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Characterization of ancient ceramic shreds: Insights into firing conditions and manufacturing technology

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ABSTRACT

Four ancient ceramic shreds from the archaeological site Leletepe in the Fizuli region of the Republic of Azerbaijan and two local raw ceramic pastes were characterized by powder X-ray diffraction (PXRD) and thermal analysis (TG-DTG) techniques. XRD analysis of ceramic sherds reveals that all investigated samples contain similar minerals: quartz, feldspar, and clay. Three samples out of four contain calcite. Based on the traditional approach, it has been assumed that the firing process in these samples stopped before 700 °C. The mass loss ratios of samples of ancient ceramics also indicate that reversible dehydroxylation took place in all four samples, thus indicating the initial mild firing conditions. The summary of all the applied methods indicates that the ceramic samples were made using a similar manufacturing technology. According to XRD analysis, samples N1 and N4 contain diopside, and samples N2 and N3 contain maghemite, indicating the different origins of the ceramic shreds. Analysis of the raw ceramic mass also did not reveal the presence of these minerals, which may indicate a discrepancy between the origin of ancient ceramic sherds and modern ones.

KEYWORDS: Thermal analysis, X-ray diffraction, Clays, Ancient ceramic, Firing temperature

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INTRODUCTION

The complex characterization of ancient ceramics most often includes a description of its mineralogical, chemical, and thermal properties (Iordanidis *et al.*, 2011; Meyvel *et al.*, 2012; Kloužková *et al.*, 2014; Molodin *et al.*, 2019; Yuan *et al.*, 2022). These data can be provided in an interdisciplinary approach using thermogravimetric (TG), thermoluminescence (TL), X-ray diffraction (XRD), and X-ray fluorescence (XRF) methods. The interpretation of the analytical results, in combination with the relevant archaeological information, can lead to essential conclusions in studies of the origin of ceramics (Ortega *et al.*, 2010) and the ancient technologies used (Arnold, 2000; Papadopoulou *et al.*, 2006; Vlase *et al.*, 2019).

During firing, ceramics' mineral components undergo phase transitions characteristic of their firing temperatures (Shoval *et al.*, 2011; Medeghini *et al.*, 2022). The results of thermal analysis in combination with powder X-ray diffraction (PXRD) are usually used to estimate the initial firing temperature of ancient ceramics (Drebushchak *et al.*, 2007; Meyvel *et al.*, 2012). XRF reveals the chemical composition of ancient ceramics and can be successfully used for the classification of ceramics into groups of similar composition (Weaver *et al.*, 2013).

This work used TG/DTG and PXRD methods to analyze four fragments of ancient pottery from the Leletepe archaeological site in the Fizuli district of Azerbaijan and two modern raw ceramic paste samples. This work aims to characterize ancient pottery in terms of its mineralogical composition, as well as to study its thermal behavior and determine the firing temperature.

The ancient settlement of Leletepe is situated in the Araz River Valley, 6 km southeast of Horadiz village in Fizuli district, Azerbaijan. The settlement, which dates back to the Late Neolithic period, covers an area of 0.9 hectares. It is one of three Late Neolithic settlements in the region. In 2019, the "Karabakh Neolithic-Eneolithic Expedition" conducted archaeological surveys and registered the site. In 2020-2021, excavations were carried out in four trenches of 16 square meters each. These excavations revealed the remains of architectural structures made of raw bricks and many artifacts of different functional purposes from the Late Neolithic period. The pottery found at Leletepe is similar to materials found at other Late Neolithic sites in the Karabakh lowland and features a variety of patterns and application techniques, including chased and relief ornamentation and painted fragments. Some specimens also have folded inlet edges of the rim and hemispherical handles with traces of mats on the bottom. Based on stratigraphic estimations, Leletepe is a settlement of the Neolithic era,

attributed to the first half of the VI millennium BC. It is one of the most significant Neolithic-Eneolithic monuments in the area known as the Caucasus. The choice of this monument as a base for our studies was because it is considerably larger than all the monuments known in this area, and we perceive it to be the center of the nest of settlements.

