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ARCHAEOMETRIC INVESTIGATION OF MIDDLE BRONZE AGE CERAMICS FROM THE MIDDLE EUPHRATES REGION

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ABSTRACT

Archaeometric analyses of ceramics yield critical insights into ancient societies, elucidating production technologies, raw material sourcing, and firing conditions. The proliferation of such studies allows for both the internal characterization of assemblages and comparative analyses with contemporary finds. This study presents a comprehensive archaeometric characterization of Middle Bronze Age comb-decorated and grooved-rim ceramics from Tilbaşar Mound (Gaziantep). To determine raw material composition and production technologies, the samples were analyzed using X-Ray Diffraction (XRD), optical microscopy (thin section), and Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM/EDX). Results indicate that the ceramics were produced using calcareous clay sources. When evaluated alongside the region's geological formation, the data suggest a local provenance for the production. Mineralogical phases identified via XRD indicate a firing temperature range of 700–950°C. Petrographic analysis reveals that the ceramic paste contains inclusions derived from basalt, siltstone, sandstone, and marl. Furthermore, SEM imaging demonstrates a generally low degree of vitrification, with only initial vitrification stages observed in select samples.

Keywords: Tilbaşar Mound, Middle Bronze Age, ceramic archaeometry, characterization

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Introduction

Archaeometry is widely used for various purposes in last decades. It serves as a multi-discipline for archaeology and conservation-restoration. The analytical data achieved through the archaeometric studies cover the chemical, mineralogical and/or structural features of the materials such as ceramic, glass, metal, plaster, mortar etc. Such information could be directive in terms of evaluating the origin of the materials, and the production technologies, as well. Use of various techniques together in archaeometry has occurred as a result of need in different perspectives in examination of the findings in detail. For instance, in ceramic archaeometry, the samples need to be exposed to both chemical and mineralogical analyses, and in some cases (e.g. glazed samples) comprehensive microscopic investigation also becomes compulsory to observe the micro structure. Colored and/or glazed samples need to be studied through advanced techniques such as electron microscopes, micro-Raman spectroscopy and/or X-ray fluorescence spectroscopy. The choice mainly depends on the aim and scope of the investigation. If the determination of the colorants in the glaze is solely sufficient, energy dispersive X-ray spectrometer would be efficient. But, if the interaction among the glaze, slip and body (e.g. thickness of the layers, grain size-shape-distribution) needs to be enlightened, then the sample should be also investigated through an electron microscope which would bring the opportunity to see the micro structure in micron (µm) levels (Issi et al., 2011; Mandrykina et al., 2024; Mangone et al., 2009).

Covering a comprehensive ceramic archaeometry work, the present paper focuses on characterization of the Middle Bronze Age comb decorated-grooved rim ceramic finds from Tilbaşar (Gaziantep, Turkey - middle Euphrates region) by means of multiple analytical techniques. The main goal of the study is to reveal the raw material types and production technologies of the ceramics. For that purpose, different techniques were applied; XRD (X-ray diffraction), thin section (optical microscope), SEM/EDX (scanning electron microscope/energy dispersive x-ray spectroscopy), FTIR (Fourier transform infrared) spectroscopy and TG-DTA (thermogravimetry-differential thermal analysis). Among these methods, FTIR and TG-DTA could be thought as the complementary techniques which provide extra information regarding mainly the presence of secondary calcite (subsequently occurs during the burial time), organic matters and/or the hygroscopic/physical water etc. (Fabbri et al., 2014; Drebushchak et al., 2005; Ravisankar et al., 2011). XRD and petrography were used to reveal the mineralogical contents, while SEM-EDX was applied to observe the micro structural features and chemical composition of the ceramics.

