A characterisation study of Ubaid period ceramics from As-Sabbiya, Kuwait, using a non-destructive portable X-Ray fluorescence (pXRF) spectrometer and petrographic analyses

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Kuwait National Council for Culture, Arts, and Letters; Kuwait University, Grant/ Award Number: 004426-2017; University of South Florida The Ubaid cultural phenomena (6500–4200 BC) of southern Mesopotamia has been used to characterise other archaeological sites in the Arabian Gulf region. The aim of this inquiry is to explore the nature of the interaction between the homeland of the Ubaid and the wider Gulf region. Through the use of a non-destructive portable X-ray fluorescence (pXRF) spectrometer, this study seeks to characterise and identify the chemical and mineralogical compositions of the ceramic assemblage from the Bahra 1 site of the As-Sabbiya region, Kuwait. The chemical results demonstrated that a combination of six trace elements [rubidium (Rb), strontium (Sr), yttrium (Y), zirconium (Zr), niobium (Nb) and barium (Ba)] occur significantly enough to delineate clay-based artifact groups—local red coarse from Ubaid, while the mineralogical analysis confirms the pXRF result and identifies the source of the raw materials and temper as well. Also, the comparison between the Ubaid and Bronze Age assemblage results suggests that potters used different clay resources within their own regions and/or production techniques.

KEYWORDS

Arabian Gulf, Bahra 1, Kuwait, pottery, pXRF, Ubaid period

1 | THE UBAID PHENOMENON IN THE ARABIAN GULF

The Ubaid phenomenon is regarded by archaeologists as one of the most influential cultures of the Late Neolithic period in the Arabian Gulf region. The "Ubaid" phenomenon has been defined as a chronological sequence, a culture, as well as a general sphere of influence defined by the distribution of ceramic assemblages whose origin is in southern Mesopotamia. Despite the diversity of ceramic assemblages and site features in the Arabian Gulf, a common thread is the presence of a distinctive black-and-buff, wheel-thrown pottery known as Ubaid.

Scholars have employed a general chronological sequence of cultural change using Ubaid ceramics, spanning nearly three millennia, such as: Ubaid 0 (Oueilli style, c.6500–5400 BC); Ubaid 1 (Eridu style, c.5400–4700 BC); Ubaid 2 (Hajji Muhammad style, c.4800–4500 BC); Ubaid 3 (Tell al-Ubaid style, c.5300–4700 BC); and Ubaid 4 (Late Ubaid style, c.4700–4200 BC) (Carter, 2010; Carter & Philip, 2010: 2). The term "Ubaid" has been used to characterise sites outside southern Mesopotamia that adhered to typological categories from Ubaid sites, such as Hajji Muhammad, Tell Abada, Ras al-Amiya, Eridu, Tell el-Oueili and Tell Songor (Smogorzewska, 2013: 565). Additionally, the term "Ubaid" has been applied to local styled wares from the sites within the Gulf region under the "Ubaid" period. Smogorzewska has suggested that provenance studies could clarify the character of the interaction between Mesopotamia and the Gulf region

(2013: 567). Specifically, provenance studies could assist in determining local wares produced from Ubaid-influenced artisans and actual imported Ubaid wares.

In the last two decades, the Ubaid-related sites have been the focus of a variety of archaeological missions. Over 60 Ubaid sites have been identified across the Gulf region (Smogorzewska, 2013: 555) suggesting an extensive trade network. Specifically, there are Ubaid sites outside of southern Mesopotamia that archaeologists have surveyed to explore the nature of the Ubaid presence. Sites, such as Bahra 1, H3, Dosariyah, Abu Khamis, Khursaniyah, Dalma, Ain as-Sayeh and Ain Qannas, are known for ceramic assemblages that are representative of the Ubaid and local ceramics (Smogorzewska, 2013: 555). For example, the British Archaeological mission, directed by Robert Carter, conducted excavations at an Ubaid-related site, called H3, in the As-Sabbyia region in 1998. Also, since 2009, extensive excavations in the As-Sabbyia region have been conducted by a Polish-Kuwaiti team, under the direction of Piotr Bieliński from the Polish Center of Mediterranean Archaeology, University of Warsaw. The latter two surveys have uncovered the largest Ubaid settlement in the region. Provenance studies of Ubaid pottery from these surveys have the potential to determine the nature of the interaction between the homeland of the Ubaid culture in southern Mesopotamia and the wider Gulf region.

2 | THE AS-SABBYIA REGION AND BAHRA 1 SETTLEMENT

As-Sabbiya is a coastal region of northern Kuwait. It stretches out for approximately 60 km to the east of Al-Jahra city. Its border extends to the east by the Khor As-Sabbiya, a tidal channel separating the mainland from Bubiyan Island, to the north by the Jal Az-Zor escarpment, and to the south by Kuwait Bay (Rutkowski, 2011). As-Sabbiya is a part of the natural overland route along the shores of the Arabian Gulf (see Figure. 1). The land consists of a desert plateau, covered by open sandy areas and rocky outcrops, and a flat coastal plain separated from the interior by cliffs and terraces. Bahra is one of four topographically defined zones in the As-Sabbiya region, the largest off the northernmost aspect of Kuwait Bay.