MATERIALS AND METHODS

The Institute of Archeology, Ethnography, and Anthropology of ANAS provided the samples of single fragments of ceramics found during the "Karabakh Neolithic-Eneolithic Expedition" in the territory of Leletepe, Fizuli region of the Republic of Azerbaijan (samples N1, N2, N3, and N4). Most of these specimens are believed to be from the Neolithic period and may have been used for cooking or preserving food. The samples were air-dried overnight at 50°C before analysis and finely powdered in an agate mortar. Two raw ceramic paste samples were also analyzed as reference material (Samples CP1 and CP2).

The D2Phaser (Bruker) diffractometer was used to conduct PXRD on samples with Ni-filtered CuKα radiation (λ=1.5406 A), and the samples were oriented randomly. Scans were performed at a 1.2°/min scanning speed within the $5 \leq 2\Theta \leq 75°$ region. Semiquantitative measures were derived from the PXRD data to estimate the abundance of mineral phases, including the intensity of specific reflections, density, and mass absorption coefficients of elements for CuKα radiation. Mineral phase identification was conducted using the DIFFRAC.SUITE software with the TOPAS non-standard quantitative phase and structure analysis program and ICDD powder databases. Semiquantitative approximations of mineral phase abundances were determined using the Reference Intensity Ratio (RIR) method, employing specific RIR values established in-house. Using the standard internal technique, the RIR method is a universally applicable constant for quantitative phase analysis in X-ray powder diffraction (Hubbard & Snyder, 1988). To assess the proportion of total amorphous material, the area of the broad background hump representing amorphous material in each sample was compared to the corresponding area of standard mixtures comprising varying concentrations of natural amorphous material. These standard mixtures were subjected to scanning under identical conditions. A semiquantitative estimation of the percentage of total amorphous material was subsequently obtained.

Thermogravimetric and differential thermal analysis of ceramic powders were carried out in a Perkin Elmer STA6000 Simultaneous Thermal Analyzer with the following parameters: heating ranges from ambient to 950 °C, heating rate 5 °C, balance sensitivity- 0.1 μg, and nitrogen gas flow-20 mL/min.

RESULTS AND DISCUSSION

Figure 1 shows the results of TG/DTG experiments carried out on a raw ceramic paste sample with two mass loss steps, indicating that the investigated material does not contain calcite. Raw ceramic pastes (CP1 and CP2) are the modern examples presented by a pottery master working near the archaeological site using local raw materials. The DTG curve is a derivative of mass loss (dm/dt) and indicates the steps of mass loss more clearly. Mass loss values for both samples are summarized in Table 1. Samples lose 8.06% (CP1) and 15.0% (CP2) upon dehydration and 3.16% (CP1) and 5.45% (CP2) upon decomposition of hydroxyls.

Generally, row ceramic paste consists of clay (smectites, kaolinites) and tempering materials like quartz, feldspar, calcite, and organic fillers. Because the tempering materials are thermally more stable than clay, one observes changes only in the clay material under mild firing (meaning the firing temperature is ≤ 700 °C) (Drebushchak *et al.*, 2011). It is generally accepted that the loss of mass during clay heating occurs due to (I) dehydration (≤ 350 °C), (II) decomposition of hydroxyls (350-600 °C), and (III) decomposition of carbonates, mainly calcite (600-850 °C) if it is present in the source material (Drebushchak *et al.*, 2018). Changes also occur at higher temperatures. Upon firing above 1000 °C, the ceramic paste becomes a glassy substance containing particles of added temper materials. This change is irreversible, and the resulting product has little in common with the initial ceramic paste. Here we should note that this confirmation is correct only if montmorillonite predominates as a clay material since it contains both bound water and hydroxyl groups in its composition.

