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*Araştırma Makalesi / Research Article*

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## **Multi-analytical Investigation of Early Bronze Age Cooking Pots with Triangular Handles (Tilbaşar, Turkey)**

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### **Abstract**

Archaeometric studies covering the investigation of archaeological findings by analytical techniques are in a great demand particularly in last decades. This type of works represents a convenient and directive data basis in terms of achieving substantial knowledge regarding the characteristic ceramics of different civilizations. Being parallel to this, it was aimed to investigate the cooking pots with triangular handles (Early Bronze Age) from Tilbaşar Höyük (Gaziantep, Turkey) in an archaeometric way using multi-analytical methods within the present study. For that purpose, 14 representative samples of cooking pot (with triangular handles) were characterized by means of scanning electron microscopy together with energy dispersive X-ray spectrometry (SEM-EDS), petrography (optical microscopy), Fourier transformed infrared (FTIR) spectroscopy, thermogravimetric-differential thermal analysis (TG-DTA) and X-ray diffraction techniques. It was intended to identify the raw material contents and production features (e.g. firing temperature range, micro structural/chemical characteristics) of the ceramics. Considering the whole results, it was seen that the evident presence of primary calcite, and also the absence (or very limited degree) of vitrification in the micro structure suggested a firing temperature range of 700-800°C for the potsherds. Additionally, determination of the carbonates through the multi analytical techniques implied that the ceramics should have been produced by calcareous clays. In this context, it was predicted that the potsherds likely belong to a local and/or regional production (considering the geological formations of the region).

**Keywords:** Archaeometry, materials characterization, cooking pot with triangular handles.

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## **Tilbaşar Höyük Erken Tunç Çağı Üçgen Kulplu Mutfak Kaplarının Çoklu Analitik Yöntemlerle İncelenmesi**

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### **Öz**

Arkeolojik buluntuların analitik tekniklerle incelendiği arkeometrik çalışmalar özellikle son yıllarda rağbet gören bir bilim alanıdır. Seramik buluntular için yapılan bu tip çalışmalar farklı medeniyetlere ait karakteristik seramikler hakkında önemli bilgilere ulaşma noktasında oldukça faydalı ve yol gösterici veriler sunmaktadır. Buna paralel olarak, bu çalışmada Gaziantep Tilbaşar Höyük'te ele geçen bazı üçgen kulplu mutfak kaplarının (Erken Tunç Çağı) çoklu analitik yöntemlerle arkeometrik olarak incelenmesi amaçlanmıştır. Çalışmada 14 adet üçgen kulplu mutfak kabı arkeometride sıklıkla tercih edilen yöntemler arasında yer alan taramalı elektron mikroskobu/enerji saçınımlı X-ray spektroskopisi (SEM/EDS), optik mikroskop (OM), Fourier dönüşümlü kızılötesi (FTIR) spektroskopisi, termogravimetrik-diferansiyel termal analiz (TG-DTA) ve X-ışını difraksiyon (XRD) teknikleri ile karakterize edilmiştir. Kullanılan bu tekniklerle seramiklerin hammadde içerikleri ve üretim özelliklerinin (pişirim sıcaklık aralığı, mikro yapısal/kimyasal karakter vb.) belirlenmesi hedeflenmiştir. Elde edilen sonuçlar göz önüne alındığında, seramiklerde belirgin biçimde tespit edilen birincil kalsit varlığı ve buna ek olarak mikro yapıda vitrifikasyonun görülmemesi veya ihmal edilebilir derecede olmasının örneklerin 700-800°C gibi bir sıcaklık aralığında pişirildiklerine işaret ettiği görülmüştür. Buna ek olarak, karbonatlı hammaddelerin belirlenmesi seramiklerin kalkerli kil ile üretildiklerini akla getirmektedir. Bu bağlamda, bölge jeolojisi dikkate alındığında örneklerin yerel veya bölgesel üretim olma olasılıklarının da yüksek olduğu öngörülmüştür.

**Anahtar kelimeler:** Arkeometri, malzeme karakterizasyonu, üçgen kulplu mutfak kabı.

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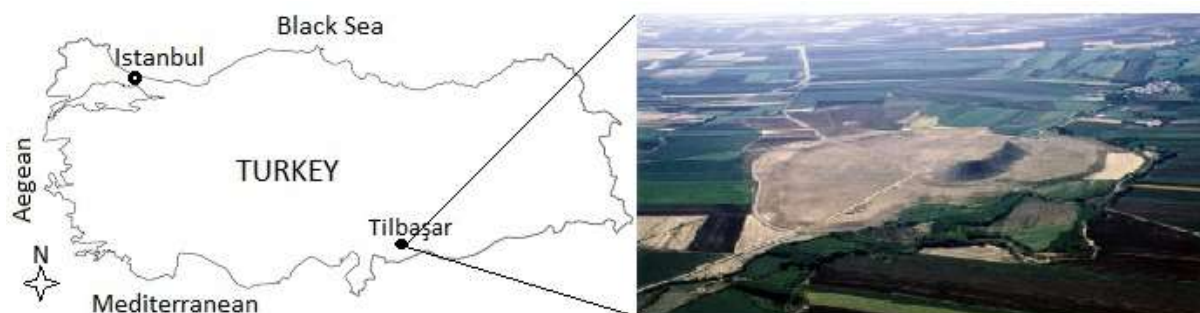
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## 1. Introduction

Archaeometry could be defined as a multi-disciplinary branch of the science using various analytical characterization methods including mostly the spectroscopic and microscopic techniques. One can enlighten the production properties of the historic materials using archaeometric methods and provide a numeric data to be evaluated within the archaeological knowledge. In this context, ceramic findings, which are one of the most abundant artifacts in archaeological areas, can be characterized by means of various analysis methods but mainly the ones revealing the chemical and/or mineralogical contents, for they are the earthen wares consisting of oxides and minerals. As traditional ceramics are also fired at relatively high temperatures (i.e. up to 900-1000°C or above which might change depending on the raw materials and production features), there can be neo-formations together with a change in terms of micro structure. So, this is why the mentioned spectroscopic and microscopic techniques are frequently preferred in ceramic archaeometry. Additionally, thermal analyses are performed for pottery, namely thermogravimetric analysis (TGA) simultaneously utilized with differential thermal analysis (DTA) which would bring a complementary information according to the mass loss and enthalpy changes, respectively [1, 2].

