.*

# Chapter 2

# EASTERN MEDITERRANEAN: FIRST STEPS IN ISLAMIC GLAZED CERAMIC MANUFACTURE

The introduction of a technique such as glazed decoration, which comes to have such a strong identity with Islamic art, holds a special place for the study of cultural and technological change. The aim of this chapter is to provide a new insight on the technical evolution of early Islamic glazed ceramics in the Eastern Mediterranean, particularly Egypt and the Levant, and how it relates to the social and political processes in the early Islamic period. Through the study of the production technology, the results provide evidence suggesting that the beginnings of Islamic glazed wares has to be sought in the 7th/8th-century AD CGW and YGF wares of Egypt and the Levant, rather than the 9th-century white glazed wares of Iraq and western Iran (Whitcomb 1999; Watson 2014).

This chapter consists of three main parts. The first part examines the chemical composition and microstructure of the CGW and YGF. The focus is on glaze properties, but the main features of bodies are also discussed. The second part describes the reproduction of the opacifying lead-tin calx (oxidised forms of lead and tin metal(s)). In the third part, the opaque yellow and white glazes are reproduced using the replicated calx and other raw materials. The resulting data of these three parts are compared in

Section 2.4. These findings set the scene for examining the next stage of Islamic glazed ceramics manufacture in Iraq and western Iran during the 9th century in Chapter 3.

## 2.1. EXAMINATION OF ARCHAEOLOGICAL SHERDS

### 2.1.1. Review of previous chemical analyses

It was discussed in Chapter 1 that little attention has been given to the CGW and the YGF. For this reason, analyses of these wares are rare. Part of the results of analysis of a small number of the CGW and the YGF wares presented in this chapter as well as analysis of a comparable wares from Kish, Samarra and Susa was given in Tite *et al.* (2015).

### 2.1.2. Glazed ceramic sample

One CGW sherd from Fustat (the Alexander Kaczmarczyk collection at the Research Laboratory for Archaeology and the History of Art, Oxford), and forty YGF sherds from Aqaba (The Oriental Institute Museum, Chicago), Raqqa (Museum für Islamische Kunst, Berlin) and Al-Mina (Victoria and Albert Museum, London) were selected for SEM examination (Appendix A). All the samples were suitable for the investigation of bodies. However, as a result of weathering, the condition of glazes of only the Fustat CGW sherd and twenty of the YGF sherds was satisfactory for analysis. All the examined sherds were classified by the typology suggested by Watson (1999) for the Raqqa wares (Table 2.1).

### 2.1.3. Analytical techniques

Polished sections were examined for their microstructure and chemical composition using SEM-EDS (Appendix A). The Si-Kα and Pb-Mα peaks in the DLH1 glass standard were used as primary standards in analysing the opaque yellow glazes, and the Si-Kα and Pb-

Mα peaks in the Corning C glass standard in analysing the opaque white glazes.

### 2.1.4. Results

#### *Body*

Table 2.1 indicates that the body of the Fustat.1 CGW ceramic was produced from aluminium-rich clay. The microstructure shows a variety of mineral inclusions derived from intermediate igneous and metamorphic rocks embedded in a compacted clay matrix (Fig. 2.1). In contrast, bodies of Aqaba, Raqqa and Al-Mina sherds were produced from calcareous clays. The chemical compositions of ceramic bodies from Aqaba, Raqqa and Al-Mina are rather consistent and similar to those of the Southern Mesopotamian bodies discussed in Chapter 3. Their microstructure shows a variety of minerals, such as quartz, muscovite, feldspars and amphibole associated with basic and intermediate igneous and metamorphic rocks (Fig.2.1). The body fabrics of the sherds from Al-Mina contain microfossils which contribute to the calcareous nature of the bodies. The sample of kiln rod from Raqqa (Raqqa.22) contains slightly higher PbO contents which reflects the impregnation of the ceramic by lead vapour during firing (Table 2.1 and Fig. 2.1). The bodies may contain a maximum of 2% PbO. The PbO concentrations are particularly noticeable in sherds with weathered glazes. This may relate to the intrusion of pieces of weathered glaze into the bodies during polishing of the sample blocks.

#### *Glaze*

The chemical compositions of opaque yellow and white glazes are given in Table 2.2 and the distinct grouping of the glazes on the basis of the PbO, $SiO_2$ and $SnO_2$ contents is illustrated in Figure 2.2. There is a notable consistency in the composition of the yellow and the white glazes. The microstructure of glazes as seen by SEM shows a distribution of bright micro-crystallites through the glaze thickness (Figure 2.3). Analysis of a selection of particles indicated that they were of the form $Pb(Sn,Si)O_3$ in the yellow glazes with an approximate average composition of 65% PbO, 25% $SnO_2$ and 10% $SiO_2$ (Appendix B). This form of lead-tin-oxide was referred to as type II by Kühn (1968) in his classification of lead-tin yellow, as opposed to $Pb_2SnO_4$, referred to as type I. The latter was never detected in the yellow glazes analysed. The particles in the white glazes, on the other hand, were of the form $SnO_2$. A further factor that contributed to the opacity of these glazes was the quartz grains and/or air bubbles that, due to under-firing, had survived in some of the samples (e.g. Figs 2.3 and 2.4).

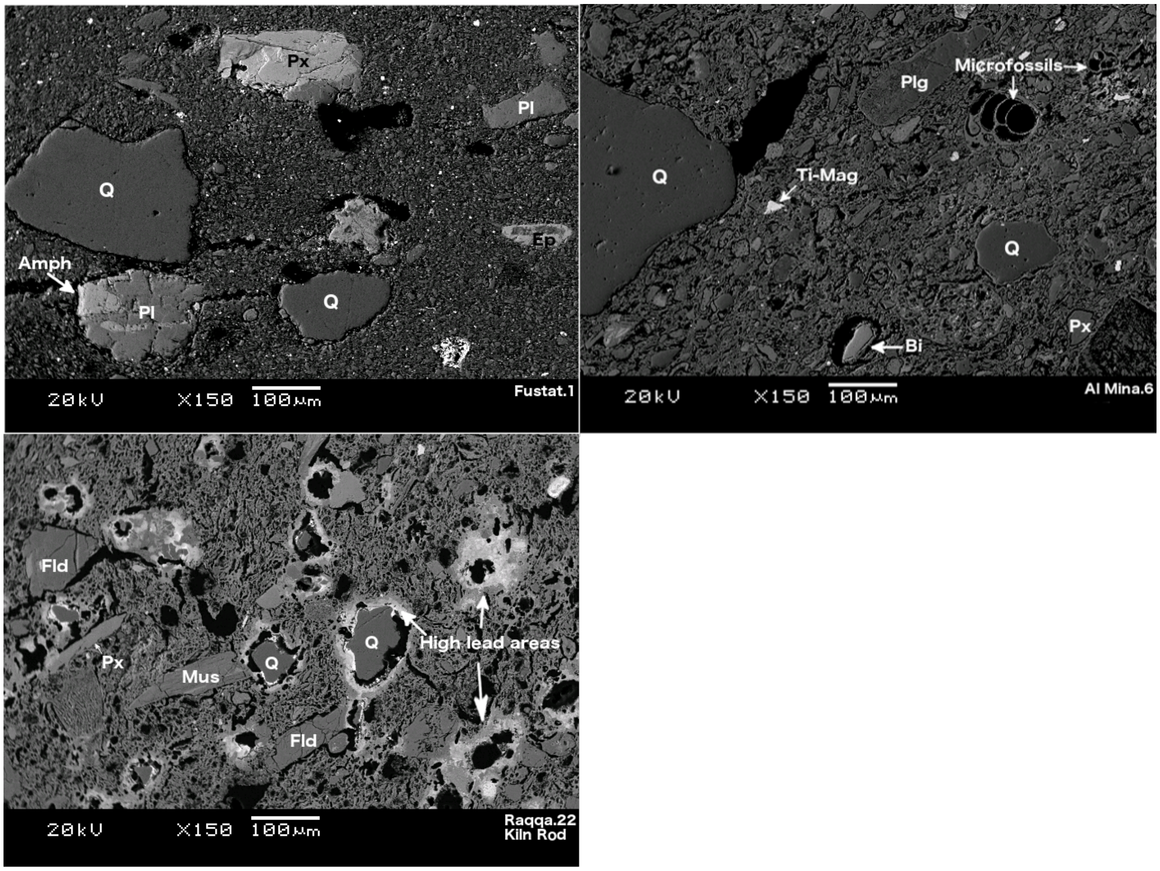
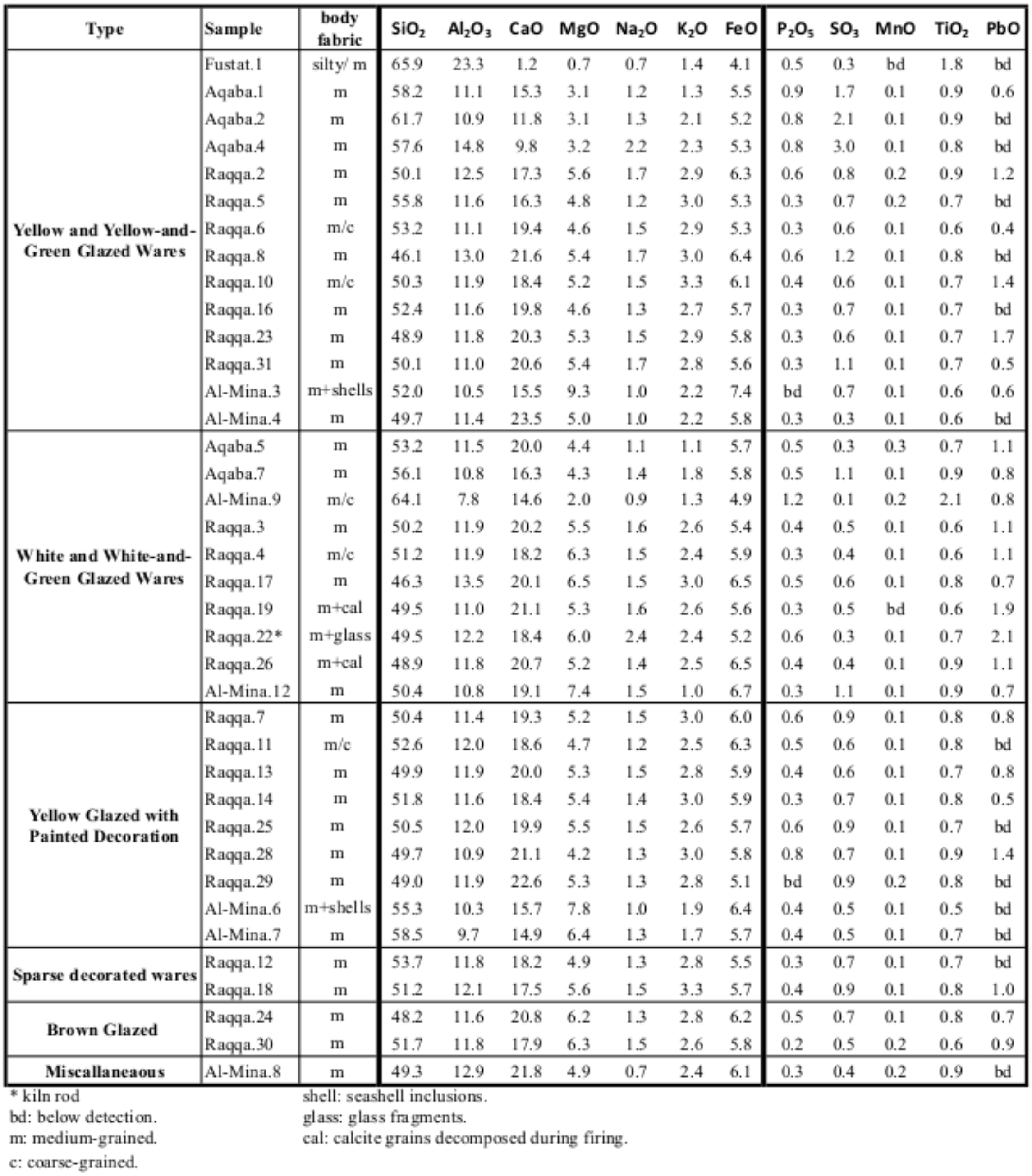


Figure 2.1. *Backscattered SEM photomicrograph of Eastern Mediterranean bodies. Fustat.1: showing compacted aluminium-rich clay with mineral inclusions associated with felsic and intermediate rocks; Al-Mina.6: showing a matrix of porous calcareous clay with felsic and intermediate mineral inclusions and microfossils; Raqqa.22: body of a kiln rod showing a variety of felsic and intermediate mineral inclusions as well as high lead glass fragments added to a calcareous clay. Q: Quartz, Px: Pyroxene, Pl: Plagioclase, Fld: Feldspar, Bi: Biotite, Mus: Muscovite, Ti-Mag: Titanium Magnetite.*

Table 2.1. *EDS analysis of Eastern Mediterranean bodies (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*



| Type | Sample | body fabric | $SiO_2$ | $Al_2O_3$ | CaO | MgO | $Na_2O$ | $K_2O$ | FeO | $P_2O_5$ | $SO_3$ | MnO | $TiO_2$ | PbO |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Yellow and Yellow-and-Green Glazed Wares** | Fustat.1 | silty/ m | 65.9 | 23.3 | 1.2 | 0.7 | 0.7 | 1.4 | 4.1 | 0.5 | 0.3 | bd | 1.8 | bd |
| | Aqaba.1 | m | 58.2 | 11.1 | 15.3 | 3.1 | 1.2 | 1.3 | 5.5 | 0.9 | 1.7 | 0.1 | 0.9 | 0.6 |
| | Aqaba.2 | m | 61.7 | 10.9 | 11.8 | 3.1 | 1.3 | 2.1 | 5.2 | 0.8 | 2.1 | 0.1 | 0.9 | bd |
| | Aqaba.4 | m | 57.6 | 14.8 | 9.8 | 3.2 | 2.2 | 2.3 | 5.3 | 0.8 | 3.0 | 0.1 | 0.8 | bd |
| | Raqqa.2 | m | 50.1 | 12.5 | 17.3 | 5.6 | 1.7 | 2.9 | 6.3 | 0.6 | 0.8 | 0.2 | 0.9 | 1.2 |
| | Raqqa.5 | m | 55.8 | 11.6 | 16.3 | 4.8 | 1.2 | 3.0 | 5.3 | 0.3 | 0.7 | 0.2 | 0.7 | bd |
| | Raqqa.6 | m/c | 53.2 | 11.1 | 19.4 | 4.6 | 1.5 | 2.9 | 5.3 | 0.3 | 0.6 | 0.1 | 0.6 | 0.4 |
| | Raqqa.8 | m | 46.1 | 13.0 | 21.6 | 5.4 | 1.7 | 3.0 | 6.4 | 0.6 | 1.2 | 0.1 | 0.8 | bd |
| | Raqqa.10 | m/c | 50.3 | 11.9 | 18.4 | 5.2 | 1.5 | 3.3 | 6.1 | 0.4 | 0.6 | 0.1 | 0.7 | 1.4 |
| | Raqqa.16 | m | 52.4 | 11.6 | 19.8 | 4.6 | 1.3 | 2.7 | 5.7 | 0.3 | 0.7 | 0.1 | 0.7 | bd |
| | Raqqa.23 | m | 48.9 | 11.8 | 20.3 | 5.3 | 1.5 | 2.9 | 5.8 | 0.3 | 0.6 | 0.1 | 0.7 | 1.7 |
| | Raqqa.31 | m | 50.1 | 11.0 | 20.6 | 5.4 | 1.7 | 2.8 | 5.6 | 0.3 | 1.1 | 0.1 | 0.7 | 0.5 |
| | Al-Mina.3 | m+shells | 52.0 | 10.5 | 15.5 | 9.3 | 1.0 | 2.2 | 7.4 | bd | 0.7 | 0.1 | 0.6 | 0.6 |
| | Al-Mina.4 | m | 49.7 | 11.4 | 23.5 | 5.0 | 1.0 | 2.2 | 5.8 | 0.3 | 0.3 | 0.1 | 0.6 | bd |
| **White and White-and-Green Glazed Wares** | Aqaba.5 | m | 53.2 | 11.5 | 20.0 | 4.4 | 1.1 | 1.1 | 5.7 | 0.5 | 0.3 | 0.3 | 0.7 | 1.1 |
| | Aqaba.7 | m | 56.1 | 10.8 | 16.3 | 4.3 | 1.4 | 1.8 | 5.8 | 0.5 | 1.1 | 0.1 | 0.9 | 0.8 |
| | Al-Mina.9 | m/c | 64.1 | 7.8 | 14.6 | 2.0 | 0.9 | 1.3 | 4.9 | 1.2 | 0.1 | 0.2 | 2.1 | 0.8 |
| | Raqqa.3 | m | 50.2 | 11.9 | 20.2 | 5.5 | 1.6 | 2.6 | 5.4 | 0.4 | 0.5 | 0.1 | 0.6 | 1.1 |
| | Raqqa.4 | m/c | 51.2 | 11.9 | 18.2 | 6.3 | 1.5 | 2.4 | 5.9 | 0.3 | 0.4 | 0.1 | 0.6 | 1.1 |
| | Raqqa.17 | m | 46.3 | 13.5 | 20.1 | 6.5 | 1.5 | 3.0 | 6.5 | 0.5 | 0.6 | 0.1 | 0.8 | 0.7 |
| | Raqqa.19 | m+cal | 49.5 | 11.0 | 21.1 | 5.3 | 1.6 | 2.6 | 5.6 | 0.3 | 0.5 | bd | 0.6 | 1.9 |
| | Raqqa.22* | m+glass | 49.5 | 12.2 | 18.4 | 6.0 | 2.4 | 2.4 | 5.2 | 0.6 | 0.3 | 0.1 | 0.7 | 2.1 |
| | Raqqa.26 | m+cal | 48.9 | 11.8 | 20.7 | 5.2 | 1.4 | 2.5 | 6.5 | 0.4 | 0.4 | 0.1 | 0.9 | 1.1 |
| | Al-Mina.12 | m | 50.4 | 10.8 | 19.1 | 7.4 | 1.5 | 1.0 | 6.7 | 0.3 | 1.1 | 0.1 | 0.9 | 0.7 |
| **Yellow Glazed with Painted Decoration** | Raqqa.7 | m | 50.4 | 11.4 | 19.3 | 5.2 | 1.5 | 3.0 | 6.0 | 0.6 | 0.9 | 0.1 | 0.8 | 0.8 |
| | Raqqa.11 | m/c | 52.6 | 12.0 | 18.6 | 4.7 | 1.2 | 2.5 | 6.3 | 0.5 | 0.6 | 0.1 | 0.8 | bd |
| | Raqqa.13 | m | 49.9 | 11.9 | 20.0 | 5.3 | 1.5 | 2.8 | 5.9 | 0.4 | 0.6 | 0.1 | 0.7 | 0.8 |
| | Raqqa.14 | m | 51.8 | 11.6 | 18.4 | 5.4 | 1.4 | 3.0 | 5.9 | 0.3 | 0.7 | 0.1 | 0.8 | 0.5 |
| | Raqqa.25 | m | 50.5 | 12.0 | 19.9 | 5.5 | 1.5 | 2.6 | 5.7 | 0.6 | 0.9 | 0.1 | 0.7 | bd |
| | Raqqa.28 | m | 49.7 | 10.9 | 21.1 | 4.2 | 1.3 | 3.0 | 5.8 | 0.8 | 0.7 | 0.1 | 0.9 | 1.4 |
| | Raqqa.29 | m | 49.0 | 11.9 | 22.6 | 5.3 | 1.3 | 2.8 | 5.1 | bd | 0.9 | 0.2 | 0.8 | bd |
| | Al-Mina.6 | m+shells | 55.3 | 10.3 | 15.7 | 7.8 | 1.0 | 1.9 | 6.4 | 0.4 | 0.5 | 0.1 | 0.5 | bd |
| | Al-Mina.7 | m | 58.5 | 9.7 | 14.9 | 6.4 | 1.3 | 1.7 | 5.7 | 0.4 | 0.5 | 0.1 | 0.7 | bd |
| **Sparse decorated wares** | Raqqa.12 | m | 53.7 | 11.8 | 18.2 | 4.9 | 1.3 | 2.8 | 5.5 | 0.3 | 0.7 | 0.1 | 0.7 | bd |
| | Raqqa.18 | m | 51.2 | 12.1 | 17.5 | 5.6 | 1.5 | 3.3 | 5.7 | 0.4 | 0.9 | 0.1 | 0.8 | 1.0 |
| **Brown Glazed** | Raqqa.24 | m | 48.2 | 11.6 | 20.8 | 6.2 | 1.3 | 2.8 | 6.2 | 0.5 | 0.7 | 0.1 | 0.8 | 0.7 |
| | Raqqa.30 | m | 51.7 | 11.8 | 17.9 | 6.3 | 1.5 | 2.6 | 5.8 | 0.2 | 0.5 | 0.2 | 0.6 | 0.9 |
| **Miscallaneaous** | Al-Mina.8 | m | 49.3 | 12.9 | 21.8 | 4.9 | 0.7 | 2.4 | 6.1 | 0.3 | 0.4 | 0.2 | 0.9 | bd |

* kiln rod
bd: below detection.
m: medium-grained.
c: coarse-grained.

shell: seashell inclusions.
glass: glass fragments.
cal: calcite grains decomposed during firing.

The composition and microstructure of the green and black decorations is given in Table 2.3 and Figure 2.6. Cu-(Fe) and Mn colourants were used respectively for the green and the black. Compared to the underlying yellow or white glazes, the green and black glazes contain fewer opacifying particles (Figure 2.6).

Table 2.2. *EDS analysis of Eastern Mediterranean opaque yellow and white glazes (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*

| Sample | Associated glazes* | $SiO_2$ | PbO | $SnO_2$ | $(Na/K)_2O$ | (Ca/Mg)O | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | $P_2O_5$ | MnO | FeO | $TiO_2$ | Pb/Sn (calx) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Yellow Glazes: opacified by $Pb(Sn,Si)O_3$ crystallites** | | | | | | | | | | | | | | | | |
| **Fustat.1** | **G** | 27.7 | 65.3 | 2.7 | 0.2 | 0.3 | 0.2 | bd | 0.2 | 0.1 | 2.9 | bd | 0.1 | 0.6 | bd | 28.1 |
| **Aqaba.4** | **-** | 24.7 | 67.0 | 4.5 | 0.7 | 1.6 | 0.4 | 0.3 | 1.4 | 0.2 | 1.1 | bd | bd | 0.4 | bd | 17.6 |
| **Raqqa.11** | **[Br]/G** | 28.5 | 66.7 | 1.9 | bd | 0.3 | bd | bd | 0.2 | 0.1 | 1.5 | bd | bd | bd | 1.0 | 40.5 |
| **Raqqa.14** | **[Br] & G** | 26.7 | 67.8 | 1.5 | 0.5 | 1.4 | bd | 0.5 | 1.2 | 0.2 | 1.0 | bd | 0.1 | 0.9 | bd | 53.7 |
| **Raqqa.16** | **G** | 25.2 | 65.7 | 3.6 | 0.5 | 2.5 | 0.2 | 0.4 | 2.0 | 0.5 | 1.3 | bd | bd | 0.9 | bd | 21.3 |
| **Raqqa.18** | **G** | 26.9 | 48.4 | 1.7 | 1.5 | 9.8 | 0.4 | 1.0 | 8.8 | 1.0 | 1.3 | 8.6 | bd | 1.5 | 0.2 | 32.8 |
| **Raqqa.25** | **Br & [G]** | 28.0 | 62.4 | 2.5 | bd | 1.2 | bd | bd | 1.0 | 0.2 | 1.0 | bd | 4.5 | 0.3 | bd | 29.0 |
| **Raqqa.29** | **[Br] & G** | 26.9 | 66.5 | 2.6 | 0.6 | 2.1 | 0.2 | 0.3 | 1.7 | 0.4 | 0.5 | bd | bd | 0.6 | bd | 29.7 |
| **Raqqa.31** | **G** | 29.9 | 62.5 | 1.6 | 0.5 | 2.6 | 0.2 | 0.2 | 2.2 | 0.4 | 1.5 | bd | bd | 1.1 | 0.2 | 44.9 |
| **Al-Mina.3** | **G** | 25.1 | 69.1 | 3.6 | 0.1 | 1.0 | 0.2 | bd | 0.8 | 0.2 | 0.8 | bd | bd | bd | bd | 22.4 |
| **A-lMina.4** | **[Br] & [G]** | 28.4 | 63.5 | 1.8 | 1.9 | 2.7 | 1.2 | 0.6 | 2.2 | 0.5 | 0.7 | bd | bd | 0.5 | 0.2 | 40.7 |
| **Al-Mina.7** | **[Br] & [G]** | 28.7 | 63.4 | 4.7 | 0.4 | 1.3 | 0.2 | 0.1 | 1.0 | 0.3 | 0.6 | bd | 0.1 | 0.6 | bd | 15.8 |
| **Average** | | 27.2 | 64.1 | 2.8 | 0.7 | 2.2 | 0.4 | 0.4 | 1.9 | 0.4 | 1.2 | 0.7 | 1.2 | 0.8 | 0.4 | 31.4 |
| **White Glazes: opacified by $SnO_2$ crystallites** | | | | | | | | | | | | | | | | |
| **Aqaba.5** | **[G]** | 37.5 | 41.1 | 10.8 | 3.2 | 3.4 | 1.1 | 2.1 | 2.7 | 0.7 | 1.6 | bd | 0.1 | 2.3 | 0.1 | 4.5 |
| **Aqaba.7** | **-** | 40.4 | 45.8 | 6.6 | 4.6 | 0.4 | 3.8 | 0.8 | 0.3 | 0.1 | 1.6 | bd | bd | 0.4 | 0.2 | 8.1 |
| **Raqqa.1** | **-** | 37.7 | 46.3 | 7.2 | 3.8 | 3.3 | 2.9 | 0.9 | 2.3 | 1.0 | 0.5 | bd | 0.1 | 1.0 | 0.1 | 7.5 |
| **Raqqa.3** | **[G]** | 40.0 | 43.6 | 7.2 | 3.8 | 4.1 | 2.4 | 1.4 | 3.0 | 1.1 | 0.8 | bd | 0.1 | 0.4 | bd | 7.2 |
| **Raqqa.20** | **-** | 45.2 | 36.9 | 7.2 | 4.6 | 4.5 | 2.6 | 2.0 | 3.5 | 1.1 | 0.9 | bd | bd | 0.6 | 0.1 | 6.0 |
| **Raqqa.22**** | **-** | 44.6 | 37.7 | 6.1 | 4.6 | 4.9 | 2.8 | 1.8 | 3.7 | 1.2 | 1.0 | bd | bd | 1.0 | bd | 7.2 |
| **Raqqa.26** | **-** | 40.4 | 41.1 | 6.5 | 4.4 | 3.7 | 2.7 | 1.7 | 2.9 | 0.8 | 2.4 | bd | 0.2 | 1.2 | 0.2 | 7.4 |
| **Al-Mina.5** | **[G] & [P]** | 38.4 | 43.5 | 8.1 | 4.7 | 3.9 | 2.3 | 2.4 | 2.9 | 1.0 | 0.6 | bd | 0.2 | 0.4 | 0.1 | 6.3 |
| **Al-Mina.9** | **-** | 36.0 | 44.2 | 12.2 | 2.8 | 2.3 | 1.8 | 1.0 | 1.8 | 0.5 | 1.9 | bd | 0.1 | 0.4 | 0.1 | 4.3 |
| **Average** | | 40.0 | 42.3 | 8.0 | 4.1 | 3.4 | 2.5 | 1.6 | 2.6 | 0.8 | 1.3 | bd | 0.1 | 0.9 | 0.1 | 6.5 |

* See Table 2.3; Glaze colours in brackets were not analaysed (either weatherd or not sampled).
** Splashes of opaque white glaze on kiln rod.
'/' indicates that glazes were applied on diiferent sides of the sherd.
'&' indicates that the glazes were applied on the same side of the sherd.

G: Green.
Br: Brown.
P: Purple.
bd: below detection.

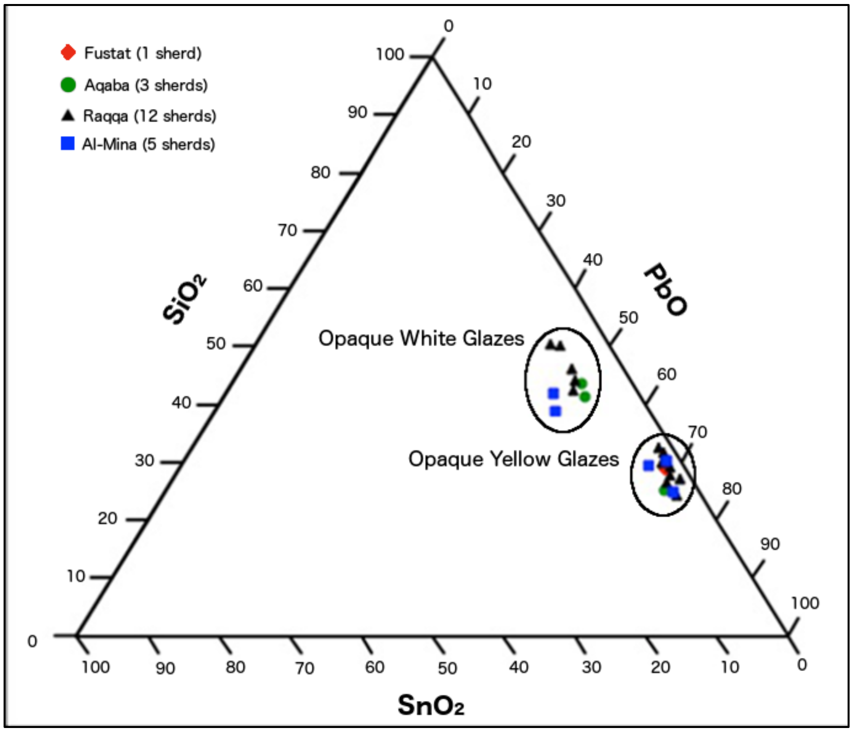


Figure 2.2. *Ternary plot of the normalised PbO-$SiO_2$- $SnO_2$ contents from the Eastern Mediterranean glaze compositions presented in Table 2.2.*

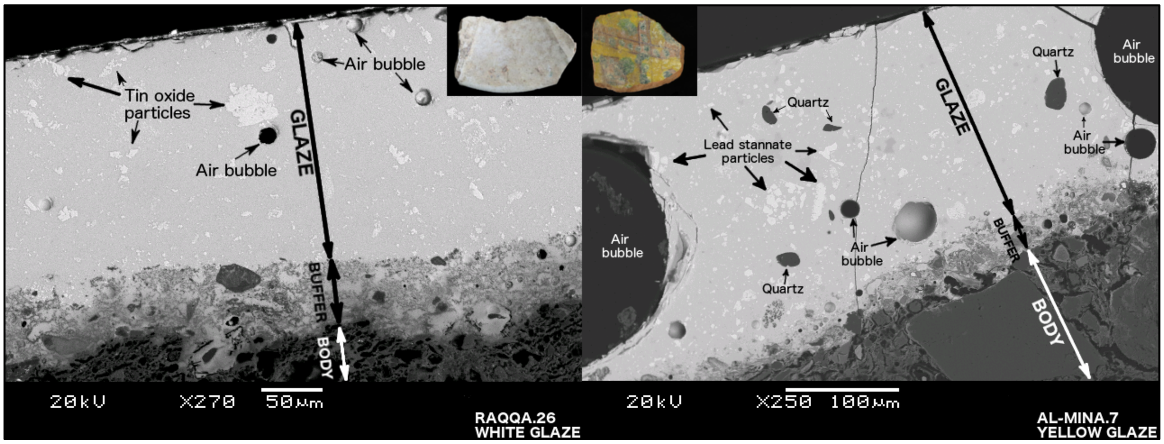
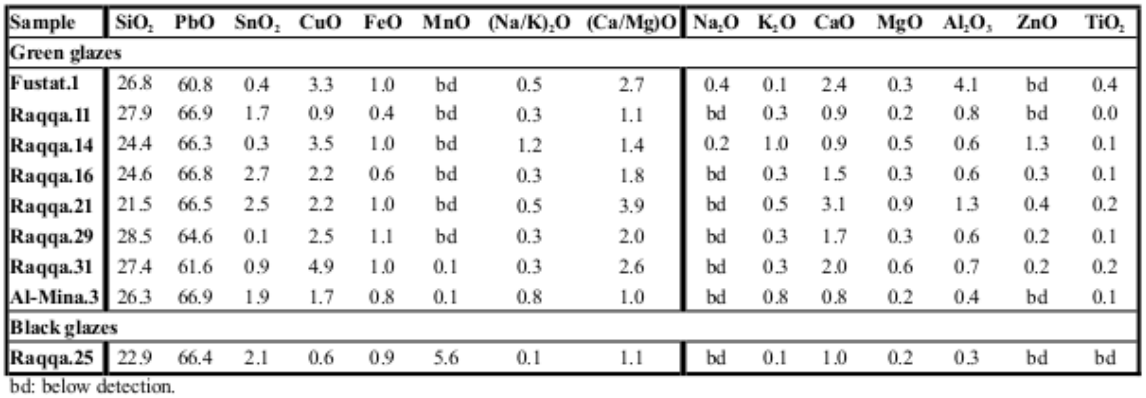


Figure 2.3. *Backscattered SEM photomicrograph of sections through glazes and bodies of Raqqa.26 white glaze showing a scatter of tin oxide micro-crystallites and air bubbles, as well as secondary crystallites of wollastonite and diopside formed in the buffer layer, and Al-Mina.7 yellow glaze showing a scatter of lead-tin-oxide micro-crystallites, air bubbles and undissolved quartz particles, as well as secondary crystallites of wollastonite and diopside formed in the buffer layer.*

Table 2.3. *EDS analysis of Eastern Mediterranean green and black glazes (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*



| Sample | $SiO_2$ | PbO | $SnO_2$ | CuO | FeO | MnO | $(Na/K)_2O$ | (Ca/Mg)O | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | ZnO | $TiO_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Green glazes** | | | | | | | | | | | | | | | |
| **Fustat.1** | 26.8 | 60.8 | 0.4 | 3.3 | 1.0 | bd | 0.5 | 2.7 | 0.4 | 0.1 | 2.4 | 0.3 | 4.1 | bd | 0.4 |
| **Raqqa.11** | 27.9 | 66.9 | 1.7 | 0.9 | 0.4 | bd | 0.3 | 1.1 | bd | 0.3 | 0.9 | 0.2 | 0.8 | bd | 0.0 |
| **Raqqa.14** | 24.4 | 66.3 | 0.3 | 3.5 | 1.0 | bd | 1.2 | 1.4 | 0.2 | 1.0 | 0.9 | 0.5 | 0.6 | 1.3 | 0.1 |
| **Raqqa.16** | 24.6 | 66.8 | 2.7 | 2.2 | 0.6 | bd | 0.3 | 1.8 | bd | 0.3 | 1.5 | 0.3 | 0.6 | 0.3 | 0.1 |
| **Raqqa.21** | 21.5 | 66.5 | 2.5 | 2.2 | 1.0 | bd | 0.5 | 3.9 | bd | 0.5 | 3.1 | 0.9 | 1.3 | 0.4 | 0.2 |
| **Raqqa.29** | 28.5 | 64.6 | 0.1 | 2.5 | 1.1 | bd | 0.3 | 2.0 | bd | 0.3 | 1.7 | 0.3 | 0.6 | 0.2 | 0.1 |
| **Raqqa.31** | 27.4 | 61.6 | 0.9 | 4.9 | 1.0 | 0.1 | 0.3 | 2.6 | bd | 0.3 | 2.0 | 0.6 | 0.7 | 0.2 | 0.2 |
| **Al-Mina.3** | 26.3 | 66.9 | 1.9 | 1.7 | 0.8 | 0.1 | 0.8 | 1.0 | bd | 0.8 | 0.8 | 0.2 | 0.4 | bd | 0.1 |
| **Black glazes** | | | | | | | | | | | | | | | |
| **Raqqa.25** | 22.9 | 66.4 | 2.1 | 0.6 | 0.9 | 5.6 | 0.1 | 1.1 | bd | 0.1 | 1.0 | 0.2 | 0.3 | bd | bd |

bd: below detection.

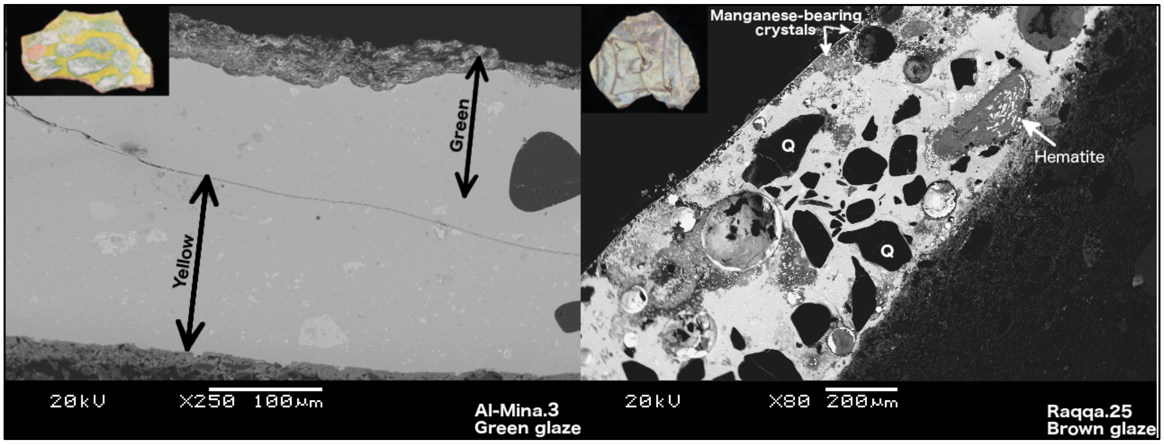


Figure 2.4. *Backscattered SEM photomicrograph of sections through glazes and bodies of Al-Mina.3: showing the green glaze with less number of lead-tin-oxide micro-crystallites compared to the underlying yellow glaze, and Raqqa.25: showing the brown glaze containing quartz grains, hematite and various oxidised forms of manganese and manganese-lead bearing crystals.*

## 2.2. CALX REPRODUCTION

Calx refers to the powder resulting from calcination (or oxidation) of metals due to heat. In the production of tin-opacified glazes (both yellow and white), a key step is the preparation of a lead-tin calx. Descriptions of calx production in some Islamic treatises, as well as reviews of anthropological observations at traditional workshops in Iran are given below. The results provide a basis for the replication experiments that follows.

### 2.2.1. Textual sources

Despite lack of reference to the production process of lead-tin calces in the early Islamic period, an Ilkhanid (1301 AD) Persian treatise by Abu'l Qasim Kāshānī, *The virtues of jewels and the delicacies of perfumes* (*Arāyes al-Jawāher va Nafāyes al-Atāyeb)*, describes the production of lead-tin calx for the production of opaque white glazes:

"One takes 3 parts of good white lead (asrab/ اسرب) and a third of a part [or: one part] of tin (rasās/ رصاص), or if one wants a better and finer mixture up to half (of a part) of tin. First they put the lead into the kiln for a time, and then they throw the tin in on top of it. They mix them at a high temperature until they are well melted. When (this mixture) brings up an earthy substance on its surface it is completely ready. They then make the fire smaller and seal the furnace door with mud. The earthy substance which collects on the melt is taken off with an iron shovel until in half a day it has all gradually changed to earth. The craftsmen call this *asranj* (اسرنج)*"* (Allan 1973, 113; Afshar 2006, 342-3).

He later goes on to describe calcination of tin alone:

"If one wants to use tin alone to get a glowing white colour, one uses two earthenware vessels. Tin is put into one and beaten with an iron pestle until it all becomes earthy and black in colour. It is cooled and sifted with a fine sieve on the end of a ladle, and put into a second vessel [which has been baked]; they leave it until the fire catches it and it rises nicely from its place. When it is cool it is a white earth and it is used for making turquoise preparations" (Allan 1973, 113; Afshar 2006, 343).

Later in the Qajar period, the master potter Ali Mohammad Isfahānī described in his Persian treatise, *On the manufacture of Modern Kashi Earthenware Tiles and Vases in Imitation of the Ancient*, the process of the production of lead-tin calx:

"Melt in the kiln one maund of lead (*surb*) and one quarter maund of tin (*gal*). But I must explain how to do this. Take an earthen vessel, on its side make two holes opposite to each other, place it in the kiln, throw in the lead and tin, stop up the mouth of the vessel, and heat the kiln so that the flame enters from the back hole of the vessel and comes out from the front hole in such a way that the fire clasps the lead and tin from above and below. Thus you keep on heating till the lead and tin melt. After melting, you decrease the fire gradually till the melted lead and give forth a froth (*kurk*), then you remove the lid of the vessel, and remove to one side the froth, again decrease the fire, froth is again formed which you remove as before until the whole of the lead and tin has turned into froth. You take it and bray it fine on a stone. Then take four parts of the previously mentioned refined paint and one part of this lead and tin (turned into froth and brayed), and mix them for a coating and varnish (*la'ab*). Keep this kind" (Isfahānī 1888, 4).