In contrast, kaolinite begins to lose mass at temperatures of about 400 °C and higher due to dehydroxylation (Drebushchak *et al.*, 2018). Mass loss after dehydroxylation also occurs at higher temperatures, but this depends on the type of clay mineral. Clay minerals are transformed into an anhydrous amorphous phase during the firing of montmorillonite and kaolinite. Even this brief review indicates that the processes under consideration are incredibly complex and that the specified temperature intervals are conditional. Nevertheless, the authors of (Drebushchak *et al.*, 2011) managed to establish some patterns in the ratios of mass loss in these temperature ranges, which will also be used in the context of this work.

The results of thermogravimetric TG/DTG measurements of four ancient ceramic samples are shown in Figure 2. Compared to the ceramic paste in Figure 1, the dehydration and dehydroxylation peaks for ceramics are smaller than for ceramic paste. However, dehydroxylation occurs when ancient pottery is heated.

Table 1 summarizes the results of TG measurements of four ceramic samples found during archaeological excavations in Leletepe. The results of TG measurements of ceramic samples are represented in terms of the mass loss in the temperature ranges of ≤350 °C (m_1-dehydration), 350÷600 °C (m_2-dehydroxylation), and 600÷850 °C (m_3-decomposition of carbonates, micas, etc.). These samples' TGs differ from each other and the ceramic paste samples.

There are different approaches in the literature for determining the firing temperature of ancient pottery (Molodin *et al.*, 2019). The basic idea of the thermogravimetric method is that only reversible

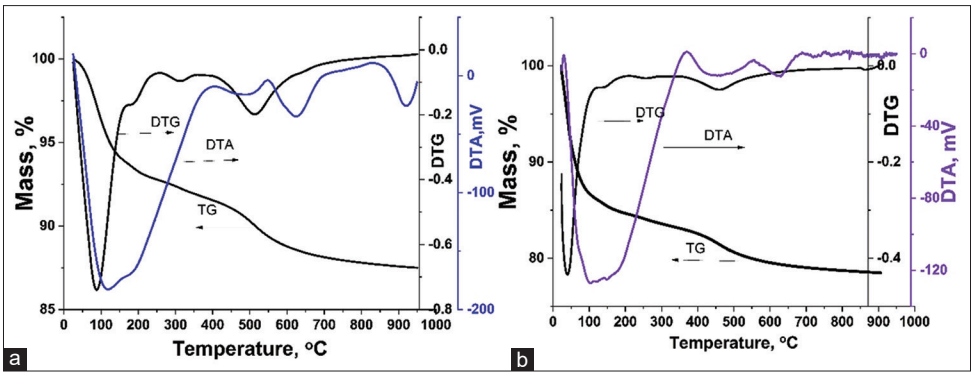

Figure 1: TG, DTG and DTA profiles of raw ceramic pastes (a-sample CP1 and b- sample CP2)