Proceeding from this point of view, and in addition to the previous archaeometric works regarding another pottery groups (tripod vessels [3], ring burnished pottery [4]), this paper focuses on archaeometric investigation of a different pottery group; cooking pots with triangular handles (Early Bronze Age) uncovered from Tilbaşar Höyük which is located at 1 km east of Gündoğan village, 12 km of Oğuzeli district, 32 km southeast of modern Gaziantep in the north of Turkish-Syrian border (Figure 1) [5]. The most striking features of the cooking pots are known as use of the additive minerals (avoiding the occurrence of cracks due to rapid heating), carbonates (acting as a heat trap so as to prevent the ware from the abrupt temperature changes during daily use) and organic temper materials (the gaps occurring after the decarbonation of organics tolerate the thermal shock stresses throughout the pottery) [6-8]. Likewise, the samples in the present study were examined in order to reveal the characteristics of the cooking pots (with triangular handles) of Tilbaşar through a comprehensive archaeometric investigation. As is known, unfortunately, the number and scope of archaeometric studies are insufficient, and should be improved. For that purpose, this study aims to provide a directive and convenient archaeometric data for the cooking pot with triangular handles samples of Tilbaşar which is believed to make a substantial contribution to the current literature, because the number of detailed archaeometric research on pottery groups of the mound is very limited. Consequently, the samples of cooking pot with triangular handles from the mound were characterized by means of scanning electron microscopy together with energy dispersive X-ray spectrometry (SEM-EDS), petrography, X-ray diffraction (XRD), thermogravimetric-differential thermal analysis (TG-DTA) and Fourier transformed infrared (FTIR) spectroscopy in order to enlighten the production features (e.g. raw materials, firing properties, textural characteristics etc.).



**Figure 1.** Location and panoramic view of Tilbaşar (shot by Nezhir Başgelen) (~36°52'31.6"N 37°33'34.0"E) [5].

## 2. Materials and Methods

The first step in the study was determination of the potsherds. The specimens were chosen by Assist. Prof. Dr. Elif Genç (Çukurova University, head of the excavation area) taking their representativeness into account. At this point the physical and textural features of the potsherds were the main decisive parameters. Consequently, 14 samples representing the cooking pot with triangular handles pottery

group of the mound were selected to be characterized. The second step was visual documentation by which the potsherds have been photographed (Figure 2) and defined (i.e. color, thickness) (Table 1). The potsherds were then soaked in distilled water so as to remove the contaminations. After these fundamental processes before the analyses, the samples were ground in a porcelain mortar and powder samples were prepared to be used in destructive methods (i.e. XRD, TG-DTA, FTIR spectrometry). Additionally, bulk samples were taken for each sample to be used in microscopic analyses (i.e. SEM-EDS, petrography).



**Figure 2.** Representative samples of the study

**Table 1.** Definition of the samples\*

Sample code	Type	Thickness (mm)			
		(min-max)	L	a	b
KW-1	Edge	0.65-1.87	38,3225	7,9337	15,2225
KW-2	Body	0.63-0.73	32,9445	6,5489	19,4508
KW-3	Edge	0.83-2.60	28,4919	4,7072	15,1966
KW-4	Edge	1.25-1.90	36,7927	11,4257	21,1605
KW-5	Edge	0.84-1.40	37,4943	13,7946	24,5800
KW-6	Edge	0.80-1.65	27,1332	6,4086	15,2989
KW-7	Edge	1.00-1.60	28,7149	7,1667	18,7377
KW-8	Edge	0.80-1.66	27,1247	9,6540	17,9482
KW-9	Edge	1.28-1.63	21,7045	8,2941	14,4447
KW-10	Edge	0.83-1.90	30,0417	10,4840	20,0077
KW-11	Edge	0.50-1.20	26,2590	6,4828	15,3884
KW-12	Edge	0.61-1.06	24,5453	5,0014	15,5852
KW-13	Edge	0.80-2.06	19,6755	6,2156	9,5366
KW-14	Edge	0.70-1.60	15,5462	6,8433	8,5807

\* L: 0/100; white/black. a: 0/-60; green and 0/+60; red. b: 0/-60; blue and 0/+60; yellow

A portable colorimeter was employed to determine the colors of the potsherds. ColorQA Pro System III was used to obtain the L, a, b values which were assessed through the Commission Internationale de L'Eclairage. The mineralogical contents of the samples were determined with a Rigaku Miniflex XRD device with Cu K $\alpha$  radiation. The goniometer speed was 2°/min and the scanning range was 5–70° 2 $\theta$ . The chemical composition of the samples was revealed by a Supra 40VP model Carl Zeiss SEM/EDS. Elemental composition was transformed to oxide concentration. The data were obtained on various scaled images of the bulk samples which were initially coated with platinum in the coating appliance of Qourum (Q150R ES model) in order to examine the micro structural properties of the potsherds. A Leica Research polarizing microscopy (DMLP model) was utilized for the petrographic investigation. A Leica DFC280 digital camera possessing single/double nicol was used to shoot the images of the samples (through the Leica Qwin imaging program) and the results were assessed by the

Point Counting method. FTIR analysis was performed with an Elmer (Spectrum Two model) FTIR spectrometry. The analysis was carried out in the range of 450-2000  $\text{cm}^{-1}$  (the finger-print region). The enthalpy changes together with the weight losses were determined using a TG-DTA device of Shimadzu (DTG-60H model). Thermal analysis was performed in the temperature range of 25-1100°C with a heating rate of 20 °C/min.