He then moves on to the production of yellow glaze using a different ratio of tin to lead and also by addition of quartz, referred to as 'brayed stone':

"If you want a yellow colour, take sixteen parts of lead and one of tin, melt them together, take the froth (*kurk*) and heat it; when it begins to melt, add a quarter of its quantity of well brayed stone, and mix thoroughly. Bricks or vases painted with this preparation and heated, will come out of the kiln a yellow colour- like a servant who has acted perfidiously, and who, as is well known, turns yellow. With an iron ladle (*sikh*), skimmer-like, you must take out the yellow colour when melting, bray it, mix it with solution of gum Arabic (*la'ab-i-ktira*), and apply it to bricks or vases. This requires only half the heat of other colours" (Isfahānī 1888, 9). Isfahānī's suggested recipe (16 Pb : 1 Sn : 5.7 $SiO_2$) translates into a glaze containing approximately 70% PbO, 5% $SnO_2$ and 25% $SiO_2$. This is rather similar to the composition of yellow glazes given in Table 2.2 and Figure 2.4.

### 2.2.2 Observations in traditional workshops

The processes described in the treatises is largely analogous to those undertaken in contemporary traditional workshops, for instance in Lālejīn (Hamedān province), Iran. In 1965, Sedigh and Karimī published a report on pottery manufacturing processes in Lālejīn, based on their interview with Ustad Sādegh Azimī, then "the greatest master of pottery in Lālejīn" (Sedigh and Karimī 1965, 1). The calcination process of lead with tin is given under 'fired lead' (*sorb-i pokhte*), translated from Persian as follows:

"They put three manns [roughly 9 kg] of lead in a pan (which is known as *tavā* in their local dialect) and put it in the furnace. Lead melts slowly and after it is entirely melted down, they add less than one mann [roughly 3 kg] of tin, so that after melting, it is mixed with lead.

On this burning mixture, something like ash emerges, which they take to a side using a stick. This gradually develops into a *zarnikh* [Persian name for realgar and orpiment] yellow colour. They carry on this until the whole mixture turns into the yellow powder. When mixture is very hot, they drop some tin in it to see if it sparks or not. If it does so, they say the lead is fired and they slowly pour it in a container with two wooden handles, which they call *chighāvān*, to cool down" (Sedigh and Karimī 1965, 5).

In April 2015, the author visited the pottery workshops located in Lālejīn in search for lead-tin calcination. The calcination of lead with tin for ceramic glaze opacification was being carried out in a number of workshops. The workshop of Mr Seyyed Mehdī Hosseinī conducted oxidation of lead with tin for application in opaque glazes produced in the same workshop. Oxidation of lead with tin was carried out in an open-air furnace located at the back of the workshop. The furnace was an updraft type, equipped with a central chimney at its roof. It comprised of a dome-shaped chamber, made of clay bricks and covered by a mixture of fireclay paste and had an open front (Fig.2.5). A stainless steel pan was placed at the bottom of the dome. The pan was connected to an electric motor beneath the chamber and turned at a constant speed. A steel rod, approximately as long as the diameter of the pan, was set at a fixed position parallel and just above the bottom of the chamber and functioned as a constant stirrer for the mixture. A gas burner was established at the open front of the chamber. The fire was burnt red hot.

A mixture of one part of metallic tin and nine parts of metallic lead was placed in the pan and the burner was switched on. According to Mr Hosseinī: "the choice of the tin:lead ratio is a matter of finance. Higher tin:lead ratios result in enhanced opacity and general quality of the glaze. However, the potters tended to decrease the amount of tin used over the years for financial considerations". After approximately fifteen minutes, the mixture started to melt and in about a further five minutes a grey earthy substance was produced. With another ten minutes, the mixture began to get a tint of yellow, but with the dominant colour being dark grey. The formation of yellow powder increased with time and after a further 40 minutes, an entirely yellow powder was produced which was left to cool down and was collected for glaze production.

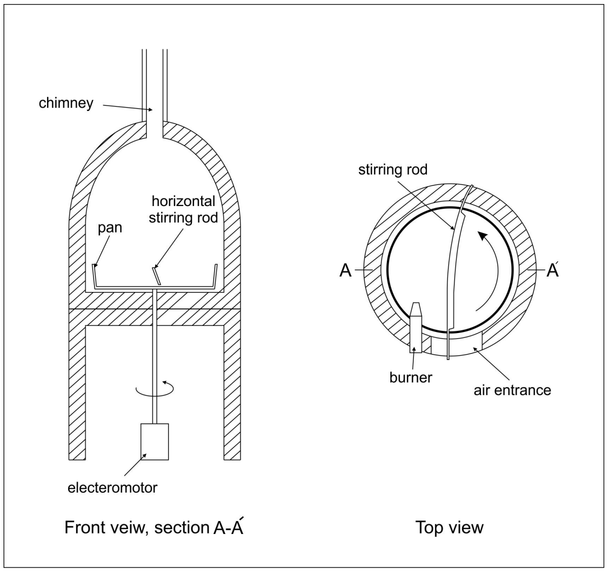


Figure 2.5. *Schematic view of the furnace at Mr Hosseinī's workshop.*

### 2.2.3. Experimental procedure

Replication experiments were undertaken on the basis of descriptions given in the historical treatises and the observations at the traditional workshop summarised above. Experiments were undertaken with three recipes given in Table 2.4.

Table 2.4. *Calx mixture recipes.*

| Calx mixtures | Pb:Sn | Pb (wt%) | Sn (wt%) |
|---|---|---|---|
| **C0** | 0 | - | 100 |
| **C7** | 7 | 87.5 | 12.5 |
| **C30** | 30 | 96.8 | 3.2 |

#### *Raw materials*

Pure lead in form of sheets and pure tin in form of granules were used in the recipes.

#### *Firing procedure*

The mixtures were put in dish-like crucibles (15 cm in diameter), made out of clay from

La Borne, France (known as grès français) which had been biscuit fired. A Hobersal MF22-134 electrical furnace at the University of Vic, Spain, was used for firing the metals. Lead was put in crucibles and fired up to 400°C. Tin was then added to the resulting melt. The mixtures were stirred using steel rods after addition of tin. Figure 2.6 shows the firing schedule and the three stages of sampling.

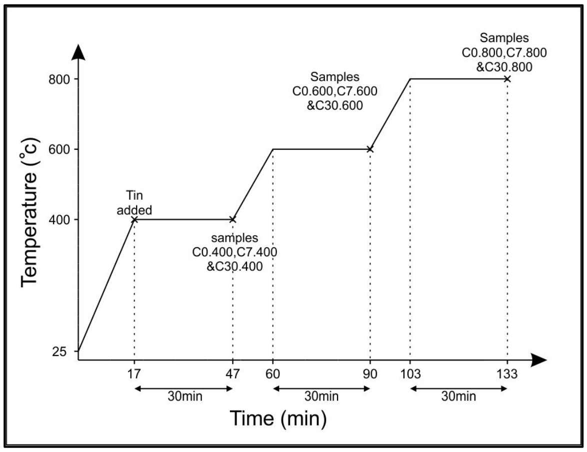


Figure 2.6. *Calx firing schedule and stages of sampling.*

### *Analytical techniques*

The resulting calx samples were observed by the naked eye and/or with a low power binocular microscope and were analysed for their crystallographic phases using XRD, and for their microstructure and chemical composition using SEM-EDS (See Appendix A).

## 2.2.4. Results for calx replicated in laboratory

### *C0*

*Appearance*. At 400°C, a rich melt was formed in the middle of the crucible surrounded by a dark grey ash-like powder (C0.400). By further heating, samples C0.600 and C0.800 turned into fine white powders (Fig. 2.7).

*Crystalline composition*. XRD analysis of the sample C0.400 showed varying amounts of tin (Sn), romarchite (SnO) and cassiterite ($SnO_2$). Samples C0.600 and C0.800 showed mainly cassiterite with negligible romarchite.

The purpose of producing C0 was to demonstrate the calcination process of metallic tin in the absence of lead. The resulting oxide was not used in any of the replica glazes discussed below in section 2.3.

### ***C7***

*Appearance*. The colour of the C7 mixtures changed considerably with temperature. Sample C7.400 exhibited a dark grey powder. As the temperature increased, the colour of the powders C7.600 and C7.800 gradually changed to yellow (Fig. 2.9).

*Crystalline composition*. XRD peaks for sample C7.400 showed lead (Pb), tin (Sn), litharge (PbO, tetragonal structure), massicot (PbO, orthorhombic structure), romarchite (SnO) and cassiterite ($SnO_2$). Sample C7.600 contained mainly lead-tin-oxide in the form of $Pb_2SnO_4$ (type I) and cassiterite, as well as massicot and litharge (for an example of XRD peaks see Appendix A, Fig. A.1). Similar crystalline forms were detected in sample C7.800.

Sample C7.600 was collected and used in the production of glazes G2 and G3 discussed below in section 2.3.

### ***C30***

*Appearance.* Samples C30.400 and C30.600 resemble C7.400 and C7.600, respectively (Fig. 2.9). Sample C30.800, however, reacted with the clay crucible and formed a layer of high lead yellow glass/glaze on the surface of the crucible. The glass/glaze layer appeared inhomogeneous and uneven.

*Crystalline composition.* The crystalline forms detected in the C30.400 and C30.600 were the same as those in the C7.400 and C7.600 samples, respectively. SEM backscattered images of the C30.800 glass showed colonies of $Pb_2SnO_4$ (lead-tin-oxide type I) and $Pb(Sn,Si)O_3$ (lead-tin-oxide type II), tin-dioxide ($SnO_2$) and crossed needle-like lead-oxide crystals embedded in the amorphous phase of the glaze .

Sample C30.600 was collected and used in the production of the G1 glaze discussed below in section 2.3.

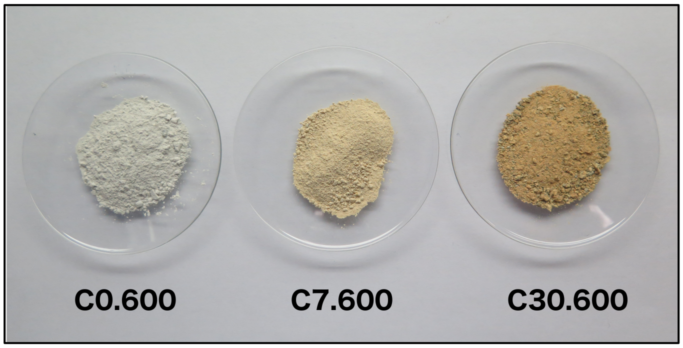


Figure 2.7. *Samples C0.600, C7.600 and C30.600.*

## 2.3. GLAZE REPLICATION

The basis for replication of yellow and white glazes was taken from two sources. Firstly, the compositions of opaque yellow and white Eastern Mediterranean glazes presented in Table 2.2 and secondly, the recipe given above by Isfahānī for opaque yellow glazes which translates approximately to 70% PbO, 5% $SnO_2$ and 25% $SiO_2$ (section 2.2.1).

### 2.3.1. Experimental Procedure

***Raw materials***

In order to imitate the calcareous clays used in the Levantine bodies, British Gault clay was used to produce body pastes (Table 2.5). The raw materials used for the production of glazes were the C7.600 and C30.600 calces, silica, alkaline frit and calcium carbonate. The chemical compositions are given in Table 2.5.

Table 2.5. *Chemical compositions of raw materials (normalised 100%).*

| Raw materials | | $SiO_2$ | $Al_2O_3$ | $Na_2O$ | $K_2O$ | CaO | MgO | FeO | MnO | $TiO_2$ | $SnO_2$ | PbO |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Body** | **Gault clay (biscuit fired)** | 41.4 | 14.8 | 4.5 | 3.1 | 28.7 | 1.8 | 4.3 | 0.1 | 0.8 | 0.4 | 0.3 |
| **Glaze** | **Silica** | 98.9 | 0.6 | - | - | 0.2 | - | 0.2 | 0.1 | - | - | - |
| | **Frit** | 71.3 | 2.7 | 16.6 | 0.8 | 7.6 | 0.4 | 0.6 | - | - | - | - |
| | **Calcium carbonate** | 1.4 | - | 0.1 | - | 97.8 | 0.7 | - | - | - | - | - |
| | **C7.600** | - | - | - | - | - | - | - | - | - | 14.8 | 85.2 |
| | **C30.600** | - | - | - | - | - | - | - | - | - | 3.9 | 96.1 |

***Body***

Clay pastes were wedged and kneaded for a few minutes. The resulting lumps were flattened and formed using a plastic mould of 35 mm in diameter. The resulting tabular discs were left to dry for 24 h and then biscuit-fired to 900°C.

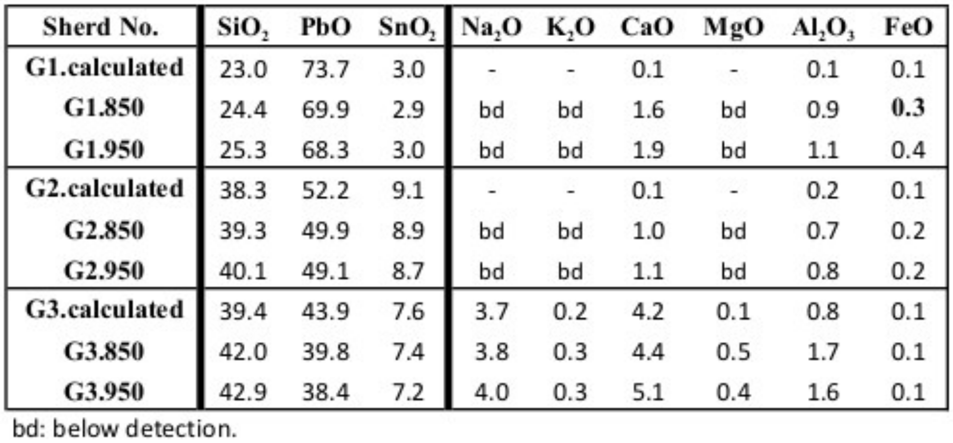
***Glaze***

The recipes and calculated chemical compositions of the glazing mixtures are given in Tables 2.6 and 2.7, respectively. Raw materials were weighed and mixed and the resulting mixtures were ground in a porcelain mortar and passed through a 240-mesh sieve (BSS 63 μm). The glazing powders were mixed with water and applied on the biscuit-fired discs using painting brushes. Firing was carried out in a Lenton electrical furnace at the RLAHA, Oxford, according to the firing schedule shown in Figure 2.8. Each glaze was fired up to 850°C and 950°C.

Table 2.6. *Recipes of glazing mixtures (wt%).*

| Glaze recipes | Silica | C7.600 | C30.600 | Frit | Calcium carbonate |
|---|---|---|---|---|---|
| **G1** | 23.3 | - | 76.7 | - | - |
| **G2** | 38.7 | 61.3 | - | - | - |
| **G3** | 23.9 | 51.5 | - | 22.1 | 2.5 |

Table 2.7. *Calculated chemical compositions of glazing mixtures (wt%) and EDS analysis of replica glazes (wt%; obtained averaging five area measurements for each sample; normalised to 100%).*



| Sherd No. | $SiO_2$ | PbO | $SnO_2$ | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | FeO |
|---|---|---|---|---|---|---|---|---|---|
| G1.calculated | 23.0 | 73.7 | 3.0 | - | - | 0.1 | - | 0.1 | 0.1 |
| G1.850 | 24.4 | 69.9 | 2.9 | bd | bd | 1.6 | bd | 0.9 | 0.3 |
| G1.950 | 25.3 | 68.3 | 3.0 | bd | bd | 1.9 | bd | 1.1 | 0.4 |
| G2.calculated | 38.3 | 52.2 | 9.1 | - | - | 0.1 | - | 0.2 | 0.1 |
| G2.850 | 39.3 | 49.9 | 8.9 | bd | bd | 1.0 | bd | 0.7 | 0.2 |
| G2.950 | 40.1 | 49.1 | 8.7 | bd | bd | 1.1 | bd | 0.8 | 0.2 |
| G3.calculated | 39.4 | 43.9 | 7.6 | 3.7 | 0.2 | 4.2 | 0.1 | 0.8 | 0.1 |
| G3.850 | 42.0 | 39.8 | 7.4 | 3.8 | 0.3 | 4.4 | 0.5 | 1.7 | 0.1 |
| G3.950 | 42.9 | 38.4 | 7.2 | 4.0 | 0.3 | 5.1 | 0.4 | 1.6 | 0.1 |

bd: below detection.

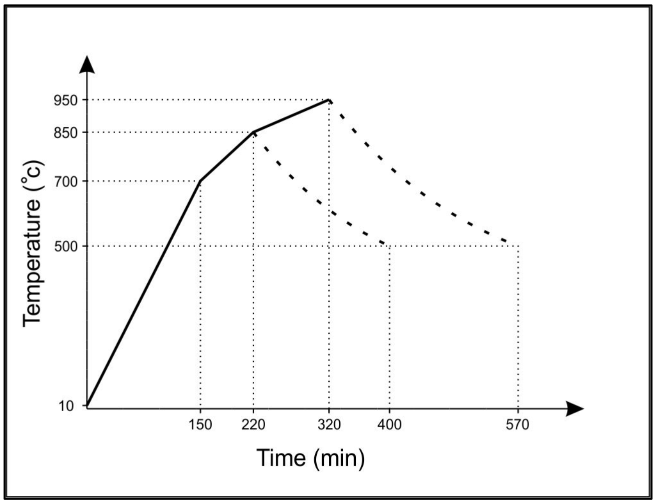


Figure 2.8. *Firing schedule for replicated glazes.*

### *Analytical techniques*

After firing, the resulting glazes were observed by the naked eye and/or binocular microscope and were examined for their chemical composition and microstructure using a SEM-EDS (Appendix A). The Si-Kα and Pb-Mα peaks in the DLH1 glass standard were used as primary standards in analysing the G1 glazes, and the Si-Kα and Pb-Mα peaks in the Corning C glass standard in analysing the G2 and G3 glazes (Tables 2.1).

### 2.3.2. Results

#### *G1*

*Glaze appearance*. The G1 glaze fired to 850°C (G1.850) exhibited deep opaque yellow colour (Fig.2.9). The G1 fired to 950°C (G1.950) produced a glossy surface which exhibited translucent brown colour with white milky opaque appearance in the middle.

*Microstructure*. SEM examination of the G1.850 glaze showed scatters of lead-tin-oxide type II particles with the formula $Pb(Si,Sn)O_3$. Air bubbles and unreacted quartz grains can also be observed throughout the glaze thickness (Fig. 2.10.a). In contrast, the microstructure of the G1.950 glaze was free of opacifying particles, but air bubbles and partially reacted quartz grains were occasionally seen (Fig. 2.10.b). The buffer layers contain a variety of secondary-formed crystals of calcium-lead-silicate.

*Chemical composition*. The chemical composition of the G1.850 and G1.950 glazes shows a shift in the PbO and $SiO_2$ contents compared to the calculated recipe (Table 2.6). Approximately 6 wt% decrease in the PbO and 3 wt% increase in the $SiO_2$ contents had taken place. The $Al_2O_3$ and CaO contents of the glazes have also increased as a result of diffusion from the body into the glaze. However, variations in the concentrations of PbO, $SiO_2$, $Al_2O_3$ and CaO from the glaze surface to the glaze-body interface of the G1-850 glaze do not follow a particular pattern (Figure 2.11).

#### *G2*

*Glaze appearance*. The G2 glaze fired to 850°C (G2-850) produced a matte surface which exhibited opaque yellow colour (Fig.2.9). The G2 glaze fired to 950°C (G2.950) showed a glossy surface with opaque white colour.

*Microstructure*. SEM examination of the G2.850 glaze showed scatters of lead-tin-oxide type II particles with the formula Pb(Si,Sn)$O_3$ and the G2.950 glaze showed tin-oxide particles ($SnO_2$) (Fig. 2.10.c and d). Microstructures of both glazes contained air bubbles and partially reacted quartz grains with round edges. The buffer layer contained a large number of secondary-formed crystals of calcium-lead-silicate.

*Chemical composition*. The chemical composition of the G2.850 and G2.950 glazes is presented in Table 2.7. The change in the contents of the PbO, $SiO_2$, $Al_2O_3$ and CaO is the same as the G1.850 and G1.950.

### *G3*

*Glaze appearance*. The G3 glaze fired to 850°C (G3.850) produced a glossy surface which exhibited a white glaze speckled with small yellow spots (Fig. 2.9). The G3 glaze fired to 950°C (G3.950) showed a glossy and smooth surface which exhibited completely white colour.

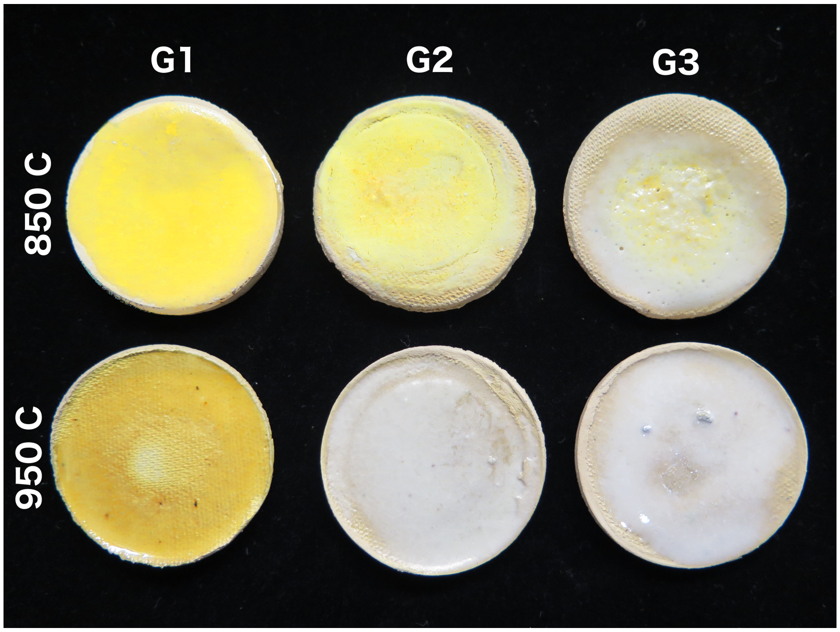


Figure 2.9. *Glazes G1.850, G2.850, G3.850, G1.950, G2.950 and G3.950.*

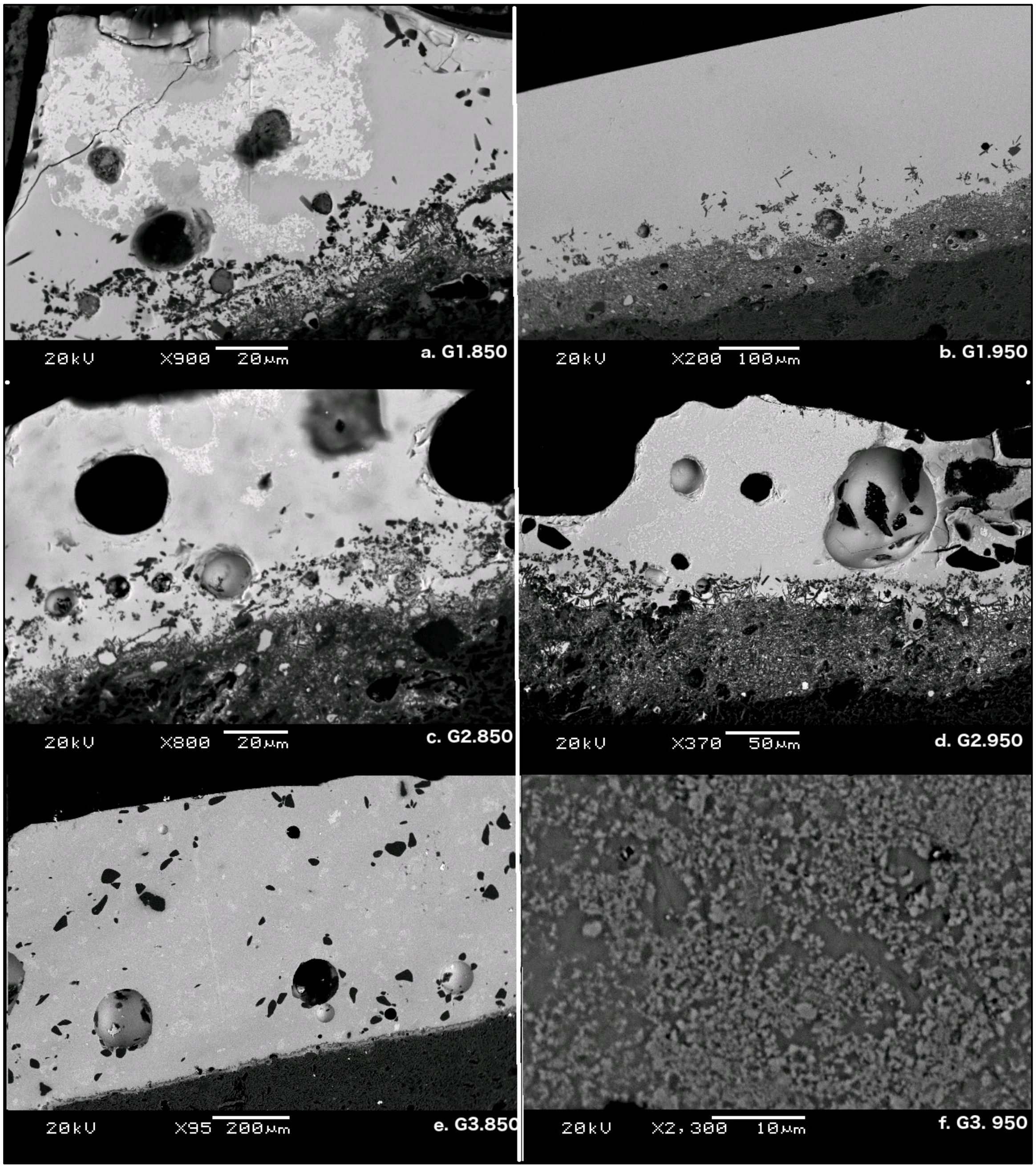


Figure 2.10. *Backscattered SEM photomicrograph of replicated glazes; (a) G1.850: lead-tin-oxide crystallites (white) with secondary crystallites of wollastonite (grey) formed in the buffer layer, (b) G1.950: glaze with no opacifying particles, secondary crystallites of wollastonite (light grey) formed in the buffer layer; (c) G2.850: lead-tin-oxide particles (white), secondary crystallites of wollastonite (grey) formed in the buffer layer; (d) G2.950: scatters of tin-oxide particles (white), secondary crystallites of wollastonite (grey) formed in the buffer layer; (e) G3.850: scatters of tin-oxide particles (white); (f) G3.950: scatters of tin-oxide particles as seen at x2300 magnification.*

*Microstructure*. Both G3.850 and G3.950 showed tin-oxide particles dispersed densely throughout the thickness of the glaze. Air bubbles and partially reacted quartz grains with round edges were also detected (Fig. 2.10.e & f). The buffer layer contained a variety of secondary-formed crystals of calcium-lead-silicate.

*Chemical composition*. The chemical compositions of the G3.850 and G3.950 glazes are presented in Table 2.7. The decrease in the concentrations of PbO and increase in the $SiO_2$, $Al_2O_3$ and CaO compared to the recipes is the same as the G1 and G2 glazes discussed above.

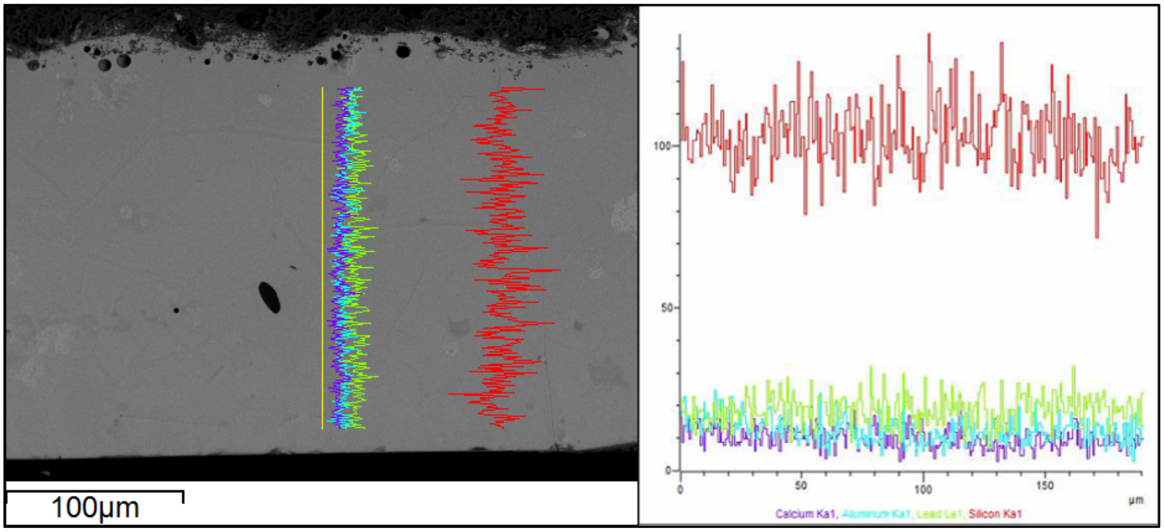


Figure 2.11. *Variation patterns of PbO, $SiO_2$, $Al_2O_3$ and CaO concentrations through the thickness of the G1-850 glaze.*

## 2.4. DISCUSSION

### 2.4.1. Body fabrics and the question of provenance

The bodies of sherds examined from Aqaba, Raqqa and Al-Mina were not sufficiently distinct to be classified into groups: in all the cases, the clay was calcareous and included minerals derived from basic to intermediate sources. The aluminium-rich body of sherd Fustat.1 indicates that it was likely produced using Nile silt. It is to be hoped that future research studies focus more on trace element analysis of clays in Egypt and the Levant in order to further differentiate body fabrics from this region.

### 2.4.2. Eastern Mediterranean ceramics: Calx and glaze recipes

Analysis of the Eastern Mediterranean opaque yellow and white glazes showed that their bulk compositions were internally consistent but distinct from one another. In particular, three differences stand out: Firstly, the PbO concentrations vary around 65% in the yellow glazes and 45% in the white glazes. Secondly, $SnO_2$ concentrations are generally higher in white glazes and finally, the combined alkali and alkaline earth content is negligible in yellow glazes but is approximately 8% in the white glazes.

Results of replication experiments help further in understanding the recipes used for the production of opaque glazes. Bulk compositions of replica glazes showed that the fired glazes contained less PbO compared to the compositions they were designed to have in the glaze recipes. This difference is approximately 3% on average and becomes slightly more significant with the increase in temperature (Table 2.6). The decrease in the PbO content can be understood in terms of the digestion of body by the lead glaze during firing, previously discussed in detail by Molera *et al.* (2001) and Walton (2004, 28-79). This reaction also leads to an increase in the CaO, $Al_2O_3$ and $SiO_2$ contents in the glaze bulk composition. The mechanism of diffusion shows that the initial recipes must have had approximately 3% more PbO than the chemical compositions of the CGW and YGF glazes show. This translates to 74% PbO, 23% $SiO_2$ and 3.0 % $SnO_2$ for yellow glazes (normalised to 100%) and 44% PbO, 29.8% $SiO_2$, 7.7% $SnO_2$, 3.9% $(Na/K)_2O$ and 4.2% (Mg/Ca)O for white glazes (normalised to 100%). The former matches closely with the recipe given by Ali Mohammad Isfahani for yellow glazes. Given the effect of diffusion, the composition of the Eastern Mediterranean yellow and white glazes corresponded respectively to calces with Pb:Sn ratios approximately 30:1 and 7:1.

### 2.4.3. Calcination of lead and tin

The essential opacifying component for lead-tin-oxide yellow and tin-oxide white glazes was the lead-tin calx. The combination and co-melting of the metallic lead and tin during calcination process provide a liquid alloy in which the reaction and oxidation of lead and tin is possible. Through this reaction, varying amounts of lead-tin-oxide (type I) as well as tin-oxide and lead-oxide were formed. These phases are crucial for the subsequent processes that take place during glaze firing.

The information given in Islamic treatises, the anthropological study of traditional workshops and the results of the calx replication experiments C7 and C30 suggested that the lead:tin ratio does not have significant effects on the calcination processes. It is merely a consideration of the degree of opacification required in resulting glazes; i.e. the lower the lead:tin ratio, the more opacified the glaze.

It was previously discussed by Kingery and Vandiver (1986, 116), and has been widely acknowledged, that when tin was heated alone, it resulted in a grey powder that was not suitable for application in glazes. However, when a combination of lead with tin was heated (e.g. Pb:Sn 3:1), the resulting mixture was a white powder. This was claimed to be the reason that the presence of lead was necessary for calcining tin for preparation of opaque glazes. However, the calcination experiment described above demonstrated that metallic lead is not necessary for calcination of tin. When heated to 400°C, the metallic tin in calces C0, C7 and C30 oxidised to tin-oxide ($SnO$) but did not proceed to form tin-dioxide ($SnO_2$) in significant quantities. The result was a dark grey powder as pointed out by Kingery and Vandiver (1986, 116). At 600°C, however, C0 was fully calcined to tin-dioxide, resulting in a fine white powder and C7 and C30 produced yellow powders containing tin-dioxide as well as other oxides. These observations demonstrate that the presence of metallic lead does not have any major impact on the calcination of tin; although it is crucial for the formation of lead-tin-oxide during firing.

### 2.4.4. Yellow Glazes: Technical aspects of production

Eastern Mediterranean yellow glazes contained lead-tin-oxide type II crystallites dispersed in the glaze substrate. These particles were responsible for both opacification and the yellow colour of the glaze. The formation of these phases during glaze firing can be explained in terms of the conversion of lead-tin-oxide type I (in the calx) to lead-tin-oxide type II. Previous studies by Rooksby (1964), Kühn (1968) and Clark *et al.* (1995) demonstrated that the factor leading to the conversion of lead-tin-oxide type I to type II was silica ($SiO_2$). Their experiments showed that the addition of $SiO_2$ between temperatures of 800°C and 950°C was necessary to convert $Pb_2SnO_4$ to $Pb(Sn,Si)O_3$. During this process $Pb(Sn,Si)O_3$ incorporated variable amounts of $SiO_2$ substituting for $SnO_2$. This process was confirmed in the present work by heating the C30 calx to 800°C. In the resulting glass layer produced as a result of reaction with the clay crucible (C30.800), both lead-tin-oxide crystals types I and II were detected. This observation testified the diffusion of $SiO_2$ from body into the glass/calx and its subsequent reaction with some of the $Pb_2SnO_4$ crystals present, leading to the formation of $Pb(Sn,Si)O_3$.

A second process for the formation of lead-tin-oxide type II was discussed by Molera *et al.* (1999) who showed that when heating different $SiO_2$-PbO-$SnO_2$ lead glaze mixtures to 700°C, $Pb(Sn,Si)O_3$ crystals were formed which produced yellow spots on the translucent glaze.

### 2.4.5. White glazes: technical aspects of production

In the Eastern Mediterranean white glazes, $SnO_2$ particles were shown to act as the main opacifying agent and the white colourant. The process of $SnO_2$ precipitation in lead ($SiO_2$-$PbO$-$SnO_2$) glazes was also discussed by Molera *et al.* (1999) and Tite *et al.* (2008). It was demonstrated that $PbSnO_3$ crystals –formed during heating- are stable at low temperature. At temperatures about 750°C or above, $PbSnO_3$ crystals decompose and subsequently stabilise by forming crystals of $SnO_2$. This conversion from $PbSnO_3$ to $SnO_2$ is crucial for production of opaque white glazes.

In the G1 glaze, at temperatures about 950°C, the $PbSnO_3$ to $SnO_2$ conversion seemed to have started, but not yet completed. The microstructure of G1.950 showed no opacifying crystals due to the complete dissolution of the $PbSnO_3$ crystals. As a result, the colour of the G1 glaze changed from yellow at 850°C to yellow with milky halos at 950°C. The milky halos seemed to represent the areas of the glaze in which conversion to $SnO_2$ particles had taken place, although no such crystals were observed using SEM. This might be due to the very small sizes of the $SnO_2$ nucleates formed. On the other hand, in the glaze G2, at temperatures about 950°C, $PbSnO_3$ particles dissolved in the glaze and $SnO_2$ particles were extensively formed. Thus, the colour of the glaze changed from yellow at 850°C to completely white at 950°C.

The temperature in which $SnO_2$ crystallites precipitate in lead glazes is the factor for determining the final colour of opaque glazes. The mechanisms controlling this conversion are poorly understood. Various factors affect this temperature including the Pb:Sn ratios of the calx, the $SiO_2$ content and the firing schedule. The role of these was not evaluated in the experiments conducted and remains to be addressed in future research. One factor that had a substantial effect on precipitation of $SnO_2$ was the addition of alkalis and alkaline earths. In the glaze G3, approximately 8 wt% alkalis and alkaline earths ($Na_2O+K_2O+CaO+MgO$) were added. When fired to 850°C, the resulting glaze exhibited white colour in most part with small yellow speckles in the centre. At 950°C, the yellow speckles disappeared and the obtained glaze was completely white. The G3 glaze experiment suggested that the addition of alkalis significantly decreased the temperature of conversion. The emergent conclusion is that white glazes can be achieved at temperatures as low as 850°C in glazes which include alkalis. This issue becomes particularly important when discussing opaque white glazes from Iraq and Iran in Chapter 3.

### 2.4.6. Tin-based opacification: history and significance

Intentional opacification of glasses began with the application of lead-antimonate ($Pb_2Sb_2O_7$) as the yellow opacifier and calcium-antimonate ($Ca_2Sb_2O_7$) as the white opacifier from the beginnings of glass production in Egypt and the Near East in the second millennium BC and continued until the Roman period (e.g. early Egyptian glasses: Mass *et al.* 2002; Shortland 2002, and Roman glasses: Mass *et al.* 1998; Freestone and Stapleton 2016). Examples of glazes opacified by lead-antimonate and calcium antimonate are the Assyrian glazed bricks from Nineveh (Hedges and Moorey 1975), the Neo-Babylonian bricks from Babylon (Fitz 1982; Matson 1985) and the Achaemenid glazed bricks from Susa (Fukai 1981, 25 ff.; Razmjou *et al.* 2004; Holakooei 2013). Subsequently, sometime during the 4th century AD, lead-tin-oxide type II ($PbSnO_3$) replaced lead-antimonate as the yellow opacifier and tin-dioxide ($SnO_2$) replaced calcium-antimonate as the white opacifier in glasses (e.g. glass pieces in Opus

Sectile panels: Brill and Whitehouse 1988; Merovingian glass beads: Heck and Hoffmann 2002; Heck *et al*. 2003; mosaic tesserae: Rooksby 1962, 24; Freestone *et al.* 1990, 274-5; Tite *et al.* 2008; Marii 2012; Nari *et al*. 2012). This change did not occur in glazes until the 7th/8th century AD. In fact, techniques of opacification were never applied in the Parthian and Sassanian glazes (see e.g. Hill 2004). Likewise, early Byzantine glazes lacked opacity. These transparent glazes were applied either directly on the body or over a slip (see e.g. Armstrong 2001; Vroom 2004). It was not until the 7th/8th century that potters carried forward in glazes the Byzantine tradition of lead-tin opacification of glasses. The study of the CGW and YGF glazing techniques suggest that in this period, the local Byzantine glass opacifying technologies were revived in glaze manufacture in novel forms. These techniques made possible the production of ceramics that held the new Islamic identity and matched the local aesthetics for fine wares. The transfer of tin-based opacification of glasses to glazes deserves to be acknowledged as a 'revolutionary step' in the history of ceramics technology: it marked the beginning of a new tradition of glazing that evolved and continued over centuries to the present day.

## 2.5. CONCLUSIONS

The results of SEM-EDS analysis of the body fabrics and glazes of Eastern Mediterranean CGW and YGF wares dated to 7th/8th century AD and deemed to be the first Islamic glazed wares have been presented. This is probably the largest corpus of data on such sherds. The majority of the body fabrics are calcareous clays with medium to coarse mixed inclusions of basic to intermediate silicate minerals. The glaze data naturally divide into two groups corresponding to the yellow and white glazes, with the yellow being differentiated chemically from the white in generally having higher PbO and lower $SnO_2$ and alkali and alkaline earth oxides. Microstructure of the yellow and white glazes showed that they were opacified respectively by lead-tin-oxide type II ($Pb(Sn,Si)O_3$) and tin-oxide ($SnO_2$) crystals formed during glaze firing. Attempts were made to construct the *chaîne opératoire* through insights gained by experimental replication. The experiments consisted of two stages: preparation of the calx and the glaze.

The results of replication of early Islamic opaque glazes are key because they establish the technical links between the production of yellow and white glazes. In both glazes, lead-tin calx was prepared and mixed with silica. In the white glazes, additional alkalis and alkaline earths were used. Because examples of early Islamic white glazed wares are rare in Egypt, and their early productions seem to have taken place further East in Jordan and Syria in the 8th century, the white opaque glazing can be considered as a later development of the yellow glazing techniques. Around the time of the arrival of Islam in the Eastern Mediterranean, potters adopt the Roman and Byzantine techniques of yellow lead-tin-oxide opacification of glasses, most commonly used in mosaic tesserae, and develop it in the form of yellow and later white opaque glazes. This change in the 7th/8th century AD marked a turning point in the history of ceramic production and gave way to a wide range of new ceramic practices.