Materials and Methods

Ceramic Samples

As the number of finds assigned to the comb decorated-grooved rim ceramic group was high, representative samples (Fig 1) were selected by Assoc. Prof. Dr. Elif Genç (Çukurova University, Türkiye), academic advisor of the excavation, taking into account their form, color and archaeological descriptions. The sample types were edge, rim and shoulder possessing the hues of red, brown and buff. Before the preparation of the powder and bulk samples, the colors of the ceramics were revealed through the colorimetric analysis which was carried out by a portable colorimeter using ColorQA Pro System III and CEI (Commission Internationale de L'Eclairage) L-a-b color system. The descriptions of the representative samples and the results of the colorimetric analysis are given in Table 1. For the purification of the ceramic fabrics, the samples were soaked in distilled water and not subjected to acid. After the soaking process, the impurities were cautiously removed from the samples' surface and ceramics were ground in a porcelain mortar after drying in order to obtain powder samples to be used in XRD. The bulk samples were also prepared for SEM-EDX and petrography.

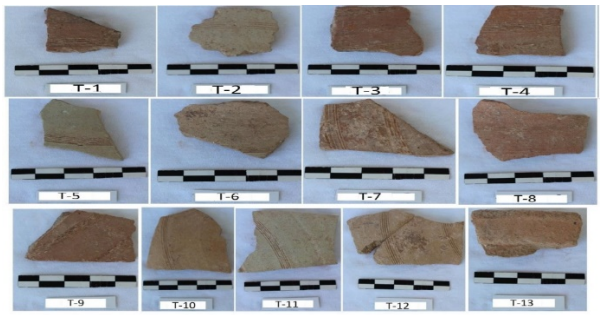

Figure 1. The images of the studied ceramic samples

Table 1. Descriptions of the ceramic samples

Code	Archaeological code	Type	Thickness (mm, min-max)	Grain size
T1	TH15.AŞD.BI 990.12-2692	Edge	8-12	Medium
T2	TH15.AŞD.BI-BJ 990.0-55	Rim	5-6	Coarse
T3	TH15.AŞD.BI 990.12-2683	Rim	3,5-5,5	Medium
T4	TH15.AŞD.BI 990.13-3050	Rim	6,5-11,5	Fine
T5	TH15.AŞD.BI 990.13-3061	Rim	6-7,5	Fine
T6	TH15.AŞD.BI 990.12-3155	Shoulder	3,5-5	Medium
T7	TH15.AŞD.BI 990.12-3106	Rim	7-9,5	Fine
T8	TH15.AŞD.BI 990.12-1899	Shoulder	05,5-10	Fine
T9	TH15.AŞD.BI-BJ 990.1272	Rim	05,5-10	Medium
T10	TH15.AŞD.BJ 990.12-3149	Rim	05,5-10	Fine
T11	TH15.AŞD.BI 990.13-2864	Rim	05,5-10	Fine
T12	TH15.AŞD.BJ 990.13-1455	Rim	05,5-10	Medium
T13	TH15.AŞD.BI-BJ 990.0-1008	Edge	05,5-10	Coarse

Applied Methods

In order to monitor the paste features of the ceramics, a DMLP model, LEICA Research Polarizan microscopy was employed for the petrographic examination. A digital camera of Leica DFC-280 with the magnification value of x25 and single/double nicol was used. The data were evaluated by the Leica Qwin digital imaging. The mineral and rock types of the ceramics were revealed by Point Counting method. The mineral and phase identification was carried out through a Panalytical-Empyrean XRD device working with Cu-Kα radiation. The scanning range was 5-70º 2-theta and the Goniometry rate was 2º/min.

A Supra 40VP model, Carl Zeiss SEM-EDX was used for the observation of the ceramic matrices with high magnifications. The SEM images were taken on the surfaces after the platinum coating process which was employed through a Q150R ES model Qourum coating device. This procedure was compulsory for the ceramics since they are not conductive, which hinders to achieve high resolution images at micron levels. The chemical compositions of the ceramics were revealed by EDX. The results were evaluated in oxide forms of the elements, as conventionally used in the available literature.