### 3. Results and Discussion

#### 3.1. XRD and petrography results

The mineral/phase contents and mineral/rock types of the samples were examined through XRD and petrography analyses, respectively (Table 2). XRD results showed that all the samples include calcite (as the major phase) and quartz. Clay minerals (illite/muscovite) were also detected for the samples except the sample KW-7 which was found to possess only calcite and quartz. It was thought that this sample likely had a very few amount of clay minerals resulting in slight intensity (cps), but not seen. Representative XRD patterns are given in Figure 3 in order to demonstrate the intensities of the minerals detected. The evident presence of calcite (primary), which decomposes around 750°C and the reaction could continue up to 900°C in case of high amounts and/or coarse grains of calcite [9, 10], indicated that the samples should have been exposed to a firing temperature lower than the decomposition temperature of calcite. This assumption was also supported by the existence of clay minerals in the samples except KW-7, since such minerals decompose about 900°C [9, 11]. Consequently, the firing temperature range of the potsherds was found as 700-800°C.

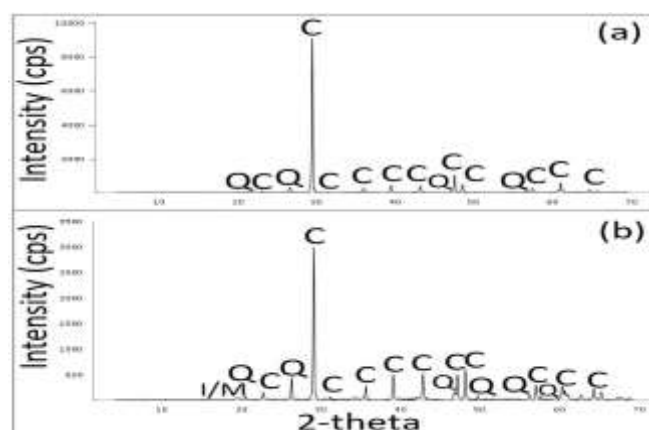
**Table 2.** XRD and petrography results of the samples

Group No*	TA (vol. %)	Mineral/rock types**	Descriptions	Mineral/phase***	Estimated Firing Temp (°C)
Gr-1	45	Q, C, L, M, G (%1)	The paste with coarse aggregates which mostly originated from marble.	C, Q, I/M	700-800
Gr-2	30	Q, C, L, Ch, Op	The paste with coarse aggregates which mostly originated from limestone.	C, Q, I/M	700-800

Fine / Medium / Coarse Aggregate (mm): <0,5 / 0,5-1,0 / >1,0; TA: Total Aggregate in ceramic matrix.

\* Gr-1: KW-1, KW-3, KW-4, KW-5, KW-6, KW-7 (excluding I/M, please see Figure 3), KW-8, KW-9, KW-10, KW-11, KW-12, KW-13, KW-14; Gr-2: KW-2, \*\* Detected by petrography; C: Calcite ( $\text{CaCO}_3$ ), Ch: Chert ( $\text{SiO}_2$ ), L: Limestone ( $\text{CaCO}_3$ ), M: Marble ( $\text{CaCO}_3$ ), Op: Opaque minerals (i.e.  $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ ) Q: Quartz ( $\text{SiO}_2$ ), G: Grog, \*\*\* Detected by XRD; C: Calcite ( $\text{CaCO}_3$ ), I/M: Illite/muscovite (clay minerals), Q: Quartz ( $\text{SiO}_2$ ).

It was seen from the petrography results that the samples mainly consisted of calcite and quartz, likewise revealed by XRD analysis. Limestone was found in all samples, while marble was detected in most of the samples except KW-2 in which chert and opaque minerals were also found. The results exhibited that most of the potsherds' paste consists of coarse aggregates originated mainly from marble (for 13 samples), and only KW-2 comprises limestone origin aggregates (Table 2). Total aggregate ratio in ceramic matrices was found relatively high (30-45 vol. %). Additionally, grog residuals were also detected in most of the samples and its existence was assigned to probable use of temper materials (production ejects or clay lumps) employed in order to strengthen the structure of the pottery [12-14]. Petrographic results were seen to be compatible with the general characteristics of the cooking pottery groups described in the available literature.