# Chapter 3

# SOUTHERN MESOPOTAMIA: THE FLOURISHING OF CERAMIC PRODUCTION

Opaque glazed wares manufactured in Southern Mesopotamia during the Abbasid period have interested researchers as well as connoisseurs of Islamic Art for almost a century. Their position as one of the most technically and aesthetically sophisticated wares makes them central to the discussions on early Islamic ceramics. This chapter attempts to shed light on the production of opaque glazed wares in Iraq and western Iran and their potential relations to the Eastern Mediterranean CGW and YGF wares. The bodies and glazes of sherds are examined for their chemical composition and microstructure and their technological and social aspects of their manufacture are discussed.

## 3.1. REVIEW OF PREVIOUS CHEMICAL ANALYSES

### 3.1.1. Bodies

Chemical analysis of the bodies of early Islamic glazed wares started in the late 20th century. In 1974, neutron activation analysis (NAA) of four lustre ware clay bodies from Fustat were conducted by Michel *et al.* (1976). The results established that “the lustred clay-bodied pottery was imported to Egypt from somewhere in Mesopotamian area” (*Ibid*, 86). Further NAA analyses of eight lustre ware bodies from Fustat and different provenances in Iran by Frierman *et al*. (1979) narrowed down the earlier results. Despite the modest number of samples, this study was the first to propose that the Fustat and Iranian lustre ware bodies share the same chemical composition with the Iraqi export wares excavated at Siraf, Iran, and are hence likely to be of an Iraqi origin. A small assemblage of pieces from Basra played a key role in the works conducted on body fabrics and provenance of early Islamic glazed wares from 1990s onwards: 27 fragments of glazed and unglazed wares, kiln rods and glassy wastes were collected at a presumed production site at Basra and given to the Metropolitan Museum by N.E. Baker in early 1950s. Petrographic analysis by Mason and Keall (1991) showed a striking match between the body fabrics of the Basra kiln materials and Abbasid cobalt blue and lustre decorated wares: a very fine grained calcareous fabric. Thus, Basra became known as the production centre for the Iraqi blue-on-white and lustrewares. Other Abbasid ceramic fabrics examined in Mason's studies showed various body fabrics and were named and then numbered after the archaeological site they were found in (e.g. Samarra 3 or Raqqa 1; see Mason 1994; 2004). These results were re-evaluated by NAA analysis of 90 Abbasid sherd bodies conducted by Hallett (2000, 50). The analysed corpus included a few guiding samples considered by the author to be credibly provenanced to Hira, Samarra and the twenty-seven Basra fragments. By cluster analysis of the compositional data, Hallett demonstrated separation of the Basra kiln materials, as well as the blue-on-white and lustre ware sherds from a single group, based on minor variations of their chemistry. These variations were suggested to possibly "reflect the utilization of clays along different canals in the Basra region, at different distances from the main conduit of the Shatt al-Arab, or stratigraphic changes in composition through a single clay bed" (Hallett 2000, 73-4). The works by Mason and Hallett established that the majority of early Islamic opaque glazed wares, the blue-on-white and lustrewares in particular, originated in Basra; subsequently these ceramics have been widely referred to in the literature as the ‘Basra wares’, and the fabric as to the ‘Basra body’.

### 3.1.1. Glazes

Among the earlier research studies on chemical composition of early Islamic glazes are those conducted on lustrewares by Crowe (1977), Jones (1980), Tite (1988) and Kleinmann (1986). The work by Kleinmann (1991) is amongst the first on the study of Islamic glazes that began to provide detailed analytical data on a comprehensive range of early Islamic wares. Main focus was laid on the early Islamic ceramics of Samarra, held at the Museum für Islamische Kunst, Berlin. On the basis of light microscopy and electron microprobe studies, her examinations suggested two major groups of tin-oxide based opaque glazes. Firstly, a group of alkali-lead-tin glazes (PbO ranging between 5 to 10 wt%). These glazes included occasional tin oxide particles, rounded quartz grains and secondary crystals, as well as round bubbles and irregularly shaped large cavities mainly situated at the base of the glazes. Due to the similarity of the composition of this group of glazes to the earlier tradition of pre-Islamic alkali glazes in the Near East, the author suggested that this might represent an experimental stage of glaze development in order to achieve white opaque glazes. The second group is of a high lead-tin type (approximately 50 wt% PbO), showing no signs of large cavities.

The most thorough and detailed work on early Islamic opaque glazed wares published to date is that conducted by Mason. His series of interconnected papers on opaque glazed wares and associated pottery from Iraq, Egypt, Iran and Syria, later put together in a single volume, concerned aspects of typology, body fabrics and manufacturing centres, as well as glaze and technological analyses (Mason and Keall 1990; 1991; Mason 1995;

1997; Mason and Tite 1997; Mason 2004). Mason's approach to the presentation of the glaze composition data was based on his own typological classification of early Islamic Iraqi wares. He proposed six groups of bowls (BOG1-6) according to the styles of rims and bases (Mason 1997, 44; 2004, 24-29). A problematic issue regarding this classification is that it cannot be regarded as a principle for seriation; i.e. the different groups do not independently correspond to a progressive sequence and do not seem to have been supported by any constant attribute, such as stratigraphy or chronology. Mason's suggested sequence, however, ties in with his theory regarding the development of opaque white glazed wares discussed in chapter 1: that is, deliberate application of a tin-oxide containing slip under an alkali glaze with air bubbles, quartz grains and secondary forming crystals in the early periods transforming progressively towards a more smooth high-lead glazes containing tin-oxide particles dispersed throughout the thickness. These results confirmed the previous observations by Kleinmann (1991). However, this idea was later questioned by Paynter *et al.* (2004) who demonstrated the main reason the concentration of opacifying particles in the interface layer to be the settling of the particles in glazes rather than the application of a tin-oxide rich slip. Various aspects of Mason's research on the early Islamic glazed wares are discussed by Priestman (2011, 95-104) and will be considered when the results are examined in this chapter.

Amongst the more recent studies on early Islamic ceramics are the works by Wood *et al.* (2007; 2009) on Abbasid blue-on-white wares and their comparison with the imported Chinese wares between the eighth and ninth centuries AD.

## 3.2. GLAZED CERAMIC SAMPLES

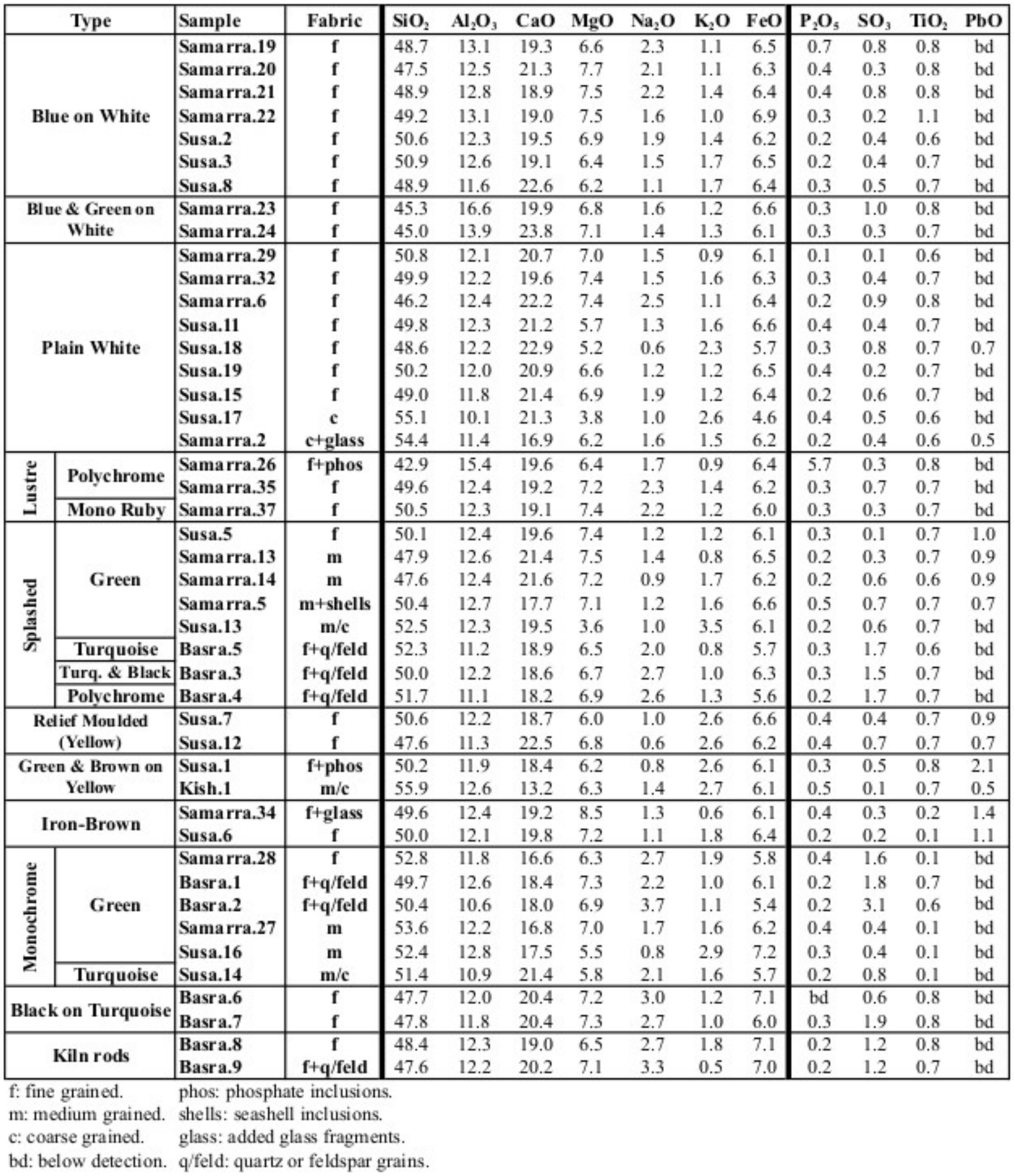
Twenty sherds from Samarra (thirteen from the Victoria and Albert Museum, London, and seven from the Museum für Islamische Kunst, Berlin collections), nineteen sherds from Susa (Musée du Louvre, Paris), nine sherds from Basra (Metropolitan Museum of Art, New York) and one sherd from Kish (Victoria and Albert Museum, London) were selected for examination. All the samples were considered for investigation of the bodies. However, as a result of weathering, the condition of glazes of only thirty-eight sherds was satisfactory for analysis.

## 3.3. RESULTS

### 3.3.1. Body

On the basis of the chemical compositions presented in Table 3.1, the bodies of the sherds from Samarra, Susa, Kish and Basra were produced from calcareous clays containing 16-26 wt% CaO. Despite the similar bulk compositions of these bodies, their fabrics vary significantly in texture. The majority of the bodies show very fine-grained fabrics. As observed in the SEM, the microstructures associated with one sample of lustreware (Samarra.26) and one sample of yellow polychrome glazed ware (Susa.1) also show dispersed inclusions of unusual peloidal phosphate phases with the composition $Ca_3(PO_4)_2$ in the very fine grained matrix. The abundance of this phase in the Samarra.26 sample raised the $P_2O_5$ content to about 6%. The bodies of about half of the samples analysed from Basra, including one kiln rod, shows the very fine grained matrix with large sized (~300 m) particles of quartz and feldspar added as temper. Bodies of some of the samples amongst the plain white wares, splashed green, black and white and the yellow polychrome glazed wares show medium to coarse sized inclusions associated with basic and intermediate igneous and metamorphic rocks. These bodies appear similar to those of the Eastern Mediterranean discussed in Chapter 2. The body of Samarra.2 shows deliberately added fragments of glass containing approximately 74% $SiO_2$, 2% PbO, 8% $(Na/K)_2O$ and 16% (Ca/Mg)O (Fig. 3.1).

Table 3.1. *EDS analysis of Southern Mesopotamian bodies (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*



| Type | | Sample | Fabric | $SiO_2$ | $Al_2O_3$ | CaO | MgO | $Na_2O$ | $K_2O$ | FeO | $P_2O_5$ | $SO_3$ | $TiO_2$ | PbO |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Blue on White** | | **Samarra.19** | **f** | 48.7 | 13.1 | 19.3 | 6.6 | 2.3 | 1.1 | 6.5 | 0.7 | 0.8 | 0.8 | bd |
| | | **Samarra.20** | **f** | 47.5 | 12.5 | 21.3 | 7.7 | 2.1 | 1.1 | 6.3 | 0.4 | 0.3 | 0.8 | bd |
| | | **Samarra.21** | **f** | 48.9 | 12.8 | 18.9 | 7.5 | 2.2 | 1.4 | 6.4 | 0.4 | 0.8 | 0.8 | bd |
| | | **Samarra.22** | **f** | 49.2 | 13.1 | 19.0 | 7.5 | 1.6 | 1.0 | 6.9 | 0.3 | 0.2 | 1.1 | bd |
| | | **Susa.2** | **f** | 50.6 | 12.3 | 19.5 | 6.9 | 1.9 | 1.4 | 6.2 | 0.2 | 0.4 | 0.6 | bd |
| | | **Susa.3** | **f** | 50.9 | 12.6 | 19.1 | 6.4 | 1.5 | 1.7 | 6.5 | 0.2 | 0.4 | 0.7 | bd |
| | | **Susa.8** | **f** | 48.9 | 11.6 | 22.6 | 6.2 | 1.1 | 1.7 | 6.4 | 0.3 | 0.5 | 0.7 | bd |
| **Blue & Green on White** | | **Samarra.23** | **f** | 45.3 | 16.6 | 19.9 | 6.8 | 1.6 | 1.2 | 6.6 | 0.3 | 1.0 | 0.8 | bd |
| | | **Samarra.24** | **f** | 45.0 | 13.9 | 23.8 | 7.1 | 1.4 | 1.3 | 6.1 | 0.3 | 0.3 | 0.7 | bd |
| **Plain White** | | **Samarra.29** | **f** | 50.8 | 12.1 | 20.7 | 7.0 | 1.5 | 0.9 | 6.1 | 0.1 | 0.1 | 0.6 | bd |
| | | **Samarra.32** | **f** | 49.9 | 12.2 | 19.6 | 7.4 | 1.5 | 1.6 | 6.3 | 0.3 | 0.4 | 0.7 | bd |
| | | **Samarra.6** | **f** | 46.2 | 12.4 | 22.2 | 7.4 | 2.5 | 1.1 | 6.4 | 0.2 | 0.9 | 0.8 | bd |
| | | **Susa.11** | **f** | 49.8 | 12.3 | 21.2 | 5.7 | 1.3 | 1.6 | 6.6 | 0.4 | 0.4 | 0.7 | bd |
| | | **Susa.18** | **f** | 48.6 | 12.2 | 22.9 | 5.2 | 0.6 | 2.3 | 5.7 | 0.3 | 0.8 | 0.7 | 0.7 |
| | | **Susa.19** | **f** | 50.2 | 12.0 | 20.9 | 6.6 | 1.2 | 1.2 | 6.5 | 0.4 | 0.2 | 0.7 | bd |
| | | **Susa.15** | **f** | 49.0 | 11.8 | 21.4 | 6.9 | 1.9 | 1.2 | 6.4 | 0.2 | 0.6 | 0.7 | bd |
| | | **Susa.17** | **c** | 55.1 | 10.1 | 21.3 | 3.8 | 1.0 | 2.6 | 4.6 | 0.4 | 0.5 | 0.6 | bd |
| | | **Samarra.2** | **c+glass** | 54.4 | 11.4 | 16.9 | 6.2 | 1.6 | 1.5 | 6.2 | 0.2 | 0.4 | 0.6 | 0.5 |
| **Lustre** | **Polychrome** | **Samarra.26** | **f+phos** | 42.9 | 15.4 | 19.6 | 6.4 | 1.7 | 0.9 | 6.4 | 5.7 | 0.3 | 0.8 | bd |
| | | **Samarra.35** | **f** | 49.6 | 12.4 | 19.2 | 7.2 | 2.3 | 1.4 | 6.2 | 0.3 | 0.7 | 0.7 | bd |
| | **Mono Ruby** | **Samarra.37** | **f** | 50.5 | 12.3 | 19.1 | 7.4 | 2.2 | 1.2 | 6.0 | 0.3 | 0.3 | 0.7 | bd |
| **Splashed** | **Green** | **Susa.5** | **f** | 50.1 | 12.4 | 19.6 | 7.4 | 1.2 | 1.2 | 6.1 | 0.3 | 0.1 | 0.7 | 1.0 |
| | | **Samarra.13** | **m** | 47.9 | 12.6 | 21.4 | 7.5 | 1.4 | 0.8 | 6.5 | 0.2 | 0.3 | 0.7 | 0.9 |
| | | **Samarra.14** | **m** | 47.6 | 12.4 | 21.6 | 7.2 | 0.9 | 1.7 | 6.2 | 0.2 | 0.6 | 0.6 | 0.9 |
| | | **Samarra.5** | **m+shells** | 50.4 | 12.7 | 17.7 | 7.1 | 1.2 | 1.6 | 6.6 | 0.5 | 0.7 | 0.7 | 0.7 |
| | | **Susa.13** | **m/c** | 52.5 | 12.3 | 19.5 | 3.6 | 1.0 | 3.5 | 6.1 | 0.2 | 0.6 | 0.7 | bd |
| | **Turquoise** | **Basra.5** | **f+q/feld** | 52.3 | 11.2 | 18.9 | 6.5 | 2.0 | 0.8 | 5.7 | 0.3 | 1.7 | 0.6 | bd |
| | **Turq. & Black** | **Basra.3** | **f+q/feld** | 50.0 | 12.2 | 18.6 | 6.7 | 2.7 | 1.0 | 6.3 | 0.3 | 1.5 | 0.7 | bd |
| | **Polychrome** | **Basra.4** | **f+q/feld** | 51.7 | 11.1 | 18.2 | 6.9 | 2.6 | 1.3 | 5.6 | 0.2 | 1.7 | 0.7 | bd |
| **Relief Moulded (Yellow)** | | **Susa.7** | **f** | 50.6 | 12.2 | 18.7 | 6.0 | 1.0 | 2.6 | 6.6 | 0.4 | 0.4 | 0.7 | 0.9 |
| | | **Susa.12** | **f** | 47.6 | 11.3 | 22.5 | 6.8 | 0.6 | 2.6 | 6.2 | 0.4 | 0.7 | 0.7 | 0.7 |
| **Green & Brown on Yellow** | | **Susa.1** | **f+phos** | 50.2 | 11.9 | 18.4 | 6.2 | 0.8 | 2.6 | 6.1 | 0.3 | 0.5 | 0.8 | 2.1 |
| | | **Kish.1** | **m/c** | 55.9 | 12.6 | 13.2 | 6.3 | 1.4 | 2.7 | 6.1 | 0.5 | 0.1 | 0.7 | 0.5 |
| **Iron-Brown** | | **Samarra.34** | **f+glass** | 49.6 | 12.4 | 19.2 | 8.5 | 1.3 | 0.6 | 6.1 | 0.4 | 0.3 | 0.2 | 1.4 |
| | | **Susa.6** | **f** | 50.0 | 12.1 | 19.8 | 7.2 | 1.1 | 1.8 | 6.4 | 0.2 | 0.2 | 0.1 | 1.1 |
| **Monochrome** | **Green** | **Samarra.28** | **f** | 52.8 | 11.8 | 16.6 | 6.3 | 2.7 | 1.9 | 5.8 | 0.4 | 1.6 | 0.1 | bd |
| | | **Basra.1** | **f+q/feld** | 49.7 | 12.6 | 18.4 | 7.3 | 2.2 | 1.0 | 6.1 | 0.2 | 1.8 | 0.7 | bd |
| | | **Basra.2** | **f+q/feld** | 50.4 | 10.6 | 18.0 | 6.9 | 3.7 | 1.1 | 5.4 | 0.2 | 3.1 | 0.6 | bd |
| | | **Samarra.27** | **m** | 53.6 | 12.2 | 16.8 | 7.0 | 1.7 | 1.6 | 6.2 | 0.4 | 0.4 | 0.1 | bd |
| | | **Susa.16** | **m** | 52.4 | 12.8 | 17.5 | 5.5 | 0.8 | 2.9 | 7.2 | 0.3 | 0.4 | 0.1 | bd |
| | **Turquoise** | **Susa.14** | **m/c** | 51.4 | 10.9 | 21.4 | 5.8 | 2.1 | 1.6 | 5.7 | 0.2 | 0.8 | 0.1 | bd |
| **Black on Turquoise** | | **Basra.6** | **f** | 47.7 | 12.0 | 20.4 | 7.2 | 3.0 | 1.2 | 7.1 | bd | 0.6 | 0.8 | bd |
| | | **Basra.7** | **f** | 47.8 | 11.8 | 20.4 | 7.3 | 2.7 | 1.0 | 6.0 | 0.3 | 1.9 | 0.8 | bd |
| **Kiln rods** | | **Basra.8** | **f** | 48.4 | 12.3 | 19.0 | 6.5 | 2.7 | 1.8 | 7.1 | 0.2 | 1.2 | 0.8 | bd |
| | | **Basra.9** | **f+q/feld** | 47.6 | 12.2 | 20.2 | 7.1 | 3.3 | 0.5 | 7.0 | 0.2 | 1.2 | 0.7 | bd |

| | |
|---|---|
| f: fine grained. | phos: phosphate inclusions. |
| m: medium grained. | shells: seashell inclusions. |
| c: coarse grained. | glass: added glass fragments. |
| bd: below detection. | q/feld: quartz or feldspar grains. |

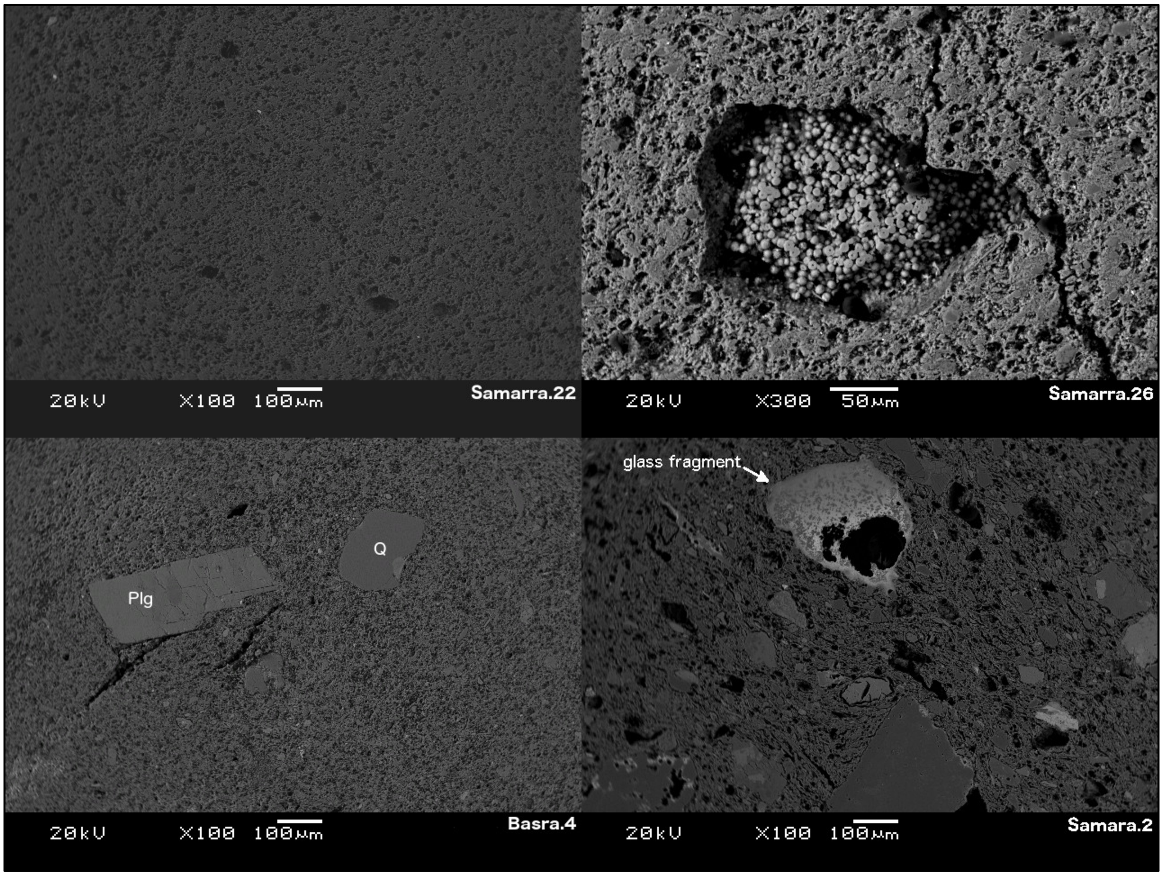
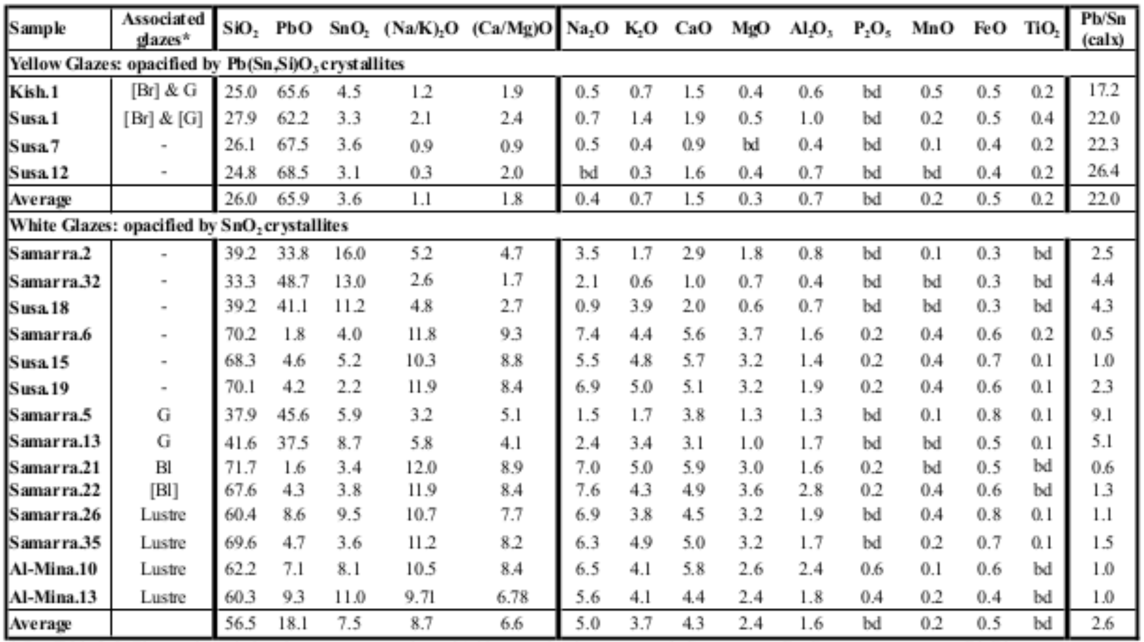


Figure 3.1. Backscattered *SEM photomicrographs of Southern Mesopotamian bodies. Samarra.22: showing fine-grained clay; Samarra.26: showing phosphate inclusions in a fine-grained clay matrix; Basra.4: showing dispersed quartz and feldspar (plg) grains in a fine-grained clay matrix; Samarra.2: showing coarse grained body fabric including added fragments of glass. Q: Quartz, Pl: Plagioclase.*

### 3.3.2. Glaze

Composition of opaque yellow and white glazes is given in Table 3.2. The Pb/Sn ratios that represent the calces used are significantly higher in opaque yellow than opaque white glazes. Similar to the Eastern Mediterranean wares, all the opaque yellow glazes are high lead. In contrast, the opaque white glazes are either lead-alkali or alkali-lead. The latter was not seen amongst the Eastern Mediterranean opaque white glazes and represents a further compositional group. The microstructures show well-formed lead-tin-oxide type II crystallites ($Pb(Sn,Si)O_3$) in the yellow glazes and a scatter of tin-oxide ($SnO_2$) crystallites in the white glazes (Figs. 3.2 and 3.3).

Table 3.2. *EDS analysis of the Southern Mesopotamian opaque yellow and white glazes (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*



| Sample | Associated glazes* | $SiO_2$ | PbO | $SnO_2$ | $(Na/K)_2O$ | (Ca/Mg)O | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | $P_2O_5$ | MnO | FeO | $TiO_2$ | Pb/Sn (calx) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Yellow Glazes: opacified by $Pb(Sn,Si)O_3$ crystallites** | | | | | | | | | | | | | | | | |
| **Kish.1** | [Br] & G | 25.0 | 65.6 | 4.5 | 1.2 | 1.9 | 0.5 | 0.7 | 1.5 | 0.4 | 0.6 | bd | 0.5 | 0.5 | 0.2 | 17.2 |
| **Susa.1** | [Br] & [G] | 27.9 | 62.2 | 3.3 | 2.1 | 2.4 | 0.7 | 1.4 | 1.9 | 0.5 | 1.0 | bd | 0.2 | 0.5 | 0.4 | 22.0 |
| **Susa.7** | - | 26.1 | 67.5 | 3.6 | 0.9 | 0.9 | 0.5 | 0.4 | 0.9 | bd | 0.4 | bd | 0.1 | 0.4 | 0.2 | 22.3 |
| **Susa.12** | - | 24.8 | 68.5 | 3.1 | 0.3 | 2.0 | bd | 0.3 | 1.6 | 0.4 | 0.7 | bd | bd | 0.4 | 0.2 | 26.4 |
| **Average** | | 26.0 | 65.9 | 3.6 | 1.1 | 1.8 | 0.4 | 0.7 | 1.5 | 0.3 | 0.7 | bd | 0.2 | 0.5 | 0.2 | 22.0 |
| **White Glazes: opacified by $SnO_2$ crystallites** | | | | | | | | | | | | | | | | |
| **Samarra.2** | - | 39.2 | 33.8 | 16.0 | 5.2 | 4.7 | 3.5 | 1.7 | 2.9 | 1.8 | 0.8 | bd | 0.1 | 0.3 | bd | 2.5 |
| **Samarra.32** | - | 33.3 | 48.7 | 13.0 | 2.6 | 1.7 | 2.1 | 0.6 | 1.0 | 0.7 | 0.4 | bd | bd | 0.3 | bd | 4.4 |
| **Susa.18** | - | 39.2 | 41.1 | 11.2 | 4.8 | 2.7 | 0.9 | 3.9 | 2.0 | 0.6 | 0.7 | bd | bd | 0.3 | bd | 4.3 |
| **Samarra.6** | - | 70.2 | 1.8 | 4.0 | 11.8 | 9.3 | 7.4 | 4.4 | 5.6 | 3.7 | 1.6 | 0.2 | 0.4 | 0.6 | 0.2 | 0.5 |
| **Susa.15** | - | 68.3 | 4.6 | 5.2 | 10.3 | 8.8 | 5.5 | 4.8 | 5.7 | 3.2 | 1.4 | 0.2 | 0.4 | 0.7 | 0.1 | 1.0 |
| **Susa.19** | - | 70.1 | 4.2 | 2.2 | 11.9 | 8.4 | 6.9 | 5.0 | 5.1 | 3.2 | 1.9 | 0.2 | 0.4 | 0.6 | 0.1 | 2.3 |
| **Samarra.5** | G | 37.9 | 45.6 | 5.9 | 3.2 | 5.1 | 1.5 | 1.7 | 3.8 | 1.3 | 1.3 | bd | 0.1 | 0.8 | 0.1 | 9.1 |
| **Samarra.13** | G | 41.6 | 37.5 | 8.7 | 5.8 | 4.1 | 2.4 | 3.4 | 3.1 | 1.0 | 1.7 | bd | bd | 0.5 | 0.1 | 5.1 |
| **Samarra.21** | Bl | 71.7 | 1.6 | 3.4 | 12.0 | 8.9 | 7.0 | 5.0 | 5.9 | 3.0 | 1.6 | 0.2 | bd | 0.5 | bd | 0.6 |
| **Samarra.22** | [Bl] | 67.6 | 4.3 | 3.8 | 11.9 | 8.4 | 7.6 | 4.3 | 4.9 | 3.6 | 2.8 | 0.2 | 0.4 | 0.6 | bd | 1.3 |
| **Samarra.26** | Lustre | 60.4 | 8.6 | 9.5 | 10.7 | 7.7 | 6.9 | 3.8 | 4.5 | 3.2 | 1.9 | bd | 0.4 | 0.8 | 0.1 | 1.1 |
| **Samarra.35** | Lustre | 69.6 | 4.7 | 3.6 | 11.2 | 8.2 | 6.3 | 4.9 | 5.0 | 3.2 | 1.7 | bd | 0.2 | 0.7 | 0.1 | 1.5 |
| **Al-Mina.10** | Lustre | 62.2 | 7.1 | 8.1 | 10.5 | 8.4 | 6.5 | 4.1 | 5.8 | 2.6 | 2.4 | 0.6 | 0.1 | 0.6 | bd | 1.0 |
| **Al-Mina.13** | Lustre | 60.3 | 9.3 | 11.0 | 9.71 | 6.78 | 5.6 | 4.1 | 4.4 | 2.4 | 1.8 | 0.4 | 0.2 | 0.4 | bd | 1.0 |
| **Average** | | 56.5 | 18.1 | 7.5 | 8.7 | 6.6 | 5.0 | 3.7 | 4.3 | 2.4 | 1.6 | bd | 0.2 | 0.5 | bd | 2.6 |

* See Table 3.5; Glaze colours in brackets were not analaysed (either weatherd or not sampled).
'&' indicates that the glazes were applied on the same side of the sherd.

Br: Brown.
G: Green.
Bl: Blue.
bd: below detection.

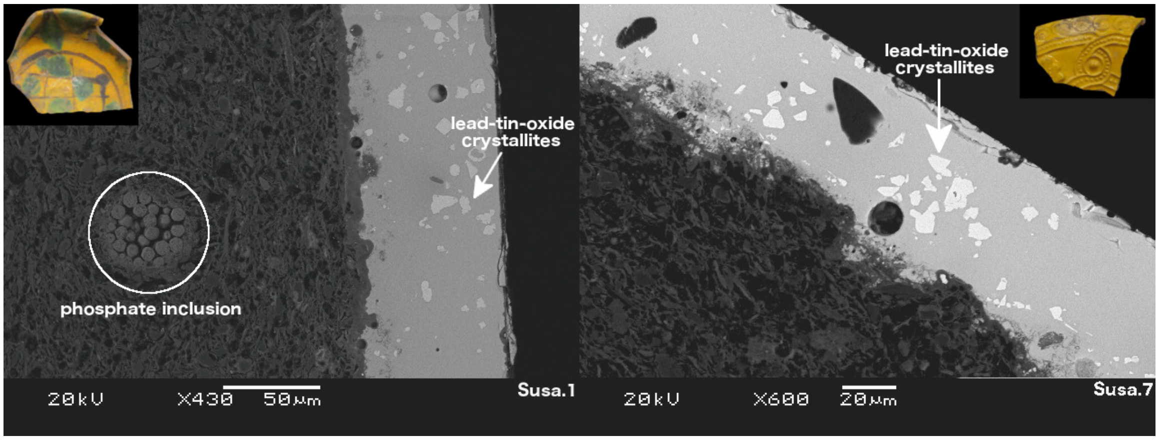


Figure 3.2. *Backscattered SEM photomicrographs of sections through Southern Mesopotamian yellow glazes and bodies. Susa.1 (green and brown on yellow glazed ware): showing scatters of well-formed lead-tin-oxide crystals (white) and air bubbles in the glaze, and phosphate inclusions in the body; Susa.7 (yellow relief-moulded ware): showing scatters of well-formed lead-tin-oxide crystals (white), quartz/feldspar particles (dark grey) and air bubbles in the glaze.*

A number of white glazes associated with various types of decorations were not opacified by tin-based crystallites (Table 3.3). Instead, they contained undissolved quartz grains, air bubbles and secondary crystals produced during firing that render a translucent quality and a whitish colour (Fig. 3.4, Samarra.19). With the exception of the high lead glaze of Susa.5, all the transluscent white glazes examined were alkali (Table 3.3). Further types of glazes not opacified by tin-based crystallites were the iron-brown, monochrome or black-on-turquoise glazes. The microstructure of these glazes show that they might or might not contain undissolved quartz grains (e.g. Fig 3.3, Basra.2). As shown in Table 3.4, these non-opacified coloured glazes have lead-alkali and alkali compositions.

The composition of coloured glazes associated with the yellow and white glazes discussed above is given in Table 3.5. As for the green glazes of the splashed decorated wares, if the oxides associated with the green colourant (CuO+ZnO) are subtracted and the composition of the green glazes are normalised to 100%, they are similar to those of the corresponding white glazes (Table 3.3; Fig 3.3, Samarra.13). EDS analysis of the relict cobalt crystals in the blue glazes indicates that they are of the Fe-Co-Zn type. Similar to the green glazes, if the oxides associated with the cobalt blue pigment (CoO+FeO+ZnO) in the blue glazes of Samarra.19 and Susa.2 are subtracted and the composition of the blue glazes are normalised to 100%, they are similar to the composition of the base white glazes (Table 3.5). None of the base glazes examined for these two sherds contained tin-oxide particles (Fig. 3.4). However, the composition of the Samarra.20, .21, and .23 glazes suggest that mixtures of cobalt pigment were either mixed or fritted with lead-oxide and then applied over the base white glazes (Fig. 3.4). The base glazes in these cases may or not have been opacified by tin-oxide.