The joint Kuwaiti–British archaeological survey effort at the H3 site, under the auspices of Robert Carter, successfully conducted one of the earliest systematic excavations in the As-Sabbiya region. The survey uncovered unique finds of significant archaeological and environmental value. The finds included ceramic wares, botanicals, boat remains and boat models. Also, there were visible features denoting the manufacture of a wide range of shell jewellery, flint tools, and a uniquely painted disc depicting a sailing boat. Additionally, there were thousands of faunal remnants, including marine fish and mammalian species (Carter & Crawford, 2010).

Later, Piotr Bieliński and his Polish team would carry out fieldwork activities throughout the As-Sabbiya zones, ultimately concentrating on the Bahra 1 settlement. In 2009, the Polish mission began regular excavations at SBH 38 (Bieliński, 2011). The site is located in Bahra, approximately 8 km from the present coastline. It had been discovered during excavations of a nearby grave site conducted by the Kuwaiti-Gulf Cooperation Council expedition. At the foot of a low sandstone promontory stretching NW-SE, at a very slight, southerly slope, regular stone alignments could be seen in the sand. Numerous shells and potsherds were found in their vicinity. Some of the pottery fragments had a painted décor, easily recognisable as belonging to the Ubaid period (Ubaid 2–Ubaid 2/3 phases). The stone features, stretching at a distance of approximately 120 m, formed straight lines, suggesting orthogonal buildings (Bieliński, 2011). Within a distance of 120 m between them, SBH 38 and SBH 35 sectors are being regarded as individual sites and as parts of one larger Bahra 1 site, settled by roughly 150 people 7000 years ago.

During subsequent seasons, the Polish team uncovered architectural features that established the site as a settlement. Bieliński's team documented the presence of a large building, a courtyard, a large furnace or kiln, as well as six building units; also, a small piece of copper, alongside bead production and workshop activities were recorded (Bieliński et al., 2016). Bieliński would later discover the remnants of a shrine, or ceremonial structure, filled with ashes and debris similar to the Eridu (P. Bieliński, personal communication). The remnants of the Bahra 1 site in As-Sabbiya have demonstrated a level of social complexity and development that has characterised other Ubaid sites.

The presence of a small community settlement at the Bahra 1 site has been established by numerous architectural units uncovered at the site. A large structure at the SHB 35 sector and a large building with other features, such as courtyard (Units 14 and 6) and units have been uncovered at sector SBH 38 by the Polish expedition. Moreover, a large building and a large kiln are located in the eastern part of SBH 38 courtyard, along with a unique tubular bead workshop; it is the only place in the Arabian Gulf identified as a centre for bead manufacturing (Bieliński et al., 2016; Reiche, 2016).

Over 35,000 lithic and stone artifacts have been recovered from the Bahra 1 site. The archaeological features and remnants include specialised tools used for shell jewellery manufacture, arrowhead and scraper workshops, as well as splintered pieces and waste debitage (Bialowarczuk, 2016). Thus, this intensive contact between Mesopotamia and the north Arabian Gulf, in particular Kuwait Bay, would suggest more circulation of Ubaid-period ceramics in the Gulf.



FIGURE 1 A map of Kuwait in the head of the Arabian Gulf, showing the area of study [Colour figure can be viewed at wileyonlinelibrary. com]

3 | ARCHAEOMETRY IN THE GULF

Archaeological studies in the Arabian Gulf region have been based essentially on typological studies. Archaeologists have focused on the Ubaid period because it is the widely accepted thread that connects the various communities across the region that were a part of the ancient Arabian Gulf trade network. Previous works were descriptive excavation reports and typological studies that reported site information and quantified recoveries (i.e. Carter, 2010; Crawford, 2010; Smogorzewska, 2013, 2015, 2016). These studies focused on describing the archaeological materials and site features, such as Ubaid and local ceramics, beads and lithic productions, workshops and architectural features. Typological studies provided generalisations about cultural affiliation and chronological schematics. However, the previous 25 years have demonstrated the value of employing archaeometric techniques to archaeological remnants.

Provenance studies in the Arabian Gulf have been accomplished using petrographic thin section analysis, instrumental nuclear activation analysis (INAA), X-ray fluorescence (XRF), laser ablation inductively couple plasma mass spectrometry (LA-ICP-MS) and, recently, portable XRF (Blackman, Méry, & Wright, 1989; Grave et al., 1996; Méry et al., 2012).

Sophie Méry and her colleagues (Blackman, Méry, & Wright, 1989; Méry & Blackman, 1999; Méry et al., 2012) used petrographic thin section analyses to characterise ceramics and to identify fabric types across the larger Gulf region,

including Mesopotamia, Oman and United Arab Emirates (UAE). The results supported a large trade network, dating from the fourth and third millennium BC. Furthermore, Méry substantiated that the network included Oman and UAE, as well as Kech-Makran (modern south-west Pakistan) ceramics. The petrographic analyses confirmed the presence of Mesopotamian vessels in Eastern Arabia, implying its participation in the larger trade network that included Iran and the Indus Valley.