**Figure 3.** XRD pattern of the sample (a) KW-7, (b) KW-10 (C: calcite, Q: quartz, I/M: illite/muscovite)

### 3.2. FTIR results

FTIR results were found to be compatible with the mineralogical contents of the potsherds (Table 3) Being detected as the major mineral in the whole sample set, calcite was identified with its characteristic band values at  $1794\text{-}1797\text{ cm}^{-1}$ ,  $1416\text{-}1419\text{ cm}^{-1}$ ,  $1426\text{-}1428\text{ cm}^{-1}$ ,  $872\text{ cm}^{-1}$ ,  $873\text{ cm}^{-1}$ ,  $847\text{-}849\text{ cm}^{-1}$  and  $712\text{ cm}^{-1}$  [15-18]. Determination of the diagnostic FTIR bands of calcite around  $1420\text{ cm}^{-1}$  was attributed to primary calcite [19, 20], and this result well-matched with the mineral/phase contents of the samples revealed through XRD. The clay minerals illite and muscovite were determined at the band values of  $1023\text{-}1025\text{ cm}^{-1}$ ,  $1028\text{ cm}^{-1}$  and also with the bands  $1015\text{-}1019\text{ cm}^{-1}$  at which kaolinite was the other possible mineral [18, 21, 22]. The bands around  $455\text{-}460\text{ cm}^{-1}$  indicated the probable existence of chlorite and illite, while the bands at  $465\text{ cm}^{-1}$  and  $474\text{ cm}^{-1}$  were assigned to illite/chlorite/kaolinite and hematite/illite/kaolinite, respectively [18, 22, 23]. Considering the XRD results, in which the clay minerals were found as illite/muscovite, it was predicted that the bands of the clay minerals in FTIR analysis were mainly related with these two, rather than chlorite or kaolinite. Quartz was detected with its characteristic band values around  $694\text{ cm}^{-1}$ ,  $777\text{ cm}^{-1}$  and  $797\text{ cm}^{-1}$  [18]. Hematite, which is thought to be the main mineral giving the colors of the samples, was detected for some of the potsherds with the bands around  $474\text{ cm}^{-1}$ ,  $520\text{ cm}^{-1}$ ,  $533\text{ cm}^{-1}$  and  $555\text{ cm}^{-1}$  [18, 24]. The band value of  $563\text{ cm}^{-1}$  was attributed to both hematite (giving the red-brown colors in oxidizing atmosphere) and magnetite (bringing the gray-black colors in reducing atmosphere), whereas  $555\text{ cm}^{-1}$  was assigned to hematite and clay mineral (muscovite) [2, 18, 24, 25]. Representative FTIR spectra are given in Figure 4.

**Table 3.** FTIR results of the samples

Sample code	Evident band values ( $\text{cm}^{-1}$ )	Related minerals*
KW-1	1795/1418/1023/873/848/797/777/712/563/520/474	Ca/Ca,I,Ms/Ca/Ca/Q/Q/Ca/H,M/H/H,I,K
KW-2	1796/1426/1024/873/848/799/780/712/695/555/533/455	Ca/Ca,I,Ms/ Ca/Ca/ Q/Q/Ca/Q/ H,Ms/H/Q,I,Cl
KW-3	1795/1418/1023/873/847/798/780/712/694/557/533/459	Ca/Ca,I,Ms/ Ca/Ca/ Q/Q/Ca/Q/ H,Ms/H/I,Cl
KW-4	1797/1417/1025/873/848/798/712/695/520/465	Ca/Ca,I,Ms/Ca/Ca/Q/Ca/Q/H/I,Cl,K
KW-5	1794/1427/1024/873/848/798/778/712/696/460	Ca/Ca,I,Ms/Ca/Ca/Q/Q/Ca/Q/I,Cl
KW-6	1796/1416/1019/872/848/798/778/712/695/465/455	Ca/Ca,I,Ms,K/ Ca/Ca/Q/Q/Ca/Q/I,Cl,K/ Q,I,Cl
KW-7	1796/1416/1015/873/849/797/778/712/694	Ca/Ca,I,Ms,K/ Ca/Ca/Q/Q/Ca/Q
KW-8	1796/1417/1022/873/848/797/779/712/695/455	Ca/Ca,I,Ms/ Ca/Ca/Q/Q/Ca/Q/Q,I,Cl
KW-9	1795/1416/1017/872/848/798/778/712/696/454	Ca/Ca,I,Ms,K/ Ca/Ca/Q/Q/Ca/Q/Q,I,Cl
KW-10	1795/1416/1017/872/848/798/778/712/695/454	Ca/Ca,I,Ms,K/ Ca/Ca/Q/Q/Ca/Q/Q,I,Cl
KW-11	1796/1428/1028/873/848/798/712	Ca/Ca,I,Ms/Ca/Ca/Q/Ca
KW-12	1794/1417/1018/873/848/798/778/712/695/455	Ca/Ca,I,Ms,K/ Ca/Ca/Q/Q/Ca/Q/Q,I,Cl
KW-13	1794/1419/1018/873/848/798/777/712/693/455	Ca/Ca,I,Ms,K/ Ca/Ca/Q/Q/Ca/Q/Q,I,Cl
KW-14	1795/1417/1016/872/848/798/779/712/695	Ca/Ca,I,Ms,K/ Ca/Ca/Q/Q/Ca/Q

\*Ca: calcite, Cl: chlorite, H: hematite, I: illite, K: kaolinite, M: magnetite, Ms: muscovite, Q: quartz