Results

Mineralogical Content (XRD and Petrography)

XRD and petrography results are given in Table 2. Quartz and feldspar/plagioclase were identified in all ceramic findings. Hematite detected in most samples indicated that these ceramics were exposed to an oxidizing atmosphere and therefore the firing process was carried out in an environment that allowed the diffusion of oxygen (Issi, 2012). It was determined that the firing temperature of the ceramics, in which calcite was clearly detected and high temperature phases were not observed, was in the range of 700-800ºC (Shoval, 2003; Broekmans et al., 2004; Fabbri et al., 2014). It has been predicted that calcite (in trace or very low amounts) observed together with the high temperature phases (e.g. pyroxene and/or melilite group minerals) is most likely secondary, and could be assumed as an impurity in the ceramic body occurring due to the burial conditions (Shoval et al., 1993; Fabbri et al., 2014). Firing temperature of ceramics vary depending on whether the calcite is primary or secondary, therefore the presence of secondary calcite indicates that the firing temperature range is not lower than 800-900ºC if high temperature minerals are present (Fabbri et al., 2014).

Different methods can be used in archaeometry to detect secondary calcite. Essentially, the characterization technique by which calcite is detected is the XRD method. When calcite shows characteristic peaks in the XRD pattern, this content is considered as primary calcite. One of the important points here is the formation of high temperature minerals. In the absence of high temperature minerals such as gehlenite, wollastonite, pyroxene and anorthite, the determination of calcite as the major mineral keeps the firing temperature in the range of 700-800ºC, and as the carbonated materials (calcite, dolomite) begin to decompose, the neo-formations start to form and give peaks on the XRD pattern, which indicates to higher firing temperatures. Another important group is clay minerals. The clay minerals lose their OH^- group at approximately 700ºC and structurally decompose at around 900ºC. Clay

minerals detected in some samples showed that these ceramics were exposed to a maximum temperature of 900ºC (Fabbri et al., 2014; Cultrone et al., 2001; Shoval, 2003; Broekmans et al., 2004).

Table 2. XRD and petrography results of the ceramics.

Code	Mineral[a]	EFT[b]	Rock/Mineral[c]	P[c]	MTA[c]	Description[c]
T1	Q,C,F/Pl,Cm,G*,Pr*	750-850	Q,Pl,Pr,B (Rc in structural voids)	7	18	Medium-sized aggregates of basalt rock origin.
T2	Q,C,F/Pl,Cm,H,G,Pr	800-900	Q,Pl,Pr,B (Rc in structural voids)	6	20	Medium-sized aggregates of siltstone, sandstone and marl rock origin.
T3	Q,C,F/Pl,Cm,H	700-800	Q,Pl,Pr,B (Rc in structural voids)	6	20	Medium-sized aggregates of siltstone, sandstone and marl rock origin.
T4	Q,C,F/Pl,Cm,H,G	800-850	Q,Pl,Pr,B,Ol	7	10	Fine-sized aggregates of basalt rock origin.
T5	Q,F/Pl,P,An	900-950	Q,Pl,Pr,B,Op	4	15	Fine-sized aggregates of sandstone rock origin.
T6	Q,C,F/Pl,Cm,H,G,Pr	800-900	Q,Pl,Pr,B (Rc in structural voids)	6	20	Medium-sized aggregates of siltstone, sandstone and marl rock origin.
T7	Q,C,F/Pl,Cm,H,G*,Pr*	750-850	Q,Pl,Pr,B (Rc in structural voids)	7	18	Medium-sized aggregates of basalt rock origin.
T8	Q,C,F/Pl,Cm,H	700-800	Q,Pl,B,Op (Rc in structural voids)	12	32	Medium-sized aggregates of basalt rock origin.
T9	Q,C,F/Pl,Cm,H	700-800	Q,Pl,Pr,B,Op	4	15	Fine-sized aggregates of sandstone rock origin.
T10	Q,C,F/Pl,Cm,H,G,Pr	800-900	Q,Pl,Pr,B,Ol	7	10	Fine-sized aggregates of basalt rock origin.
T11	Q,C*,F/Pl,H,G,Pr	900-950	Q,Pl,Pr,B (Rc in structural voids)	6	20	Medium-sized aggregates of siltstone, sandstone and marl rock origin.
T12	Q,C,F/Pl,Cm,H,G*,Pr*	750-850	Q,Pl,Pr,B (Rc in structural voids)	6	20	Medium-sized aggregates of siltstone, sandstone and marl rock origin.
T13	Q,C,F/Pl,H,G,Pr	800-900	Q,Pl,Pr,B,Op	5	28	Coarse-sized aggregates of ophiolitic basalt rock origin.