Stremtan et al. (2014) utilised multiple analytical methods to study the provenance of Dilmun pottery from Kuwait. Samples of raw materials from the surrounding areas of known archaeological sites were analysed by means of petrographic thin sections, non-destructive portable X-ray fluorescence (pXRF), and high precision powder X-ray diffractometer to study the bi-phase composition of ceramics (matrix and clasts). Stremtan et al. (2014) wanted to use this data to establish the recipe of raw materials used in the manufacturing process (e.g. clays and tempering materials) of Dilmun wares, and also to establish the physical parameters of the techniques of manufacturing. The results have allowed archaeologists to characterise Dilmun ceramics. For instance, the firing temperature for Dilmun wares was between 800°C and 850°C. Furthermore, the typical mineralogical composition of the matrix includes the presence of the mineral gehlenite, a partial decomposition of micas, advanced fissuring in quartz and the melting of quartz rims. Also, there are discernible textural changes, such as the developments of secondary pores and contraction voids around large clasts, later filled by melted quartz as well (Stremtan et al., 2014). Because these aspects were observed within the same sample and throughout the sample set, Stremtan et al. argued that these wares were manufactured in kilns that could sustain relatively high firing temperatures and maintain oxidising conditions.

Ashkanani (2014) utilised petrographic thin-section analysis to determine the use of clays by particular production centres and to determine if any scale of standardisation of raw materials existed. The study consisted of a set of 25 samples analysed from Dilmun sites in Kuwait and Bahrain. Based on the matrix activity (i.e. fired clay type and inclusions) and ceramic technology, the 25 samples were divided into three distinct petrographic groups: Group A consisted of Dilmun ceramic sherds; Group B consisted of Mesopotamian-tradition ceramic sherd; and Outliers consisted of non-local sherds. The results suggested that ceramic recipes can be distinguished using this technique. The wider study (Ashkanani, 2014) yielded similar results to the initial smaller inquiry (Ashkanani & Tykot, 2013) characterising the chemical composition of Dilmun pottery by the presence of specific trace elements obtained from pXRF. Ashkanani and Tykot (2013) utilised non-destructive pXRF on various types of early Bronze Age pottery (n = 75) from Failaka Island, Kuwait and Bahrain. Later, the same technique was used on a larger sample size (n = 304) from more sites in Kuwait and Bahrain, including Bronze Age sites in Iran and the Indus Valley (Ashkanani, 2014). The chemical composition of Dilmun pottery has been established to be homogeneous and was apparently made from a single source and possibly produced at a centralised location. Interestingly, the pXRF results revealed that the so-called Dilmun imitated greenish wares could be grouped chemically with the Old Babylon period assemble.

In terms of the Ubaid period, Oates et al. (1977) conducted the earliest archaeometric analysis of an Ubaid ceramic assemblage from Dosariyah, a site in the eastern province of the Kingdom of Saudi Arabia (KSA). Using neutron activation analysis, the study established that there are two distinct chemical composites of Ubaid ceramics the Ubaid Fine ware and the Red Coarse ware, known as Gulf ware.

Provenance studies have allowed archaeologists to discuss ceramic production and distribution in more meaningful ways. Chemical and mineralogical analyses have proven to be vital to ceramic studies for the Arabian Gulf region. Smogorzewska (2013: 567) suggested that provenance studies could help clarify "the character of the interaction between Mesopotamia and the Gulf region in the Ubaid period" for the Bahra 1 site.

4 | RESEARCH AIM

This study seeks to explore whether the non-destructive pXRF technique would support the typological categories suggested by previous research, mainly southern Mesopotamian and local Red Coarse ware (Table 1). Secondly, we seek to answer whether the pXRF can examine non-destructively the homogeneity of the sample surfaces and slip composition as well. Thirdly, this study aims to explore the mineralogical compositions of the Ubaid-period pottery (n = 20) from Kuwait using petrographic thin-section analysis. Additionally, results from the latter will be compared to existing pXRF-generated data for sites in the region (n = 123) to determine if there is a continuity in the provenance used for local production.

The two typological groups at the Bahra 1 site are Ubaid 2–Ubaid 2/3 ware, Hajji Muhammad styled, and Coarse Red ware. The local Coarse Red ware type is considered the earliest local-made pottery in the Arabian Gulf, and it has been recovered from an Ubaid context, suggesting the inspiration of Gulf tradition by the Ubaid ceramics (Smogorzewska, 2013). The Arabian Gulf ware is distinguished by its red-colour vessel, and less often range between greyish to light-brownish colour. Another telling feature is its soft and brittle ware with grey to carbonised or black cores due the open kiln and low firing temperature (Smogorzewska, 2013, 2016).