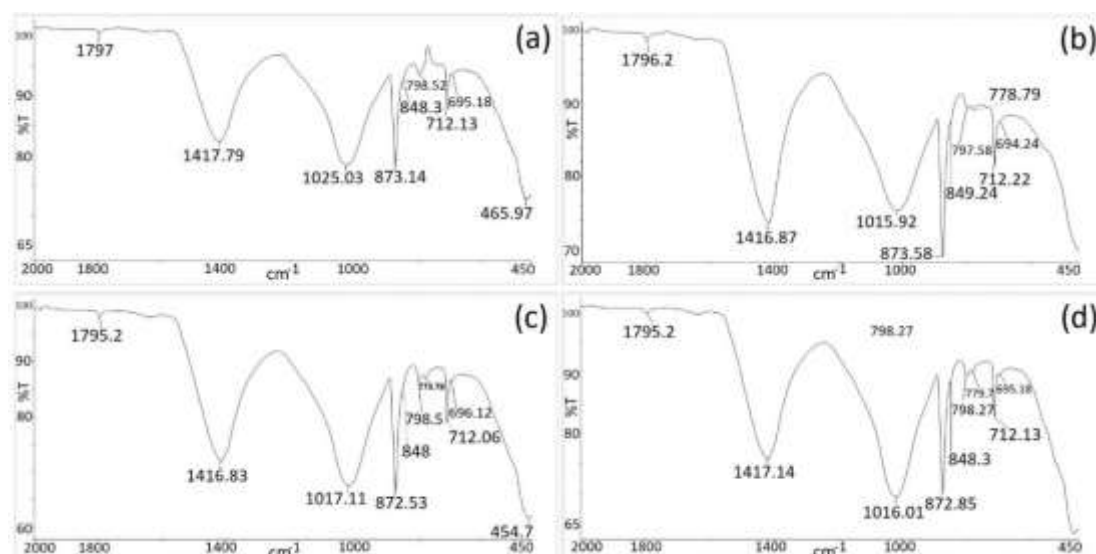


Figure 4. FTIR spectrum of the sample (a) KW-4, (b) KW-7, (c) KW-9, (d) KW-14

### 3.3. TG-DTA results

TG-DTA analysis is a convenient technique which allows examining the enthalpy changes together with the weight loss values during a gradual increase in temperature [26]. The TG curves show the mass loss, while DTA curves indicate the enthalpy changes occurred in the sample in the course of heating process [27]. The results (Table 4) showed that the weight loss values change between 27.84 and 31.37 wt. % for the sample set, and the majority of such losses have emerged owing to the presence of carbonates (i.e. calcite) which decompose at about 700-850°C resulting in an endothermic peak on DTA curves (representative TG-DTA diagrams are given in Figure 5) [26, 27]. The evident existence of such peaks around 800°C on DTA diagrams proved that calcite was present in the samples and its decomposition reaction did not happen. This results supported the firing temperature ranges (700-800°C) revealed by XRD. Additionally, the appearance of characteristic endothermic effects indicating the decomposition of carbonates around 800°C (and not lower than that temperature) showed that calcite was primary rather than secondary which is accepted as a burial residual and/or reformed calcite [20].

The endothermic peaks on DTA curves at 25-200°C together with the gradual weight loss were attributed to the hygroscopic water [27]. The absence of any evident endothermic or exothermic effects on DTA curves of the samples after 900-1000°C suggested that the polymorphic transformations were likely limited, and this result indicated that the firing temperature was lower than that range [28]. These data, likewise the FTIR results, verified the firing temperature range of the samples.

Table 4. Weight loss values detected in the range of 25-1100 °C

Code	Initial weight (mg)	Total weight loss (mg)	Total weight loss (wt. %)
KW-1	18,08	5,48	29,95
KW-2	18,65	5,32	28,52
KW-3	18,38	5,31	28,91
KW-4	18,25	5,65	30,98
KW-5	18,32	5,73	31,30
KW-6	17,77	5,38	30,27
KW-7	18,15	5,20	28,65
KW-8	18,23	5,13	28,17
KW-9	18,14	5,69	31,37
KW-10	18,32	5,70	31,13
KW-11	18,08	5,03	27,84
KW-12	18,22	5,09	27,95
KW-13	18,42	5,40	29,35
KW-14	18,22	5,41	29,69

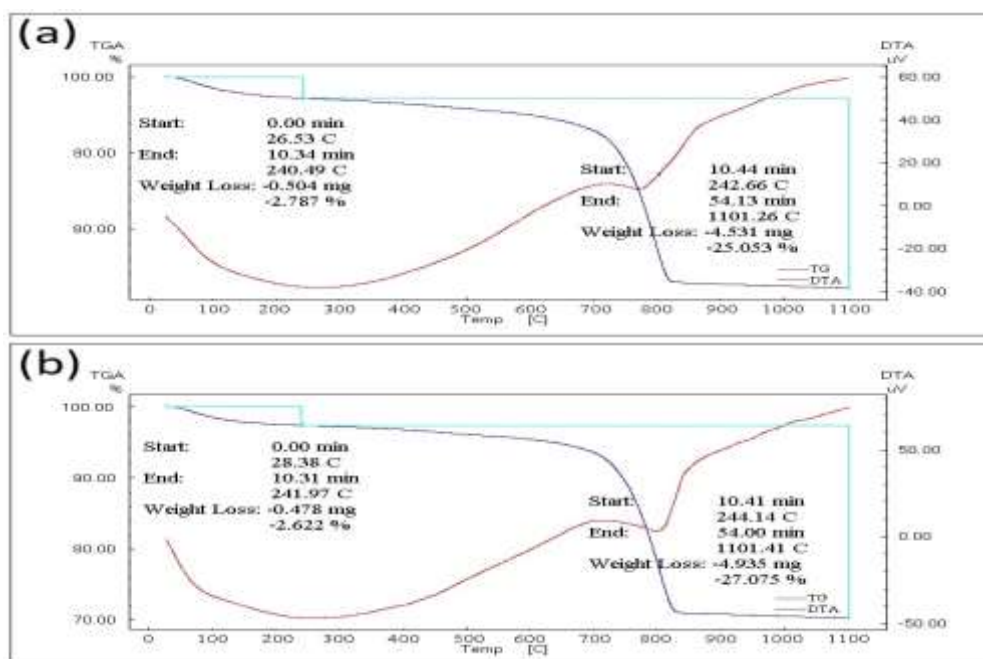


Figure 5. TG-DTA diagrams of the sample (a) KW-11, (b) KW-14

### 3.4. SEM/EDS results

SEM/EDS analysis was carried out to enlighten the micro structural and micro chemical properties of the samples. EDS results (Table 5) showed that the amount of CaO changes in the range of 18.34-40.22 wt. % indicating calcareous raw materials. The relatively higher amounts of CaO (up to 40.22 wt. %) detected for few of the samples (e.g. KW-3, KW-9) were attributed to the fact that the analysis was performed on  $\mu\text{m}$  scaled sectional areas of the samples (maximum 100  $\mu\text{m}$  sized SEM images), and the abundant presence of calcite together with its relatively coarser grains would have occasionally affected the chemical composition of the selected image. This situation was proved through the sectional EDS analysis of the representative samples (Figure 6 and Figure 7).