[a]Detected by XRD; Q: Quartz, C: Calcite, F/Pl: Feldspar/Plagioclase, Cm: Clay mineral, H: Hematite, G: Gehlenite, P: Pyroxene, An: Anorthite. *: Indicates to trace/very low amounts.
[b]Specified by XRD results; EFT: Estimated Firing Temperature (ºC).
[c]Identified by Petrography; Q: Quartz, Pl: Plagioclase, Pr: Pyroxene, B: Basalt, Rc: Recrystallized Calcite, Op: Opaque minerals, Ol: Oligoclase / P: Porosity (vol.%), MTA: Matrix Total Aggregate (vol.%).

According to XRD data, the firing temperature range of the ceramics was estimated between 700-950ºC. It was deduced that the samples in which high temperature phases were seen at low intensity and primary calcite was partially present were exposed to 800-900ºC, and the samples where new phases (pyroxene, gehlenite, etc.) were dominant and calcite was secondary (due to its low abundance, mostly in trace amounts) or not detected were exposed to 900-950ºC. In addition to pyroxene determined as the major phase in the sample T-5 within the sample set, the presence of anorthite also indicated that this ceramic was fired at relatively high temperature. Different firing temperature ranges suggested that ceramics would have been exposed to various firing conditions which depend mainly on the parameters of the firing technology (e.g. heating rate, maximum temperature, soaking time, atmosphere, location of the ware during firing etc.) (Cultrone et al., 2001; Bayazit et al., 2014).

Considering the petrography results (Table 2), the ceramics could be evaluated in six groups according to the minerals, rock origins, porosity and the matrix total aggregate. It was observed that the samples occasionally possess fine, coarse and/or medium-sized aggregates and the minerals were siltstone, sandstone, marl and basalt rock origin. While quartz and plagioclase were detected in all samples, other minerals seen in different samples were opaque minerals, oligoclase and pyroxene. Recrystallized calcite (in structural voids) was detected in some

of the ceramic fabrics. The porosity and matrix-total aggregate ratios in the ceramics were determined to be in the range of 4-12 vol.% and 10-32 vol.%, respectively.

It was observed from the microphotographs of the ceramics (Fig 2) that the samples generally possess fine and/or medium-sized aggregates. Some ceramics contain residues that could be assigned as grog which could be dried clay or ground ceramic/brick added to strengthen the ceramic body (Rice, 1987). It was predicted that the possible secondary calcite detected in the XRD analysis could have formed due to the burial conditions. The re-crystallized calcite detected in the structural voids of the ceramic samples confirmed this prediction. The different colors of the paste observed in the microphotographs of the ceramics suggested a heterogeneous raw material mixture, while also indicating an irregular firing process. Another important parameter that would most likely affect the red and black tones, which may occur due to the redox reactions of the iron mineral, is the organic matter content. While bonfiring or pit firing techniques were frequently used in the production of traditional ceramics in prehistoric times, with the advent of ceramic kiln technology, the changes in physical properties of the ceramics, in particular, could be controlled. Considering the microphotographs in the present study, the inhomogeneous distribution in the paste structure and the wide color scale in the ceramic matrices suggested an irregular and/or inattentive firing technique (Emami et al., 2009; Bong et al., 2008).