Sample group name	Site	Typology category	Morphology/Vessel type	Fabric/Colour	Sample (<i>n</i>)
Arabian-Local Coarse Red Group (pXRF Group A)	Sabbyia	Red Arabia Coarse-ware associ- ated Ubaid 2-Ubaid 2/3	Lugged pots, bowls	Red, light-brownish-grey colour; clay tempered w/ chaff, sand, various inclusions; fired low temp	n = 60 (pots $n = 15$ and bowls $n = 45$)
Mesopotamian Ubaid Group (pXRF Group B)	Sabbyia	Ubaid Hajji Muhammad (fine)	Goblet, carinated bowls	Well-levigate clay w/ no inclusions; 0.2–0.5 cm thick; undecorated, smooth surface	n = 19
	Sabbyia	Ubaid Hajji Muhammad style (Common)	Plates, jars, big bowls Mesopotamia Green Group A, including pale-yellow and pale- yellow-slip sherd group	Clay w/ mineral temper, voids present, painted	<i>n</i> = 91
	Sabbyia	Ubaid Hajji Muhammad Style (coarse)	Jars Mesopotamian Green Group A, including pale-brown sherd group	Wall thickness +2 cm; painted; heavy chaff temper	n = 17
pXRF Outlier Group	Sabbyia		Mixture of Ubaid pale brown, pale yellow and brown sherds		n = 6
Reference Group; Failaka Island (Kuwait)	F6	Barbar tradition Period 1 (1950 BC)	Jar	Barbar Red ridged hand-made, sand-tempered.	<i>n</i> = 15
	al-Khidr	Barbar tradition Period 2 (1850 BC)	Jar, cooking pots, flat plate	Barbar-tradition Red ridged hand-made, sand-tempered.	n = 90
	F6	Ur III period (2100 BC) (Mesopotamian House structure	Jar	Mesopotamian-tradition thick red and green hand-made jars	<i>n</i> = 18

TABLE 1 Site sample groups

A total of 316 ceramic wares used for the study representing Ubaid-period wares (Ubaid and local group) from Kuwait (n = 193) and Bronze Age ceramic sherds from Kuwait (n = 123).

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The main feature of southern Mesopotamia Ubaid wares is a greenish to buff background surface and the black to olive paint on this background surface. The painted Ubaid assemblage is distinguished by painted decoration, which was made by a technique of incision, making some common motifs featured identifiably Ubaid 2, Hajji Muhammadstyled (Carter, 2010; Smogorzewska, 2015). The motifs occur as dense oblique grids, triangle, date-pits, as well as a variety of horizontal, vertical, zigzag, wavy and diagonal lines (Smogorzewska, 2015). An oblique grid decoration which leaves a tiny square was the most popular motif on Ubaid pottery at the Bahra 1 site (Figure. 4, t). According to Smogorzewska's (2015) study on Ubaid pottery at Bahra 1 site, the Ubaid pottery follows three main technological groups: (1) fine ware is characterised by well-fired clay with no visible inclusions on thin-walled surfaces; (2) the common ware pot is the most common Ubaid technological group in which mineral temper is presented; and (3) coarse ware is characterised by heavy chaff temper.

5 | METHODS OF ANALYSIS

5.1 | Portable X-ray fluorescence spectrometer analysis

Samples were from the Bahra 1 settlement within the As-Sabbyia region of Kuwait (Figure. 1), specifically from the two sectors, known as SBH 35 and SBH 38. After cleaning of the potsherds by a brush and water to avoid any contamination of dust and soil residues, the elemental composition of the surfaces was analysed non-destructively using a Bruker Tracer III-SD portable X-ray fluorescence spectrometer.

Portable XRF instrumentation has its inherent limitations related to both sample (e.g., moisture content, matrix type, size of the particulate components, heterogeneity, surface conditions, sample geometry; see the discussion below) and method (e.g., the type and resolution of the detector, excitation source, matrix effects, fluorescent yields of the elements to be measured, etc.). Furthermore, the limits of detection and limits of quantification tend to be instrument-dependent, while the accuracy and precision of the measurements are element-related. Our analytical approach was designed to minimise those limitations, both by choosing the samples which seemed more appropriate (matrix and surface wise) and by selecting the analytical parameters that would allow us to measure relevant chemical elements. The elements that are abundant in the paste (e.g., Ba, Sr-mostly hosted in the clay minerals), or characteristic to tempering material (e.g., Zr, Y, Nb) have been used for discriminating compositional groups.

The instrument was set up with a filter (12 mm Al, 1 mm Ti, 6 mm Cu) designed to enhance data measurements of

mid-Z elements in the spectrum, and to reduce continuum radiation. In order to maximise the accuracy and precision of trace elemental analysis, 40 kV and 11 μ A were used. The instrument was set up to provide quantitative results for trace elements including, rubidium (Rb), strontium (Sr), yttrium (Y), zirconium (Zr), niobium (Nb) and barium (Ba), which have been shown in many studies to be successful in determining groups and sub-groups.

Generally, the elements commonly used for sourcing studies based on instrumental neutron activation analysis (INAA) are caesium (Cs), barium (Ba), lanthanum (La), hafnium (Hf), tantalum (Ta), cerium (Ce), praseodymium (Pr), neodymium (Nd) and other rare earth elements. Though INAA and other chemical analyses have produced results for the latter trace elements, the trace elements in this study have been found to successfully distinguish different groups and sub-groups in the Gulf region, thus allowing us to address questions about local production versus imports (Ashkanani & Tykot, 2013; Tykot, 2016, 2018). The value of using the pXRF is that the device can distinguish between groups using these trace elements.