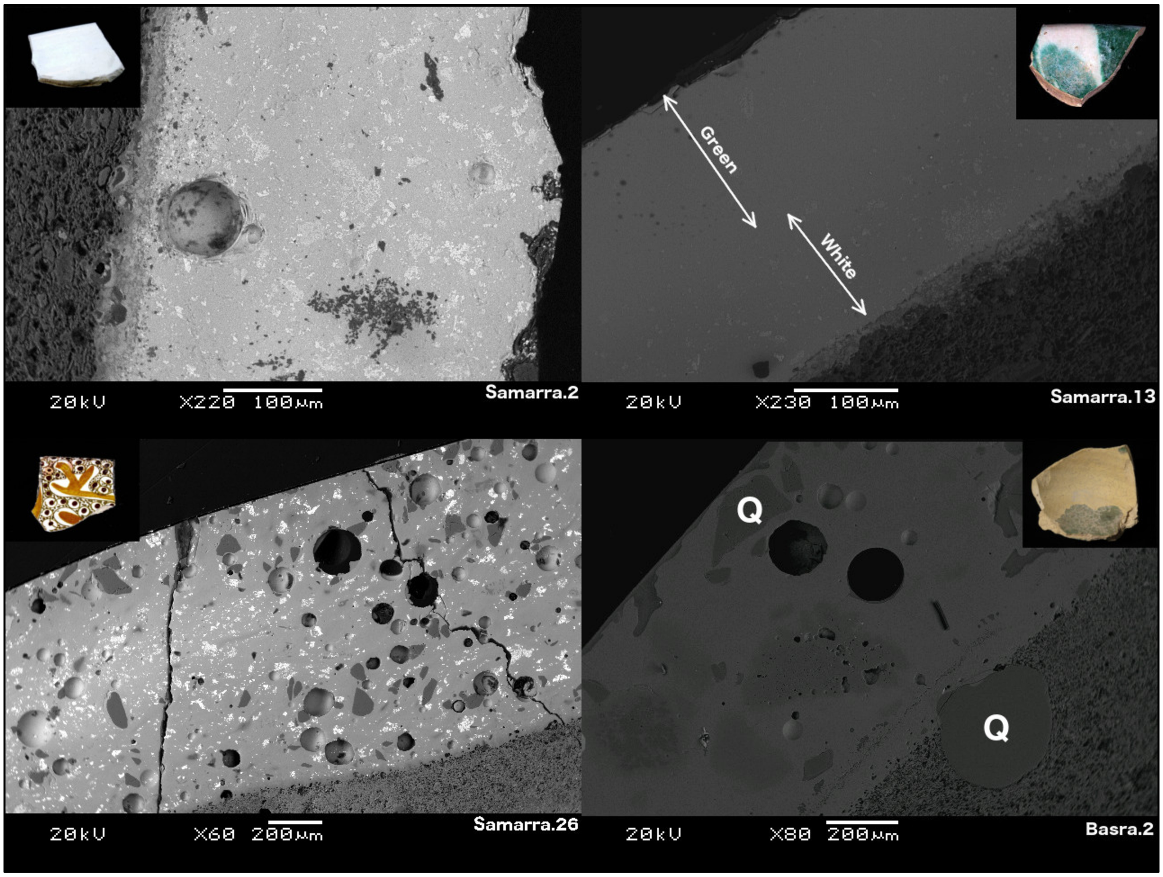
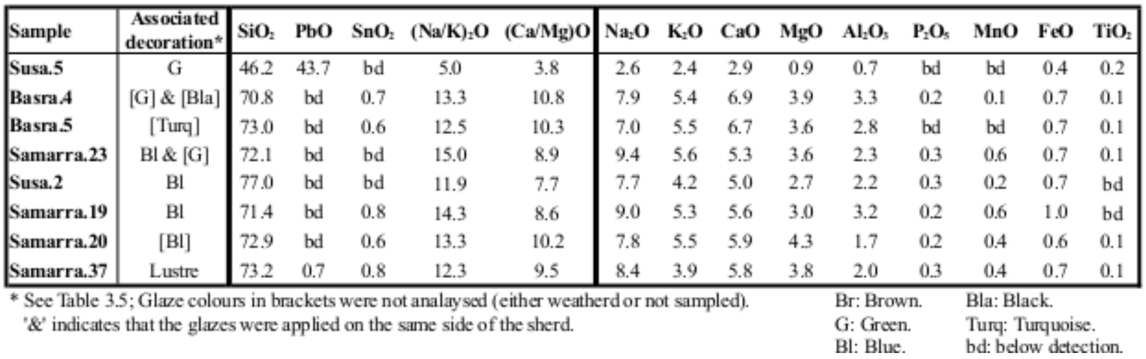


Figure 3.3. *Backscattered SEM photomicrographs of sections through Southern Mesopotamian glazes. Sherds Samarra.2 (plain white): showing a scatter of tin-oxide particles (white), and secondary crystallites of wollastonite and diopside (light grey) formed in the buffer layer; Samarra.13 (splashed green): showing double glaze layer with inner glaze containing a scatter of tin-oxide particles (white) corresponding to the white glaze and outer glaze being essentially free of particulate phases corresponding to the green glaze, secondary crystallites of wollastonite and diopside (light grey) formed in the buffer layer; Samarra.26 (polychrome lustreware): showing high concentration of tin-oxide particles (white), air bubbles and quartz/feldspar particles (dark grey) distributed through glaze; Basra.2 (monochrome green): showing a scatter of relatively large quartz/feldspar particles. Q: quartz.*

Table 3.3. *EDS analysis of non-opacified white glazes from Southern Mesopotamia (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*



| Sample | Associated decoration* | $SiO_2$ | PbO | $SnO_2$ | $(Na/K)_2O$ | (Ca/Mg)O | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | $P_2O_5$ | MnO | FeO | $TiO_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Susa.5** | G | 46.2 | 43.7 | bd | 5.0 | 3.8 | 2.6 | 2.4 | 2.9 | 0.9 | 0.7 | bd | bd | 0.4 | 0.2 |
| **Basra.4** | [G] & [Bla] | 70.8 | bd | 0.7 | 13.3 | 10.8 | 7.9 | 5.4 | 6.9 | 3.9 | 3.3 | 0.2 | 0.1 | 0.7 | 0.1 |
| **Basra.5** | [Turq] | 73.0 | bd | 0.6 | 12.5 | 10.3 | 7.0 | 5.5 | 6.7 | 3.6 | 2.8 | bd | bd | 0.7 | 0.1 |
| **Samarra.23** | Bl & [G] | 72.1 | bd | bd | 15.0 | 8.9 | 9.4 | 5.6 | 5.3 | 3.6 | 2.3 | 0.3 | 0.6 | 0.7 | 0.1 |
| **Susa.2** | Bl | 77.0 | bd | bd | 11.9 | 7.7 | 7.7 | 4.2 | 5.0 | 2.7 | 2.2 | 0.3 | 0.2 | 0.7 | bd |
| **Samarra.19** | Bl | 71.4 | bd | 0.8 | 14.3 | 8.6 | 9.0 | 5.3 | 5.6 | 3.0 | 3.2 | 0.2 | 0.6 | 1.0 | bd |
| **Samarra.20** | [Bl] | 72.9 | bd | 0.6 | 13.3 | 10.2 | 7.8 | 5.5 | 5.9 | 4.3 | 1.7 | 0.2 | 0.4 | 0.6 | 0.1 |
| **Samarra.37** | Lustre | 73.2 | 0.7 | 0.8 | 12.3 | 9.5 | 8.4 | 3.9 | 5.8 | 3.8 | 2.0 | 0.3 | 0.4 | 0.7 | 0.1 |

* See Table 3.5; Glaze colours in brackets were not analaysed (either weatherd or not sampled).
'&' indicates that the glazes were applied on the same side of the sherd.

Br: Brown.
G: Green.
Bl: Blue.
Bla: Black.
Turq: Turquoise.
bd: below detection.

Table 3.4. *EDS analysis of non-opaque glazes including iron-brown, monochrome and black-on-turquoise glazes (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*

| Type | | Sample | $SiO_2$ | PbO | $SnO_2$ | FeO | CuO | $(Na/K)_2O$ | (Ca/Mg)O | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | MnO | $TiO_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Iron-Brown | | Samarra.34 | 44.9 | 39.2 | 0.5 | 4.8 | 0.2 | 5.1 | 4.0 | 2.0 | 3.1 | 3.1 | 0.9 | 1.0 | 0.1 | 0.2 |
| | | Susa.6 | 52.8 | 18.0 | bd | 10.8 | bd | 9.5 | 6.8 | 2.9 | 6.6 | 5.1 | 1.6 | 1.9 | 0.1 | 0.1 |
| Monochrome | Green | Susa.16 | 32.4 | 55.3 | bd | 0.5 | 6.3 | 1.3 | 2.9 | 0.3 | 0.9 | 2.4 | 0.5 | 0.9 | 0.1 | 0.3 |
| | | Samarra.27 | 64.4 | 2.8 | 0.5 | 1.1 | 4.6 | 12.6 | 11.1 | 7.4 | 5.2 | 8.3 | 2.8 | 2.5 | 0.1 | 0.2 |
| | | Samarra.28 | 66.0 | 1.6 | bd | 1.2 | 3.5 | 12.9 | 9.8 | 8.9 | 4.0 | 7.2 | 2.6 | 4.7 | 0.1 | 0.2 |
| | | Basra.1 | 68.3 | bd | bd | 0.8 | 0.7 | 15.4 | 10.0 | 10.4 | 5.0 | 6.7 | 3.3 | 4.1 | 0.6 | 0.2 |
| | | Basra.2 | 69.9 | bd | bd | 1.3 | 3.2 | 12.7 | 9.4 | 7.4 | 5.3 | 6.6 | 2.8 | 3.4 | bd | 0.1 |
| | Turquoise | Susa.14 | 69.6 | bd | 0.5 | 0.8 | 2.7 | 13.5 | 9.0 | 9.1 | 4.4 | 6.8 | 2.3 | 3.7 | 0.1 | 0.1 |
| Black on Turquoise | | Basra.6 | 68.7 | bd | 0.5 | 0.9 | 0.2 | 13.1 | 12.3 | 8.7 | 4.4 | 8.4 | 3.9 | 4.1 | 0.1 | 0.2 |
| | | Basra.7 | 68.3 | bd | bd | 0.8 | 0.7 | 15.4 | 10.0 | 10.4 | 5.0 | 6.7 | 3.3 | 4.1 | 0.6 | 0.2 |

f: fine grained.
m: medium grained.
c: coarse grained.

glass: added glass fragments.
q/feld: quartz or feldspar grains.
bd: below detection.

Table 3.5. *EDS analysis of associated green and blue glazes (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*

| Sample | $SiO_2$ | PbO | $SnO_2$ | CuO | CoO | $(Na/K)_2O$ | (Ca/Mg)O | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | MnO | FeO | ZnO | $As_2O_3$ | $TiO_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Green glazes** | | | | | | | | | | | | | | | | | |
| Kish.1 | 24.8 | 65.7 | 4.8 | 0.3 | bd | 0.9 | 1.7 | 0.3 | 0.6 | 1.4 | 0.3 | 0.7 | 0.3 | 0.4 | 0.2 | bd | 0.2 |
| Samarra.5 | 37.0 | 44.4 | 6.1 | 1.6 | bd | 2.7 | 5.3 | 1.2 | 1.4 | 3.7 | 1.7 | 1.5 | 0.1 | 0.8 | 0.1 | 0.3 | 0.1 |
| Samarra.13 | 38.3 | 43.4 | 5.4 | 1.6 | bd | 4.5 | 3.8 | 2.1 | 2.4 | 2.9 | 0.9 | 1.2 | 0.3 | 0.7 | 0.4 | 0.3 | 0.1 |
| Samarra.14 | 37.4 | 45.1 | 0.5 | 5.6 | bd | 5.1 | 4.0 | 1.7 | 3.4 | 3.1 | 0.9 | 1.2 | 0.1 | 0.6 | 0.3 | bd | 0.1 |
| Susa.5 | 41.2 | 45.2 | 0.3 | 2.7 | bd | 4.3 | 3.7 | 2.1 | 2.2 | 2.5 | 1.2 | 0.7 | bd | 0.7 | 0.9 | bd | 0.3 |
| Susa.13 | 63.0 | 0.3 | 1.4 | 4.8 | bd | 15.5 | 9.7 | 6.3 | 9.2 | 6.3 | 3.4 | 1.7 | 1.3 | 0.9 | 1.1 | 0.2 | 0.1 |
| **Blue glazes** | | | | | | | | | | | | | | | | | |
| Samarra.19 | 63.8 | bd | 0.4 | 0.3 | 1.5 | 13.8 | 5.5 | 8.5 | 5.3 | 3.6 | 1.9 | 3.2 | 0.2 | 9.8 | 1.1 | bd | 0.2 |
| Samarra.20 | 68.1 | 3.1 | 0.6 | 0.2 | 0.3 | 13.1 | 8.6 | 8.1 | 5.0 | 5.2 | 3.4 | 2.3 | 0.2 | 2.4 | 0.9 | bd | 0.2 |
| Samarra.21 | 62.0 | 17.5 | 0.7 | 0.2 | 1.3 | 6.6 | 4.7 | 2.5 | 4.0 | 3.5 | 1.2 | 1.8 | bd | 4.2 | 0.9 | bd | 0.2 |
| Samarra.23 | 62.3 | 1.4 | 0.5 | 0.2 | 1.3 | 13.7 | 7.9 | 8.9 | 4.8 | 5.1 | 2.8 | 3.1 | 0.1 | 8.6 | 0.8 | bd | 0.1 |
| Susa.2 | 71.9 | 0.1 | 0.3 | 0.2 | 0.6 | 12.3 | 7.2 | 7.8 | 4.5 | 4.8 | 2.4 | 2.2 | 0.2 | 4.0 | 0.6 | 0.2 | 0.2 |

bd: below detection.

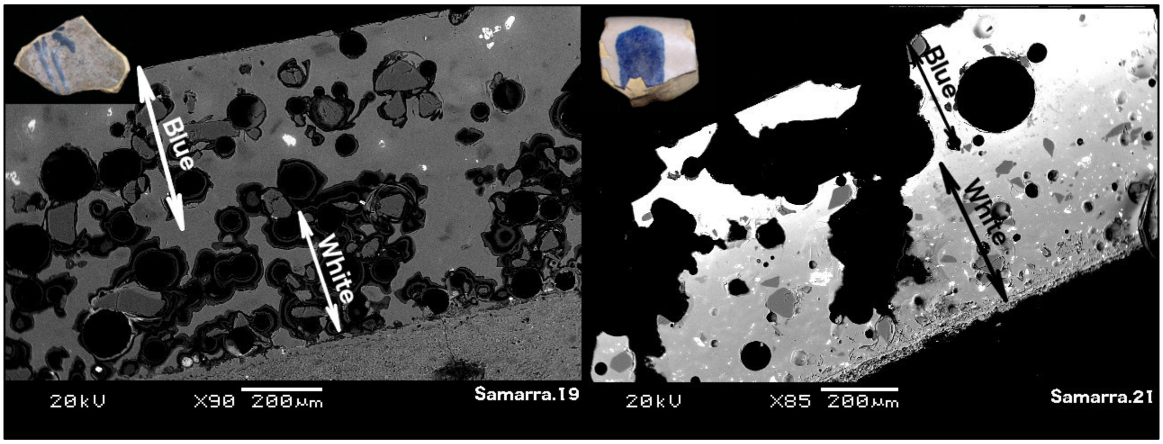


Figure 3.4. *Backscattered SEM photomicrographs of sections through blue-on-white glazes. Samarra.19 (blue-on-white glazed ware): showing a single layer glaze with a scatter of cobalt-bearing minerals (white) in the alkali glaze, quartz/feldspar particles (dark grey) and air bubbles; Samarra.21 (blue-on-white glazed ware): showing double glaze layer with inner glaze containing a scatter of tin-oxide particles (white), quartz/feldspar particles (dark grey) and air bubbles corresponding to the white alkali glaze, and outer glaze (bright white) corresponding to the blue lead glaze.*

## 3.4. HISTORICAL TREATISES

Textual evidence on the production of white tin-oxide glazes is not known from the early Islamic period. However, medieval treatises hold descriptions on the processes of preparation and application of tin-oxide white glazes. Although these documents are chronologically later than the sherds examined in this study, they provide valuable technical insight into the different aspects of their production. In the discussion that follows, three historical treatises are examined; namely *Nizāmī's book on precious stones* (*Jawāhirnāma-yi Nizāmī)* by Mohammad b. A.B. Jōwhari Nayshābūrī, dated AD 1196, *The virtues of jewels and the delicacies of perfumes (Arāyes al-Jawāher va Nafāyes al-Atāyeb)* by Abu'l-Qāsim A. Kāshānī, dated AD 1301, and *The three books of the potter's art (Li tre libri dell'arte del vasajo)* by Cipriano M. Piccolpasso, dated AD 1557. Based on the processes described, the recipes can be divided into two main categories: direct application and fritted glazing recipe.

### 3.4.1. Direct application glazing recipes

The accounts by *Nayshābūrī* and *Piccolpasso* describe processes in which glazing mixtures were applied directly on biscuit fired bodies.

***Mohammad Jōwhari Nayshābūrī, AD 1196***

Under the section *Minā va Talāvihāt-e Mokhtalef*, Nayshābūrī *states*:

"And before [the body] is completely dry, they paint on it and place it in the kiln and fire it; and once it is fired, they let it cool down and take it out of the kiln; and they cover the interior and exterior evenly with a wetted mixture of sintered tin calx (*kals-i qal'i* كلس /قلعى) and ground quartz (*hajar-i mahā*/حجر مها), and they leave it to dry. They make each vessel a case [saggar] (*qalāf*/غلاف) out of clay, of the appropriate size, and make it a lid, and put the vessel in the case and cover the lid with clay, and again they put it in the fire

[kiln] and increase the fire gradually until the colour becomes melted red; and then they let it cool down and take it out of fire*”* (Afshar 2004, 362).

#### ***Cipriano M. Piccolpasso, AD 1557***

Since the preparation of the glaze and the raw materials is explained in length and detail by Piccolpasso, only a brief summary is given here: A mixture of *marzacotto* (sintered mixture of sand, wine lees and sometimes common salt) with lead-tin calx, and sometimes further sand was painted over a biscuit fired body. *Coperta* (a transparent glaze) or other types of decoration were applied over the unfired glaze and all fired together for a second time (Lightbrown and Caiger-Smith 2007, 16-17).

### 3.4.2. Fritted glazing recipe

#### ***Abu’l-Qāsim Kāshānī, AD 1301***

An English translation of the Kāshānī’s treatise was published by Allan (1973) which, together with the original Persian text (Afshar 2006), provides the basis for the following accounts and discussions. Slight additions or alterations to Allan’s translation have been made.

After explaining the production of lead-tin calx (*asranj*; see Chapter 2), Kāshānī carries on to describe the firing of the glazing mixture into a frit which will be subsequently applied over clay bodies to produce the glaze during the second firing:

“After this one roasts one part of *qamsarī* stone (سنگ قمسری) and one part of *shakhār* (شخار) together, leaves it to cool, and for every three mans of the above-mentioned frit (*jowhar*/جوهر) one adds one man of this *asranj* (اسرنج). One then puts it back into the frit kiln (*fern-i jowhar*/فرن جوهر) for 12 hours brewing until the frit becomes a uniform opaque (*moṣmat*/مصمت) glassy mass. When it has become properly melted it is put into water by the ladleful and is stored, broken up and ground" (Allan 1973, 113; Afshar 2006, 343).

In order to follow Kāshānī's recipe, it is necessary to first identify the nature of the raw materials *qamsarī*, *shakhār and asranj.* There has been a wide agreement that *shakhār* designates the ash of a range of halophytic plants that provide the source of soda in the glaze, and that *asranj* refers to the lead-tin calx discussed in Chapter 2. However, the nature of *qamsarī* has been controversial. Wulff (1966,147) suggested that *qamsarī* was boro-calcite whereas Allan (1973, 115-6) suggested magnesium-silicate. These two proposals do not completely tie in either with the geological setting of the Qamsar region, or with the Kāshānī's earlier mention of *qamsarī* in his treatise as a mixture "attributed to a village of that name [in the district of Kāshān]. It is burned and broken up until it becomes a powder like white sugar" (Afshar 2006, 339). This description matches closely with the calcination process of limestone or dolomite. After being heated, mixtures of limestone and/or dolomite turn into a very fine white powder that runs smoothly. The Qamsar village of Kāshān, Iran, is rich in limestone and dolomitic rocks. These rocks which are geologically part of the Qom limestone formation constitute the main geological unit in the region (Radfar 1993; Mohammaddoost 2010; Matin and Pollard 2016). The limestones and dolomites of the Qamsar region exhibit a finely crystallised and extremely white appearance in general and contain various amounts of sand/silt (Matin 2013, 23, Fig. 3.3.b). When used in the glaze recipe, *qamsarī* should be considered as the main source of calcium, magnesium (alkaline earths) and silica (sand/silt).

## 3.5. DISCUSSSION

### 3.5.1. Body fabrics and the question of provenance

#### *Geological setting*

Before discussing body fabrics and their provenance, it is necessary to look at the sedimentation process by the Tigris and Euphrates Rivers in the Mesopotamian plain (see Chapter 1, Fig. 1.3). The Tigris and Euphrates rise out of the Taurus Mountains of southern Turkey, pass through modern Syria and Iraq, and, together with the Karun River, run into the Persian Gulf. The Tigris flows, compared to that of the Euphrates, are generally known to be less predictable and characterised by more violent flooding and discharging (Morozova 2005).

Our knowledge of the sediments of the Tigris and Euphrates Rivers and their variations is primarily based on the research studies conducted by the Geology Department of the Baghdad University during late 1960s. The results were published in two separate articles. The publication by Philip (1968) focused on the mineralogical variations of the sand-sized fraction of samples (between 0.25 mm and 125 μm in size) from 35 localities along the rivers. He identified seven minerals in the light fraction, i.e. quartz (dominant), cryptocrystalline silica (opal or chert), carbonates (consisting variable amounts of calcite, Mg-calcite and dolomite), chlorite, biotite, muscovite, and plagioclase feldspar, and four main mineral species making up more than 80 per cent of the heavy fraction, i.e. iron ores, epidotes, amphiboles and pyroxenes. The resulting conclusion was that "sediments of the Euphrates and Tigris (main channel) contained nearly the same mineral species in about the same relative frequencies" (Philip 1948, 43). The work by Berry *et al*. (1970) concentrated on the clay-sized fraction (< 3.91 μm) of suspended sediment samples from

36 localities. Based on their observations, the authors argued that "variations in clay mineralogy with time and distance are apparently very random", and were only able to suggest "general trends" in the clay mineral distribution of the sediments (Berry *et al.* 1970, 135).

As a general rule in the study of ceramic production sites, one of the main presumptions is that the distinctive characteristics (i.e. fingerprints) of ceramic sherds vary measurably between geological sources that have geographical significance. The above-mentioned investigations confirm the fairly consistent sediment types along the Tigris and Euphrates Rivers, as well as the several factors that might disrupt sedimentation processes with time, such as seasonal climatic variations and changes in the location of river courses. The question therefore arises as to what extent, if at all, the provenance study of ceramic sherds from the rather geologically homogenous alluvial plain of Mesopotamia is plausible.

The above considerations lead us to a discussion on the body fabrics of the Southern Mesopotamian sherds. Comparison of the sherd bodies with the Tigris-Euphrates type sediments shows a strong correlation. Despite variable body fabrics, the chemical composition of all the thirteen examined Samarra sherds showed a mainly calcareous clay with elevated MgO and FeO contents. The grain sizes, however, varied within the samples, from coarse to medium to fine-grained. One may assume as a general model that as the Tigris and Euphrates Rivers flow towards the south, their sedimentation becomes finer grained. Thus, the coarse to medium-grained bodies, similar to those presented in Chapter 2, can be associated to Eastern Mediterranean and Northern Mesopotamia, and the fine-grained bodies to Southern Mesopotamia (Table 3.2). There are some exceptions to the above criteria, such as seasonal changes in river flows and floods, and therefore any generalisations must be made cautiously.

Despite the general consistency of the Tigris-Euphrates type body fabrics, a few anomalous inclusions were identified. Fragments of glass were characterised in the coarse-grained body of the plain white sherd Samarra.2, presumably deliberately added by the potter to enhance the extent of vitrification (for comparable body fabrics see, Mason and Tite 1994). The coarse-grained body of the splashed green sherd Samarra.5 contained a variety of microfossils (for comparable body fabrics see, Chapter 2, Fig. 2.3). Peloidal phosphate phases were identified in the fine-grained bodies of Samarra.26 and Susa.1. Iraq is rich in phosphorite deposits of Late Cretaceous-Early Tertiary (Paleocene and Eocene) age, composing part of the giant extensive Tethyan phosphorite province. According to the geological investigations by Al-Bassam (2007), the Late Cretaceous deposits are generally poor grade and thin, and mainly contain bioclastic and lithoclastic phosphorites. The Paleocene deposits, on the other hand, are relatively high grade and thick, and contain phosphorite beds of peloidal and ooidal texture. The Eocene phosphorites are of thin to medium thickness, showing either peloidal or intraclastic textures. A quick look at the geological map reveals the best development of these deposits, particularly those of the high grade Paleocene, in the Western Desert of Iraq (Akashat and Swab phosphorite deposits). The phosphate inclusions suggest that the primary source of the clay used could have been located somewhere in western Iraq, where the Paleocene and Eocene peloidal phosphorite units are present. The description of the peloids of the Western Desert phosphorite deposits closely matches those seen on the sherd bodies. Thus, peloidal phosphates suggest a plausible indicator of the Western

Desert Iraqi deposits as the primary source of clays. These clays might have been collected in-situ in western Iraq or from the deposits washed down in southern Iraq. The quartz/feldspar inclusions characterised with the Southern Mesopotamian bodies of Basra propose another different body fabric. Due to their fundamental significance in provenance studies of early Islamic ceramics, the Basra body fabrics are discussed below in a separate subsection.

### ***Textual sources***

Leaving the scientific examination of body fabrics aside, based on the textual sources there have been two main schools of thoughts regarding the production centre of Samarra-type ceramics. The first, supported by Sarre (1925), suggested that Samarra itself was the production centre. This is based on the text given in Kitab al-Buldan by the 9th-century Arab historian, al-Yaqubi, who stated that: "he brought … from al-Basra people who make glass, pottery and mats, and he brought from al-Kufa people who make pottery" (Al-Yaqubi 1968, 164). This vision was challenged by Northedge (1994), amongst others, who noted that no kiln sites were recovered from the west bank of the Tigris, where Yaqubi located the pottery manufacturing site. The second school of thought believed that Samarra was not necessarily the production centre, and that it is plausible to suppose that the quality pottery of other workshops across the Abbasid caliphate were brought to Samarra, the capital city, for sale (e.g. Northedge 1994, 210). One of the most likely locations of a production centre was at Basra. Al-Azdi, a writer from Basra, records production of cups in Basra from "a white layer from Sawad [an area near Basra]" (Al-Azdi 1902, 46; cited in Tamari 1995, 137).

### *Basra body fabric*

The collection of ceramic fragments from Basra at the Metropolitan Museum of Art has held a key position in the provenance study of Early Islamic ceramics. Based on the findings of kiln rods and waste materials, Basra has been considered as a production centre. Results of previous studies demonstrated that Basra bodies have the typical fine-grained fabrics discussed above (Mason 1991; 2004; Hallett 2000). Thus, early Islamic sherds with fine-grained bodies, the lustre and the blue-on-white decorated wares in particular, were deemed to have been almost exclusively attributed to the production site of Basra; insofar as to the fine-grained bodies – labelled 'Southern Mesopotamian' in this study – became widely referred to as the 'Basra body' or 'Basra fabric'. Although this assumption may hold true to some extent, there are serious problems in associating Samarra type wares with fine-grained body fabrics exclusively to Basra. Firstly and most importantly, the glazed sherds found in Basra do not easily or convincingly fit into the Samarra ceramic typology (see Chapter 1 and Appendix D for photographs). Secondly, unlike previous reports, the examinations conducted in the present study demonstrated two types of body fabrics. The body of the majority of the Basra pieces examined (six out of nine), including one kiln rod, contained large grains of quartz or feldspar in the fine-grained matrix which might have been either naturally present in the clay or deliberately added by the potter. The rest of the sherds examined showed fine-grained bodies. On this basis, it appears that the body of the products of Basra went beyond a single type and were not confined to the fine-grain body wares.Finally, for the reasons discussed above, it is difficult to attribute fabric types to a certain production centre in Mesopotamia. The homogenous geology of this region means that the same clay types could be used in various production centres over wide geographic areas. Therefore, one may expect the possibility that the same clay used in the Basra workshop could have been as well used in other neighbouring production centres.

***Synthesis***

Bringing together the geological characteristics of the Mesopotamian plain, the historical textual accounts and the examination of body fabrics, a more realistic insight into the provenance study of Samarra ceramics can be achieved. The variations in the sediments of the Tigris and Euphrates do not show clear time- or distance-dependency and therefore the distinction of clay types across the rivers by common methods of provenance studies is unlikely.  The clay types used do not seem to have been particularly part of potters' technological choice but rather as determined geologically in a certain location for a specific time. The mentions of pottery workshops in the literary documents, however, highlight some of the possible production sites. Although the attribution of fabric types to some potential production sites has been conducted by means of provenanced materials in previous research studies, the fabric types might have been the same as those occurring on sherds of other- perhaps neighbouring- sites and should therefore not be assumed to be exclusive. For the reasons discussed above the general terms Northern Mesopotamian and Southern Mesopotamian will be used to refer respectively to the typical medium/coarse-grained and fine-grained calcareous bodies in the present study. This attribution is however subject to exemptions and should not be taken as a rigid rule. The use of site-based labels such as Samarra or Basra to refer to body fabrics will be strictly avoided.

### 3.5.2. Yellow glazes: technical aspects of production

The Southern Mesopotamian fabrics of the three samples Susa.1, .7 and .12 (Table 3.2) suggest that the Eastern Mediterranean yellow glazing technology continued into Iraq and western Iran and was practised locally. The composition of the Susa yellow glazes is similar to the Eastern Mediterranean yellow glazes reported in Chapter 2. The main difference is, however, in their microstructure. The lead-tin-oxide crystallites of the Susa glazes are comparatively more well-developed, their cubic forms can clearly be distinguished in the SEM photomicrographs, and their sizes reach up to 20 microns (Fig. 3.2). The glaze layers appear rather smooth with minimum quartz/feldspar grains and air bubbles. On the other hand, the lead-tin-oxide crystallites in the Eastern Mediterranean glazes and the replicated yellow glazes discussed in Chapter 2 exhibited scattered nucleation with predominantly irregular forms in general, often accompanied with quartz/feldspar grains and large air bubbles. This observation suggests that the Susa glazes were either given longer soaking times at the temperature where lead-tin-oxide crystallites formed, or their raw materials, i.e. calx and silica, were first pre-fired and then used as a frit to produce the glaze. Considering that fritting has been a long-established practice in Mesopotamia – due to the common use of alkali frits in glazes – the latter case is the more probable.

### 3.5.3. White glazes: technical aspects of production

Based on the results of replication experiments, it was discussed in Chapter 2 that addition of alkalis and alkaline earths to essentially lead glazes reduced the temperature at which tin-oxides formed from disassociation of lead-tin-oxide crystals (see sections 2.3.2 and 2.4.5). This in turn resulted in producing white glazes at lower temperatures. This observation leads us to argue that alkali and alkaline earths were essential ingredients for producing opaque white lead glazes. The $PbO:SnO_2$ ratios in these cases were dictated by the composition of the calces used. Textual documents suggest that the glazing mixtures could have been applied to bodies in different ways. The recipes given by Nayshābūrī and Piccolpasso suggest direct application of the glazes to the bodies, whereas Kāshānī described the fritting of the calx and its subsequent application to the bodies.

Based on the EDS analysis, plain opaque white glazes associated with fine-grained Southern Mesopotamian bodies can be classified in terms of their composition into two groups: lead-alkali and alkali-lead. The lead-alkali group is similar to the Eastern Mediterranean white glazes reported in Chapter 2. On the other hand, the alkali-lead group appears exclusive to the Southern Mesopotamian industry based on their fine body fabrics. It should be noted that the fragments of plain opaque glazes examined might in fact be a fragment of a blue (and green)-on-white decorated piece where no blue (or green) decoration was applied, or a piece of lustreware where the lustre decoration had disappeared through weathering. This is a cautionary note for the plain white sherds with alkali-lead glazes in particular, since their composition is similar to the blue-on-white and lustre decorated wares.

A key question that follows from the results reviewed so far relates to the interactions between the Eastern Mediterranean and Southern Mesopotamian potters. Under the Umayyad and Abbasid caliphates, cultural, social and technological interactions were facilitated. However, it is difficult to establish whether the technique of white glazing (regardless of its lead-alkali or alkali-lead composition) was originally developed in Eastern Mediterranean and subsequently transferred to Mesopotamia, or it was born out of an interaction between Eastern Mediterranean and Southern Mesopotamian potters. Production of alkali glazed ceramics has been an established tradition in Mesopotamia since about 1500 BC (Tite and Shortland 2008, 187-196) and can be regarded as a signature of the pre-Islamic Mesopotamian glaze making. The high contents of alkalis and alkaline earths in plain opaque white glazes might lead us to suggest potential impacts from the Mesopotamian tradition. However, in view of correspondence between glaze and glass making technologies, this argument seems to lose validity once we consider that the Late Roman/Byzantine tradition produced soda-lime silica glass that was also rich in alkalis and alkaline earths (e.g. Freestone 2006). The composition of Late Roman and Byzantine lead-tin-oxide opacified yellow glasses are alkali-lead and that of tin-oxide opacified white glasses are essentially alkali and continue into Islamic glass being alkali plus a few % PbO (Turner and Rooksby 1959; Brill 1999; Mark Wypyski, pers. comm). This suggests that the source of alkalis in white glazes might have been an inspiration from the Roman/Byzantine glassmaking tradition.

### 3.5.4. Islamic opaque white glazes and Chinese imported wares

It has been widely argued that the technology of tin-oxide opacification emerged in Iraq or western Iran in the 9th century in response to the Chinese imported stoneware and porcelain of the Tang dynasty (e.g. Lane 1947; Fehérvári 2000; Watson 2004). This ‘invention’ was considered an attempt by Islamic potters to imitate the whiteness and fine surface of the Chinese wares. Contrary to this popular belief, the results presented in Chapter 2 established that the application of lead-tin calx for opacification of glazes had begun in the 7th/8th century in the Eastern Mediterranean. While initially yellow glazes were produced by using mixtures of calx and silica, white glazes seem to have emerged subsequently by the addition of a few percentages of alkalis and alkaline earths to the glaze formula. Regardless of the uncertain dates for the beginnings of the yellow and white glazes, the technical links between their manufacture remains undeniable. The new evidence seems sufficient to revise the old story of Islamic-Chinese ceramic encounter.

At the time that Chinese wares arrived in the Mesopotamian region, the technology of white opaque glazing was well developed and ready to seek for more advanced methods of application and decoration. Under the newly increased demand for fine wares in the region, the technique of opaque glazing nurtured and flourished. Islamic potters made use of this already existing technology to produce a wide range of delicate and fine ceramics.

### 3.5.5. Blue-on-white and lustre decorated wares: standardised production?

The analytical and microstructural data presented above, as well as in a number of previous studies on early Islamic ceramics of Iraq and western Iran (Mason 2004, Wood *et al*. 2007; 2009), suggest several potential links between the production of blue-on-white and lustre decorated wares. Firstly, both types of wares were always exclusively associated with the fine-grained Southern Mesopotamian body fabrics. Secondly, the composition of the white glazes applied on the blue-on-white and lustre decorated wares appears similar in all cases. White glazes with alkali compositions (negligible PbO and $SnO_2$ contents), as well as alkali-lead compositions (tin-opacified) appear on these wares. The presence of alkalis is essential for the mechanisms of the ionic exchange that take place during the formation of the lustre layer (Pradell *et al*. 2008; Pradell 2016). However, no such necessity seems to be associated with the production of blue-on-white decorated wares. The PbO, on the other hand, improves the refractive index of the glaze (see section 3.4.5 below). These observations suggest that blue-on-white and lustre decorated wares could have been the products of related industries with standardised manufacture in Iraq or western Iran. However, it has remained obscure whether single or multiple workshops were involved in the production of these wares. The study of blue-on-white wares appears more enlightening in this respect. The general picture that emerges from the results suggests that several aspects of the manufacture of blue-on-white wares varied significantly.

EDS analysis of relicts of cobalt minerals showed that an Fe-Co-Zn colourant was used for the blue (also detected by Kleinmann 1991 and Wood *et al*. 2007). The question as to what the possible sources of these minerals could have been remains to be addressed by future research. Examination of cobalt ores from Kashan, Iran, demonstrated that the cobalt bearing minerals were rich in As and contained negligible Zn or Cr (Matin and Pollard 2015; 2016). Hence, it is unlikely that the source of Abbasid blue pigments was in the cobalt mine of Kashan.

On the basis of the composition and microstructure of the blue glazes, it seems that two main methods of application were used. The cobalt pigment was either applied directly over the base glaze, or was first mixed or fritted with lead-oxide and then applied over the base glaze. The base glazes might or not have been opacified by tin-oxide. Three methods of application of blue glazes, including those mentioned here, were also previously discussed by Wood *et al*. (2007). It was argued that the relatively wide range of technical variations applied on the blue-on-white wares "suggest either that a great deal of experimentation was taking place at this time, or that the sherds examined represent a number of separate workshops that were following rather different approaches to the manufacture of Abbasid blue-on-white earthenware" (Wood *et al*. 2007, 679).

Further variation of blue-on-white wares can be identified on the grounds of their decorative patterns. Tamari (1995, 127-45) characterised four broad styles, namely palmette designs, half-moon borders, hatched designs and epigraphic designs. These groups were tentatively ascribed to different workshops according to the inscriptions they bore. However, whether the names referred to the potters, decorators or both has remained controversial.

### 3.5.6. Grey glazed wares

Grey glazes appear in place of white glaze in some of the lustre and the blue (and green)-on-white decorated wares. It has been unclear whether their application was a deliberate effect made by the potters or a result of weathering during subsequent burial of the sherds. Crowe (1975-7) argued that grey glazes featured a further development in the production of early Abbasid glazed wares to provide a grey background in the place of white. She suggested that the grey tint could be the result of addition of MnO to the glaze mixtures.

The grey glazes of the lustre and blue (and green)-on-white decorated sherds examined in the current study (sherds Samarra.19, .20, .37) contained negligible concentrations of MnO. Compared to the white glazes of the rest of the lustre and blue (and green)-on-white decorated sherds, the grey glazes always contained negligible PbO contents and always showed single, rather than double, layer glazes (see also, Wood *et al*. 2007, 669, 672, sherds P8744 and P8760 vs. P8740; Molina *et al*. 2014, 2565,7). This feature is represented in Figure 3.4 where the microstructure of the grey and white glazes of two blue-on-white decorated sherds is compared.

Investigation of the corrosion mechanisms of alkali glazes compared to alkali-lead glazes is complex and requires a thorough study of the roles of each oxide. Ignoring for current purposes the corrosion processes, based on the observations discussed above, we can often anticipate chemical compositions and microstructures similar to that of Samarra.19 when recognising a grey glaze in hand specimen (Table 3.3 and Fig. 3.4).

## 3.6. CONCLUSIONS

Chemical and microstructural analysis of the bodies of sherds from Iraq and western Iran characterised a fine-grained calcareous fabric as typical Southern Mesopotamian paste. The results suggested that due to the homogenous geology of the Mesopotamian region, further differentiation of production sites on the basis of body fabrics is not possible. Trace analysis of clays in the future may be useful to distinguish between different workshops.

The results of EDS analysis of glazes indicated that opaque yellow and white glazes with similar composition to those of Eastern Mediterranean were produced in Southern Mesopotamia. Although the composition of the opaque yellow glazes remained unchanged, the new methods of application and firing, i.e. fritting, seem to have been developed in Southern Mesopotamia. In the case of the opaque white glazes, however, major changes took place in terms of chemical composition, application techniques and decorative designs. In a context where there was a particular need for fine wares, partially satisfied by the Chinese imported wares, Islamic potters creatively embraced the technique of opaque white glazing to produce a wide range of new ceramic types.

# Chapter 4

# EASTERN IRAN AND CENTRAL ASIA: FURTHER CONTINUATION OF ISLAMIC OPAQUE GLAZED WARES

Early Islamic opaque glazed wares from Eastern Iran and Central Asia were known since excavations in the early twentieth century. However, few studies investigated the production of these wares and the technical aspects of their manufacture. This chapter presents the results of scientific analysis of the bodies and glazes of 9th/10th-century ceramics from three sites in Eastern Iran and Central Asia, namely Nishapur, Merv and Samarqand. Focused on a small number of samples, the current study represents an interim assessment of the production of these wares and their relations with the Eastern Mediterranean and Southern Mesopotamian wares.

## 4.1. REVIEW OF PREVIOUS CHEMICAL ANALYSIS

Chemical analyses of the opaque glazes and bodies of sherds from Eastern Iran and Central Asia has been rare and in most cases comprise too small a data set compared to those of Southern Mesopotamia, for instance. By presenting a review of previous analytical studies on opaque glazed sherds from Nishapur, Samarqand and Merv, this section aims to summarise some of the data relevant to the development and continuation of opaque glazing techniques in Eastern Iran and Central Asia.

### 4.1.1. Nishapur

In the most comprehensive work conducted to date on early Islamic glazed ceramics from Nishapur, Rante and Collinet (2013, 68-90) examined body fabrics of forty-seven sherds under optical microscope. Around 90% of the fabrics comprised mainly of intermediate volcanic inclusions and occasionally contained fragments of metamorphic rocks. Composition of one opaque yellow glaze was determined by SEM-EDS as part of this study (Rante and Collinet 2013, 116, sample 48). The results are re-produced in Table 4.1.

### 4.1.2. Merv

In a study by Casellato *et al.* (2007), eight samples of 9th/10th-century AD ceramics from Merv were investigated using an optical microscope, SEM-EDS and WDS. All the body pastes were manufactured using fine-grained clay with the main inclusions being quartz and feldspar (plagioclase and K-feldspar). The only opaque yellow glaze (TF) examined in this work was identified to have high-lead composition and included lead-tin-oxide ($PbSnO_3$) crystals as confirmed by XRD, EDS and WDS investigations. WDS data for the body and glaze of this sherd is reproduced in Table 4.1.

### 4.1.3. Samarqand

Published analytical data on the bodies and glazes of early Islamic sherds from Samarqand are extremely rare. One sample from Samarqand was analysed by Mason (2004, 133 and 135, sample ROM.18). However, the glaze was not opacified by tin-based particles and the data are irrelevant to the discussion here.

Table 4.1. *Published data on opaque yellow glazes from Nishapur and Merv.*

| Sample | Provenance | Technique | $SiO_2$ | PbO | $SnO_2$ | $(Na/K)_2O$ | $(Ca/Mg)O$ | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | $P_2O_5$ | $SO_3$ | MnO | $Cr_2O_3$ | FeO | CuO | ZnO | $TiO_2$ | Cl |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **48*** | Nishapur | SEM-EDS | 25.1 | 68.2 | 3.4 | 0.2 | 1.1 | 0.1 | 0.1 | 1.0 | 0.1 | 0.4 | bd | 0.6 | 0.1 | 0.1 | 0.3 | 0.1 | 0.1 | na | 0.3 |
| **TF**** | Merv | SEM-WDS | 10.82 | 65.03 | 23.96 | 0.25 | 0.33 | 0.1 | 0.15 | 0.3 | 0.03 | 0.2 | na | na | 0.01 | na | 0.15 | bd | na | na | na |

*Bouquillon *et al.* (2013, 116)
**Caselllato et al. (2007, 418)
na: not analysed.
bd: below detection.

## 4.2. GLAZED CERAMIC SAMPLES

Four sherds from Nishapur (Metropolitan Museum of Art), three sherds from Merv (two provided by Paul Wordsworth and one provided by Tim Williams) and two sherds from Samarqand (Victoria and Albert Museum) were sampled and selected for examination. All the sherds were considered for investigation of their bodies. However, the condition of glazes of only six sherds was sufficient for chemical analysis.

## 4.3.RESULTS

### 4.3.1. Body

EDS data presented in Table 4.2 indicate that the bulk composition of all the bodies examined have calcareous clays with CaO wt% ranging between 6-16 and do not show meaningful differences in clay composition with provenance. Their fabrics, however, can be distinguished by texture and different inclusions.