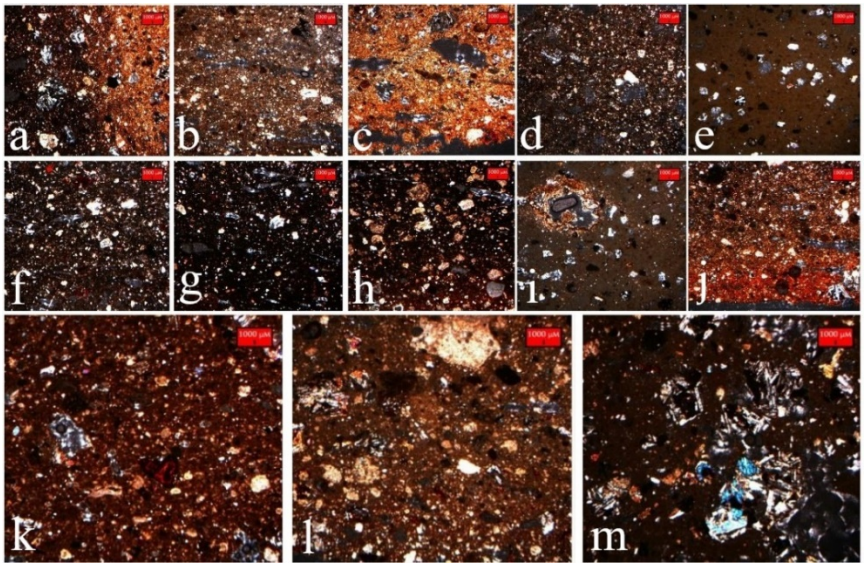

Figure 2. Microphotographs of the ceramics (a-m: T1-T13)

Micro Structural and Micro Chemical Features (SEM-EDX)

The chemical composition of the ceramics pointed out high amounts of CaO (Table 3) indicating that calcareous raw materials were used in the production. Thus, the presence of calcite detected as a mineralogical content through XRD was also confirmed by EDX analysis. FeO is detected as a natural oxide that gives color to the ceramics. This revealed that the color formation in the ceramics was mostly related to the firing atmosphere. The presence of feldspar/plagioclase, quartz and clay minerals was revealed by the alkali-earth alkali oxides, SiO_2 and Al_2O_3 contents detected in the EDX spectra. The performance of EDX analysis in selected areas on the SEM images caused some oxide contents to be much higher than expected. While the fact that the amount of CaO in some samples is very high (e.g. OT-2; CaO amount is 55.69 wt.%) indicates that CaO is high in this sample, on the other hand, it should not be ignored that there may be a fluctuation in the EDX data as a result of the heterogeneous distribution of the minerals containing this oxide in the ceramic matrix.

In detailed SEM examinations of the samples (Fig 3), the formation of glassy phase was rarely observed in most of the findings. In some samples, partial vitrification behavior was seen (e.g. ET-4, ET-6, ET-8, OT-6) suggesting that the ceramics were not exposed to high temperatures, not higher than 1000°C. SEM images of some samples showed that the clay structure was preserved indicating that the clay minerals did not decompose and the firing temperature of such samples did not exceed 900°C, which is the decomposition temperature of the clay. The vitrification behavior, which can be graded as initial, continuous and extended, provides information about the firing temperature. The limited observation of sintering behavior in ceramic bodies, which occurs under the sufficient temperature and appropriate firing environments, and the resulting poor vitrification degree confirmed

the firing temperature ranges determined for the ceramics (Cultrone et al., 2001; Emami et al., 2009; Bong et al., 2008).

Table 3. Chemical composition of the ceramics identified by EDX (wt.%).