For this research, the 2008 University of Missouri Research Reactor (MURR) calibration, which was originally designed by Robert Speakman and Michael Glascock for obsidian and other silicate materials, was conducted on the raw data obtained from pXRF at the Laboratory for Archaeological Science at the University of South Florida. In 2006, Speakman and Glascock began using empirical calibration based on obsidian references to calibrate an ElvaX XRF and further developed it for the Bruker pXRF, along with an obsidian "green" filter, to be used since 2008 (Speakman & Shackley, 2013). Thus, the data obtained by pXRF in this study is valid for the purpose of the current study and may be re-calibrated in the future with other software for comparison with other studies.

The matrix effect in pXRF is expected when dealing with the calibrated values. It requires standards of similar major element composition to produce accurate values, which Bruker has not developed for analysis of different archaeological materials. An obsidian calibration created by MURR is therefore used for ceramics that are silica-based. Each sample was set on the top of the exit window for 120 seconds to obtain elemental composition in parts per million (ppm) concentrations.¹ The ceramic fragments tested are approximately 1-3 cm, and completely cover the beam size of this instrument, which is about 3×5 mm in diameter. Because there is some concern about analysing pottery that does not have a smooth, flat surface and thus affecting the actual X-ray angle, the sherd's spot of X-ray exposure has been carefully selected, avoiding a non-flat area and visual temper inclusions. This method was utilised on the Arabian Gulf ancient pottery

¹The data values are available from the authors on request.

to overcome the non-homogenised samples and to ensure the consistency of the whole sample (Ashkanani & Tykot, 2013).

The inner and outer surfaces of the 193 samples were analysed and the edge as well for thick samples to ensure that the results are consistent. Multiple points in a single sample were taken to ensure "representativeness" and homogeneity. The analysis was designed to run multiple pXRF points similar to Chayes' (1956) modal analysis for thin section analysis in which shots are pointed along the length and width of the potsherd and then one can calculate the average percentage of different elements. It can also be analysed by a traditional method that measures the percentage of variant runs along a set of lines, as followed in the Bronze Age ceramic studies (Ashkanani, 2014). However, following the Chayes method or a set of lines presents some difficulty in this study due to the sensitivity of the pXRF to a non-flat surface. Thus, it is a challenge to set a line on each surface avoiding any apparent inclusion or décor. Due to this limitation and in order to avoid measuring large single mineral inclusions, surfaces of painted ceramic potsherds were excluded. Moreover, cooking ceramic potsherds that are diagnostic pieces with a blackcarbonised inner and/or outer surface were also excluded.

As many Ubaid ceramic potsherds are painted with black lines and due to the small size of the sherds, some outer surfaces are excluded from the analysis to obtain only the elements that represent the clay either through some unpainted outer surface or through inner and edges of thicker samples (Figure. 2). The data obtained from pXRF were subject to statistical analysis. Multivariate statistic applications using "SPSS statistics 25" software was employed to find which elements can be informative in differentiating ceramic groups and showing variations in chemical composition between samples even if they are visually distinguishable. Principal component analysis (PCA) was employed as an exploratory method to examine the correlation between chemical elements, to suggest which variables or groups of elements are meaningful, and to account for the maximum variance in the data set. The SPSS component matrix was useful because it contains the loading of each variable onto each factor.

5.2 | Petrographic thin-section analysis

A set of 20 thin section slides were prepared for examination. After smoothing and mounting the sample on the glass slide, all observations were recorded including visible inclusions, main minerals, secondary minerals, grain shape, mineral features and alteration, and textural descriptions as well. The samples represent Ubaid buff/green and brown group (n = 10) and Arabian coarse red group (n = 10). All the samples were examined under a polarising microscope under 10×, 20×, and 100× magnification, depending on the visibility of inclusions and homogeneity of the sample paste.



FIGURE 2 A biplot of Rb vs. Zr for ceramics data collected using pXRF for multiple shoots within each sample. The result confirms the outer surface of 29161 is not consistent with edge and inner plain surfaces. The inner and outer surfaces of 29185 are not consisted with the edge due to the paint

5.3 | Multivariate statistical analyses

The results showed that only two component factors could explain the variance, 70.6%. Also, it showed how Nb contributed highly (0.810) in the first component but was lower in the second component (0.327). Rb and Zr showed their high contribution in the loading variable onto first and second factor components.

The principal component analysis was followed by a cluster analysis using PCA scores, which included all six trace elements (Nb, Sr, Y, Zr, Rb and Ba), for identifying natural groupings and evaluating PCA results. K-means cluster analysis was utilised for the clustering method based on presuming three clusters to group all samples and then finding clusters. The ANOVA output showed that PCA factor 1 and 2 scores were statistically significant (p < 0.05), which confirmed the PCA scatterplot in Figure 3.

Discriminant function analysis (DFA) was employed as a different statistical technique to discriminate between groups and classify the samples into different production centres. For DFA, the average of the groups was 3 as it was supposed to represent three production centres that the samples might have been made in: Mesopotamia, As-Sabbyia and an unknown area for an outlier group.