Table 5. EDS results of the samples.

Code	Oxide						
	SiO <sub>2</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	FeO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O
KW-1	42.38	33.26	13.14	7.80	2.36	1.06	-
KW-2	54.08	18.34	16.38	5.77	2.96	2.47	-
KW-3	40.50	36.80	14.62	5.00	1.70	1.39	-
KW-4	44.39	29.71	12.91	9.40	2.82	0.76	-
KW-5	46.05	32.29	13.49	3.55	3.42	1.19	-
KW-6	43.36	31.11	13.47	7.70	2.39	1.63	0.63
KW-7	41.99	35.38	13.42	5.44	2.34	1.43	-
KW-8	45.10	32.91	13.16	5.86	2.23	0.75	-
KW-9	39.80	40.22	12.13	4.65	1.89	1.28	-
KW-10	44.20	33.29	13.72	5.18	1.62	1.98	-
KW-11	42.94	35.72	11.24	7.00	2.00	1.10	-
KW-12	42.37	33.62	12.47	6.91	2.50	1.57	-
KW-13	44.19	33.39	13.64	5.41	2.09	1.28	-
KW-14	49.19	24.18	16.34	5.56	3.48	1.25	-

(-): not detected or under the detection limit.

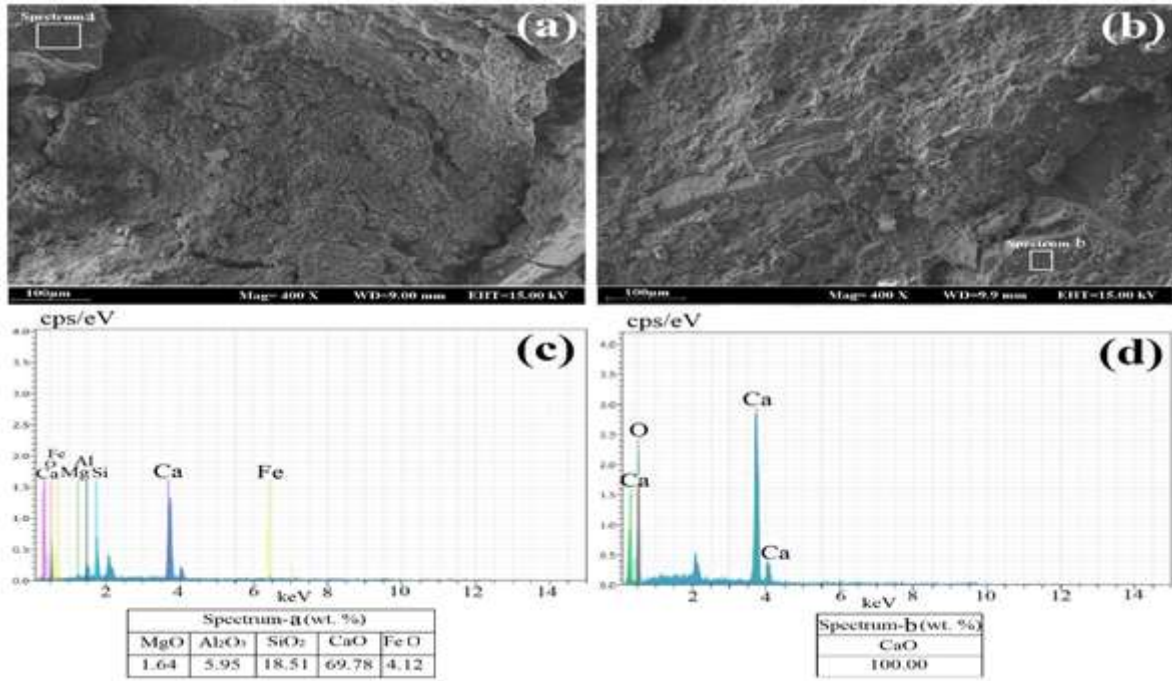


Figure 6. SEM images and EDS results of the selected areas of (a-c) KW-3 and (b-d) KW-6