Table 4.2. *EDS analysis of Eastern Iranian and Central Asian bodies (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*

| Type | Sample | Fabric | $SiO_2$ | $Al_2O_3$ | CaO | MgO | $Na_2O$ | $K_2O$ | FeO | $P_2O_5$ | $SO_3$ | MnO | $TiO_2$ | PbO |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Opaque Yellow-Ware (overall)** | **Nishapur.1** | **immature** | 62.7 | 13.7 | 6.7 | 2.5 | 1.8 | 3.1 | 5.2 | 1.1 | 2.1 | 0.1 | 0.9 | bd |
| | **Nishapur.2** | **immature** | 60.8 | 13.7 | 10.0 | 3.3 | 1.7 | 3.1 | 5.0 | 0.5 | 1.1 | 0.1 | 0.7 | bd |
| | **Nishapur.4** | **immature** | 60.7 | 13.3 | 9.5 | 4.0 | 1.7 | 2.7 | 5.6 | 0.8 | 0.8 | 0.1 | 0.6 | bd |
| | **Nishapur.9** | **immature** | 61.2 | 13.5 | 10.7 | 3.6 | 1.6 | 2.9 | 4.6 | 0.5 | 0.7 | 0.1 | 0.6 | bd |
| **Opaque Yellow-Ware (painted)** | **Merv.1** | **silty alluvial** | 55.7 | 14.0 | 14.8 | 3.7 | 1.2 | 3.1 | 5.5 | 0.4 | 0.2 | 0.1 | 0.6 | 0.5 |
| | **Merv.2** | **silty alluvial** | 53.3 | 15.6 | 15.9 | 3.9 | 2.1 | 1.5 | 5.7 | 0.5 | 0.6 | 0.1 | 0.7 | bd |
| | **Merv.3** | **silty alluvial** | 53.7 | 15.4 | 15.2 | 3.7 | 2.4 | 2.9 | 5.3 | 0.4 | 0.3 | 0.1 | 0.6 | bd |
| **Opaque White-Ware** | **Samarqand.1** | **f** | 61.8 | 11.4 | 13.0 | 4.4 | 1.2 | 2.7 | 4.0 | 0.5 | 0.2 | 0.1 | 0.7 | bd |
| | **Samarqand.3** | **f** | 62.4 | 12.0 | 10.9 | 5.2 | 1.2 | 2.6 | 4.3 | 0.4 | 0.2 | 0.1 | 0.7 | bd |
| | **Samarqand.6** | **f** | 64.3 | 12.2 | 8.8 | 4.3 | 1.4 | 2.6 | 4.6 | 0.5 | 0.3 | 0.1 | 0.7 | bd |

f: fine grained.

### *Nishapur*

The bodies of sherds from Nishapur constituted of highly immature fabrics, dominated by sub-angular fragments of quartz, plagioclase, potassium-feldspar, mica, calcite and talc, and rock fragments showing assemblages of sphene, magnetite/titanium magnetite, and chlorite. Geologically, it can be hypothesised that the Nishapur bodies correspond to sediments associated with intermediate igneous and metamorphic rocks deposited not far from their source. Given that Nishapur is situated at the foot of the Binalud Mountains, the mineral assemblages probably represent sediments of the andesites of the Eocene volcanic rocks in the Binalud Range.

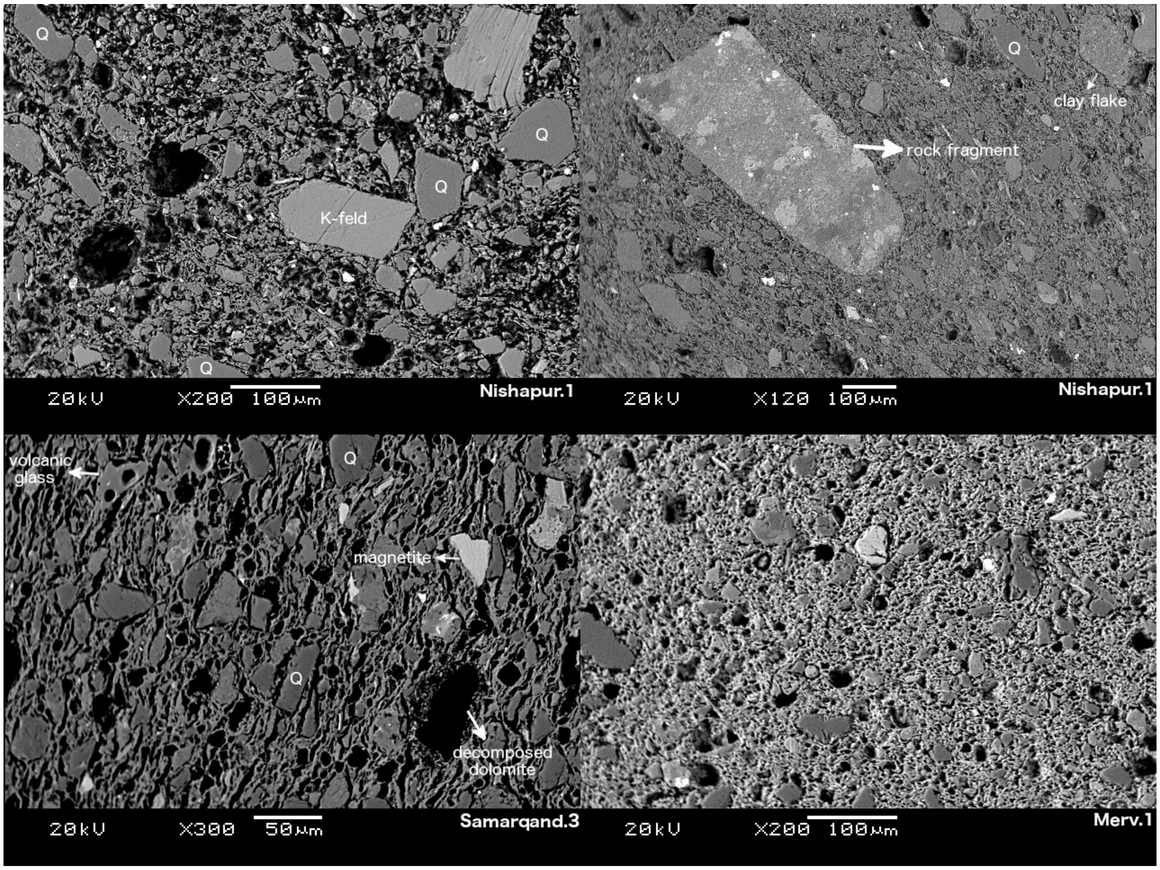


Figure 4.1. *Backscattered SEM photomicrographs of bodies. Sherds Nishapur.1: showing immature, unsorted fabric constituted of minerals and rock fragments corresponding to intermediate igneous and metamorphic units; Samarqand.3: showing medium-grained alluvial silty clay containing minerals corresponding to basic to intermediate igneous units, the body also includes grains of dolomite decomposed during firing; Merv.1: showing fine-grained clay. Q: Quartz, K-feld: Potassium feldspar.*

### *Merv*

The bodies of Merv sherds are very fine-grained and seem to reflect sediments that were deposited distant from their primary sources. The most probable source could have been the local clay from the delta fan created by the Murghab River on which the ancient city of Merv was located.

### *Samarqand*

Samarqand body fabrics represent alluvial silty clays and show a general linear orientation (Fig. 4.1). It comprises a high density of angular inclusions of quartz, plagioclase, mica, amphibole, volcanic glass (tuff), and dolomite grains that were decomposed during firing. The mineral types and the oriented texture of the fabric lead us to point to the highly deformed granodiorite-granite complexes in the Zarafshan Mountains, south of Samarqand, as the primary source of the sediments (Brookfield 2000).

Table 4.3. *EDS analysis of opaque glazes from Eastern Iran and Central Asia (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*

| Sample | Associated glazes* | $SiO_2$ | PbO | $SnO_2$ | $(Na/K)_2O$ | (Ca/Mg)O | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | $P_2O_5$ | MnO | FeO | $TiO_2$ | Pb/Sn |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Yellow Glazes: opacified by $Pb(Sn,Si)O_3$ crystallites** | | | | | | | | | | | | | | | | |
| **Nishapur.1** | G | 24.0 | 67.2 | 2.8 | 0.8 | 3.0 | 0.2 | 0.6 | 2.5 | 0.5 | 1.7 | bd | bd | 0.5 | bd | 28.3 |
| **Nishapur.2** | [G] | 24.7 | 68.1 | 4.9 | 0.7 | 0.8 | 0.3 | 0.4 | 0.5 | 0.3 | 0.4 | bd | 0.1 | 0.2 | bd | 16.4 |
| **Nishapur.9** | [G] | 26.8 | 65.4 | 4.7 | 0.5 | 1.2 | 0.4 | 0.1 | 1.0 | 0.2 | 0.8 | bd | bd | 0.6 | bd | 16.4 |
| **Merv.1** | [Br] | 27.6 | 65.8 | 3.1 | 0.7 | 1.1 | 0.2 | 0.5 | 0.9 | 0.2 | 1.1 | bd | 0.2 | 0.3 | 0.1 | 25.0 |
| **Average** | | 25.8 | 66.6 | 3.9 | 0.7 | 1.5 | 0.3 | 0.4 | 1.2 | 0.3 | 1.0 | bd | 0.1 | 0.4 | 0.1 | 21.5 |
| **White Glazes: opacified by $SnO_2$ crystallites** | | | | | | | | | | | | | | | | |
| **Samarqand.3** | G | 62.9 | 14.7 | 3.4 | 7.8 | 6.4 | 4.9 | 2.9 | 3.9 | 2.5 | 4.1 | bd | bd | 0.5 | 0.2 | 5.0 |
| **Samarqand.6** | [Br] & [Am] | 55.9 | 18.8 | 6.0 | 9.5 | 5.5 | 6.5 | 3.0 | 3.9 | 1.6 | 3.6 | 0.3 | bd | 0.4 | bd | 3.7 |
| **Average** | | 59.4 | 16.8 | 4.7 | 8.6 | 5.9 | 5.7 | 2.9 | 3.9 | 2.0 | 3.9 | 0.3 | bd | 0.4 | 0.2 | 4.4 |

* See Table 4.4; Glaze colours in brackets were not analysed (either weathered or not sampled).
G: Green. Am: Amber.
Br: Brown. bd: below detection.

Table 4.4. *EDS analysis of green glazes (wt%; obtained averaging three to five area measurements for each sample; normalised to 100%).*

| Sample | $SiO_2$ | PbO | $SnO_2$ | CuO | $(Na/K)_2O$ | (Ca/Mg)O | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | $P_2O_5$ | MnO | FeO | $TiO_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Nishapur.1** | 23.6 | 64.5 | 1.7 | 2.6 | 1.0 | 2.8 | 0.2 | 0.7 | 2.3 | 0.5 | 1.6 | bd | bd | 0.8 | 0.1 |
| **Samarqand.3** | 59.3 | 17.1 | 2.8 | 1.2 | 8.5 | 5.8 | 5.6 | 2.9 | 4.0 | 1.8 | 3.9 | 0.5 | 0.1 | 0.5 | bd |

bd: below detection.

### 4.3.2. Glaze

Table 4.3 indicates that the composition of the yellow glazes from Nishapur and Merv are all high lead and appear consistent with those of the Eastern Mediterranean and Southern Mesopotamia (Chapters 2 and 3). The white glazes from Samarqand, on the other hand, contain slightly more lead than combined alkalis and alkaline earths and are categorised as lead-alkali. The microstructures of the yellow and white glazes show respectively dispersion of lead-tin-oxide type II and tin-oxide particles throughout thicknesses. The two green glaze types from Nishapur and Samarqand (Table 4.4) are similar to those of the yellow and white glazes respectively except for addition of CuO and reduced $SnO_2$ concentrations.

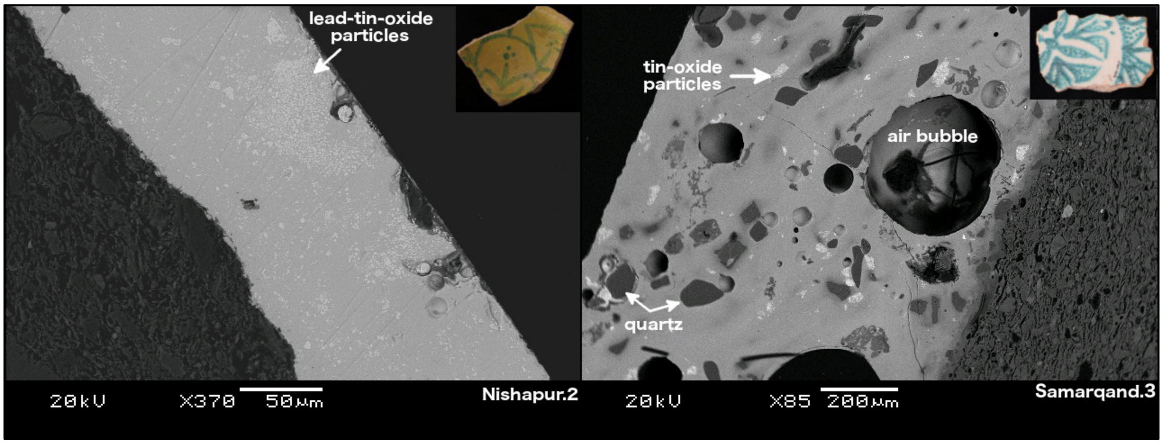


Figure 4.2. *Backscattered SEM photomicrograph of sections through glazes and bodies of Nishapur.2: showing a dense scatter of lead-tin-oxide micro-crystallites in the yellow glaze, and Samarqand.3: showing dispersed tin-oxide micro-crystallites, air bubbles and undissolved quartz grains in the white glaze.*

## 4.4. DISCUSSION

### 4.4.1. Body fabrics and the question of provenance

Unlike the general homogeneity of the Eastern Mediterranean and Southern Mesopotamian body fabrics, the bodies of sherds from Nishapur, Samarqand and Merv are consistent internally but differ significantly from each other. Therefore, the results of the study of bodies indicate that they are the products of three distinct centres.

### 4.4.2. Yellow glazes: technical aspects of production

The compositions of the opaque yellow glazes are high lead and thus consistent with those of the western Islamic world. The results suggest a further phase of continuation of this technology. The microstructure of the opaque yellow wares indicates that the lead-tin-oxide crystallites appear poorly developed, commonly with irregular forms, and are extensively scattered. This mechanism of formation suggests that the glazing techniques used in Eastern Iran and Central Asia were more similar to the Eastern Mediterranean tradition. Mixtures of calx and silica was most likely applied directly - rather than fritted - and was fired with limited soaking time; although this needs to be confirmed by further replication experiments. Ample historical evidence suggests direct links between the Eastern Mediterranean and the Eastern Iranian worlds. During the reign of Harun al-Rashid, for instance, substantial troops were recruited from Khurasan for military settlement in the city of Raqqa (Kennedy 1986). One may, therefore, assume that such human mobility that had roots between the Eastern Mediterranean and Khurāsān, could have led to transfer of knowledge resulting in the production of opaque yellow high lead glazed wares during the 9th-10th centuries AD in Nishapur and Merv, among other sites in Eastern Iran and Central Asia.

In addition to the opaque yellow wares, yellow decorations on the so-called Buffwares seem related to the tin-based opacification technology. No sample of Buffwares have been analysed as part of current study as the yellow decorations were mostly concentrated in the middle of the sherds at the Metropolitan Museum and were hence not reachable for sampling. Analysis of the yellow decoration of a single sherd of Nishapur Buffware by Mason (2004, 135, sherd ROM.20) detected 59.0 wt% PbO, 34.8 wt% $SiO_2$

and 1.1 wt% $SnO_2$, consistent with the compositions of the opaque yellow glazes reported in the present study. A comprehensive evaluation would require the study of the microstructure and chemical composition of the decorations on Buffwares and their comparison with the Opaque Yellow Wares.

### 4.4.3. White glazes: technical aspects of production

The chemical composition of the opaque white glazes of Samarqand contains significantly less PbO contents compared to the lead-alkali white glazes of Eastern Mediterranean (~ 40% PbO). In virtue of both their composition and microstructure (i.e. including several quartz grains, air bubbles and tin-oxide crystals dispersed throughout the thickness), these glazes appear most similar to those of the white glazes applied on Southern Mesopotamian lustrewares (~ 10% PbO) reported in Chapter 3. However, the Pb/Sn ratios representing the primary calx used are significantly higher in the Samarqand glazes (~ 4-5) than those in the lustre glazes (~1).

## 4.5. CONCLUSIONS

The techniques of lead-tin-oxide type II opacified yellow glazes continued in Nishapur and Merv and the tin-oxide opacified white glazes in Samarqand, all adopting distinct local decorative patterns and forms. It was discussed in Chapter 3 (section 3.5.3) that the lead-tin-oxide crystallites in opaque yellow glazes from Mesopotamia are often well-formed with sizes up to 20 microns. This suggests that the glaze mixture might have been fritted. The shapes of these particles are significantly different from lead-tin-oxide crystals in opaque yellow glazes from Eastern Mediterranean. These are smaller in size and their crystal nucleuses are more widely dispersed throughout the glaze thickness. The irregular shapes and dispersion patterns of the lead-tin-oxide crystallites in yellow glazes from Nishapur and Merv suggests that their production was more related to the techniques applied in the Eastern Mediterranean, rather than Southern Mesopotamia. In order to further understand the production of these glazes and their variations, a cohesive typological study on ceramics from a range of Iranian and Central Asian sites is required as the first step. The findings of the present preliminary study on Eastern Iranian and Central Asian ceramics provide direction for future investigations regarding these wares.

# Chapter 5

# DISCUSSION

As presented in Chapter 1, the initial purpose of this thesis was two-fold. First, it was intended to investigate the relationship between early Islamic opaque yellow and opaque white glazed ceramics by studying their techniques of production. Second, it was designed as a scientifically driven study into ceramic production that depended less upon traditional definitions and typologies of early Islamic ceramics and that provided evidence to suggest a new version of the evolution of early Islamic wares. Both of these topics require more research before any conclusive models can be constructed, but the data presented and discussed in this thesis provide an important corpus of material that speaks to both issues. It is to be hoped that these data will inspire future research into Islamic ceramics that will further challenge long-standing ideas about their origins and development and the societies in which they operate.

## 5.1. CHALLENGING THE IRAQI ORIGIN

For almost a century, Iraq has been deemed the birthplace of Islamic glazed ceramics. The large number of glazed ceramics excavated in Samarra attested to the importance of Iraq as the producer of some of the most delicate and technically complicated ceramics developed during the Abbasid Caliphate. However, there are some difficulties in accepting that Islamic glazed ceramics originated in 9th-century Iraq. First, by following this approach, one is going to deny the existence of an earlier stage of Islamic ceramics production that began in the Eastern Mediterranean around the 7th/8th century AD. The Eastern Mediterranean products, including CGW and YGF wares, represent a precursor to so called Samarra type wares but have close links in shape and decorative styles both with early Byzantine and Samarra pottery. Second, the invention of tin-based opacification in Iraq under the influence of imported Chinese wares has been widely studied, but no conclusive evidence has been given to explain this 'sudden' ceramic breakthrough. Viewing the Eastern Mediterranean CGW and YGF as the first phase in Islamic ceramic production offers a more complete picture of the situation. Yellow glazes that were coloured and opacified by using lead-tin-oxide crystals were employed on Islamic CGW and YGF. Previously, the technique was widely used in glass production during the Late Roman and early Byzantine periods (Tite *et al.* 2008; Marii 2012; Neri *et al.* 2012). The arrival of Islam to the Eastern Mediterranean provided the foundations for old technologies to re-emerge through products that held the new Islamic identity. The technique of glaze opacification by lead-tin-oxide particles not only originated in the Eastern Mediterranean but also developed further to result in a second technical revolution: white tin-oxide glaze opacification. Replication experiments summarised in Chapter 2 demonstrated the very close links between the production of lead-tin-oxide yellow and tin-oxide white opaque glazes. It was explained that the addition of alkalis and alkaline earths to an essentially yellow opaque glaze mixture results in a white opaque glaze after firing. This technological link between the opaque yellow and white glazing traditions has important implications on the way the development of Islamic ceramics is viewed. Based on this new evidence, the production of tin-oxide white glazes in Iraq was no ‘sudden’ invention. In contrast, it was rooted in an earlier tradition of opaque glaze production in the Eastern Mediterranean that was later embraced by potters in Iraq and flourished in the 9th century.

## 5.2. TRANSFER OF TECHNOLOGY

This study encompasses wide geographical areas from Egypt to Central Asia and it therefore allows potential links between different ceramic technologies and cultures to be considered. Lead-tin-oxide as a yellow colourant and opacifier was used in glasses from about the 4th century AD in Late Roman and Byzantine periods. The use of lead-tin-oxide yellow colourant and opacifier in glasses continued into the Islamic period (Early Islamic glass from Nishapur: Pilosi *et al.* 2012 and Wypyski 2015; Early Islamic glass from Samarra: M. Wypyski 2015, pers. comm., 18 November) but reached its zenith when used in glaze production in the 7th/8th century AD.

By focusing specifically on CGW and YGF wares originating in the Islamic Eastern Mediterranean of the Umayyad and Abbasid Caliphates, the class becomes a vehicle for examining commercial and technological interactions during the early Islamic period. Despite the relatively small number of sites from which the yellow glazed wares were studied in this thesis, the resulting picture conveys an eastwards transfer of this technology from the Eastern Mediterranean to Mesopotamia, Iran and Central Asia during the 9th to 10th centuries AD. One may relate the initial spread of Islamic glazed wares to Iraq in the 9th century to the movement of the Islamic capital from Damascus, Syria, to Baghdad, Iraq, through which potters and other craftsmen might have been migrated to the new capital. The use of lead-tin-oxide yellow glazes did not stop in the

10th century. Chemical analyses of some of the Medieval Islamic yellow glazes from Iran, Central Asia and India, amongst other sites, have shown evidence of lead-tin-oxide opacification (Fabbri *et al.* 2002; Gill and Rehren 2011; Gill *et al.* 2014). The 19th-century treatise by Ali Mohammad discussed in Chapter 2 also described the use of lead-tin-oxide to produce yellow glazes in Qajar Iran.

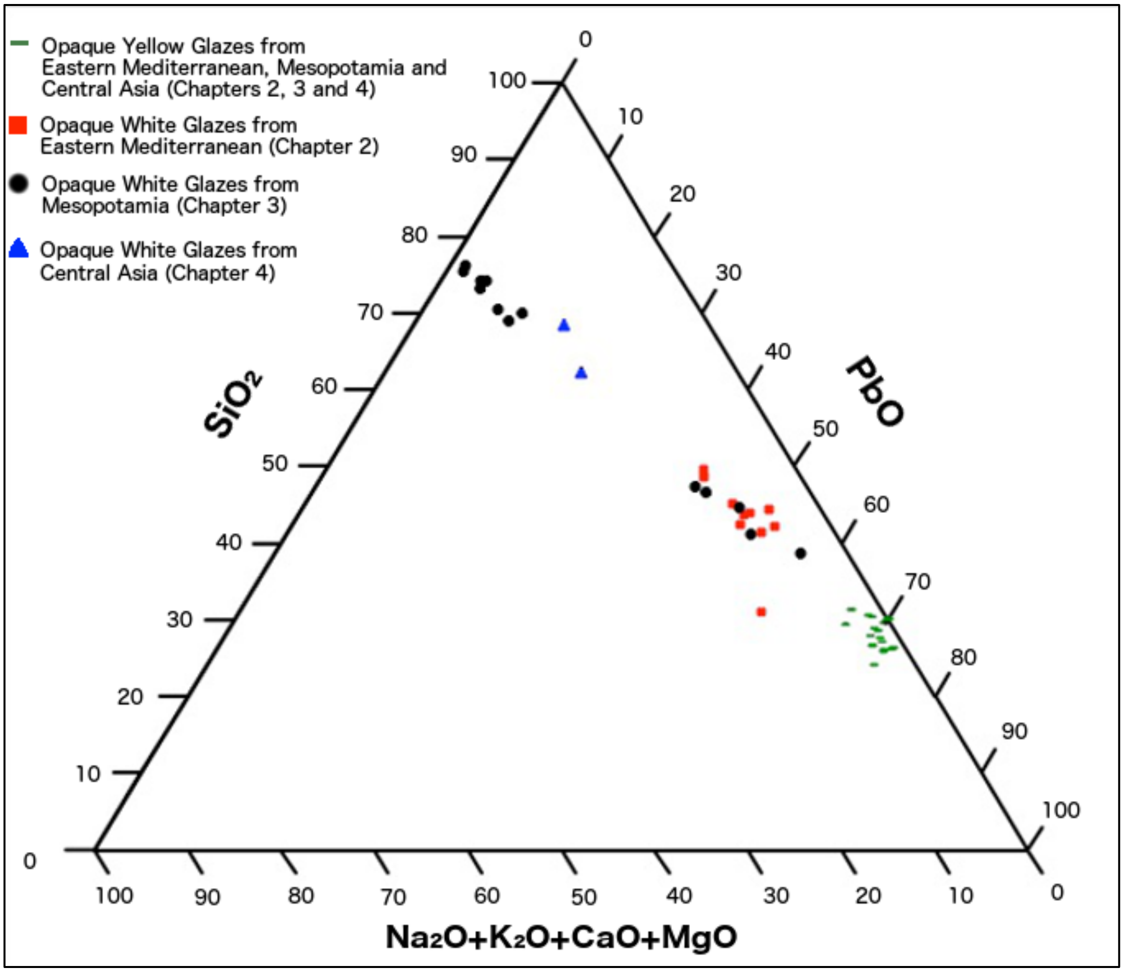


Figure. 5.1. *Ternary plot of the normalised PbO-$SiO_2$-($Na_2O$+$K_2O$+CaO+MgO) contents in opaque glaze compositions from Eastern Mediterranean, Mesopotamia, Eastern Iran and Central Asia (data from Tables 2.2, 3.2 and 4.3.*

Opaque white glazes seem to represent a further development of opaque yellow glazes. As discussed above, chemical analyses given in Chapters 2, 3 and 4 demonstrated that in contrast to the opaque yellow glazes, opaque white glazes contain significant contents of alkalis and alkaline earths. The results of replication experiments conducted in Chapter 2 established that the addition of alkalis decreases the temperature at which tin-oxide forms from dissolution of lead-tin-oxide crystals and the white colour emerges. However, other factors might also be involved in this conversion which remain to be identified by further replication experiments. Based on this evidence, it appears that Islamic potters had the composition of their glazes well under control and modified this depending on the colour features they were after: high lead compositions for opaque yellow glazes, lead-alkali and alkali-lead compositions for opaque white glazes (Fig. 5.1). While opaque white glazes with lead-alkali compositions were produced in the Eastern Mediterranean, those with alkali-lead compositions were mainly produced in Southern Mesopotamia.

In contrast to the opaque yellow glazes that were transferred only eastwards, opaque white glazes continued from somewhere in Mesopotamia eastwards to Iran and Central Asia and westwards to North Africa and Europe and acquired dominance as a main glazing technology wherever they reached. In Europe, white tin-oxide glazed pottery of different periods and provenances became known by different names. By the beginning of the 13th century a type of tin-glazed pottery known as ‘Maiolica’ started production in Italy (Caiger-Smith 1973, 65-199; Whitehouse 1978; 1980). The production of Maiolica wares was influenced by the Spanish imports through the islands of Majorca and Sicily and was later traded northwards to other European cities. By the late 16th century in France, tin glazed pottery centres, known as ‘faiencerie’, were widely developed (Caiger-Smith 1973, 111-27). In the Netherlands, tin-glazed pottery became known as ‘delftware’ throughout the 17th and 18th centuries and was eventually spread to Germany, Eastern Europe, and Britain (Caiger-Smith 1973, 127-140 and Fourest 1980). In today’s ceramic manufacture, tin oxide is chiefly replaced by zirconium and titanium compounds, but is still employed by artists and craftsmen for its particular opacification and glossy effect. The invention of tin-oxide opaque glazes is therefore considered as one of the most long lasting and wide spreading technological innovations and ceramic traditions.

## 5.3. CALX: A NEW WAY OF VIEWING TIN-BASED OPAQUE GLAZES

While there has been relatively extensive research on tin-based opacification of glazes, little attention has been given to the technical and practical aspects of its production. The results of anthropological observations of a traditional workshop in Iran, as well as the descriptions of the processes given in Islamic treatises, summarised in Chapters 2 and 3, suggested that the calcination process of tin with lead was a key stage before the actual glaze production began. Therefore, calculating the Pb/Sn ratio of these glazes would represent a rough estimate of the calces used. As a general observation, the Pb/Sn ratios in opaque yellow glazes are consistently higher compared to opaque white glazes (Fig. 5.2).

## 5.4. TECHNOLOGICAL CHOICES

The production of glazed ceramics requires the artisan to make a series of ‘choices’ selecting from a range of possible raw materials and techniques. These particular choices are the determining factors that make up the final products.

### 5.4.1. Raw materials

*Clay*

The features of the clay bodies from Eastern Mediterranean and Southern Mesopotamia have proved to be internally consistent. For this reason, and due to the homogenous geology of these regions, it is difficult to establish which exact sediments were used as sources of clay. It is likely that the potters used the local clay in manufacturing ceramic bodies rather than seeking for clay from a particular source, as the clay features were almost similar all across the regions. On the other hand, the clay body textures examined from three sites in Eastern Iran and Central Asia, well separated from the geology of Mesopotamia, were significantly different from each other (although the chemical compositions were very similar), and were consistent with the local sedimentations in each case. This provides evidence supporting the use of local clay sediments for ceramic body manufacture.

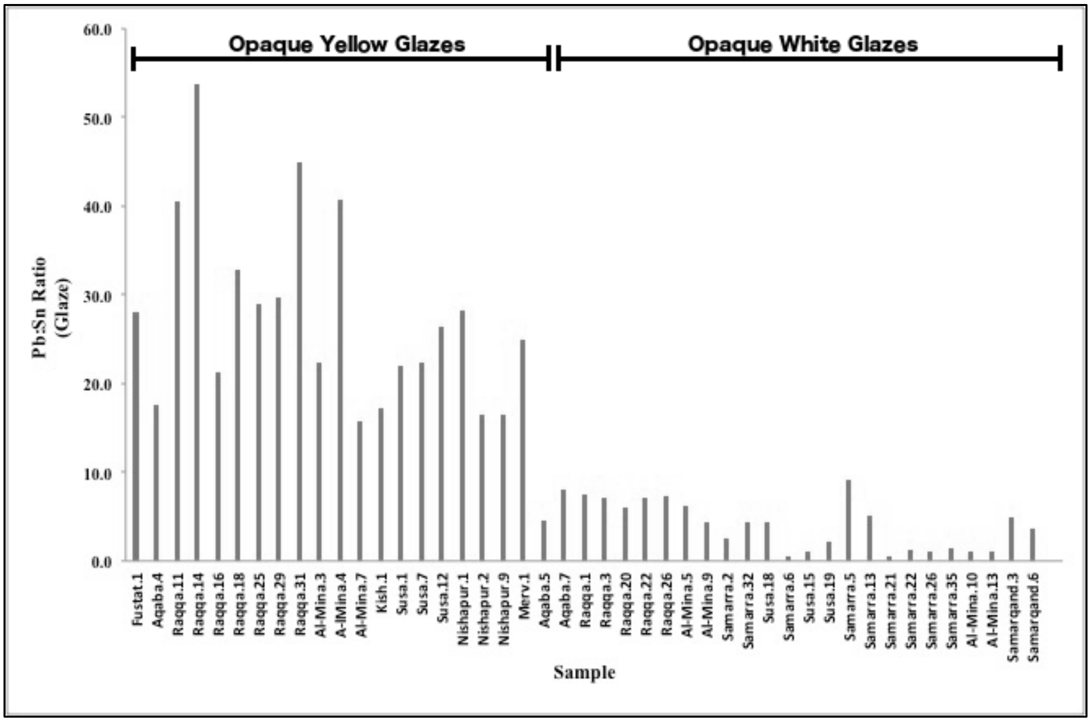


Figure 5.2. *Histogram of Pb:Sn ratios for opaque yellow and white glazed wares using data from Tables 2.2, 3.2 and 4.3.*

### *Quartz*

Two main sources of silica are crushed quartz pebbles or quartz sand. In glazing mixtures, crushed quartz pebbles appear to have been chosen mainly because of their higher purity compared to sand. Higher contents of impurity in sand, particularly iron, would have a direct adverse effect on the colour of the glaze.

*Lead*

According to the textual sources lead in the metallic form (Pb) or in the sulphide form (PbS) was used as a primary raw material and was converted to an oxide before being used in the glaze mixture. No data are available on the source of lead used in the production of Early Islamic glazes. However, lead isotope analysis can provide information on the source of lead to produce lead glazes of the Umayyad and Abbasid periods (Wolf *et al.* 2003).

*Tin*

Similar to the situation with lead, no data are available on sources of tin minerals used in the production of Early Islamic ceramics. Islamic textual sources suggest that tin, combined with lead, was oxidised before being used in the glaze mixture. A medieval text by Kashani also described the process of oxidising tin in the absence of lead to achieve a turquoise coloured glaze (Chapter 3).

Tin might have also been used as a naturally occurring tin-oxide mineral ($SnO_2$) known as cassiterite. However, there are difficulties in accepting this theory. Firstly, this method of glaze opacification is not consistent with the descriptions given in the Islamic historical texts, detailing the calcination of tin and lead (see Chapters 2 and 3). Secondly, the notion of cassiterite used directly in the glaze mixture with no former preparation processes such as calcination with lead does not convincingly explain the origins of Islamic opaque glazing and its development particularly in relation to the earlier opaque yellow glazes. Finally, by assuming that opaque white glazes were produced by addition of naturally occurring cassiterite into the mixture, the reasons behind lead contents being always present in the opaque white glazes would not be explained.

*Alkalis and alkaline earths*

Alkalis and alkaline earths were used only in opaque white glazes. The negligible amounts of alkalis and alkali earths present in the opaque yellow glazes were most likely the result of migration from the body or were introduced by other raw materials as an impurity. The two possible sources of alkalis and alkaline earths in opaque white glazes are the naturally occurring mineral soda deposits such as so-called 'natron' from Egypt (Shortland *et al.* 2006), and the ashes derived from salt-tolerant, halophytic plants growing in coastal, salt marsh and desert regions (Sayre and Smith 1974; Tite and Shortland 2008, 38). Natron consists of various sodium carbonates and has very low $K_2O$, CaO and MgO contents (Shortland *et al*. 2006). On the other hand, plant ash as a source of alkalis and alkaline earths is compositionally complex and contains higher levels of $K_2O$, CaO and MgO. The generally high potash ($K_2O$) contents of all the opaque white glazes analysed provide clear evidence that plant ashes were the source of the alkali fluxes (see also Chapter 2, Table 2.2).

*Colourants*

Copper- The concentrations of copper in green glazes examined vary from around 1 to 5 wt% (Tables 2.3, 3.5 and 4.4). Because most of the glazes have high contents of tin and lead, the correlation of these elements with copper cannot be established, and therefore it is impossible to distinguish whether scale from copper alloys (with high lead or tin contents) or a relatively pure copper ore was used as the source of the copper colourant. Chemical composition of green glazes of two Susa sherds showed elevated ZnO contents with high amounts of CuO contents (Table 3.5, Susa.5 and Susa.13). This suggests that copper scale from brass might have been used in these cases.

Cobalt- Chemical composition of the relics of the cobalt pigments used in the blue glazes from Southern Mesopotamia contained Fe-Co-Zn. Little is known about the mines in Mesopotamia and the neighbouring areas used as the source of cobalt colourants. Perhaps the only one that has been studied more thoroughly is the cobalt mine in Kashan, Iran. Previous examinations of Kashan ore demonstrated that the cobalt ore is rich in As and contains negligible Zn (Matin and Pollard 2016). Therefore, it is unlikely that ores from this mine were used as the source of the blue pigment in Early Islamic glazes.

Managanese- Black line decorations were extensively used on the opaque yellow glazed wares of the Eastern Mediterranean and Sourthern Mesopotamia. The results of chemical composition of one sample of this black decoration (Raqqa.25, Table 2.3) showed significant concentrations of MnO, as well as elevated FeO contents. The microstructure of the glaze showed that the primary manganese colourants were transformed during firing and a variety of secondary manganese crystals were formed, including manganese oxides, manganese-containing diopsides and lead-bearing manganese silicates. The most common source of manganese colourant is pyrolusite ($MnO_2$) and the microstructure and chemical composition of the black glaze suggests that this mineral might have been used. However, this cannot be firmly established due to the transformation of the primary manganese colourants into secondary phases during firing.

# Chapter 6

# CONCLUSIONS AND FUTURE RESEARCH

This study resulted in a number of conclusions concerning the beginnings of Islamic ceramics and the technique of glaze opacification which are set forth in the following paragraphs. The project encompassed a wide range of samples and used a comprehensive methodology: body analyses providing information on provenance; glaze analyses providing information on technology; laboratory replication and the survey of historical texts both of which are crucial to interpreting the technological implications of glaze analysis. The results provided new information on glazing technology from Egypt to Central Asia during the 7th/8th to 10th centuries AD, and the links between them.

1. Deliberate opacification of glazes in the Islamic period began in the 7th/8th century AD in the Eastern Mediterranean, best represented in the Coptic Glazes Wares (CGW) and Yellow Glaze Family (YGF) wares. Here, Islamic potters used a mixture of calcined tin with lead (tin-lead calx), together with silica, to produce opaque yellow glazes. The opacifier and colourant in these glazes have been identified as lead-tin-oxide type II ($Pb(Sn,Si)O_3$). The composition of the glazes in all cases was high-lead, consistently containing around 60% PbO and only negligible amounts of alkalis and alkaline earths.

2. Opaque yellow glaze technology continued during the 9th to 10th centuries AD in Mesopotamia and Eastern Iran, particularly in Susa, Nishapur and Merv, amongst other sites.
3. Addition of alkalis and alkaline earths to the mixture of tin-lead calx and silica results in the formation of tin-oxide ($SnO_2$) particles from disassociation of lead-tin-oxide ($Pb(Sn,Si)O_3$) crystals at temperatures around 950°C. This crystal conversion results in the production of opaque white glazes containing tin-oxide. On this ground, Islamic opaque white glazes may be considered the result of the development of earlier opaque yellow glazes. This piece of evidence is pivotal in studying the links between the Eastern Mediterranean and Mesopotamian ceramic traditions and in investigating the origins of Islamic ceramics. Tin-oxide opaque glazing makes up one of the key strands of ceramic production technology that has been widely employed from Asia to Europe and which has continued until the modern period.
4. The composition of opaque white glazes in Eastern Mediterranean and Mesopotamia fall into two general classes: I) lead-alkali glazes, associated to medium/coarse-grained body fabrics of Eastern Mediterranean and Northern Mesopotamia; II) alkali-lead glazes, associated to fine-grained body fabrics of Southern Mesopotamia. Regarding the opaque white glazes from Eastern Iran and Central Asia, the number of ceramics studied has been limited and do not form a cohesive picture; however, those studied from Samarqand fall into lead-alkali compositional group.

5. After the importation of Chinese wares to Mesopotamia in the 9th century AD, the Islamic potters seized the opaque white glazing technology, which was ready and well-developed by that time, to produce ceramics with white surface that resembled those of Chinese porcelain and stoneware. The development of these new range of ceramics, referred to as the Samarra type wares, marked a turning point in the history of ceramics.

Several topics have arisen in the course of this work on the subject of tin-based opaque glazing with potential for further research. These are highlighted below:

1. A thorough study of typology and examination of the chemical composition and microstructure of Early Islamic CGW and YGF wares from sites illustrated in Figure 1.2 to provide a more comprehensive data on these ceramics and to investigate the variations.
2. Analysis of the black manganese lines extensively used in the CGW and YGF wares and studying their potential links with the later Cuerda Seca technology.
3. Examination of opaque yellow glazes after the 10th century AD to investigate the development of this technology until the modern time and thus establish the relationship between the 10th century yellow (and white glazes) and those produced at a much later date (e.g. Fabbri *et al.* 2002; Fabbri *et al.* 2004, Gill and Rehren 2011).
4. Examination of the chemical composition and microstructure of various types of Early glazed ceramics from sites in Eastern Iranian and Central Asia, such as Nishapur, Merv and Samarqand followed by their comparison with each other.

The lack of information on these ceramics is a major problem in studying the development of Early Islamic ceramics.

5. Further experimental replication to investigate factors affecting the formation of lead-tin-oxide and tin-oxide crystals during the firing process.
6. Analysis of trace elements in clays from Eastern Mediterranean and Mesopotamia to further differentiate pottery workshops in these regions.

# Appendix A

# ANALYTICAL TECHNIQUES

In the investigation of the production technology of early Islamic glazes, the primary technique has been the examination of polished cross-sections through samples from the glaze layer into the body in a scanning electron microscope (SEM) with attached energy dispersive spectrometer (EDS) at the Research Laboratory for Archaeology and the History of Art (RLAHA), University of Oxford. X-ray diffraction (XRD) was conducted at the Department of Physics and Nuclear Engineering, Polytechnic University of Catalonia (UPC), on samples of replicated calx to identify the obtained crystals after heating. The two analytical techniques are outlined below.

## A.1. SCANNING ELECTRON MICROSCOPY-ENERGY DISPERSIVE SPECTROMETRY (SEM-EDS)

### A.1.1. Sample preparation

Polished sections through the glaze and into the bodies were prepared for investigation under scanning electron microscope (SEM). The sections were first mounted in Caldofix (Struers) epoxy resin and were kept under vacuum for an hour to ensure a full impregnation with resin. The samples were then left in the oven at 75°C for two hours. A flat surface was obtained on the mounts using 320, 800, 2500 and 4000 grit SiC abrasive discs and 9, 3,1 and 0.25 μm diamond polishing pastes.