Original data were used for all six trace elements (Nb, Sr, Y, Zr, Rb and Ba) as variables and colour type names as a grouping variable. DFA produces a matrix of function, based on the presumed groups in order to show the contribution of each variable in the DFA. The result showed that three discriminant functions were obtained. The canonical discriminant function showed that the ceramic sherds are separated into two main groups, with a third being potentially differentiated. The Wilk's output showed that the two discriminant functions are statistically significant (p < 0.05).



FIGURE 3 A biplot derived from PCA of 193 sherd samples measured by pXRF, showing variation within the Bahra 1 site in As-Sabbyia. At least two distinct groups of ceramics, local Red Coarse wares (Group A) and Mesopotamian tradition (Group B), known as Ubaid fine and standards wares. A possible third group identified as outliers

6 | RESULTS

6.1 | pXRF result

The results of this study are consistent with the typological categories designated by earlier archaeological studies (Smogorzewska, 2013, 2015). There are two main distinguishable chemical groups, one from southern Mesopotamia and a local group. Additionally, there are marginal sherds that appeared to be local, a sub-group of the imports.

A scatterplot was performed using PCA factor scores 1 and 2 that included Nb, Sr, Y, Zr, Rb and Ba, which previously showed their high contribution in the component matrix. The results show two distinct groups of ceramics and a few outlier samples (Figure. 3). Group A consists of almost all red ceramic sherds associated with the local red ware, while Group B consists of all buff and green ceramic sherds associated with Mesopotamian tradition in addition to brown and pale sherds (Figure. 4). The chemical composition of Group A is characterised by high Rb (average of 69 ppm, max. of 93 ppm) and Zr (average of 207 ppm, max. of 357 ppm), as well as an overall noticeable spread of the data (Figure. 2). Group B, on the other hand, is characterised by overall lower and less variable concentrations (e.g., Rb: average of 35 ppm, max. of 63 ppm; Zr: average of 124 ppm, max. of 151 ppm). Statistically, other than local red sherds, none overlapped and clustered in Group A. Within Group B, a total of six brown sherds overlapped with this group (Figure. 4, g, h, i, k and l). The main feature of these wares is the chaff-tempered surfaces. Also, a total of four of the six pale-brown sherds overlapped with Group B

(Figure. 4, m, o and r). A total of 14 pale-yellow and paleyellow slip sherds overlapped with Group B, as representing Mesopotamian clay source (Figure. 4, n, p and q). There is a total of six of the 193 ceramic sherds that are not clustered to either Group A or B, making themselves as an outlier group (Figure. 4, a–e). They represent the Ubaid brown, pale-brown, pale-yellow and pale-yellow slip wares.

Additionally, to determine if any of the Bahra 1 site wares could be linked chemically to other sites in the region, the pXRF data was compared to pXRF data from Bronze Age ceramics from sites on Failaka Island, Kuwait (n = 123). The Bronze Age reference ceramic groups were subjected to the same analytical methods, settings and calibration procedure (Ashkanani, 2014). The reference group consisted of samples from several Dilmun sites, such as: 15 ceramic sherds from F6 site, known as the Governor's Palace site (c.1950 BC); 90 ceramic sherds from al-Khidr site, serving as Dilmun port (c.1950–1800 BC); and 18 ceramic sherds from the Mesopotamian House structure, located within the F6 site (Ur-III Dynasty period, 2100 BC).

The PCA was carried out and the results show four compositional groups, one sub-group, and a few outliers (Figure. 5). Interestingly, the results confirmed the difference between local coarse red ware and local Dilmun ceramic potsherds. One of the two pale-yellow slip sherds that were clustered closely with the Group A (see Figure. 3), is now grouped with al-Khidr site ceramic sherds, confirming the local provenance for its clay. However, the other pale-yellow slip and brown sherd are now clustered as a sub-group of

FIGURE 4 A variety of ceramic sherds overlapped with the main two groups (Mesopotamian Group A, and Gulf/local Group B). Sherds labelled (a–e) represent outlier group; sherd (f) represents creamy-colour in Gulf/local Group A; sherds labelled (g–r) represent cream-colour, brownish and reddish colour in Mesopotamia Group B; and the sherd labelled (s) represents the Gulf/local tradition of the late Neolithic period, while the sherd labelled (t) represents the Mesopotamian tradition of Ubaid culture in the late Neolithic period





FIGURE 5 A biplot derived from PCA of 316 sherd samples measured by pXRF, showing variation within the Gulf ceramic group and the Mesopotamian-origin groups. A total of four compositional groups are identified, representing Ubaid ware from Kuwait (green, pale brown, pale yellow, pale-yellow slip and brown types) and Ur-III dynasty sherds from the Mesopotamian House structure in Failaka Island (F6 MH site type), as well as Ubaid-period local Red Coarse ware and Bronze Age Dilmun potsherds (al-Khidr and F6 sites type). The identical separation between assemblages suggests a difference of clay resources utilised and/or the ancient technique used

Group B, after including Bronze Age ceramic sherds. Only one of the 90 Bronze Age al-Khidr sherds overlaps with local red coarse ware of Group A. It is a typical "Barbar tradition" jar sherd used in the Dilmun sites during the Bronze Age. The firing temperature and/or clay source could be the reason for the overlap with the red coarse ware group.