Code	SiO_2	CaO	Al_2O_3	FeO	MgO	K_2O	Na_2O
T1	60,4	5,85	17,19	11,97	0,69	3,9	-
T2	28,2	55,69	7,05	6,57	1,25	-	1,24
T3	54,25	17,24	17,9	5,68	1,74	3,21	-
T4	47,57	16,67	15,69	12,53	5,89	0,77	0,88
T5	49,93	30,26	12,42	-	4,75	2,63	-
T6	44,26	29,93	13,07	10,26	1,62	0,86	-
T7	53,17	21,34	14,4	6,71	2,9	2,24	-
T8	45,13	28,11	14,15	8,2	3,01	0,81	0,6
T9	53,72	14,36	17,84	9,84	2,8	1,43	-
T10	38,73	41,17	11,72	6,91	1,08	0,38	-
T11	46,39	21,89	13,5	11,71	5,83	0,68	-
T12	35,91	46,01	10,28	5,72	2,08	-	-
T13	45,79	30,07	14,66	5,77	2,67	1,03	-
Mean	46,41	27,58	13,83	7,83	2,79	1,38	0,20
Minimum	28,2	5,85	7,05	0	0,69	0	0
Maximum	60,4	55,69	17,9	12,53	5,89	3,9	1,24

-: not detected or under the detection limits.

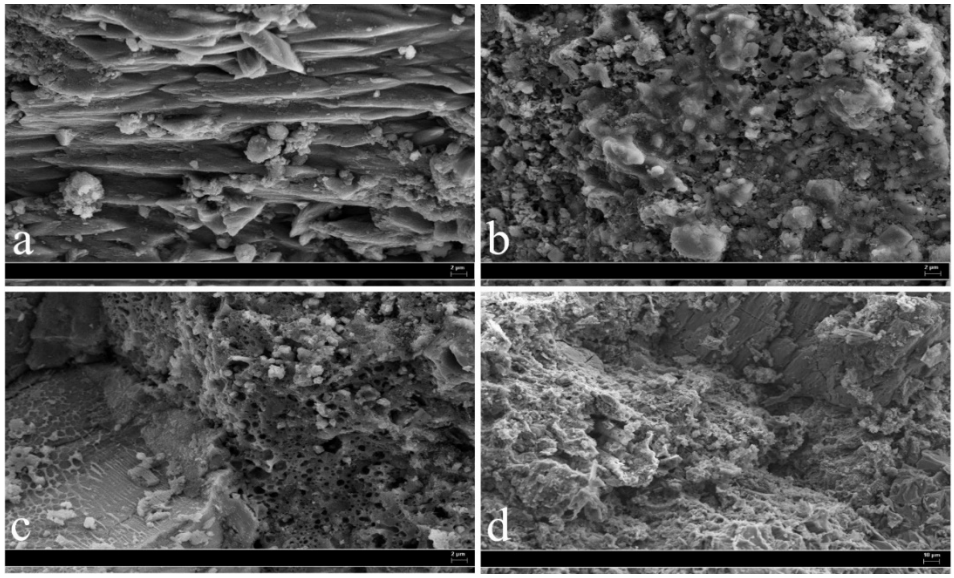

Figure 3. Electron microscope images of the samples (a) T2 (partially preserved clay structure), (b) T5 (distribution of the glassy phase), (c) T6 (regional vitrification, (d) T9 (no evident traces of vitrification)

Conclusions

The mineralogical and chemical content of the samples indicated that ceramics were produced from raw material sources containing calcareous clay. The intensive presence of carbonated raw materials (limestone, calcite, marble, etc.) in the vicinity of the mound pointed out use of local raw materials (MTA 2018 a-g). Therefore, it could be predicted that the ceramics were likely to be locally produced. As far as the firing conditions are concerned, the firing temperature of the samples in which high temperature phases were not detected was in the range of 700-800°C, the samples in which high temperature phases were seen with low intensity and primary calcite was partially present were in the range of 800-900°C, and finally the samples in which high temperature phases (pyroxene, gehlenite, anorthite, etc.) were dominant and calcite was secondary (low intensity) or absent

were fired in the range of 900-950°C. Hematite found in most of the samples indicated that such ceramics were fired in an oxidizing environment or that oxygen was present in the final stage of the firing process. According to the petrographic data, it was seen that the ceramics were divided into six groups, and ceramics possess fine, coarse and/or medium-sized aggregates of siltstone, sandstone, marl and basalt rock origin. These results may suggest use of local raw materials but from different sources.