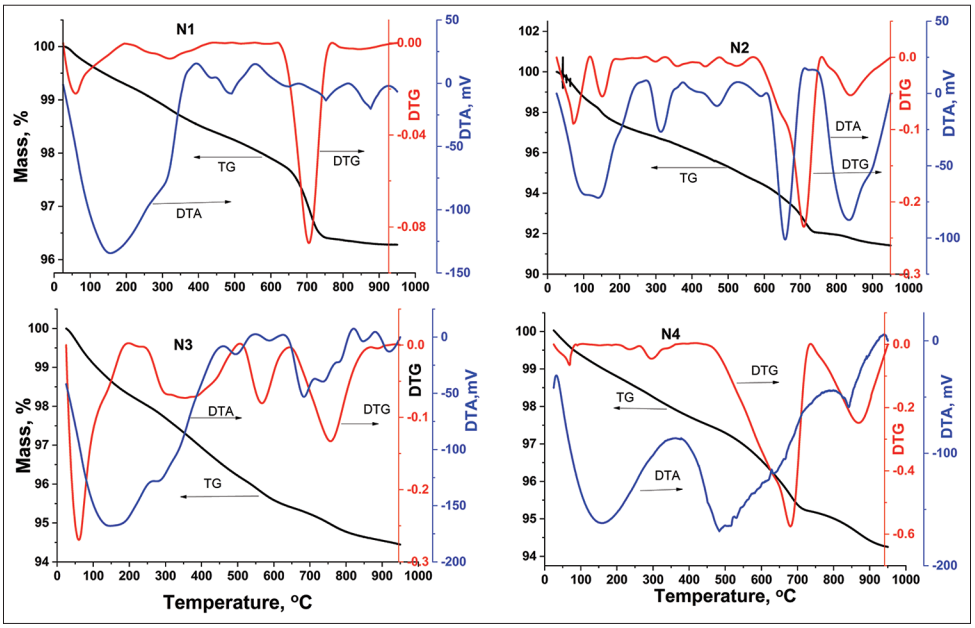

Figure 2: TG, DTG and DTA profiles of four (N1, N2, N3, and N4) ancient ceramic sherds from Leletepe archeological site

Table 1: Mass-loss of ancient ceramic samples and raw ceramic pastes in the temperature intervals

Sample	Mass loss, %			m1	m2	m3	m2/m1
	≤ 350 °C	≤ 600 °C	≤ 850 °C				
N1	98.7	97.9	96.32	1.3	0.8	1.58	0.62
N2	96.47	94.4	91.72	3.53	2.07	2.68	0.59
N3	97.33	95.59	94.64	2.67	1.74	0.95	0.65
N4	97.98	96.56	94.75	2.02	1.42	1.81	0.70
CP1	91.94	88.78	87.68	8.06	3.16	1.1	0.39
CP2	85	79.55	78.61	15	5.45	0.94	0.36

thermal transformations will be detected if the sample is heated a second time. Upon reheating, transformations not observed in the previous heating will be detected only at temperatures above the upper-temperature limit of the first heating. The irreversibility of thermal transformations in clay occurs due to chemical transformations with the release of gaseous products, the formation of new minerals, or irreversible phase transformations.

The endothermic peaks in differential thermal analysis enable the identification of the upper limit of temperature intervals of loss of chemically combined hydroxyl groups by clay minerals such as smectites and kaolin. It has been assumed that (Meyvel *et al.*, 2012) "when this peak is present, it shows that the pottery has not been heated above this temperature previously; when it is absent, it has been fired above this temperature. "However, this statement is confirmed only for freshly prepared ceramic products. In the DTA of these products, no endothermic peaks are detected at temperatures from 400 °C to 600 °C (Drebushchak *et al.*, 2005). Numerous experimental data indicate that most samples of ancient ceramics lose water, i.e., the hydroxyl group, when heated to 550 °C (Drebushchak *et al.*, 2018). In this case, one has to assume that the firing temperature of ancient ceramics was less than 550 °C or that the decomposition of hydroxyls is reversible. However, many works indicate that the process of dehydration and dehydroxylation of clay fired up to 700 °C (so-called mild firing) is a partially reversible process (Fajnor & Jesenák, 1996; Shoval *et al.*, 2011; Gallet & Le Goff, 2015). Barrett (2015) have shown that the dehydroxylation of clay heated up to 700-800 °C is reversible and can even be used for dating ancient ceramics after re-hydroxylation at ambient

conditions. Therefore, dehydroxylation upon reheating ancient pottery indicates a low or short-term firing temperature.

X-ray diffraction patterns can indicate the presence of amorphous phases in a sample. Amorphous phases using CuKα radiation can be identified as one or more broad background humps between approximately 10 and 50°, depending on their chemical composition. In inorganic samples, amorphous phases usually appear between 10 and 18 °C (Papadopoulou *et al.*, 2006).