Also, the results show a separation between all Ubaid ceramic sherds of Group B (i.e., pale-yellow, pale-brown, pale-yellow slip, green/buff) and the Bronze Age Ur-III Dynasty assemblages. In a previous study, Ashkanani (2014) noted that the analytical results implied that the green chaff-tempered sherds from the al-Khidr site matched the Ur-III Dynasty group (see Figure. 5). Comparing Ubaid Group B wares, it appears that different clay resources were used by the Mesopotamian potters; or different production centres that made pottery differed in technique in terms of temper preparation and firing control. Interestingly, a total five of Ubaid-period outliers in Figure 3 are now clustered as a sub-group of Ubaid wares, called sub-group B.1 (see Figure. 4, a-d and Figure. 5), while the other sherd has overlapped chemically with Group B (see Figure. 4, e). The so-called outlier sherds can be confirmed as Mesopotamian in origin through performing pXRF and combining them with other samples from other sites, maximising statistically the difference between ceramic compositions.

6.2 | Petrographic result

The results of the petrographic analyses also determined that there is a mineralogical distinction between the two typological groups (Table 2). The petrographic data also supports the geochemical pXRF data, in which the higher contributions of mafic (ferromagnesian) rocks can discriminate between the two groups. Ba and Sr ratios may reflect an association with calcium, common in carbonate rock fragments (limestone) and carbonate matrix detected in most of the studied samples; other trace elements reflect their association with clay particles.

According to petrographic components of the studied samples, it seems that the source rocks included sedimentary, igneous and metamorphic rocks from a variety of sources. The local red ware of Group A are differentiated by more rounded and polycrystalline quartz with subordinate plant remains, limestone, rare volcanic rock fragments, muscovite and olivine, than those of Group B, which may refer to mafic and ultramafic igneous rocks (mainly volcanic) and metamorphic sources.

Specifically, Group A, representing local red ware, included ten samples of red to pale brown silty and sandy clay which are commonly porous (Table 2, Nos. 1–10). Petrographically, this group is heterogenous in composition and is characterised by common rounded sand-sized quartz grains with polycrystalline quartz, silt-sized subangular quartz grains, alkali and plagioclase feldspar with traces of chert, plant remains, limestone and rare volcanic rock fragments; secondary carbonate and secondary gypsum are also present. Muscovite, biotite, olivine, pyroxene and amphiboles are the main accessory heavy minerals present in the studied samples of this group. This group could be further divided into sub-groups according to the occasional presence or absence of specific mineral grains, such as the absence of **TABLE 2** Thin-section micrographs of local red coarse ware (Group A) and Ubaid (Group B) specimens, in plane polarised light (PPL) and crossed Nichols (XPL)



TABLE 2 (Continued)



TABLE 2 (Continued)

No.	PPL photo Petrographic group	XPL photo	Sample photo
15			
	Group B: Ubaid ware		
16	Group B: Ubaid ware		
17			
18	Group B: Ubaid ware Group B: Ubaid ware		
19	Group B: Ubaid ware		
20			
	Group B: Ubaid ware		Setta State 1

A total of 20 ceramic sherds selected for petrographic thin-section analysis, showing the two main mineralogical groups (Group A of Gulf/Sabbyia vs. Group B of Mesopotamian clay), with a further division into sub-groups with the Gulf group.

olivine and secondary gypsum, and the presence of chert and marlstone (Table 2, Nos. 7–10).

Group B, representing Ubaid ware, included ten samples of green silty clay samples finer than those in the previous group (see Table 2, Nos. 11–20). Accordingly, this group is more homogeneous in its mineralogical constitution than the previous group. It is composed of common chert, subangular silt-sized quartz grains, both alkali and palgioclase feldspar distributed in carbonate-rich matrix. Biotite and pyroxene are dominant heavy mineral inclusions in the studied samples.

Metamorphic and igneous rock debris including mafic volcanic rocks is the accepted provenance for the common polycrystalline quartz, olivine and volcanic rock fragments, as has been noted in the Dibdibba formation (late Miocene-Pleistocene), northern Kuwait (Milton, 1967). Group A had dominated-rounded quartz grains, polycrystalline quartz and lithic fragments, including igneous, vein quartz and volcanic glass fragments. The heterogenity of the composition may be from the recycling of materials. Additionally, the long distance transportation of raw materials from older geological formations, such as Oligocene Ghar formation, was common in southern Iraq and included such a suite of minerals (Al-Juboury et al., 2010). In Kuwait, the Ghar formation forms the basal part of the Kuwait Group which includes the Lower Fars and Dibdibba formations (Al-Awadi et al., 1997) and forms one of the surface formations in Kuwait. The presence of volcanic inclusions in the local pottery was noted in three Bronze Age ceramic sherds from the al-Khidr site, Failaka Island (Ashkanani, 2014: 228). Also, the Bronze Age assemblage had a similar heterogenous nature.