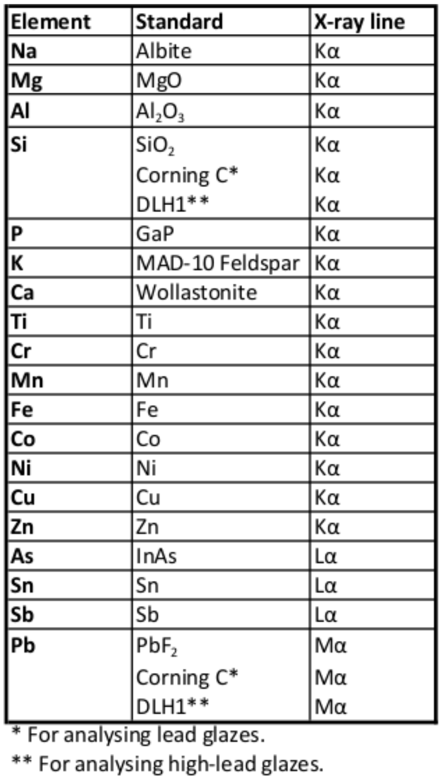
### A.1.2. Analytical procedure

A JEOL JSM- 5910 SEM with Oxford Instruments EDS (INCA 300 System) was used for examination of polished sections. The system was operated at 20 kV and 120s measuring time. Count rate on metallic cobalt was around 3000 cps. Glaze and body microstructures were studied and recorded in backscattered electron mode (BSE), in which the different phases present could be distinguished on the basis of their atomic number contrast. The system was calibrated using appropriate primary standards by the Oxford Instrument system as shown in Table A.1. Both before and after a series of analyses, the accuracy of the system was checked by analysis of Corning A and Corning B glass standards. Table A.2 shows the most probable values for these standards.

The main focus of the present research has been the examination of glazes. However, compositions of bodies and main inclusions have been also included in order to enable a rough association of different types of glazes to body fabrics. EDS semi-quantitative measurements were performed by analysing three areas of approximately 1 mm x 0.8 mm. Depending on the degree of porosity, the totals ranged between 68-75%. The average of the resulting normalised totals were reported as the chemical composition of sherd bodies.

As for the glazes, the bulk semi-quantitative EDS compositions were determined by analysing areas up to about 0.4 mm x 0.4 mm. A major problem in the study of early Islamic ceramics was inhomogeneity of glazes due to the presence of scattered tin-oxide/lead-tin-oxide particles, undissolved quartz/feldspar grains, air bubbles and extensive weathering in some cases. The latter tended to be more severe for the thin glaze layers of the painted yellow glazed wares (see Chapter 2; the painted glazes were most likely applied using a brush). To help moderate this effect, at least five areas were analysed in each glaze and the average normalised compositions were reported.

Table A.1. *Primary standards for calibration on JEOL-5910 SEM.*



| Element | Standard | X-ray line |
|---|---|---|
| **Na** | Albite | Kα |
| **Mg** | MgO | Kα |
| **Al** | $Al_2O_3$ | Kα |
| **Si** | $SiO_2$<br>Corning C*<br>DLH1** | Kα<br>Kα<br>Kα |
| **P** | GaP | Kα |
| **K** | MAD-10 Feldspar | Kα |
| **Ca** | Wollastonite | Kα |
| **Ti** | Ti | Kα |
| **Cr** | Cr | Kα |
| **Mn** | Mn | Kα |
| **Fe** | Fe | Kα |
| **Co** | Co | Kα |
| **Ni** | Ni | Kα |
| **Cu** | Cu | Kα |
| **Zn** | Zn | Kα |
| **As** | InAs | Lα |
| **Sn** | Sn | Lα |
| **Sb** | Sb | Lα |
| **Pb** | $PbF_2$<br>Corning C*<br>DLH1** | Mα<br>Mα<br>Mα |

* For analysing lead glazes.

** For analysing high-lead glazes.

A further difficulty emerged with high-lead (>60 wt% PbO) and lead-alkaline (35-50 wt% PbO) glazes with compositions close to the DLH1 and Corning C glass standards. As Table A.2 indicates, the ‘measured values’ for PbO and $SiO_2$ in the DLH1 and Corning C glass standards deviated from the ‘most probable values’ by ±6 wt%, resulting in totals of around 90-100%. The issue was found to be related to the high matrix corrections as would be the case of quantifying a heavy element in light matrix (Pb in a Si matrix) or vice versa. Minimising such effect would have required using a primary standard for Pb and Si in a similar matrix as seen in the glazes. Therefore, the Pb-Mα and

Si-Kα peaks in the DLH1 were used as the primary standards for the calibration in measuring the composition of the high-lead glazes (see opaque yellow glazes in Chapters 2,3 and 4) and the Pb-Mα and Si-Kα peaks in the Corning C standards as the primary standards for the calibration in measuring the composition of lead-alkaline glazes (see opaque white glazes in chapters 2 and 3). The resulting totals ranged between 98-102%.

Table A.2. *Composition of glass standards; measured values represent the average of three EDS analyses (wt%).*

| Glass Standards | Values | | $SiO_2$ | PbO | $SnO_2$ | $Na_2O$ | $K_2O$ | CaO | MgO | $Al_2O_3$ | MnO | FeO | $P_2O_5$ | $Cr_2O_3$ | CoO | $As_2O_3$ | NiO | CuO | $TiO_2$ | ZnO | $Sb_2O_3$ | Totals |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| CORNING A | Most Probable Values* | | 66.56 | 0.12 | 0.19 | 14.3 | 2.87 | 5.03 | 2.66 | 1.00 | 1 | 1.21 | 0.13 | 0.001 | 0.17 | | 0.02 | 1.17 | 0.79 | 0.044 | 1.75 | 99.02 |
| | Meseared Values | Avg | 67.54 | 0.15 | 0.40 | 14.28 | 3.20 | 5.17 | 2.70 | 0.85 | 1.02 | 0.94 | 0.06 | bd | 0.23 | bd | bd | 1.21 | 0.98 | bd | 1.73 | 100.46 |
| | | Sd | 0.11 | 0.10 | 0.33 | 0.30 | 0.03 | 0.13 | 0.01 | 0.08 | 0.06 | 0.05 | 0.01 | - | 0.06 | - | - | 0.06 | 0.04 | - | 0.25 | |
| CORNING B | Most Probable Values* | | 61.55 | 0.61 | 0.04 | 17.00 | 1 | 8.56 | 1.03 | 4.36 | 0.25 | 0.38 | 0.82 | 0.005 | 0.046 | | 0.1 | 2.66 | 0.09 | 0.19 | 0.46 | 99.15 |
| | Measured Values | Avg | 62.46 | 0.73 | 0.20 | 15.80 | 1.18 | 8.43 | 0.97 | 3.77 | 0.19 | 0.26 | 0.85 | 0.05 | bd | 0.12 | 0.09 | 2.53 | 0.18 | 0.12 | 0.53 | 98.46 |
| | | Sd | 0.73 | 0.04 | 0.27 | 0.25 | 0.03 | 0.11 | 0.15 | 0.13 | 0.01 | 0.06 | 0.09 | 0.08 | - | 0.09 | 0.11 | 0.06 | 0.07 | 0.09 | 0.17 | |
| CORNING C | Most Probable Values* | | 34.87 | 36.7 | 0.19 | 1.07 | 2.84 | 5.07 | 2.76 | 0.87 | 0.82 | 0.38 | 0.14 | 0.001 | 0.18 | | 0.02 | 1.13 | 0.79 | 0.052 | 0.03 | 87.91 |
| | Measured Values | Avg | 34.11 | 35.85 | 0.52 | 0.91 | 3.10 | 5.17 | 2.47 | 0.70 | 0.07 | 0.36 | bd | bd | 0.17 | 0.16 | 0.08 | 1.16 | 5.20 | 0.10 | bd | 90.14 |
| | | Sd | 0.19 | 0.15 | 0.23 | 0.08 | 0.04 | 0.12 | 0.03 | 0.09 | 0.11 | 0.12 | - | - | 0.10 | 0.01 | 0.08 | 0.13 | 0.08 | 0.05 | - | |
| DLH1 | Most Probable Values** | | 24.96 | 67.04 | na | 1.01 | 0.96 | 1.04 | 0.33 | 3.98 | na | 0.98 | na | na | na | na | na | na | na | na | na | 100.30 |
| | Meseared Values | Avg | 26.55 | 64.54 | bd | 0.92 | 0.98 | 1.02 | 0.23 | 3.82 | bd | 1.11 | bd | bd | bd | 0.11 | bd | 0.05 | bd | bd | bd | 99.33 |
| | | Sd | 0.39 | 0.44 | - | 0.11 | 0.09 | 0.06 | 0.09 | 0.05 | - | 0.08 | - | - | - | 0.21 | - | 0.11 | - | - | - | |

* Brill 1999.

** British Museum Research Laboratory, I. Freestone, pers. comm.

Detection limits for minor oxides at 3 sigma are given in Table A.3. Typical standard deviations based on five analyses for bodies, opaque yellow glazes and opaque white glazes are given in Table A.4.

Table A.3. *Typical detection limits for minor oxides (wt%).*

| $Na_2O$ | MgO | $Al_2O_3$ | $P_2O_5$ | $SO_3$ | $K_2O$ | CaO | $TiO_2$ | MnO | FeO | CoO | CuO | ZnO | $As_2O_3$ | $SnO_2$ | PbO |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.2 | 0.2 | 0.1 | 0.2 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.2 | 0.2 | 0.3 | 0.5 | 0.5 |

Table A.4. *Typical standard deviations for bodies, opaque yellow glazes and opaque white glazes based on five analyses (wt%).*

| Sample | $SiO_2$ | $Al_2O_3$ | PbO | $SnO_2$ | $Na_2O$ | $K_2O$ | CaO | MgO | FeO | $P_2O_5$ | $SO_3$ | MnO | $TiO_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Body** | 2.2 | 0.6 | bd | - | 0.2 | 0.1 | 2.0 | 0.5 | 0.8 | bd | 0.2 | bd | 0.1 |
| **Opaque Yellow Glaze** | 0.2 | 0.1 | 0.6 | 0.5 | bd | bd | 0.1 | bd | bd | 0.1 | - | bd | bd |
| **Opaque White Glaze** | 0.3 | bd | 0.8 | bd | bd | 0.1 | 0.1 | bd | 0.2 | bd | - | 0.1 | 0.1 |

## A.2. X-RAY DIFFRACTION (XRD)

### A.2.1. Sample preparation

Calx powders were ground using an agate mortar and pestle and passed through a 240-mesh sieve (BSS 63 μm). The resulting powders were then pressed into pellets and mounted on a holder. The sample preparation approach provides a more homogenous representation of the sample with no void spaces and little sample dilution.

### A.2.2. Analytical procedure

Qualitative X-ray diffraction analysis was conducted using a Bruker D8 Advance XRD with a vertical goniometer and Bragg-Brentano θ-2θ configuration for powder diffraction measurements. Identification of the compounds has been performed based on the Powder Diffraction File (PDF) database from the International Centre for Diffraction Data (ICDD) (Fig A.1).

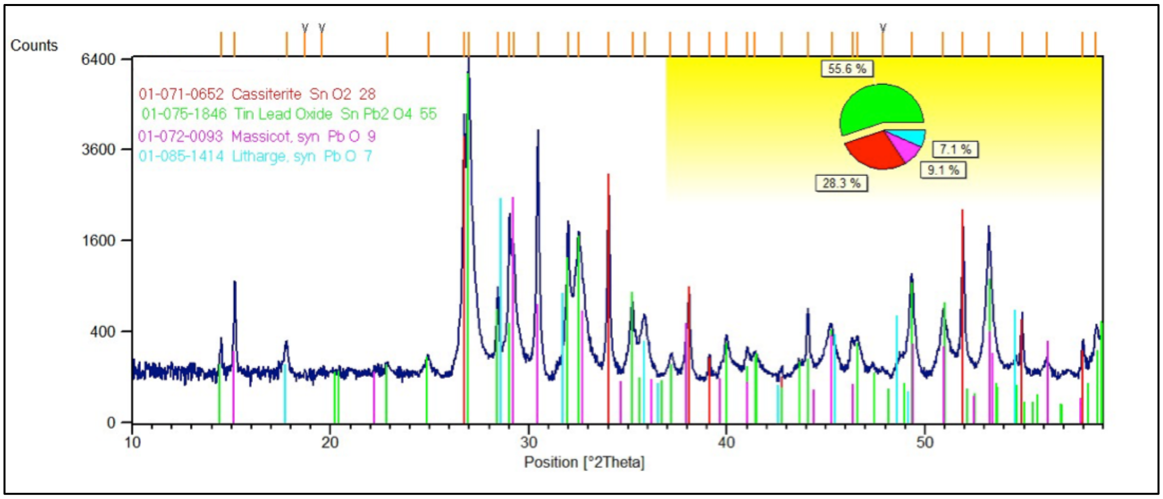


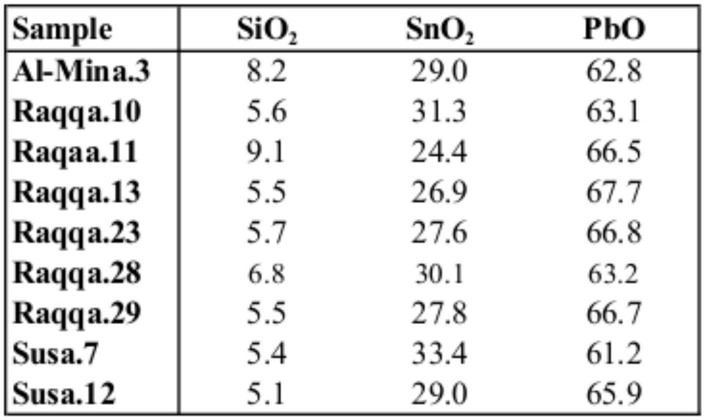
Fig. A.1. *Example of XRD peaks (calx sample C.700).*

# Appendix B

# ANALYTICAL DATA FOR LEAD-TIN-OXIDE CRYSTALS

Lead-tin-oxide crystals in opaque yellow glazes were identified using two methods: SEM-EDS and SR-Micro-XRD. SEM-EDS analyses of crystals were carried out at the RLAHA, Oxford, using spot analysis targeting the centre of the crystals to minimise the effect of the background glaze on the results (more details given in Appendix A). Table B.1 indicates the chemical composition of these crystals. The analytical totals ranged between 82 and 98 wt%.

Table B.1. *EDS analysis of lead-tin-oxide crystals (normalised to 100%).*



| Sample | $SiO_2$ | $SnO_2$ | PbO |
|---|---|---|---|
| **Al-Mina.3** | 8.2 | 29.0 | 62.8 |
| **Raqqa.10** | 5.6 | 31.3 | 63.1 |
| **Raqaa.11** | 9.1 | 24.4 | 66.5 |
| **Raqqa.13** | 5.5 | 26.9 | 67.7 |
| **Raqqa.23** | 5.7 | 27.6 | 66.8 |
| **Raqqa.28** | 6.8 | 30.1 | 63.2 |
| **Raqqa.29** | 5.5 | 27.8 | 66.7 |
| **Susa.7** | 5.4 | 33.4 | 61.2 |
| **Susa.12** | 5.1 | 29.0 | 65.9 |

Synchrotron Radiation X-ray Diffraction (SR-Micro-XRD) was performed at the Xaloc, beam line 13 of the ALBA Light Facility, Barcelona, Spain, using 12.6 keV energy and 7*50 $\mu m^2$ spot size. The small beam size facilitated the acquisition of information from submicron crystalline compounds. Figure B.1 shows the Micro-XRD spectrum corresponding to the yellow glaze of sherd Fustat.1. Similar XRD data for lead-tin-oxide crystals are also presented in Tite *et al.* (2015).

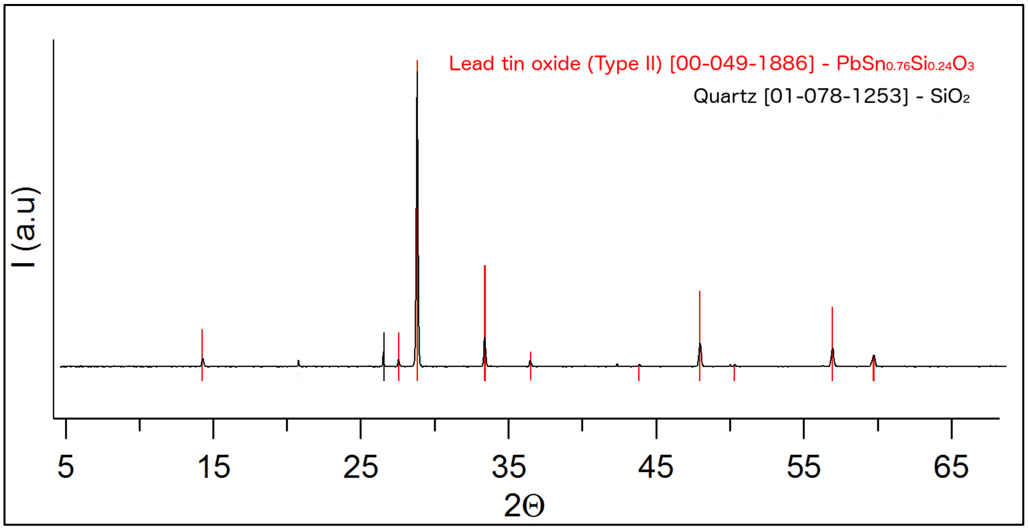


Figure B.1. *Micro-XRD spectrum corresponding to the Fustat.1 yellow glaze.*

# Appendix C

# LIST OF SHERDS

| Sherd No. | Acc. No. | Collection |
|---|---|---|
| Fustat.1 | ALX.202 | Alexander Kaczmarczyk* |
| Aqaba.1 | 10d-42/RN88-669 | Oriental Institute Museum, Chicago |
| Aqaba.2 | F10a-5/RN87-79 | Oriental Institute Museum, Chicago |
| Aqaba.4 | F10a-10/RN87-75, 23.3.87 | Oriental Institute Museum, Chicago |
| Aqaba.5 | G10A-1/RN87-284, 29.3.87 | Oriental Institute Museum, Chicago |
| Aqaba.7 | E9a-3/RN87-214 | Oriental Institute Museum, Chicago |
| Raqqa.2 | Ra I.170-7745-13 | Museum für Islamische Kunst, Berlin |
| Raqqa.3 | Ra I.170-F735-31 | Museum für Islamische Kunst, Berlin |
| Raqqa.4 | Ra I.170-4130-29-1/12 | Museum für Islamische Kunst, Berlin |
| Raqqa.5 | Ra I.170-F746-16 | Museum für Islamische Kunst, Berlin |
| Raqqa.6 | Ra I.170-A145-13 | Museum für Islamische Kunst, Berlin |
| Raqqa.7 | Ra I.170-As.42-41-3/9 | Museum für Islamische Kunst, Berlin |
| Raqqa.8 | Ra I.170-F789-13 | Museum für Islamische Kunst, Berlin |
| Raqqa.10 | MM1 (shelf 7) | Museum für Islamische Kunst, Berlin |
| Raqqa.11 | NK (shelf 7) | Museum für Islamische Kunst, Berlin |
| Raqqa.12 | AS46.713 (shelf 7) | Museum für Islamische Kunst, Berlin |
| Raqqa.13 | Ra I.160-1-MM3-148 | Museum für Islamische Kunst, Berlin |
| Raqqa.14 | A143 (shelf 7) | Museum für Islamische Kunst, Berlin |
| Raqqa.16 | AS.Gl.11/2 (shelf 7) | Museum für Islamische Kunst, Berlin |
| Raqqa.17 | AS.Gl.813-A145-25 (shelf 7) | Museum für Islamische Kunst, Berlin |
| Raqqa.18 | AS.Gl.5/5 (shelf 7) | Museum für Islamische Kunst, Berlin |
| Raqqa.19 | AS.Gl.12/7 (shelf 7) | Museum für Islamische Kunst, Berlin |
| Raqqa.22 | Kiln rod M1-R167.1 (shelf 4) | Museum für Islamische Kunst, Berlin |
| Raqqa.23 | R167.1-A115-15 (shelf 4) | Museum für Islamische Kunst, Berlin |
| Raqqa.24 | R167.1-4103-26 (shelf 4) | Museum für Islamische Kunst, Berlin |
| Raqqa.25 | Ra I.168.13.1-MM4 | Museum für Islamische Kunst, Berlin |
| Raqqa.26 | Ra I.168-Tell Aswad g2-15 | Museum für Islamische Kunst, Berlin |
| Raqqa.28 | Ra I.162-AS.Gl.4/4-F739 (shelf 2) | Museum für Islamische Kunst, Berlin |
| Raqqa.29 | Ra I.162-AS.Gl.3/11-A130 (shelf 2) | Museum für Islamische Kunst, Berlin |
| Raqqa.30 | Ra I.171-Sh 501 (shelf 5) | Museum für Islamische Kunst, Berlin |
| Raqqa.31 | Ra I.171-97 (shelf 5) | Museum für Islamische Kunst, Berlin |
| Al-Mina.3 | C244B-1937 | Victoria and Albert Museum, London |
| Al-Mina.4 | C242G-1937 | Victoria and Albert Museum, London |
| Al-Mina.6 | C242-1937 | Victoria and Albert Museum, London |
| Al-Mina.7 | C242J-1937 | Victoria and Albert Museum, London |
| Al-Mina.8 | C243A-1937 | Victoria and Albert Museum, London |
| Al-Mina.9 | C234C-1937 | Victoria and Albert Museum, London |
| Al-Mina.12 | C249B-1937 | Victoria and Albert Museum, London |
| Samarra.5 | C.692-1922 | Victoria and Albert Museum, London |
| Samarra.6 | C.640-1922 | Victoria and Albert Museum, London |
| Samarra.13 | C.644-1922 | Victoria and Albert Museum, London |
| Samarra.14 | C.635-1922 | Victoria and Albert Museum, London |
| Samarra.19 | C.679-1922 | Victoria and Albert Museum, London |
| Samarra.20 | C.711-1922 | Victoria and Albert Museum, London |

| **Sherd No.** | **Acc. No.** | **Collection** |
|---|---|---|
| Samarra.21 | C.709-1922 | Victoria and Albert Museum, London |
| Samarra.22 | C.680-1922 | Victoria and Albert Museum, London |
| Samarra.23 | C.678-1922 | Victoria and Albert Museum, London |
| Samarra.24 | C.720-1922 | Victoria and Albert Museum, London |
| Samarra.26 | C.769-1923 | Victoria and Albert Museum, London |
| Samarra.27 | sam I.164.1-8 | Museum für Islamische Kunst, Berlin |
| Samarra.28 | sam I.159.1-6 | Museum für Islamische Kunst, Berlin |
| Samarra.29 | sam I.274.1-11 | Museum für Islamische Kunst, Berlin |
| Samarra.32 | sam I.273.1-4 | Museum für Islamische Kunst, Berlin |
| Samarra.34 | sam 1068 | Museum für Islamische Kunst, Berlin |
| Samarra.35 | sam I.184.1-17 | Museum für Islamische Kunst, Berlin |
| Samarra.37 | sam I.281.1-4 | Museum für Islamische Kunst, Berlin |
| Kish.1 | C.262-(A)-1931 | Victoria and Albert Museum, London |
| Susa.1 | MAO S 495 | Musée du Louvre, Paris |
| Susa.2 | MAO S 670 | Musée du Louvre, Paris |
| Susa.3 | MAO S 702 | Musée du Louvre, Paris |
| Susa.5 | MAO S 838 | Musée du Louvre, Paris |
| Susa.6 | MAO S 839 | Musée du Louvre, Paris |
| Susa.7 | MAO S 781 | Musée du Louvre, Paris |
| Susa.8 | MAO S 1169 | Musée du Louvre, Paris |
| Susa.11 | MAO S 1265 | Musée du Louvre, Paris |
| Susa.12 | MAO S 579 | Musée du Louvre, Paris |
| Susa.13 | MAO S 1429 ab | Musée du Louvre, Paris |
| Susa.14 | MAO S 1432 | Musée du Louvre, Paris |
| Susa.15 | MAO S 1629 | Musée du Louvre, Paris |
| Susa.16 | MAO S 2066 | Musée du Louvre, Paris |
| Susa.17 | MAO S 2422 | Musée du Louvre, Paris |
| Susa.18 | MAO S 2082 a | Musée du Louvre, Paris |
| Susa.19 | MAO S 1405 | Musée du Louvre, Paris |
| Basra.1 | 52.130.1 | Metropolitan Museum of Art, New York |
| Basra.2 | 52.130.3 | Metropolitan Museum of Art, New York |
| Basra.3 | 52.130.4 | Metropolitan Museum of Art, New York |
| Basra.4 | 52.130.5 | Metropolitan Museum of Art, New York |
| Basra.5 | 52.130.6 | Metropolitan Museum of Art, New York |
| Basra.6 | 52.130.9 | Metropolitan Museum of Art, New York |
| Basra.7 | 52.130.12 | Metropolitan Museum of Art, New York |
| Basra.8 | 52.130.23 | Metropolitan Museum of Art, New York |
| Basra.9 | 52.130.25 | Metropolitan Museum of Art, New York |
| Nishapur.1 | 38.40.105 | Metropolitan Museum of Art, New York |
| Nishapur.2 | 40.170.465 | Metropolitan Museum of Art, New York |
| Nishapur.4 | 40.170.505 | Metropolitan Museum of Art, New York |
| Nishapur.9 | Not registered | Metropolitan Museum of Art, New York |
| Merv.1 | KRS02-2 27- Bag 20 | Paul Wordsworth** |
| Merv.2 | KRS02-2/2011 (4)- Bag 28 | Paul Wordsworth |

| **Sherd No.** | **Acc. No.** | **Collection** |
|---|---|---|
| Merv.3 | 1037 | Tim Williams*** |
| Samarqand.1 | 1775-1898 | Victoria and Albert Museum, London |
| Samarqand.3 | 1780-1898 | Victoria and Albert Museum, London |
| Samarqand.6 | 1628-1898 | Victoria and Albert Museum, London |

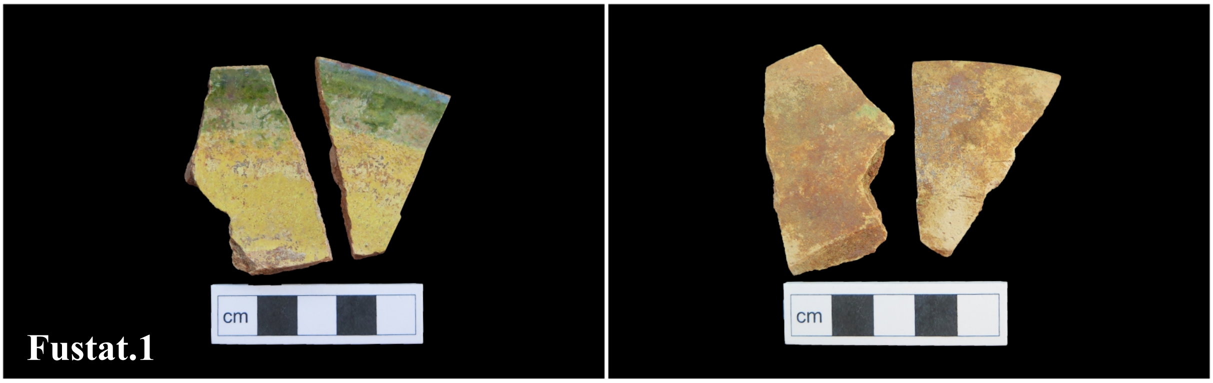
*Research Laboratory for Archaeology and the History of Art (RLAHA), University of Oxford, Oxford, UK. See Tite 2011.

** Oriental Institute, University of Oxford, Oxford, UK.

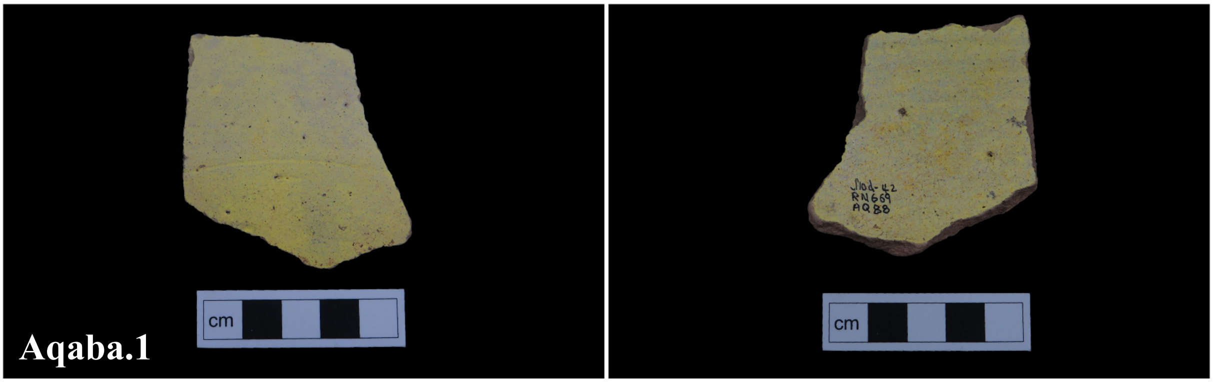
***Institute of Archaeology, UCL, London, UK.