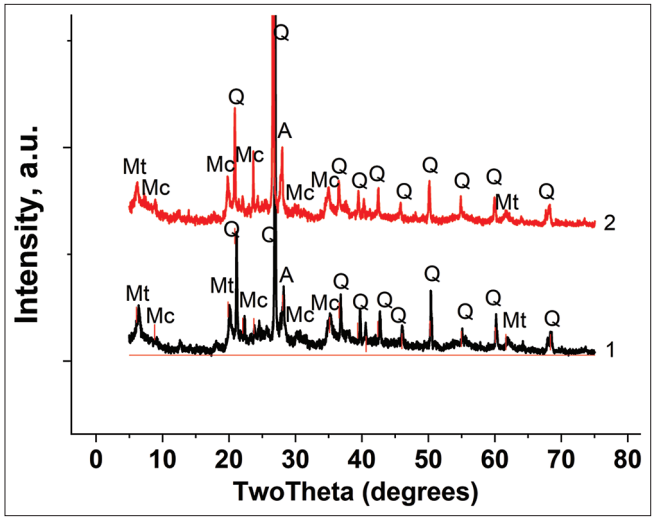

Figure 3: XRD patterns of two raw ceramic pastes; 1-sample CP1 and 2-sample CP2; Mt-montmorillonite; Mc-muscovite; Q-quartz; A-albite (feldspar)

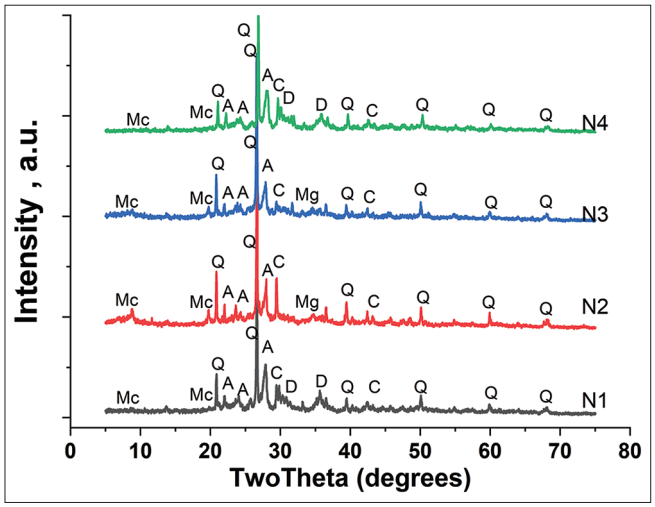

Figure 4: XRD patterns of four ceramic shreds (samples N1, N2, N3, and N4). Mc-muscovite; Q-quartz; A-albite (feldspar), Mg- Maghemite, D- diopside

XRD patterns of raw ceramic paste indicate the presence of clay minerals like montmorillonite (PDF 00-060-0318) and muscovite, quartz, and feldspars as a tempering material (Figure 3). Table 2 shows that both samples of raw ceramic paste do not contain calcite in their composition. Figure 4 illustrates XRD patterns of ceramic shreds, showing that the samples contain Mc-muscovite (PDF 00-007-0025), Q-quartz (PDF 01-070-7344), and A-albite (feldspar, PDF 00-041-1480)). XRD analysis of ceramic sherds reveals that all investigated samples contain minerals like quartz and feldspar in different quantities (Table 2). Calcite (PDF 01-089-1304) is present in samples N2, N3 and N4 but not contained in sample N1. According to XRD analysis, samples N1 and N4 contain diopside, and samples N2 and N3 contain maghemite. Analysis of the raw ceramic mass did not reveal the presence of these minerals, which may indicate a discrepancy between the origin of ancient ceramic shards and modern ones.

Feldspars (in our case, albite and microcline (PDF 01-076-0830)) can be introduced into the ceramic mass as a hardening agent or be present in the composition of the original clay as a natural admixture since the clays themselves are considered to be the weathering products of feldspar. Feldspars are stable when heated to 950 °C (Stubna *et al.*, 2013). Alkaline feldspars remain glassy when melted, while anorthite crystallizes relatively quickly. So, feldspars' presence says little about ceramics' firing temperature.