In comparison, these petrographic results have common mineralogical components to Ubaid pottery from the Dosariya site, Saudi Arabia (Oates et al., 1977) such as, quartz, chert, feldspars, olivine, pyroxene, micas (biotite and muscovite) and amphiboles. Scholars have reported that Ubaid Pottery analysed from Arabian Gulf countries (i.e Saudi Arabia, Qatar, and Bahrain) was made in Mesopotamia because the petrographic suite was detected in Ur and Eridu of the southern Mesopotamian region. Silty clay sediments rich in quartz, feldspar and chert rock fragments with biotite and pyroxenes common in Group B are similar to several sources in the Mesopotamian area, such as those found in marsh soils from southern Iraq (Al-Juboury & Al-Juburi, 2002); to sandy sediments from the Mesopotamian floodplain of southern Iraq (Garzanti et al., 2016); and to the clastic sediments of the late Miocene-Pleistocene Dibdibba formation of northern Kuwait (Table 2) and southern Iraq (Al-Sulaimi & Pitty, 1995; Awadh & Al-Ankaz, 2016; Khalaf, Gharib, & Al Kadi, 1982). The latter mineralogical features and depositions can be attributed to the fluviatile environment that covered a wide area of the Arabian Gulf, particularly Saudi Arabia, Kuwait, Qatar and Iraq.

7 | DISCUSSION AND CONCLUSION

The results showed the success of pXRF for examining the homogeneity of ceramic potsherds and supports the categories designated by earlier typological studies. The pXRF instrument was able to make a quantitative distinction between the wares and to identify two distinct chemical groups (A and B) within the Bahra 1 site. Importantly, this device allowed the samples to be tested on multiple points per surface of each sherd without sample loss. The multivariate analyses also confirmed that there are two distinct groups (A and B) and an Ubaid sub-group, a mixture of brown, pale-brown and pale-yellow wares. Group A consisted of local ware, also known also as Arabian coarse ware (see Figure. 4, s), and was distinguished from Group B, greenish to buff coloured, Ubaid 2, Hajji Muhammad-styled (see Figure. 4, t).

Additionally, the results from the comparison with data from Failaka Island suggests that the use of raw materials and/or the technique of pottery production was not as static as it appeared. Through the millennia, potters were altering their wares within southern Mesopotamia as well as among Kuwait, or Gulf potters. When Failaka Island wares were compared to the Bahra 1 groups, there was a distinction between the local Coarse Red ware and the local Dilmun ware. Interestingly, Ubaid-period outliers were clustered with Ubaid wares. Furthermore, the outliers seem to suggest that even Mesopotamian potters did not use the same clay sources or had followed different production techniques. Also, the results of the pXRF analyses show a consistency between slip outer surface and inner surface, suggesting a single clay type was used for the Arabian coarse ware and the Ubaid slip ware. The inconsistency of the painted surfaces justified our exclusion of taking readings from these surfaces for this study. In future, studies will be done using alternative instrumental settings and filters to determine major elements (e.g. Fe, Ca, Mn) as well as trace elements to delineate the clay composition from paint composition.

The differences between Mesopotamian and Gulf wares suggests that the chemical and mineralogical composition of wares may be related to socio-cultural factors, in addition to the technological aspects, related to ceramic production. Petrographic and pXRF results suggest that there was a consistent selection and preparation of raw materials that lent to the final pottery production for Ubaid wares. The homogeneity of the Ubaid wares could reflect that the scale of production involved more than a small class of specialists in the Gulf during the late Neolithic period. Stein (1999) suggested that the homogeneity of material preparation, the firing temperature and the clay paste was due to the control over raw materials and production efficacy by a possible centralised or administrative authority. On the other hand, the presence of heterogenous wares, like the Arabian coarse red ware, would suggest either less control by a centralised authority on local ceramic production; or a small-scale household-level industry; or individual artisans that devoted part of their time to ceramic production.

In the future, heterogeneity versus homogeneity of wares could be used to establish the presence or absence of an administrative authority and the scale of ceramic production from small household-level to the more complex large-scale level over ceramic production. This aspect can be explored in the future by including more samples from the Ubaid-period sites of the Gulf. Also, chemical outliers could be used to explore shifts in ceramic production, such as recipe changes, a change in the availability of raw materials, and the possibility of competing production centres. Oates et al. (1977) wrote that Eridu and Uruk were examples of production centres that co-existed as original Mesopotamian production centres for Ubaid pottery. Also, samples that are outliers could be used to explore consumption patterns. The nature of material consumption includes human preferences, craft specialisation and the availability of goods. According to Carter's model (2010: 203–212), the increased demand for more products can foster more social complexity.

The demand for Ubaid-period products could have supported an increase in social stratification outside as well as inside southern Mesopotamia. This study has shown that Ubaid wares and cultural materials existed alongside local productions. It is highly probable that the control of the availability of local prestigious goods, such as a boat figurine from the H3 site, and Hajji Muhammad pottery, may have been done by craft specialists or an emerging social class that is associated with distribution of ceramics. These inferences would support the notion that an increase in social complexity accompanies material consumption and a sedentary lifestyle.

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