# Appendix D

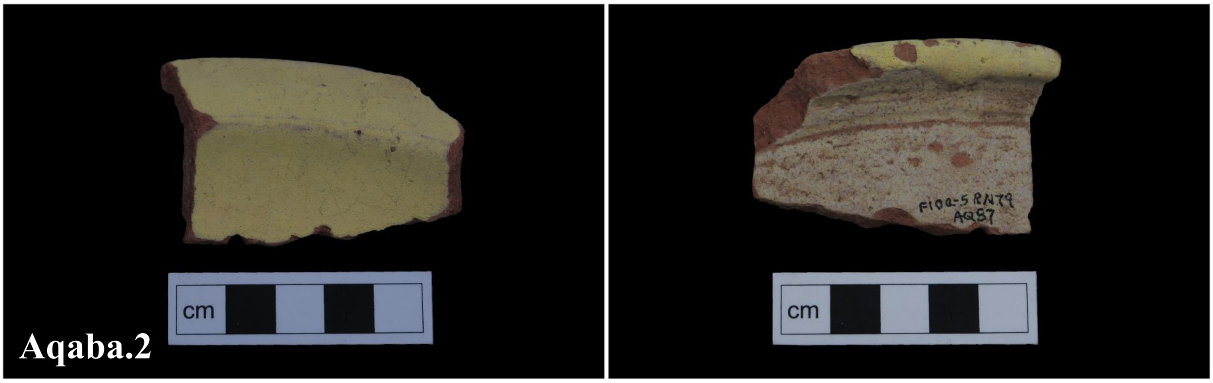
# PHOTOGRAPHS OF SHERDS



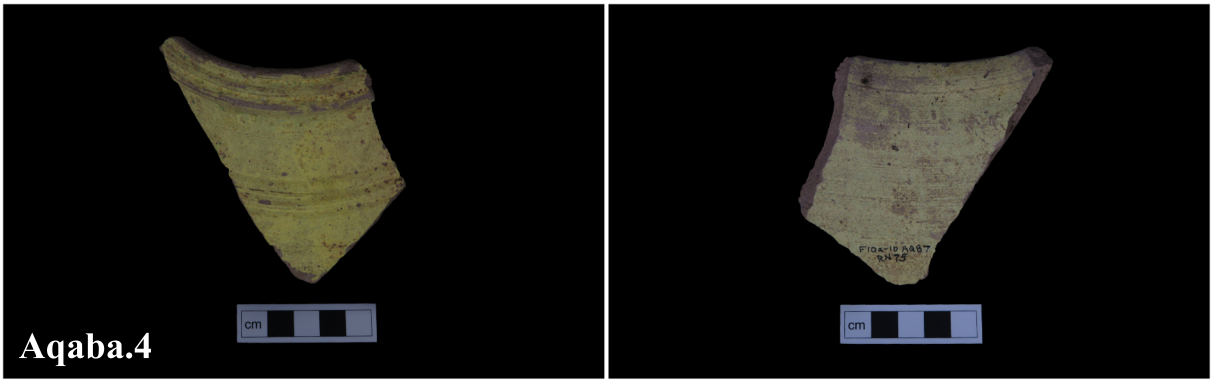
Fustat.1



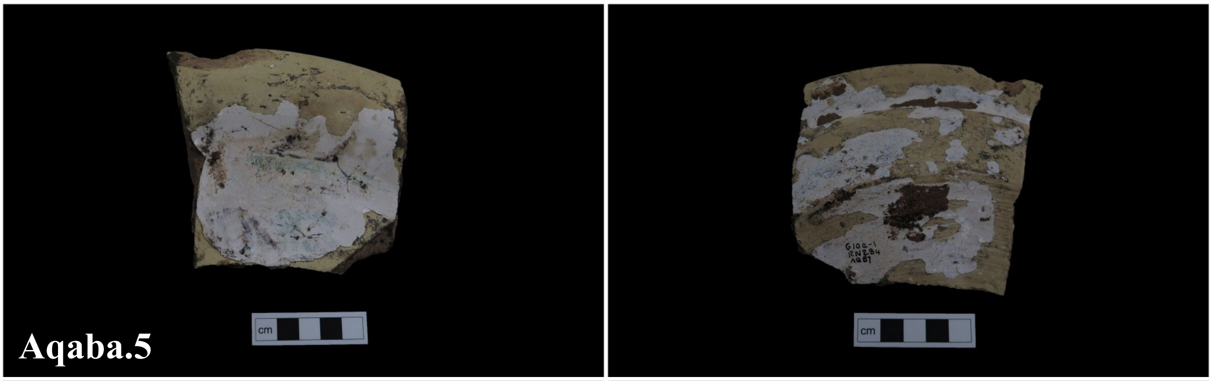
Aqaba.1

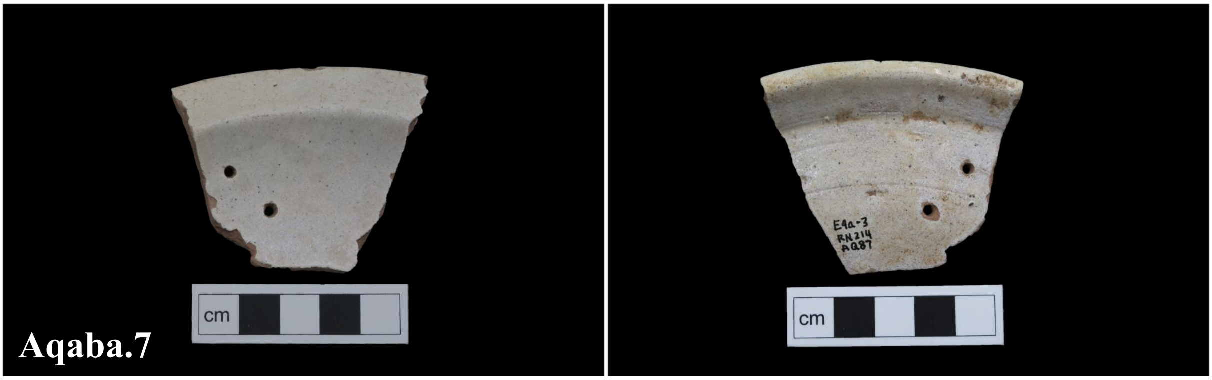


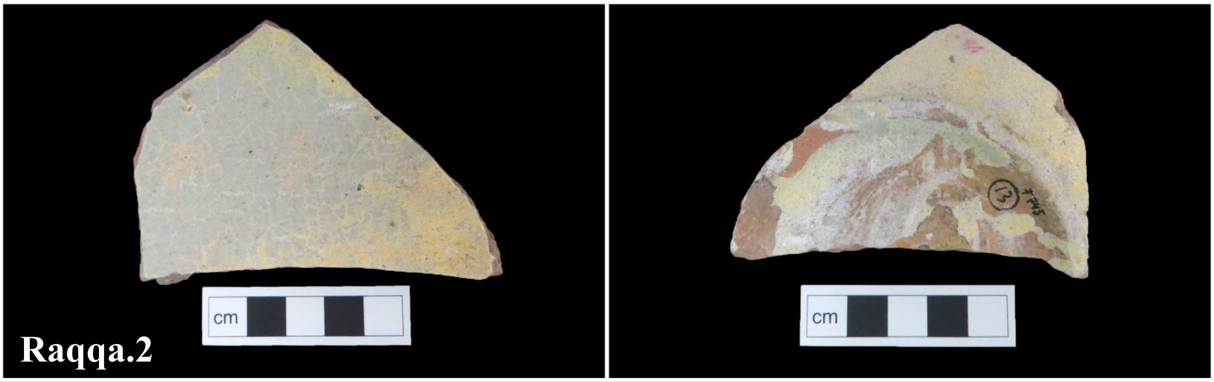
Aqaba.2

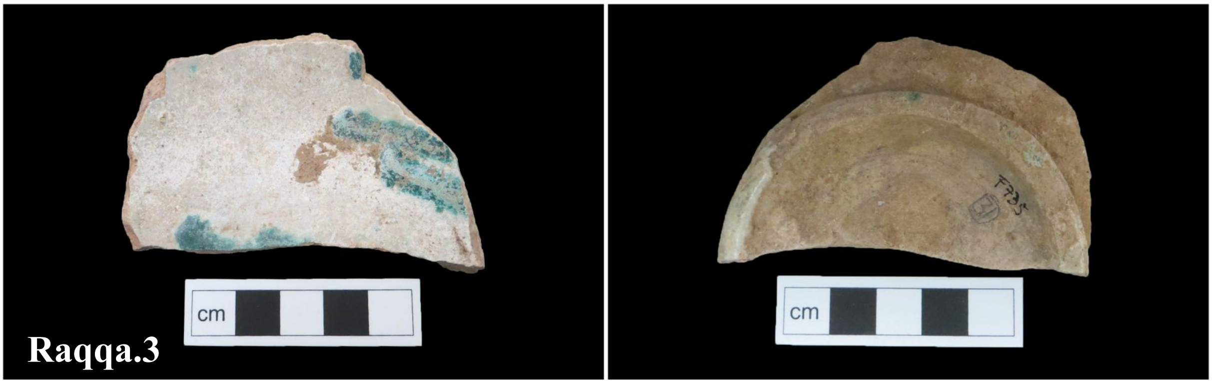


Aqaba.4

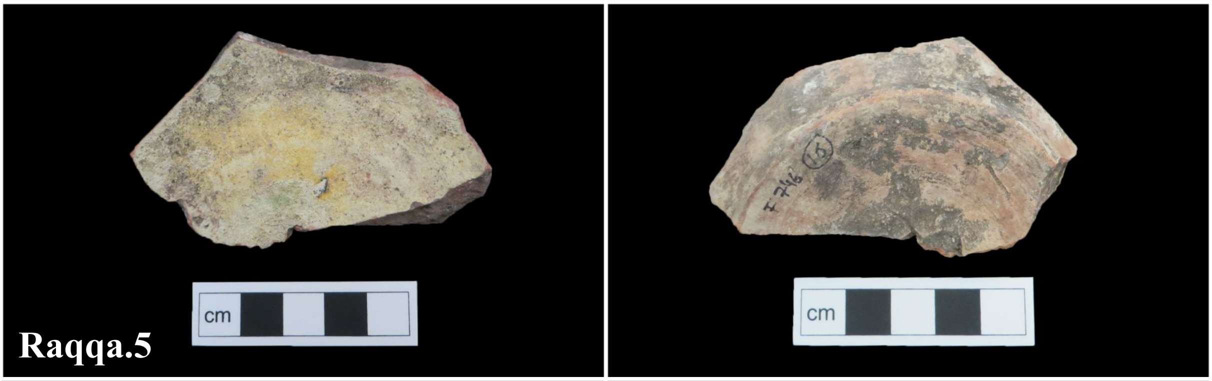

**Aqaba.5**


**Aqaba.7**

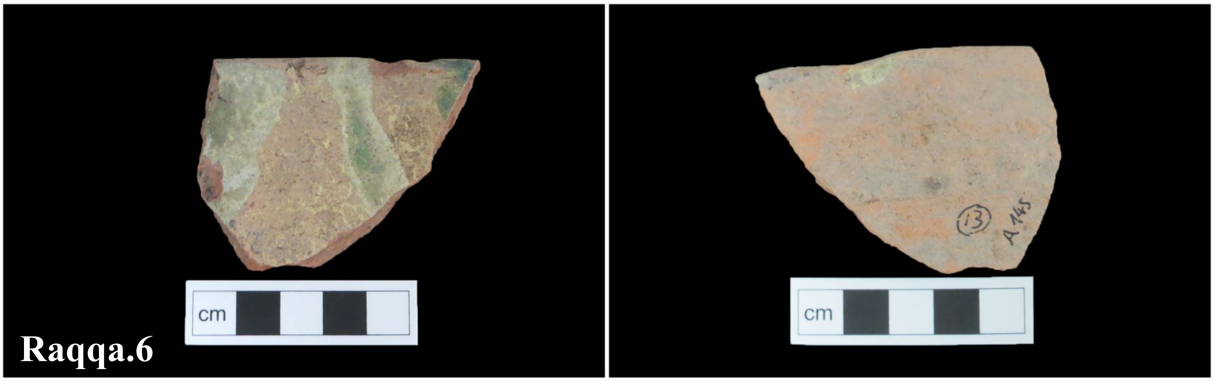

**Raqqa.2**


**Raqqa.3**

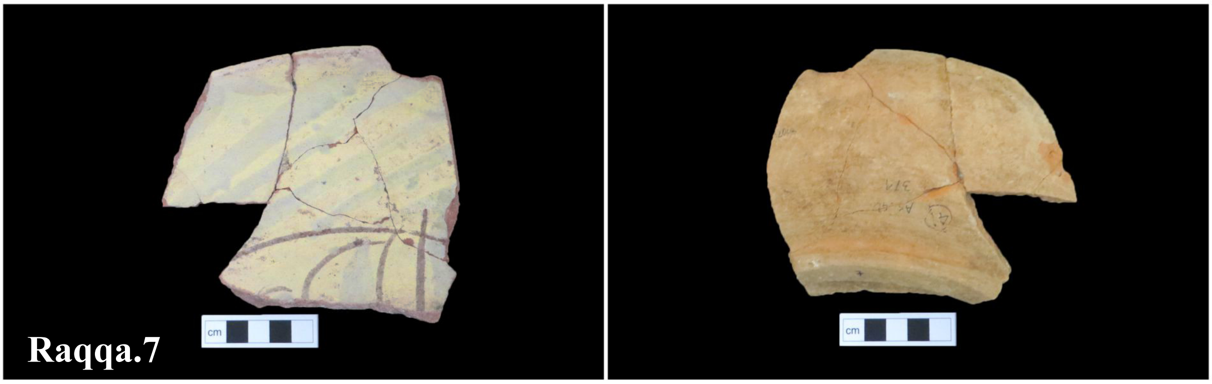
Raqqa.4



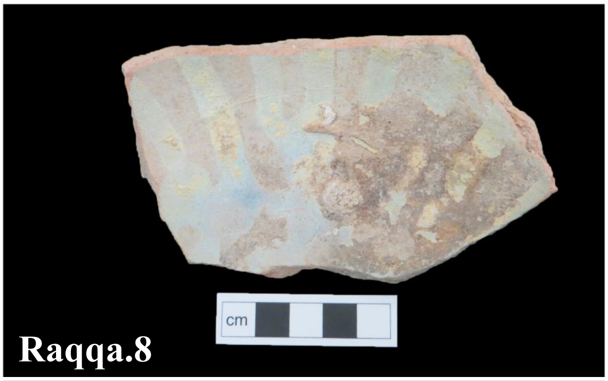
Raqqa.5



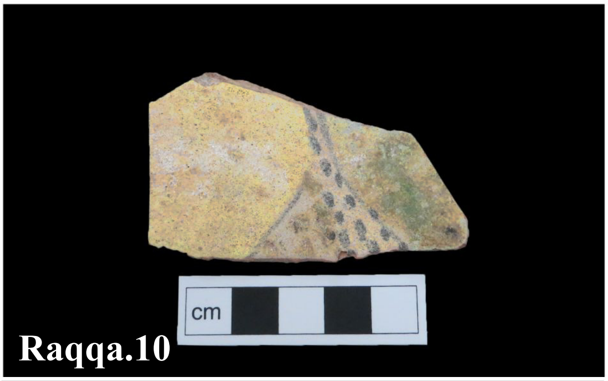
Raqqa.6



Raqqa.7

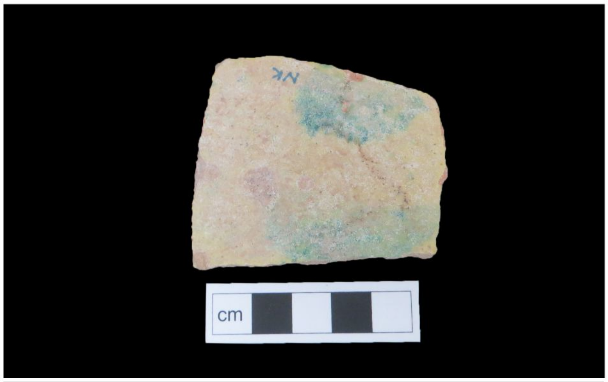


Raqqa.8

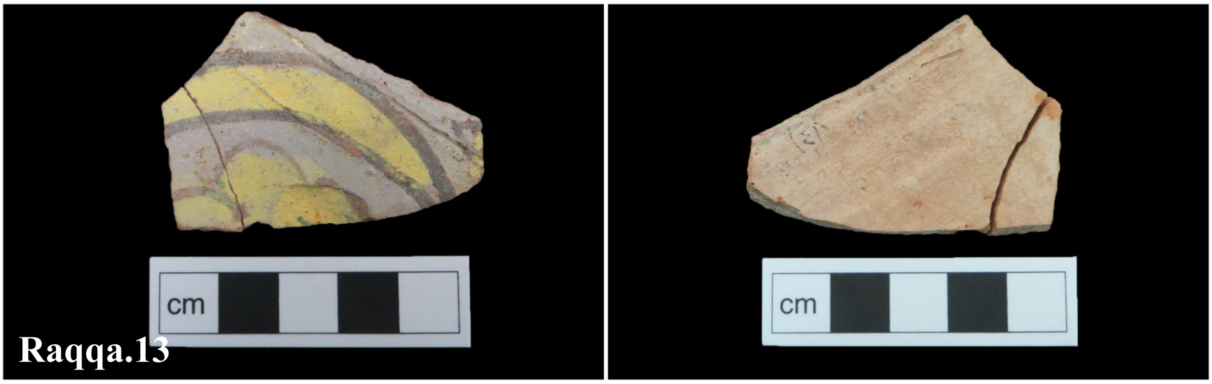


Raqqa.10

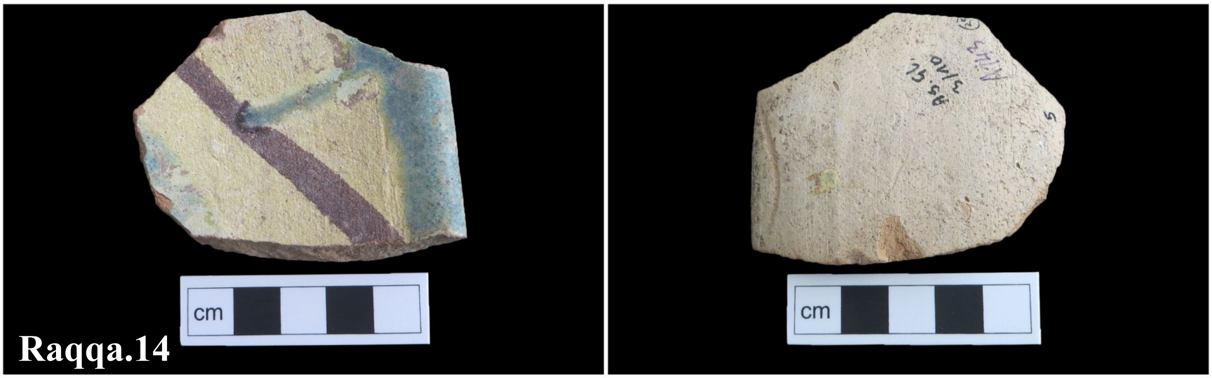
Raqqa.11



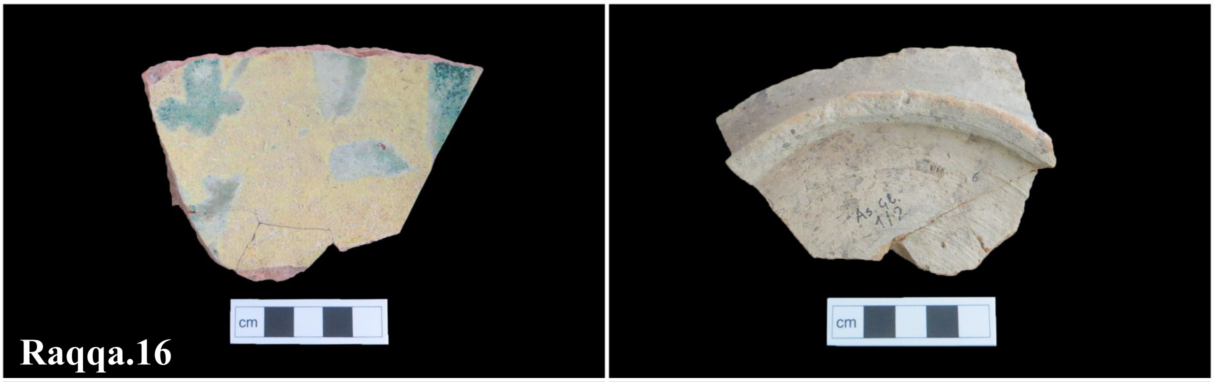
Raqqa.12



**Raqqa.13**



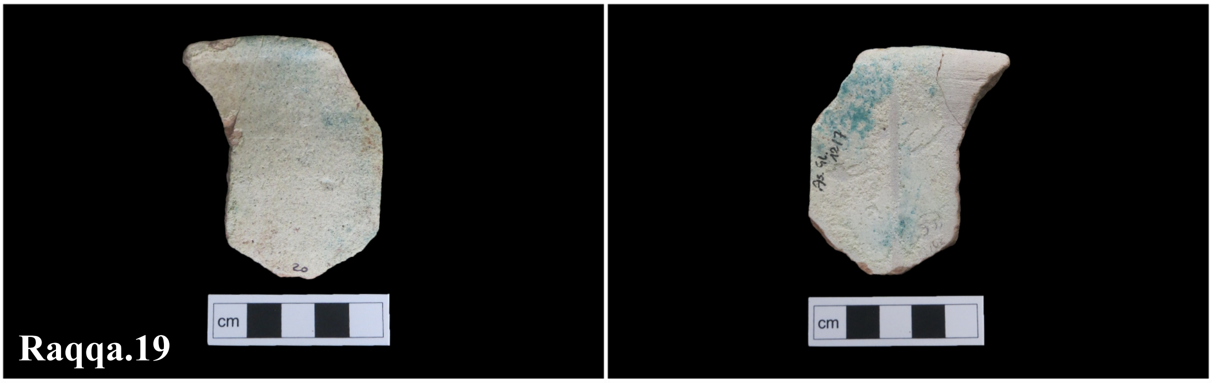
**Raqqa.14**



**Raqqa.16**

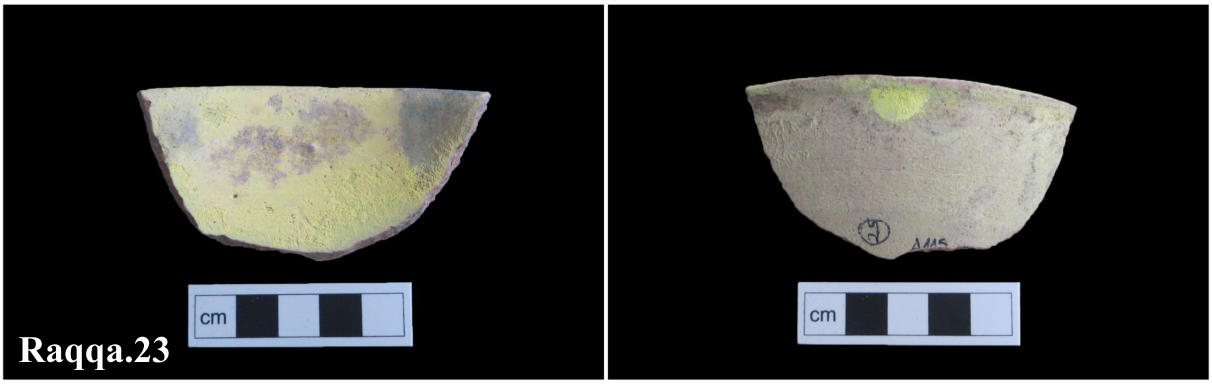
**Raqqa.17**

**Raqqa.18**



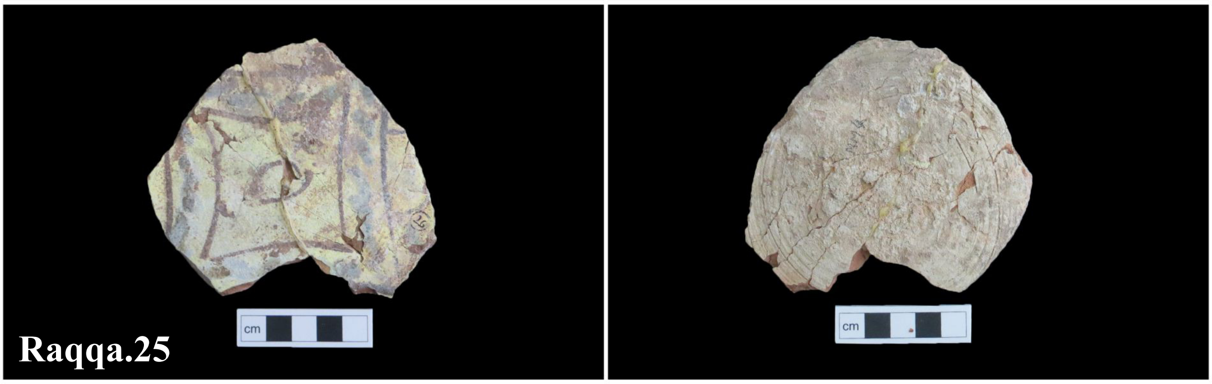
**Raqqa.19**

**Raqqa.22**



**Raqqa.23**

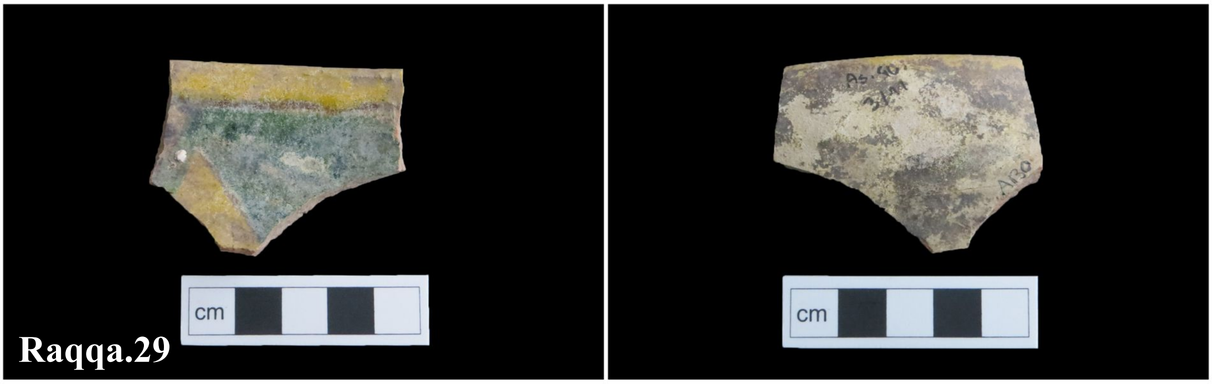
Raqqa.24



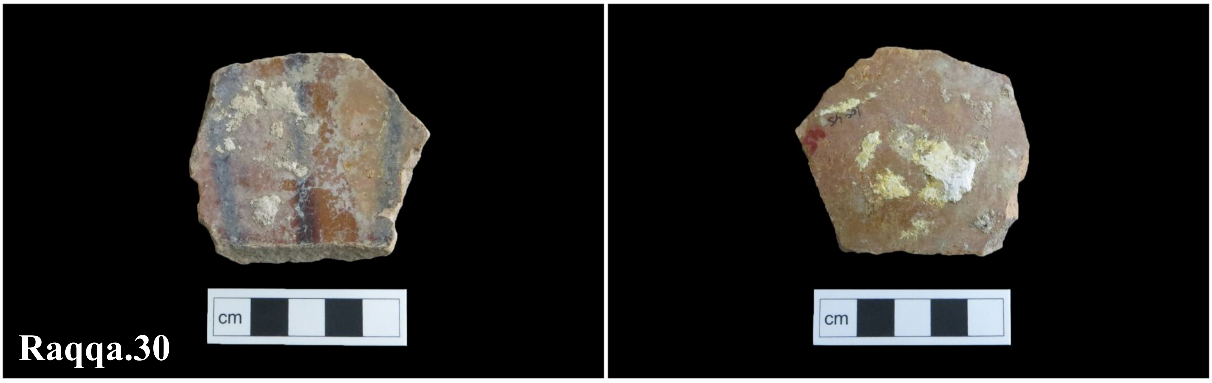
Raqqa.25

Raqqa.26

Raqqa.28

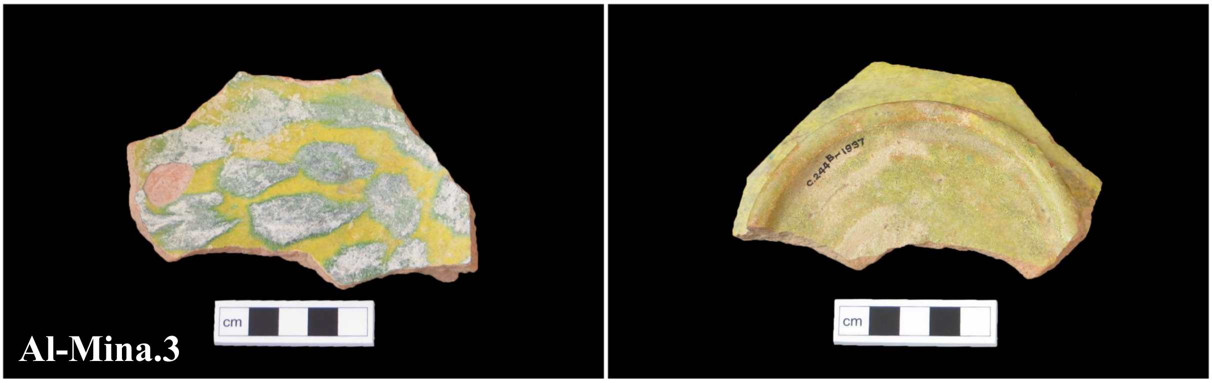


Raqqa.29

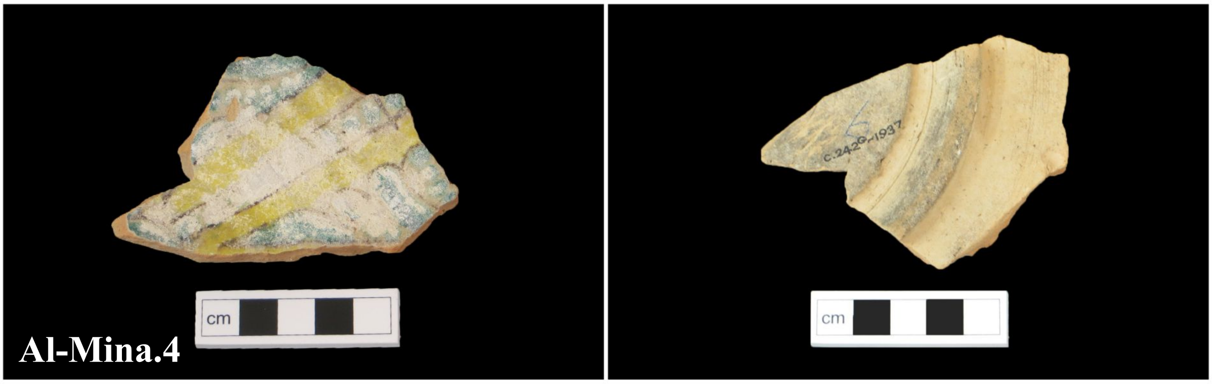


Raqqa.30

Raqqa.31



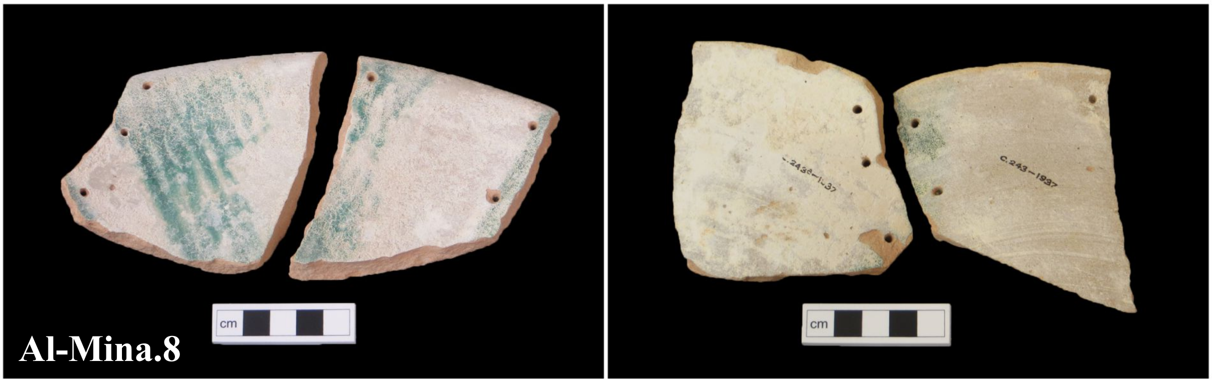
Al-Mina.3



Al-Mina.4

Al-Mina.6

Al-Mina.7



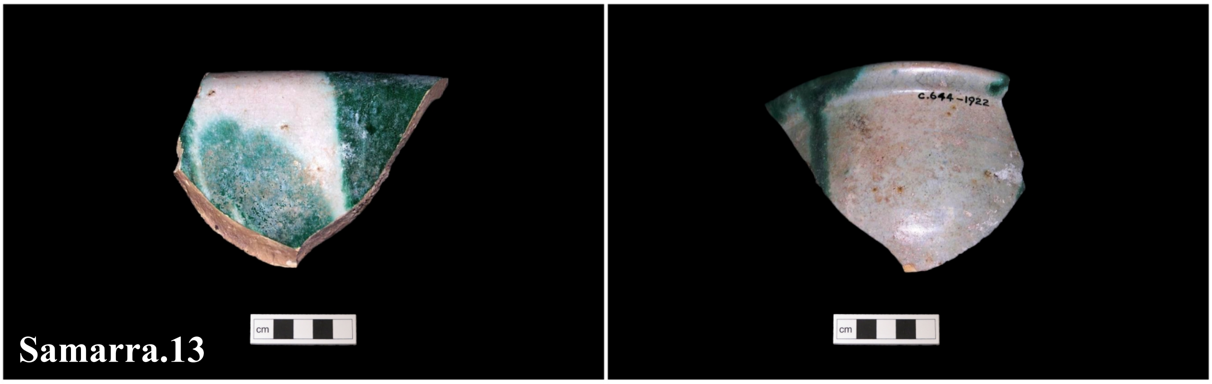
Al-Mina.8

Al-Mina.9

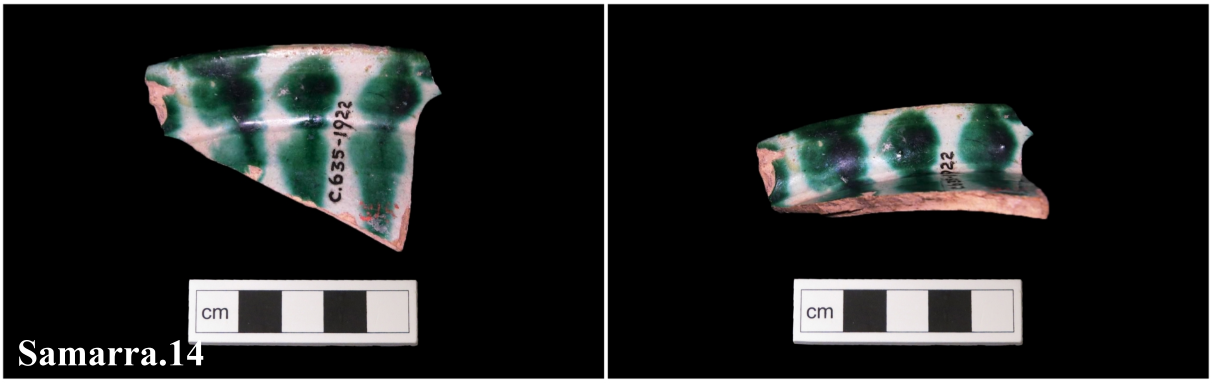
Al-Mina.12

Samarra.5

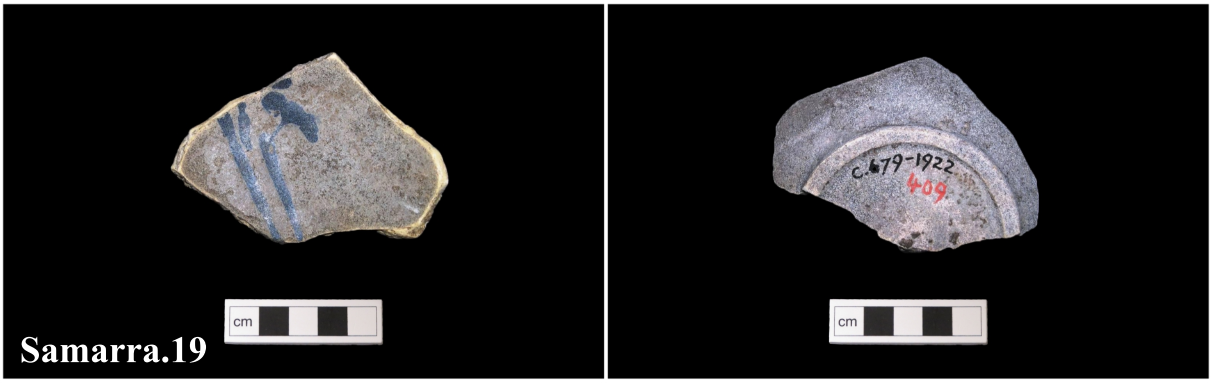
Samarra.6



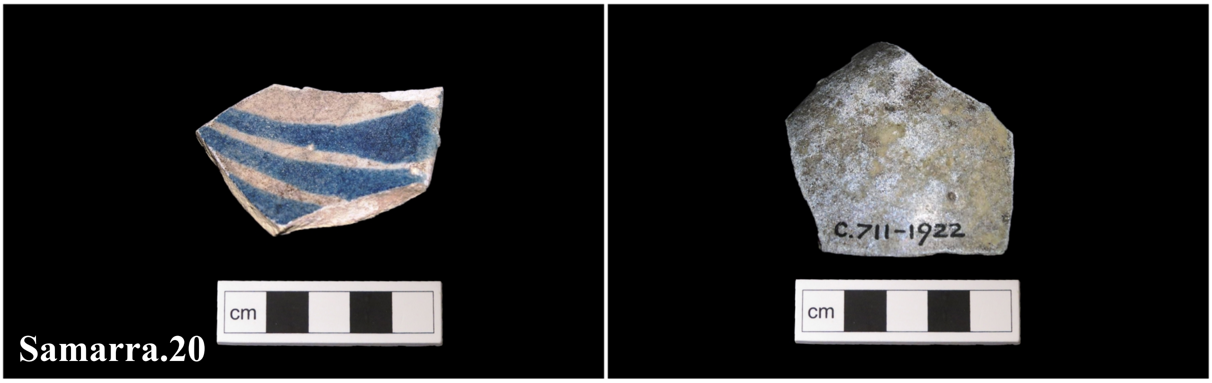
Samarra.13



Samarra.14



Samarra.19



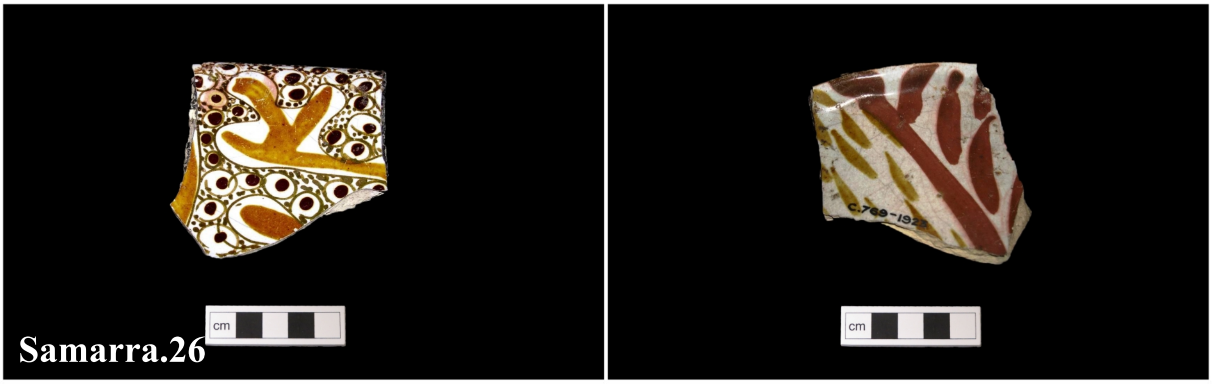
Samarra.20

**Samarra.21**

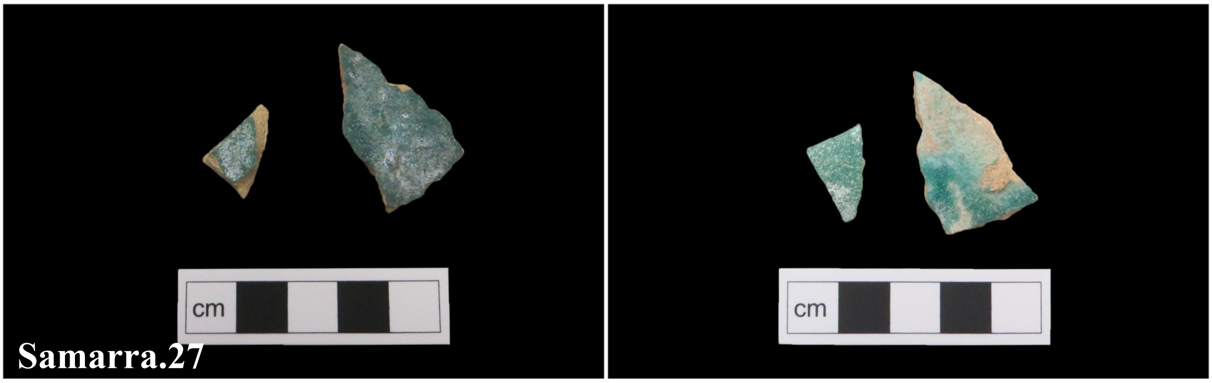
**Samarra.22**

**Samarra.23**

**Samarra.24**



**Samarra.26**

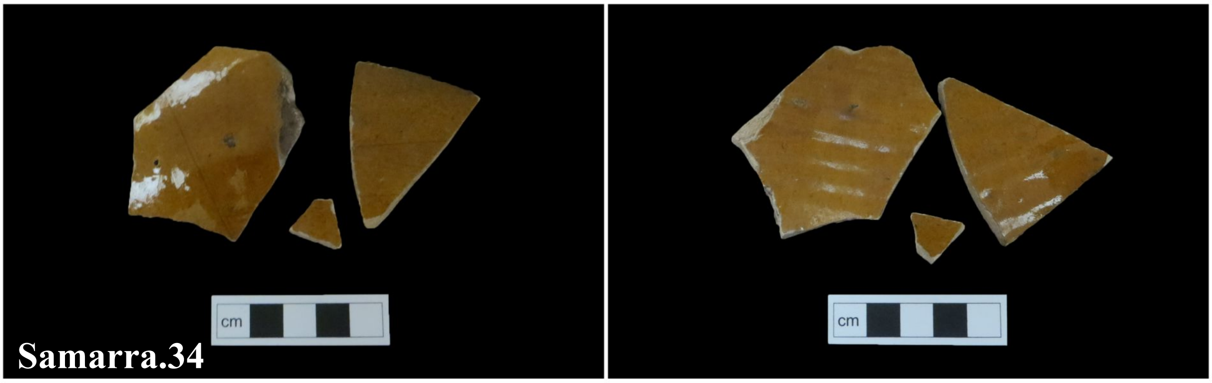


**Samarra.27**

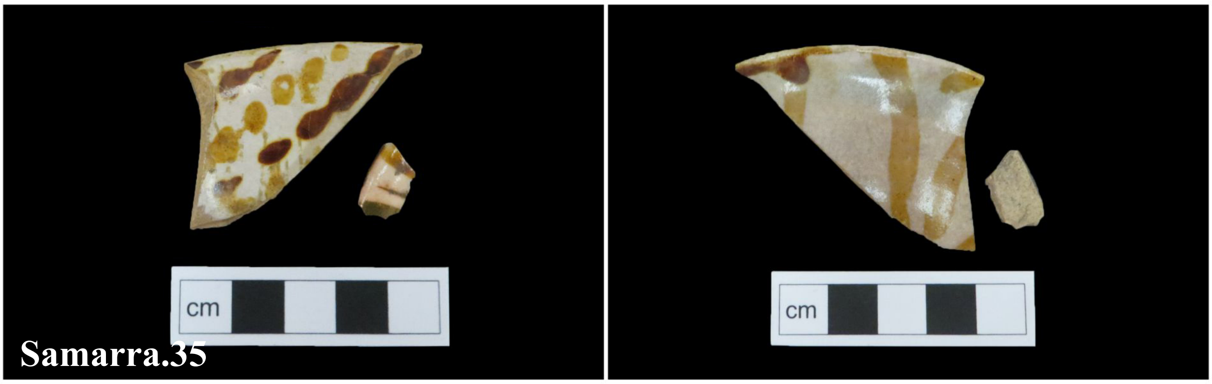
**Samarra.28**

**Samarra.29**

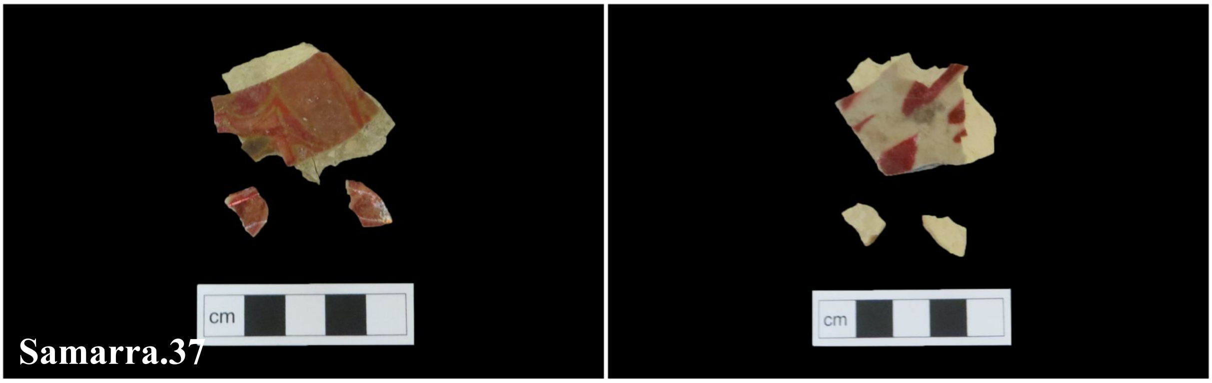
**Samarra.32**



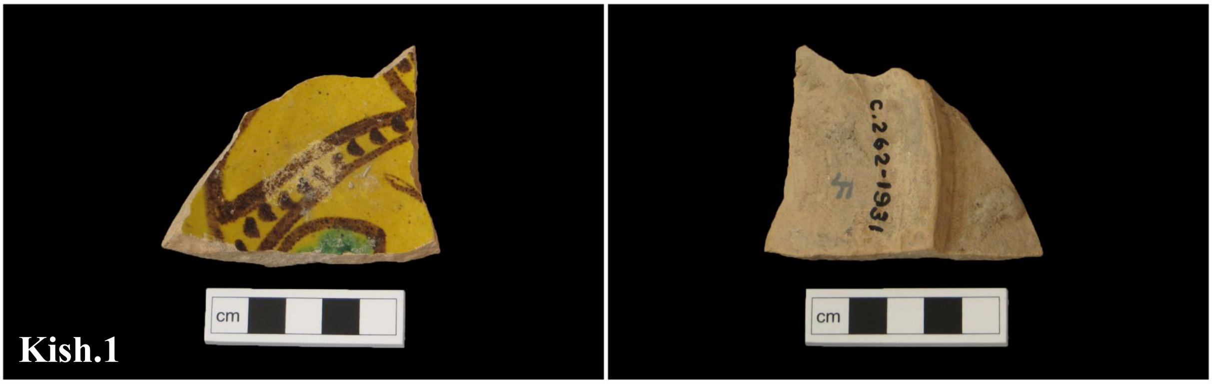
**Samarra.34**



**Samarra.35**

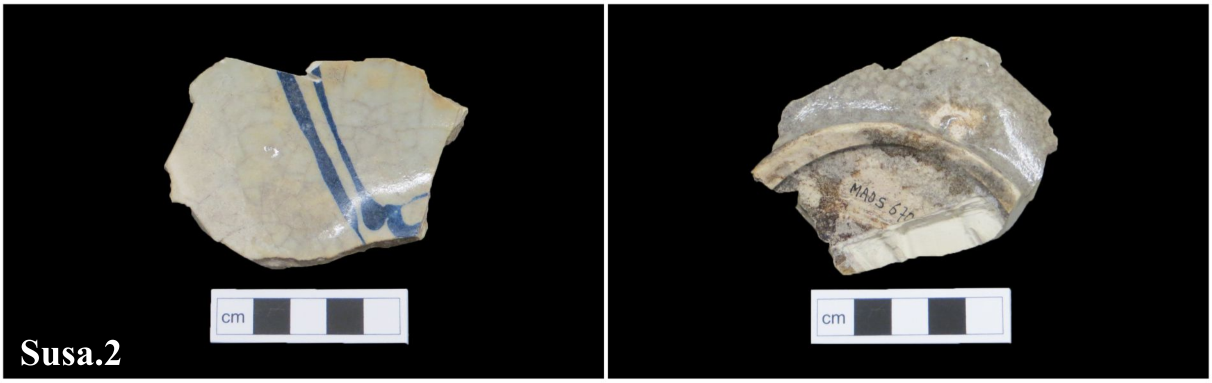


**Samarra.37**

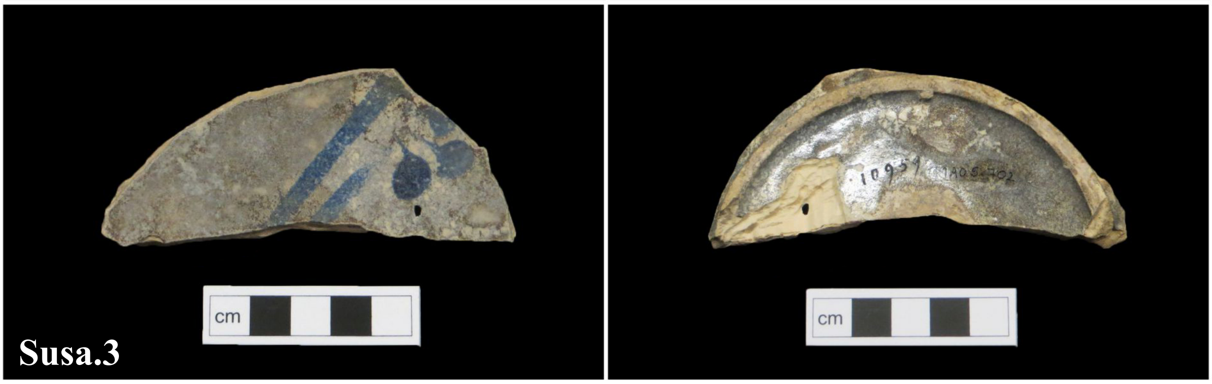


**Kish.1**

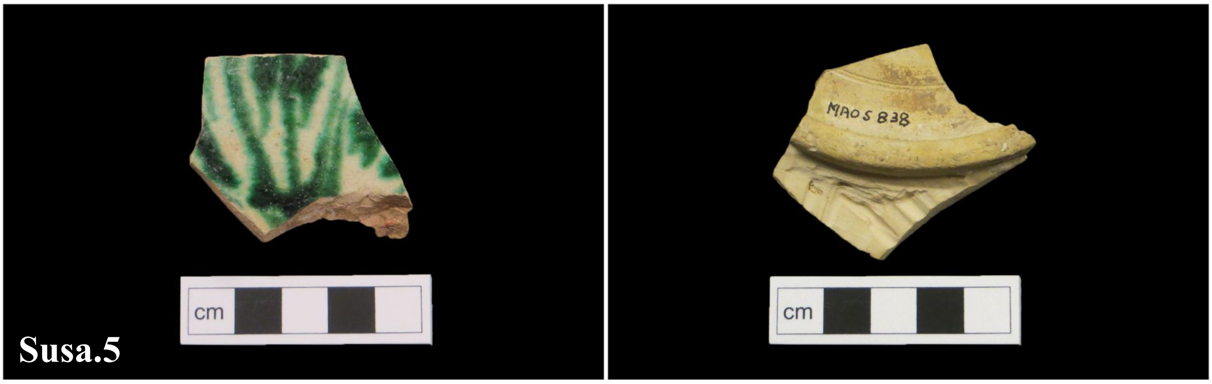
**Susa.1**



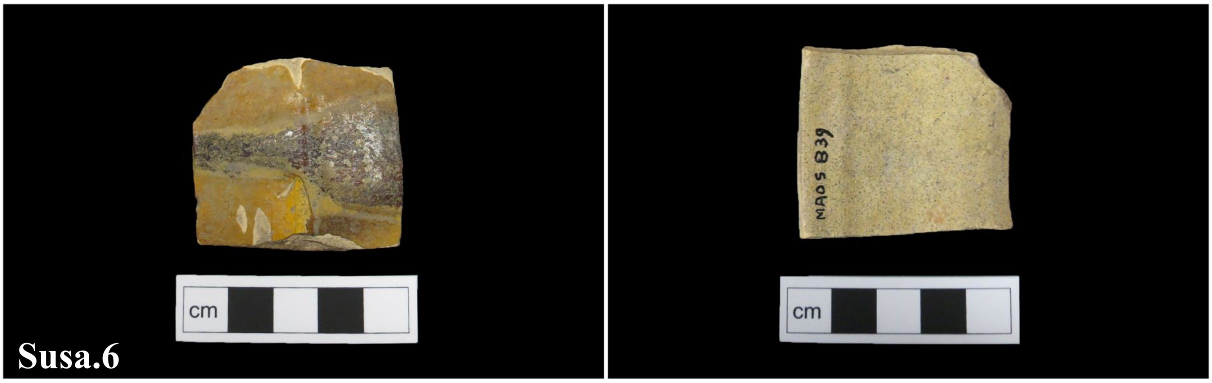
**Susa.2**



**Susa.3**



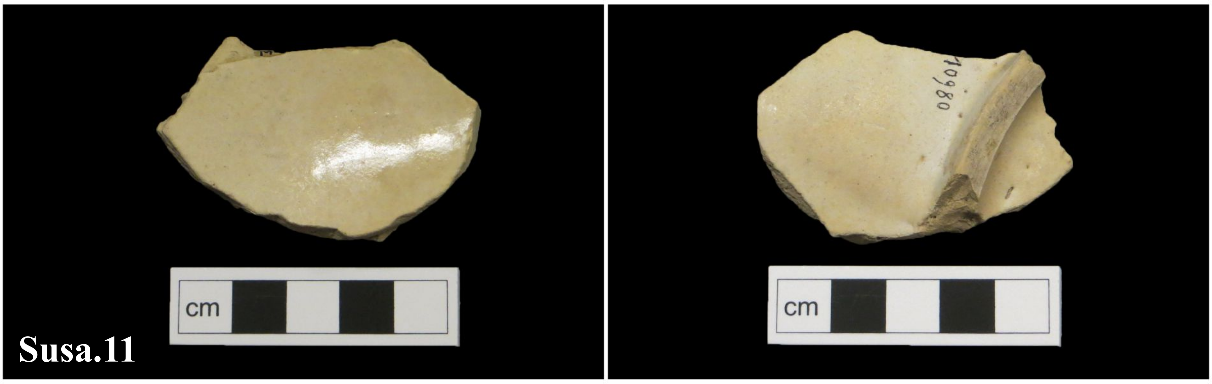
Susa.5



Susa.6

Susa.7

Susa.8



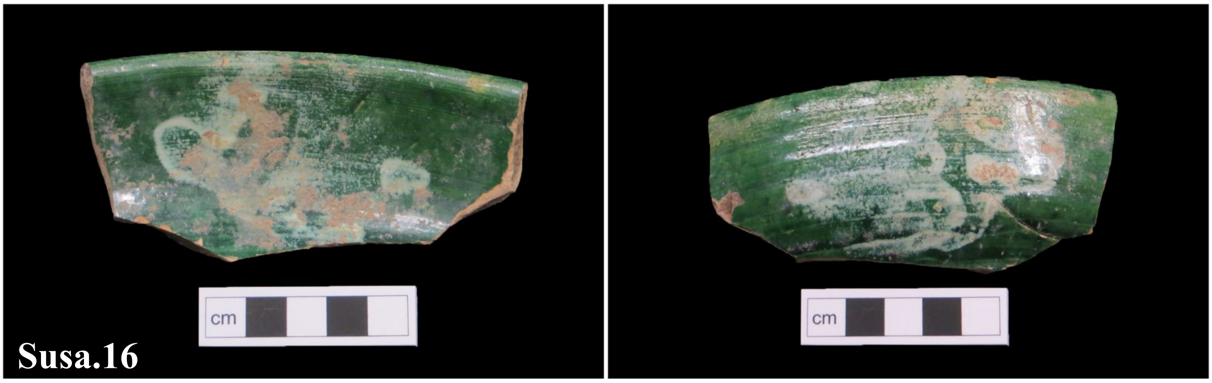
**Susa.11**

**Susa.12**

**Susa.13**

**Susa.14**

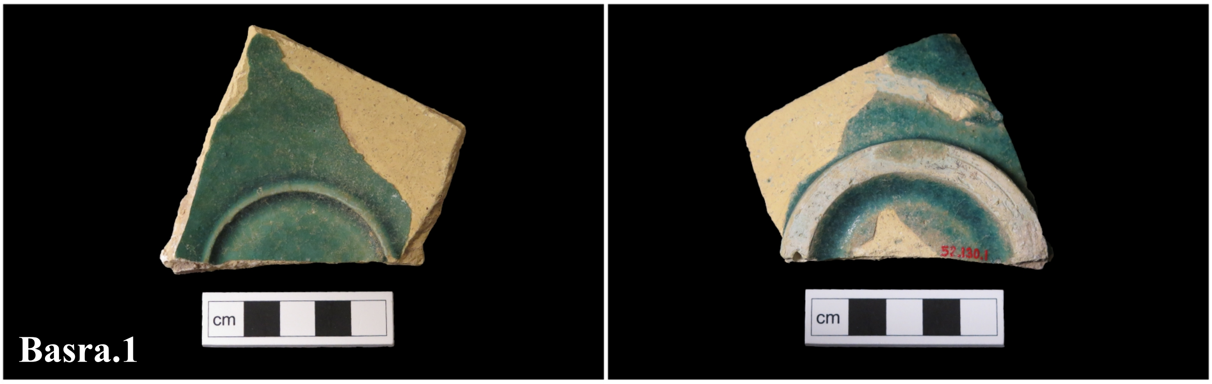
Susa.15



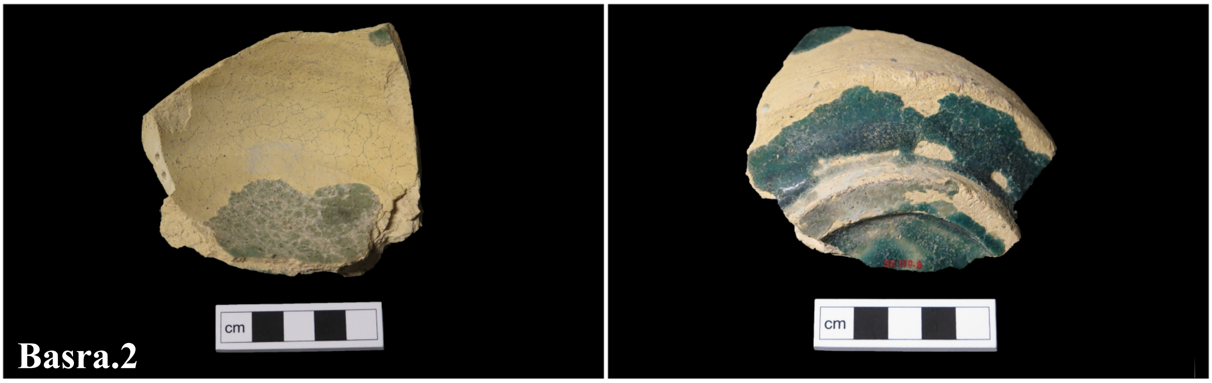
Susa.16

Susa.17

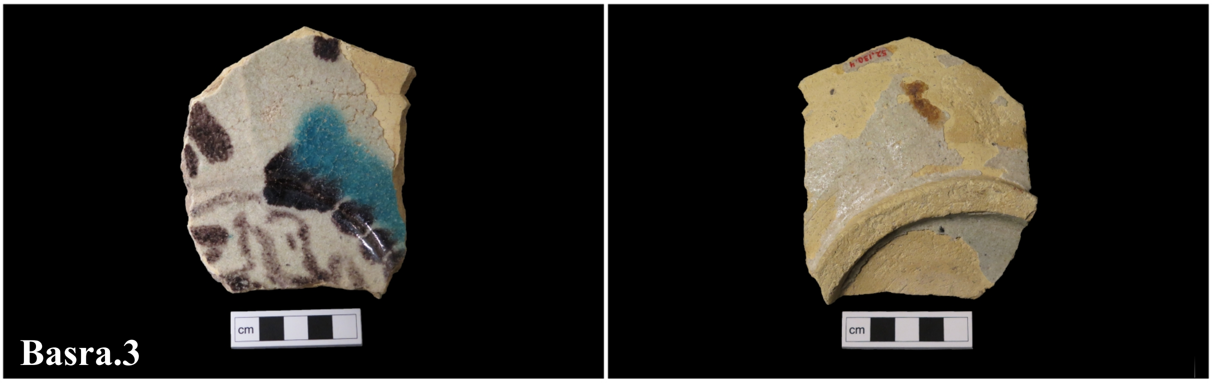
Susa.19



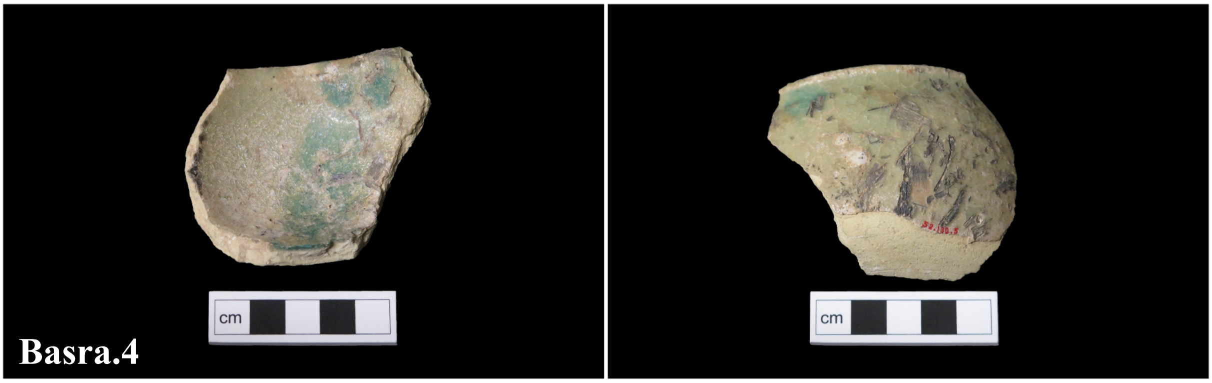
**Basra.1**



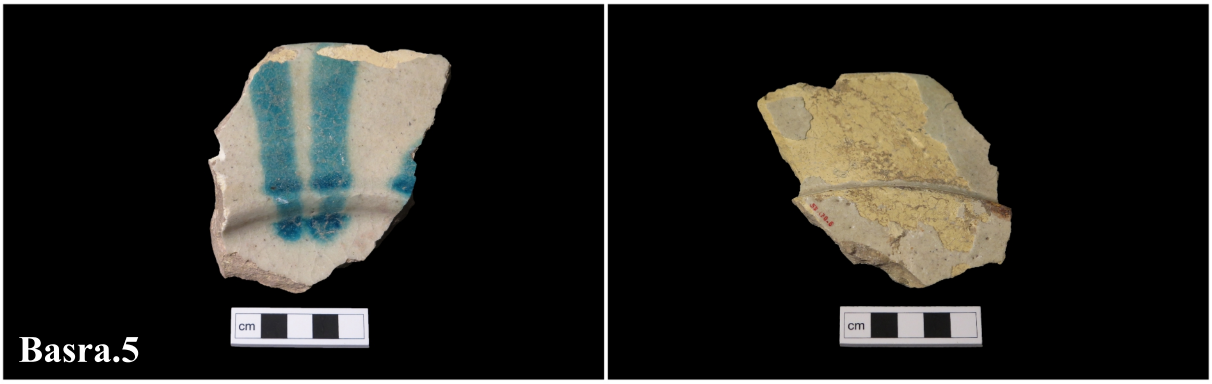
**Basra.2**



**Basra.3**

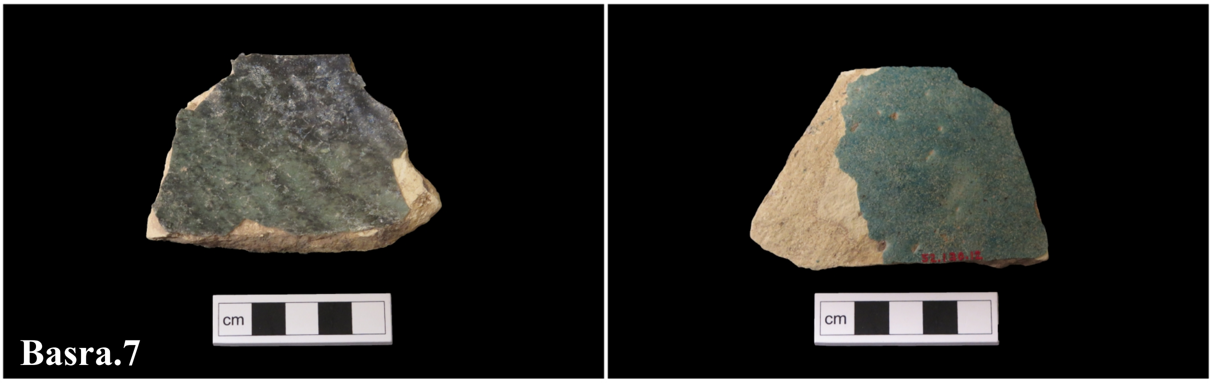


**Basra.4**



Basra.5

Basra.6



Basra.7

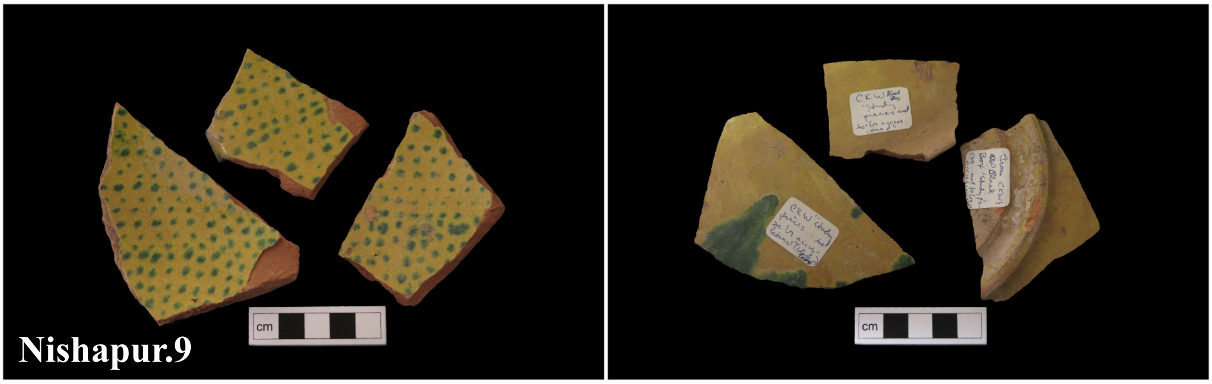
Basra.8

Basra.9

Nishapur.1

Nishapur.2

Nishapur.4



**Nishapur.9**

**Merv.1**

**Merv.2**

**Merv.3**

Samarqand.1

Samarqand.3

Samarqand.6

# BIBLIOGRAPHY

- Afshar, I., 2006, *Arāyes al-Jawāher va Nafāyes al-Atāyeb*, Abu'l-Qāsim A. Kāshānī, Tehran: Entesharat-i al-Maie [Persian].

-Afshar, I. and Daryagasht, M.R., 2004, *Jawāhirnāma-yi Nizāmī,* Mohammad b. A.B. Jōwhari Nayshābūrī, Tehran: Miras-i Maktub Press [Persian].

- Al-Bassam, Kh., 2007, Swab Phosphate Deposit, a Giant Discovery in the Western Desert, Iraq, *Iraqi Bulletin of Geology and Mining*, **3**(1), 25-40.

- Allan, J.W., 1973, Abu'l-Qasim's treatise on ceramics, *Iran*, **11,** 111-20.

- Al-Yaquibi, A. b. A.Y., 897?, Al-Buldan, Translated by M.I. Ayati, Tehran : Bungāh-i Tarjumah va Nashr-i Kitāb.

- Armstrong, P., From Constantinople to Lakedaimon: Impressed White Wares, *British School at Athens Studies,* **8**, MOSAIC: Festschift for A.H. S. Megaw, 57-67.

- Arnon, Y.D., 2008, *Caesarea Maritima, the Late Periods (700–1291 CE)*, BAR International Series 1771, Oxford: Archaeopress.

- Avissar, M., 1996, The Medieval Pottery, In: A. Ben-Tor, M. Avissar and Y. Portugali (eds.), *Yokne'am I, the late period*, Qedem Reports, 3, Jerusalem: The Institute of Archaeology, The Hebrew University of Jerusalem, 75-262.

- Bagci, Y., 2016, *Colours of the Caliphs, Shades of the Thughur al Sham: Revisiting Early Islamic Ceramics from 1935-1948 Gozlukule Excavations in Tarsus (Southern Turkey)*, unpublished PhD thesis, Leiden: University of Leiden.

- Brill, R.H., Whitehouse, D., 1988, The Thomas Panel, *Journal of Glass Studies*, **30**, 34-50.

- Brookfield, M.E., 2000, Geological development and Phaneroozoic crustal accretion in the western segment of the southern Tien Shan (Kyrgyzstan, Uzbekistan and Tajikistan), *Tectonophysics*, **328**, 1-14.

- Brusenko, L.G., 1986, *Glasurevannia Keramika Tashkenta IX-XII Veko*, Tashkent: FAN.

- Burjakov, J., Filanobich, M., 1999, Tashkent and Ilak, In: Srednia Azia v rannem srednevekov'e, G.M., Brykina (ed.), 78-92.

- Caiger-Smith, A., 1973, *Tin-glaze pottery in Europe and the Islamic world: the tradition of 1000 years in maiolica, faience and delftware*, London: Faber.

- Casellato, U., Fenzi, F., Pia Riccardi, M., Osmida, G.R., Vigato, P.S., 2007, *Journal of Cultural Heritage*, **8**, 412-422.

- Clark, R., Cridland, L., Kariuki, B., Harris, K., and Withnall, R., 1995, Synthesis, structural characterisation and Raman spectroscopy of the inorganics pigments lead tin yellow types I and II and lead antimony yellow: their identification on medieval paintings and manuscripts, *Journal of the Chemical Society, Dalton Transactions*, **16**, 2577-82.

- Crowe, Y., 1977, Early Islamic Pottery and China, *Transactions of Oriental Ceramics*, **41**, 263-75.

- Engeman, J., 1990, Early Islamic Glazed Pottery of the Eight Century AD from the Excavations at Abu Mina, In: W. Godlewski (ed.), *Coptic and Nubian pottery, Part I: international workshop, Nieborów, August 29-31, 1988*, Warsaw: National Museum in Warsaw, 63-70.

- Fabbri, B., Gualtieri, S., Ruffini, A., Movassaghi, E., Alavi, M., 2004, Compositional and technological characteristics of the inlay wall facing ceramics of the Jamé Mosque in Esfahan, Iran, *Key Engineering Materials*, vols. **264-268**, 2403-2406.

- Fehérvári, G., 2000, Ceramics of the Islamic World in the Tareq Rajab Museum, London: Tauris.
- Fourest, H.P., *Delftware: faience production at Delft*, translated by K. Watson, London: Thames and Hudson.

- Freestone, I.C., 2006, Glass production in Late Antiquity and the Early Islamic period: a geochemical perspective, In: M. Maggetti and B Messiga (eds.), *Geomaterials in Cultural Heritage,* 201-216.

- _____, Bimson, M., Buckton, D., 1990, Compositional categories of Byzantine glass tesserae, In: *Annales du 11e Congrès de l'Association Internationale pour l'Histoire du Verre (Bâle, 29 août -3 septembre 1988)*, 271-279.

- _____, Stapleton, C.P., 2016 (in press), Composition, technology and production of coloured glasses from mosaic vessels of the Early Roman Empire, In: J. Bayley, I.C. Freestone and C. Jackson (eds.), Glass of the Roman Empire, Oxford: Oxbow books.

- Frierman, J.D., Asaro, F., and Michel, H.V., 1979, The provenance of early Islamic lustre wares, *Ars Orientalis*, **11**, 111-26.
- Fitz, S.C., 1982, The coloured glazes of Neo-Babylonian Wall Facings, *Ceramic Forum International, Berichte Deutch Keramischen Gesellschaft*, **59**, 179-85 [German].
- Fukai, S. 1981, *Ceramics of Ancient Persia*, New York: Weatherhill; Kyoto: Tankosha.

- Gayraud, R.P., 2009, Assemblages de ceramiques egyptiennes dates pae les fouilles d'Istabl Antar (milieu VIIe-2ere moitie du Xe s.), In: J. Zozaya et al. (eds.), *Actas del VIII Congreso Internacional de Cerámica Medieval en el Mediterráneo: Ciudad Real-Almagro, del 27 de febrero al 3 de marzo de 2006*, **1**, Asociación Española de Arqueología Medieval, 171-192.

- Gill, M.S., Rehren, Th., 2011, Material characterization of ceramic tile mosaic from two 17th-century Islamic monuments in Northern India, *Archaeometry*, **53** (1), 22-36.

- _____, Rehren, Th., Freestone, I., 2014, Tradition and indigeneity in Mughal architectural glazed tiles, Journal of Archaeological Science, 49, 546-555.

- Guillermina, J., Peli, A., Makariou, S., 2005, *Suse: terres cuites islamiques*, Gand: Snoeck, Paris: Musée du Louvre.

- Hallett, J., 2000, *Trade and Innovation: The Rise of the Pottery Industry in Abbasid Basra*, unpublished DPhil thesis, Trinity College, University of Oxford.

- Heck, M., Hoffmann, P., 2002, Analysis of Early Medieval Glass Beads- The Raw Materials to Produce Green, Orange and Brown Colours, *Microchimica Acta*, **139**, 71-76.

- Heck, M., Rehren, Th., Hoffmann, P., 2003, The Production of Lead-Tin Yellow at Merovingian Schleitheim (Switzerland), *Archaeometry*, **45**(1), 33-44.

- Hedges, R.E.M., Moorey, P.R.S., 1975, Pre-Islamic Ceramic Glazes at Kish and Nineveh in Iraq, *Archaeometry*, **17**, 25-43.

- Henshaw, C.M., 2010, *Early Islamic Ceramics and Glazes of Akhsiket, Uzbekistan*, unpublished PhD thesis, London: University College London.

- Herrmann, G., Kurbansakhotov, K., Simpson, St J., 1997, The International Merv Project, preliminary report on the fifth season (1996), *Iran*, **35**, 1-33.

- _____, 1999, The International Merv Project, preliminary report on the fifth season (1998), *Iran*, **37**, 1-24.

- _____, 2000, The International Merv Project, preliminary report on the fifth season (1999), *Iran*, **38**, 1-31.

- _____, 2001, The International Merv Project, preliminary report on the fifth season (2000), *Iran*, **39**, 9-52.

- Hill, D.V., 2004, Chemical and Mineralogical Characterization of Sasanian and Early Islamic Glazed Ceramics from the Deh Luran Plain, Southwestern Iran, *Archaeometry*, **46**(4), 585-605.

- Holakooei, P., A multi-spectroscopic approach to the characterization of early glaze opacifiers: studies on an Achaemenid glazed brick found at Susa, south-western Iran (mid-first millennium BC), *Spectrocimica Acta Part A: Molecular and Bionolecular Spectroscopy*, **116**, 49-56.

- Jones, R.E., 1980, Analysis of some Islamic lustre ware from Egypt, in H. Philon (ed.), *Early Islamic ceramics: ninth to late twelfth centuries*, catalogue of Islamic art, **1**, 303-5, Athens: Benaki Museum, Islamic Art Publications, S.A.

- Kervran, M., 1977, Les niveaux islamiques du secteur oriental de l'Apadana, II. - Le matériel céramique, *Cahiers de la Délégation Archéologique Française en Iran*, **7**, 75-161.

- _____, 1979, Une sucrerie d'époque islamique sur la rive droite du chaour à Suse: II, le material archaeologique, *Cahiers de la Délégation Archéologique Française en Iran*, **10**, 177-237.

- Kleinmann, B., 1986, History and development of early Islamic pottery glazes, in *Proceedings of the 24th international archaeometry symposium*, J.S. Olin and M.J. Blackman (eds.), PP. 73-84, Washington DC: Smithsonian Institution Press.

- _____ , 1991, Cobalt pigments in the early blue Islamic glazes and the reconstruction of the way of their manufacture, in *Archaeometry '90, Proceedings of the 27th international Archaeometry symposium*, E. Pernicka and G.A. Wagner (eds.), 327-36, Basle: Birkhäuser Verlag.

- Isfahānī, A.M., 1888, *On the Manufacture of Modern Kashi Earthenware Tiles and Vases In Imitation of the Ancient*, Museum of Science and Art, Edinburgh.

- Joel, G., 1992, Céramique glacurées d'époque Islamique trouvées à Tod, *Annales Islamologiques*, **26**, Cairo: Institute Francais d'Archéologie Orientale du Caire (IFAO), 1-18.

- Kennedy, H., 1986, *The Early Abbasid Caliphate; A Political History*, London: Croom Helm.

- Kingery, W.D., Vandiver, P.B., 1986, *Ceramic Masterpieces: art, structure, and technology*, New York: Free Press, London: Collier Macmillan.

- Koechlin, R., 1928, Les céramiques Musulmanes de Suse au Musée du Louvre, *Syria*, **9**(1), 40-58.

- Kühn, H., 1968, Lead-tin yellow, *Studies in Conservation*, **13**, 1, 7-33.

- Lane, A., 1947, Early Islamic Pottery: Mesopotamia, Egypt and Persia, London: Faber and Faber.

- Lightbown, R.W., and Caiger-Smith, A., 1980, The three books of the potter's art, by Cipriano Piccolpasso, London: Scolar Press.

- Marii, F., 2012, Glass Tesserae from the Petra Church, In: C. Entwistle and L. James (eds.), *New Light on Old Glass: Recent Research on Byzantine Mosaics and Glass*, London: British Museum.

- Mason, R.B., and Keall, E.J., 1990, Petrography of Islamic Pottery from Fustat, *Journal of the American Research Centre in Egypt*, **27**, 165-184.