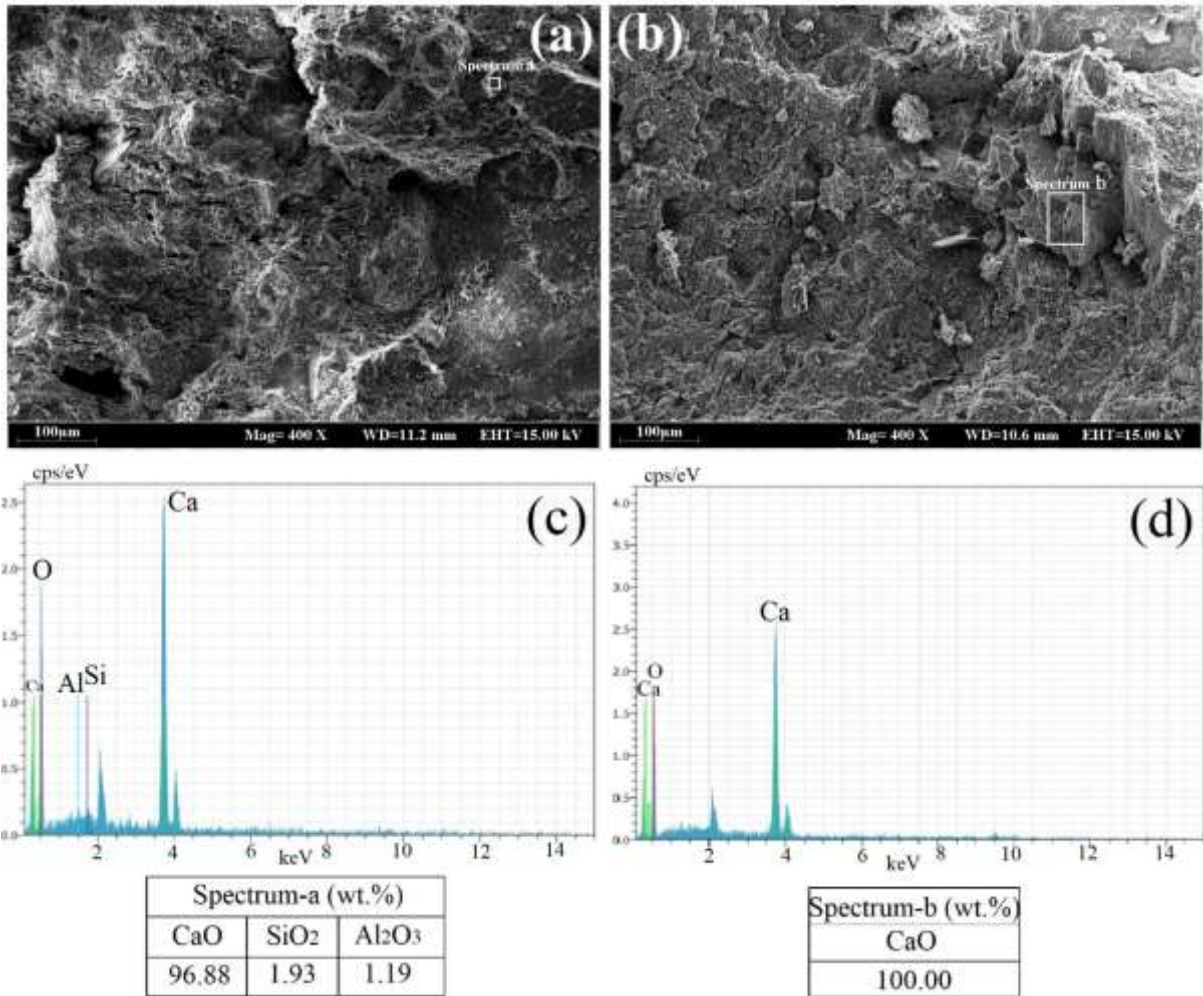
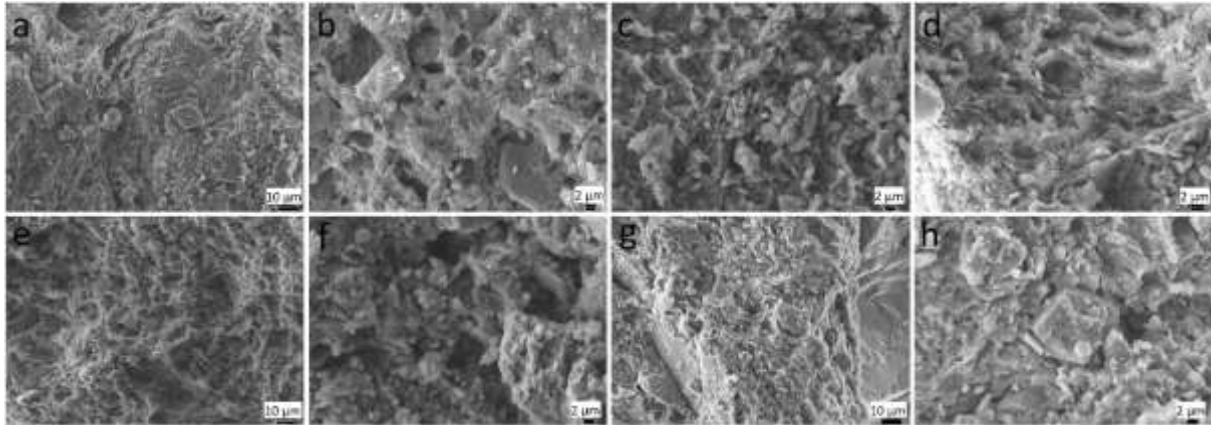


Figure 7. SEM images and EDS results of the selected areas of (a-c) KW-9 and (b-d) KW-12