The electron microscope images revealed weak vitrification in most of the findings. In some of the samples where the glassy phase was more evident or relatively extended than others, the occurrence of vitrification was local in the matrix. This suggested that the ceramics should have not been exposed to high temperatures such as 1000°C. Similarly, in samples where the clay structure was preserved, it was determined that the firing temperature of such samples did not exceed 900°C (the decomposition temperature of the clay minerals). The overall results in the present study suggested a diversity in production of the Middle Bronze Age comb decorated and grooved rim ceramic finds from Tilbaşar Mound.

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ORTA FIRAT BÖLGESİ'NDE ELE GEÇEN ORTA TUNÇ ÇAĞI SERAMİKLERİNİN ARKEOMETRİK İNCELENMESİ

Murat Bayazit
Didem Çağine

ÖZ

Seramik buluntular için yapılan arkeometrik incelemeler seramiklerin ait olduğu toplumlar hakkında yol gösterici bilgiler sağlamaktadır. Özellikle hammadde içerikleri ve pişirim şartları gibi üretim teknolojisine ışık tutan bilgilere analitik analiz teknikleri ile ulaşmak mümkündür. Son dönemde sayıca artmaya başlayan seramik arkeometrisi çalışmaları sayesinde buluntuların hem kendi içlerinde hem de diğer çağdaş seramiklerle karşılaştırmalı olarak ele alınması mümkün olmaktadır. Seramik örneklerin mineralojik ihtivaları ve kimyasal kompozisyonları hammadde hakkında bilgi verirken ayrıca provenans çalışmalarına da katkı sunmaktadır. Bu bilgiler ışığında, mevcut çalışmada Tilbaşar Höyük (Gaziantep) Orta Tunç Çağı tarak bezemeli ağzı yivli seramik buluntuları için kapsamlı bir arkeometrik karakterizasyon gerçekleştirilmiştir. Seramiklerin hammadde içerikleri ve üretim teknolojilerini ortaya çıkarabilmek amacıyla ileri analiz tekniklerinden faydalanılmıştır. Çalışmada XRD (X-ışını difraksiyon), ince kesit (optik mikroskop) ve SEM/EDX (taramalı elektron mikroskobu/enerji saçınımlı X-ışını spektroskopisi) verileri üzerinden değerlendirmeler yapılmıştır. Bu teknikler seramik buluntular için kullanılan başlıca metotlardan bazılarıdır. Elde edilen arkeometrik veriler örnek grubunun kalkerli kil içeren hammadde kaynaklarından üretildiğine işaret etmektedir. Bölgenin jeolojik formasyonuna bakıldığında, incelenen seramiklerin yerel üretim olma olasılığının yüksek olduğu düşünülebilir. Seramiklerin bünyesinde tanımlanan mineral ve faz içerikleri örnek seti için pişirim sıcaklık aralığının 700-950°C olduğunu göstermiştir. Petrografik analiz sonuçları seramiklerdeki minerallerin bazalt, silt taşı, kum taşı ve marn kökenli olduğuna işaret etmiştir. Seramiklerin SEM görüntülerinde genel olarak zayıf vitrifikasyon saptanırken, bazı numunelerde kısmi olarak bölgesel vitrifikasyon davranışı olduğu gözlemlenmiştir.

Anahtar Kelimeler: Tilbaşar Höyük, Orta Tunç Çağı, seramik arkeometrisi, karakterizasyon