- _____, and Keall, E.J., 1991, The Abbasid glazed wares of Siraf and the Basra connection: petrographic analysis, *Iran*, **29**, 51-66.

- _____, and Tite, M.S., 1994, The beginnings of Islamic stonepaste technology, *Archaeometry*, **36** (1), 77-91.

- _____, and Tite, M.S., 1997, The beginnings of tin opacification of pottery glazes, *Archaeometry*, **39** (1), 41-58.

- _____, 1994, *Islamic Glazed Pottery: 700-1250*, DPhil Thesis, University of Oxford.

- _____, 1995, New looks at old pots, results of recent multidisciplinary studies of glazed ceramics from the Islamic world, *Muqarnas*, **12**, 1-10.

- _____, 1997, Early Medieval Iraqi lustre-painted and associated wares: typology in a multidisciplinary study, *Iraq*, **59**, 15-61.

- _____, 2004, *Shine Like the Sun: lustre-painted and associated pottery from the medieval Middle East*, Costa Mesa and Toronto: Mazda Publishers in association with Royal Ontario Museum.

- Mass, J.L., Stone, R.E., Wypyski, M.T., 1998, The mineralogical and metallurgical origins of Roman opaque colored glass, In: W.D. Kingery and P. McCray (eds.), the Prehistory and history of glassmaking technology, Ceramics and civilization, Westerville, Ohio: American ceramic society,121-144.

- _____, Wypyski, M.T., Stone, R.E., 2002, Malkata and Lisht glassmaking technologies: towards a specific link between second millennium BC metallurgists and glassmakers, *Archaeometry*, **44**, 67-82.

- Matin, M., 2013, An experimental study of cobalt ore, treatment processes from Kashan, Iran, based on historical accounts, unpublished MSc thesis, University of Oxford.

- _____ and Pollard, M., 2015, Historical accounts of cobalt ore processing from the Kashan Mine, Iran, *IRAN (Journal of the British Institute of Persian Studies)*, 53, 171-183.

- _____, 2016, From ore to pigment: A description of the minerals and an experimental study of cobalt ore processing from the Kashan Mine, Iran, *Archaeometry*.

- Matson, F.R., 1985, The brickmakers of Babylon, In: W.D. Kingery (ed.), *Ancient technology to modern science: ceramics and civilisation I*, Columbus, Ohio: American Ceramics Society.

- McCarthy, B.E., 1996, *Microstructural and Compositional Studies of the Technology and Durability of Ceramic Glazes from Nippur, Iraq, ca. 250 BC to 1450 AD*, unpublished PhD Dissertation, Baltimore: John Hopkins University.

- Michel, H.V., Frierman, J.D., and Asaro, F., 1976, Chemical composition of ceramic, wares from Fustat, Egypt, *Archaeometry*, **18**(1), 85-92.

- Michel, H.V., Frierman, J.D., and Asaro, F., 1976, Chemical composition of ceramic, wares from Fustat, Egypt, *Archaeometry*, **18**(1), 85-92.

- Mohammaddoost, H., 2010, *Kanishenassi, geochimi va jenez kansar cobalt Qamsar, djonub e Kashan*, Unpublished MSc dissertation, Tarbiat Moddares University, Tehran, Iran.

- Molera, J., Pradell, T., Salvadó, N., Vendrell-Saz, M., 1999, Evidence of tin oxide recrystallization in opacified lead glazes, *J. Am. Ceram. Soc.*, **82**(10), PP. 2871-75.

- Molina, G., Tite, M.S., Molera, J., Climent-Font, A., Pradell, T., 2014, Technology of production of polychrome lustre, *Journal of the European Ceramic Society*, **34**, 2563-2574.

- Morzova, G.S., 2005, A review of Holocene avulsions of the Tigris and Euphrates Rivers and possible effects on the evolution of civilizations in lower Mesopotamia, *Geoarchaeology: An International Journal*, **20** (4), 401-23.

- Neri, E., Verita, M., Conventi, A., 2012, Glass Mosaic Tesserae from the 5th to 6th Century Bapistery of San Giovanni alle Fonti Milan, Italy; Analytical Investigation, In: C. Entwistle and L. James (eds.), *New Light on Old Glass: Recent Research on Byzantine Mosaics and Glass*, London: British Museum.

- Northedge, A., 2001, Thoughts on the introduction to polychrome glazed pottery in the Middle East, in E. Villeneuve and P.M. Watson (eds), *La ceramique Byzantine et proto-islamique en Syrie-Jordaine (IVe-VIIIe sciecles apr. J.-C.)*, Beirut: Institut francais d'archaeologie du Proche-Orient), 207-14.

- Pace, M., Bianco Prevot, A., Mirti, P., Venco Riccardi, R., 2008, The Technology of Production of Sasanian Glazed Pottery from Veh Ardašir (central Iraq), *Archaeometry*, **50**(4), 591-605.

- _____, and Kennet, D., 1994, The Samarra Horizon, In: E. Grube (ed.), *Cobalt and Lustre*, The Nasser D. Khalili Collection of Islamic Art, V IX, Oxford: Khalili Research Centre, University of Oxford, 21-35.

- Paynter, S., Okyar, F., Wolf, S., Tite, M.S., 2004, The production technology of Iznik pottery – a reassessment, *Archaeometry*, **46**(3), PP. 421-37.

- Philip, G., 1968, Mineralogy of Recent Sediments of Tigris and Euphrates Rivers and Some of the Older Detrital Deposits, *Journal of Sedimentary Petrology*, **38**(1), 35-44.

- Pilosi, L., Stamm, K., Wypyski, M.T., 2012, An Islamic cameo glass fragment in the Metropolitan Museum of Art, In: Annales du 18e Congrés de l'Association Internationale pour l'Histoire du Verre (Thessaloniki 2009), 341-345.

- Pradell, T., Lustre and Nanostructures- Ancient Technologies Revisited, In: Philippe Dillmann *et al.* (eds.), *Nanoscience and Cultural Heritage*, Atlantis Press.

- _____, Molera, J., Smith, A.D., Tite, M.S., 2008, The invention of lustre: Iraq 9th and 10th centuries AD, *Journal of Archaeological Science*, **35**, 1201-1215.

- Priestman, S.M.N., 2011, Opaque Glazed Wares: the definition, dating and distribution of a key Iraqi ceramic export in the Abbasid period, *Iran*, **49**, PP. 89-113.

- Radfar, J., Alaee-Mahabadi, S., Emami, M.H., 1993, *Geological map of Kashan, scale 1:100,000*, Tehran: Geological Survey of Iran.

- Rante, R., Collinet. A., 2013, Nishapur Revisited: Stratigraphy and Ceramics of Qohandez, Oxford: Oxbow Books.

- Razmjou, Sh., Tite, M.S., Shortland, A.J., Jung, M, Hauptmann, A., 2004, Glazed Bricks in the Achaemenid Period, In: T. Stoellner, R. Slotta and R. Vatandoust (eds.), Persien Antike Pracht, Bochum: Deutches Bergbau-Museum, 382-393.

- Rice, D.T., 1930, *Byzantine Glazed Pottery*, Oxford: Clarendon Press.

- Rodziewicz, M., 1976a, *Alexandrie I: La céramique romaine tardive d'Alexandrie*, Warsaw: Editions scientifiques de Pologne.

- _____, 1976b, Un quartier d'habitaion Graeco-Romain a Kom el Dikka, *Études et Traveaux*, **9**, 169-210.

- _____, 1978, La céramique émaillée copte de Kom el Dikka, *Études et Traveaux*, **10**, 337–345.

- _____, 1983, Egyptian glazed pottery of the eighth to ninth centuries, *Bulletin de la société d'archéologie copte*, **25**, 73–75.

- Rosen-Ayalon, M., 1972, Niveaux islamiques de la Ville Royale, *Cahiers de la Délégation Archéologique Française en Iran*, **2**, 169-201.

- _____, 1974, *La poterie islamique, Ville Royale de Suse IV*, Mémoires de la Délégation archéologique en Iran, 50, Paris: P. Geuthner.

- Rooksby, H.P., 1962, Opacifiers in opal glasses through the ages, GEC Journal of Science and Technology, **29**, 20-26.

- _____ 1964, A yellow cubic lead tin oxide opacifier inancent glasses, *Physics and Chemistry of Glasses*, **5**, 20-5.

- Sarre, F., 1925, *Die Keramik von Samarra*, Berlin: D. Reimer.

- Sedigh, M., and Karimi, A., 1965, Sofālgarī dar Lālejīn (Pottery in Lālejīn), *Majale-i Honar va Mardom*, **3**, 30, 1-7 [Persian].

- Syre, E.V. and Smith, R.W., 1961, Compositional Categories of Ancient Glass, *Science*, **133**, 1824-6.

- Scanlon, G.T., 1998, Slip-Painted Early Lead-Glazed Wares from Fustat: A Dilemma of Nomenclature, In: R.P. Gayraud, *Colloque International d'Archéologie Islamique 3-7 février 1993*, Cairo: Institute Francais d'Archéologie Orientale du Caire (IFAO), 21-53.

- Shishkina, G.V., Pavchinsakaja, L.V., 1993, *Terres Secrètes de Samarcande, Ceramiques du VIII au XIII Siècle, Toulouse: M*{Shortland:2006dm}*usée des Augustines.*

- _____, 1979, *Glazed ceramics of Sogd*, Tashkent.

- _____, 2002, *The use and origin of antimonate colorants in early Egyptian glass*, *Archaeometry*, **44**, 517-31.

- Shortland, A., Schachner, L., Freestone, I., Tite, M., 2006, Natron as a flux in early vitreous materials industry: sources, beginnings and reasons for decline, *Journal of Archaeological Science*, **33**, 521-30.

- Stacy, D.A., 1995, *The Archaeology of Early Islamic Tiberias*, unpublished PhD thesis, London: University of London.

- Vorderstrasse, T., 2005, Al-Mina: A Port of Antioch from Late Antiquity to the End of the Ottomans, Nederlands Instituut voor het Nabije Oosten.

- Tamari, V., 1995, Abbasid Blue-on-White Ware, In: J. Allan (ed.), Islamic Art in the Ashmolean Museum, Part two, Oxford: Oxford University Press.

- Tite, M.S., 1988, Inter-relationship Between Chinese and Islamic Ceramics from 9th to 16th Century A.D., Proceedings of the 26th International Archaeometry Symposium: Toronto: Archaeometry Laboratory, 30-34.

- _____, 2011, The technology of glazed Islamic ceramics using data collected by the late Alexander Kaczmarczyk, *Archaeometry*, **53**(2), 329-339.

- _____, Shortland, A., 2008, *Production Technology of Faience and Related Early Vitreous Materials*, Oxford University School of Archaeology, Monograph 72, Oxford: Oxford School of Archaeology.

- _____ Pradell, T., Shortland, A., 2008, Discovery, production and use of tin-based opacifiers in glasses, enamels and glazes from the Late Iron Age onwards: a reassessment, *Archaeometry*, **50**(1), 67-84.

- _____, Watson, O., Pradell, T., Matin, M., Molina, G., Domoney, K., Bouquillon, A., 2015, Revisiting the beginnings of tin-opacified Islamic glazes, *Journal of Archaeological Science*, **57**, 80-91.

- Vroom, J., 2004, Late Antique Pottery, Settlement and Trade in the East Mediterranean: A Preliminary Comparison of Ceramics from Limyra (Lycia) and Boeotia, In: W. Bowden, L. Lavan and C. Machado (eds.), *Recent Research on the Late Antique Countryside*, Leiden, Boston: Brill, 282-330.

- Waage, F.O., 1948, Antioch-on-the-Orontes IV, Part One: Ceramics and Islamic Coins, Princeton: Princeton University Press.

- Walmsley, A.G., 1995, Tradition, Innovation and Imitation in the Material Culture of Islamic Jordan: The First Four Centuries, In: A. Hadid (ed.), *Studies in the History and Archaeology of Jordan*, **5**, Amman, 657-668.

- _____, 1997, Ceramics and the Social History of Early Islamic Jordan: The Example of Pella (Tabaqat Fahl), *AL-‘USUR AL-WUSTA, THE BULLETIN OF MIDDLE EAST MEDIEVALISTS*, **1**,9, 1-3 & 12.

- Watson, O., 1999, VIII. Report on the Glazed Ceramics, In: P.A. Miglus (ed.), *Ar-Raqqa I: Die Frühislamische Keramik von Tell Aswad*, Mainz, 81-87, Taf. 94-99.

- _____, 2004, *Ceramics from Islamic Lands*, London: Thames and Hudson.

- _____, 2014, Revisiting Samarra: the rise of Islamic glazed pottery, In: J. Gonnella, R. Abdellatif and S. Struth (eds.), *Beiträge zur Islamischen Kunst und Archäologie,* **4**, 125-144.

- Whitcomb, D., 1989, Glazed Ceramics from the Excavations at Aqaba, Jordan, *Journal of the American Research Centre in Egypt*, **26**, 167-182.

- _____, 1991, Glazed Ceramics of the Abbasid Period From the Aqaba Excavations, *Transactions of the Oriental Ceramic Society (1990-91)*, **55**, 43-65.

- _____, 1999, Hadir Qinnasrin, The *Oriental Institute of the University of Chicago, 1998-1999 Annual Report*, 76-83.

- Whitehouse, D.M., 1978, The origins of Italian maiolica, *Archaeology*, **31**, 42-9.

- _____, 1979, Islamic Glazed Pottery in Iraq and the Persian Gulf: the ninth and tenth centuries, *Annali dell'Instituto Orientale di Napoli*, **39**, 45-61.

- _____, 1980, Proto-maiolica, *Faenza*, 66, 77-89.

- Wilkinson, C.K., 1959, Kilns of Nishapur, *The Metropolitan Museum of Art Bulletin*, **17**(9), 235-240.

- _____, 1973, *Nishapur: Pottery of the Early Islamic Period*, Metropolitan Museum of Art.

- Wolf, S., Stos, S., Mason, R., Tite, M.S., 2003, *Archaeometry*, **45**(3), 405-420.

- Wood, N., Tite, M.S., Doherty, C., and Gilmore, B., 2007, A technological examination of ninth-tenth century AD Abbasid blue-and-white ware from Iraq, and its comparison with eight century AD Chinese blue-and-white *Sancai* ware, *Archaeometry*, **49**(4), 665-84.

- _____, Tite, M.S., Rosser Owen, M., 2009, Blue and white- the early years: Tang China and Abbasid Iraq compared, *Colloquies on Art and Archaeology in Asia*, **24**, Percival David Foundation of Chinese Art, London: School of Oriental and African Studies (SOAS).

- Wulff, H.E., 1966, *The traditional crafts of Persia: their development, technology and influence on eastern and western civilizations*, Cambridge, Mass. And London: M.I.T. Press.

- Wypyski, M.T., 2015, Chemical analysis of early Islamic glass from Nishapur, *Journal of Glass Studies*, **57**, 121-136.