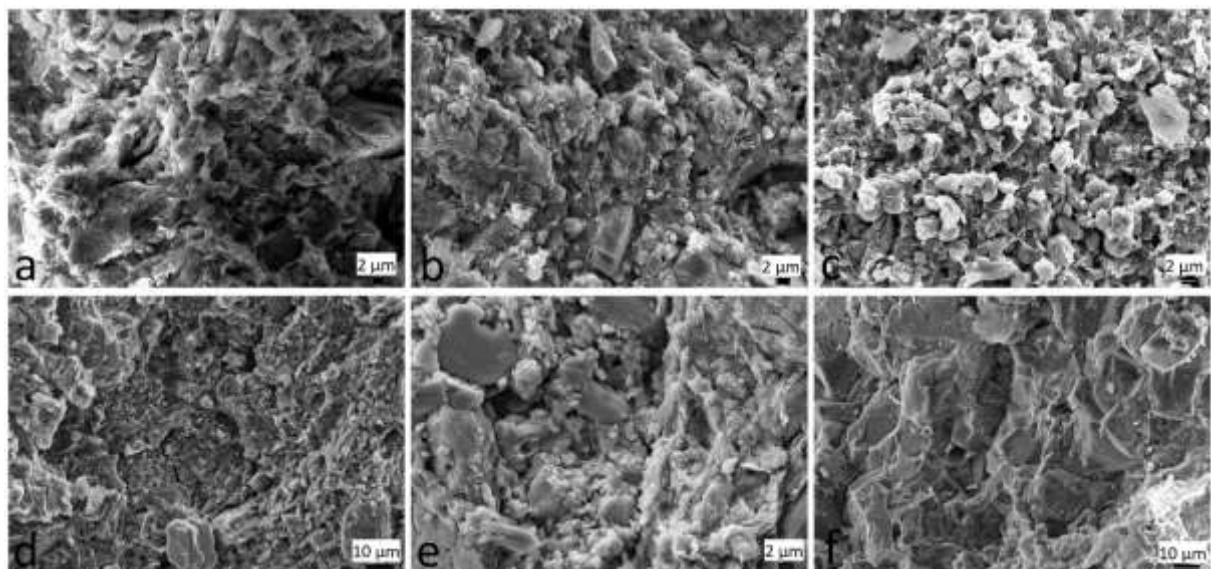


The amount of  $\text{SiO}_2$  was found in the range of 39.8-54.08 wt. % and its presence was mainly attributed to quartz and secondly clay minerals, two of which have been identified on XRD patterns of the potsherds.  $\text{Al}_2\text{O}_3$  was found as 11.24-16.38 wt. % (13.58 wt. % in average) which suggested relatively low amount of clay minerals. The amount of  $\text{K}_2\text{O}$  was seen to change between 0.75 and 2.47 wt. %,  $\text{Na}_2\text{O}$  was encountered for one sample (KW-6; 0.63 wt. %). The source of these two oxides was thought to be the plagioclase and/or feldspars, but such minerals were not seen on XRD patterns (or their intensity was under the detection limit). Thus, it was assumed that  $\text{K}_2\text{O}$  and  $\text{Na}_2\text{O}$  were mainly derived from the clay minerals. The amount of  $\text{MgO}$  changes in the range of 1.62-3.48 wt. % (2.41 wt. % in average). The origin of this oxide is presumed as the Mg-bearing minerals (e.g. chlorite) and/or dolomite. Found as the only possible colorant for the potsherds, iron oxide changes in the range of 3.55-9.40 wt. % (6.08 wt. % in average). The comparative evaluation of the EDS results together with the former data, it was seen that the chemical compositions of the samples were substantially compatible with the mineralogical assemblages.

SEM images were also shot in 2-10  $\mu\text{m}$  scale so as to see the microstructure of the potsherds (Figure 8, Figure 9). It was observed that there was a limited vitrification which suggested that the firing temperature range of the samples were low, thus the maturation and sinterization behaviors of the potsherds were likely inhibited throughout the ceramic matrices. This assumption approved the firing temperature ranges revealed by means of XRD analysis in which there were no traces of neo-formations (new phases occurring at higher temperatures).



**Figure 8.** SEM images of the samples (KW-1/KW-8 respectively showed by a-h)



**Figure 9.** SEM images of the samples (KW-9/KW-14 respectively showed by a-f)

#### 4. Conclusions

The archaeometric data achieved within this study by using multiple analytical techniques showed that the representative samples of Early Bronze Age cooking pots with triangular handles belonging to Tilbaşar Höyük (Gaziantep, Turkey) were mainly produced with clays rich in carbonates (particularly calcite). The presence of the primary calcite was revealed by both spectroscopic (XRD, FTIR), microscopic (SEM-EDS, petrography) and thermal analysis (TG-DTA) methods. In the context of the geological formations of the mound and its vicinity [29-35], it could be seen that the region covering Antakya, Şanlıurfa, Adıyaman, Osmaniye, Kilis, Kahramanmaraş and Gaziantep is occasionally comprised of carbonates (i.e. dolomite, limestone, marble). Consequently, the origin of the raw materials used for such ceramic group would be local.

The dominant existence of calcite on XRD patterns together with no traces of high temperature minerals, and also the evidences of carbonates identified through FTIR (with characteristic band values) and TG-DTA (with the enthalpy changes and weight loss values at certain degrees) suggested that the firing temperature range of the studied potsherds was presumably 700-800°C. This estimation was also proved by SEM images in which a very limited vitrification degree was seen throughout the micro structure of the samples. It could be concluded from the results that the characteristic features of the studied samples were, in general, consistent with the diagnostics of the ancient cooking pots as in the available literature. Additionally, considering the whole data, it could be deduced that the samples of cooking pots with triangular handles in this work are probably of a single type product group. It is predicted that the outcome of this study would be a convenient scientific step for the further archaeometric studies regarding the same or other ceramic groups of the mound and/or the region.

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#### Authors' Contributions

Murat BAYAZIT and Osman Ekinçi analyses, evaluation of the archaeometric data and interpretation of the results, Elif GENÇ achievement of the samples, archaeological literature, assessment of the results.

#### Statement of Conflicts of Interest

There is no conflict of interest between the authors.

#### Statement of Research and Publication Ethics

The author declares that this study complies with Research and Publication Ethics.

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