Quartz is a significant component of tempering materials and also exists in raw clay as a natural mixture. A characteristic high intensity peak of quartz was detected at 27.19° was present in all the other samples. Quartz undergoes a phase transition around 573 °C when heated, but this process is reversible, and no signs of previous heating could be detected after cooling. Small endothermic peaks of phase transformation of quartz were reported by (Papadopoulou *et al.*, 2006) as well.

Calcite is the most common "fingerprint" for determining the provenance of ceramics and, to some extent, for determining the firing temperature since it can be added to ceramic paste or found in the original clays as a natural impurity. The presence of calcite in ancient pottery is considered today the sign of low-temperature firing at about 700 °C (Papadopoulou *et al.*, 2006). But here, too, not everything is so clear. Silicates that were produced from the clay minerals possess the ability to absorb carbon dioxide selectively from the atmosphere (Kalinkin *et al.*,

Table 2: Mineral composition of ancient ceramic samples and raw ceramic pastes

Sample	Quartz, mass %	Feldspar, mass %	Calcite, mass %	Clay minerals, mass %	Other minerals, mass%
N1	48.9	Albite -31.3	0	Muscovite -8.9	Diopside -10.4
N2	54.8	Albite -29.3	3.7	Muscovite -10.3	Maghemite -1.9
N3	40.0	Albite-29.8	1.9	Muscovite -27.1	Maghemite -1.2
N4	29.1	Albite-39.0	8.6	Muscovite -7.3	Diopside -16.0
CP1	42.6	Albite -9.5	0	Muscovite -40.5 Montmorillonite -7.3	
CP2	42.0	Albite -14.7	0	Muscovite -36.2 Montmorillonite -7.3	

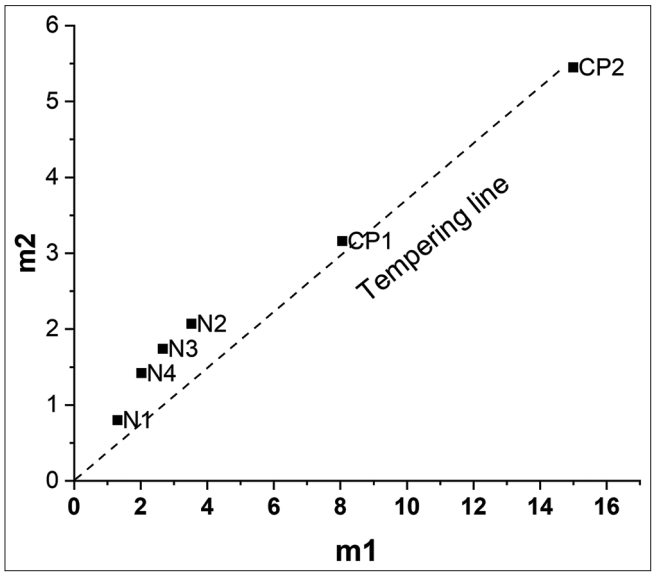

Figure 5: Mass-loss diagram for the ancient ceramic samples and raw ceramic pastes

2008, 2009); in other words, it is quite a reversible process over a long time and, thus, cannot be used for the estimation of firing temperature (Drebushchak *et al.*, 2018).

The concentration of calcite in samples N2, N3, and N4 is 3.7, 1.9, and 8.6%, respectively (Table 2); therefore, according to the traditional interpretation, the firing temperature of these samples was 700 °C (Cultrone *et al.*, 2001). X-ray phase analysis did not reveal the calcite in sample N1, and it can erroneously be assumed that this sample was fired at a temperature above 800 °C. But the analysis of the raw ceramic pastes CP1 and CP2 showed that calcite might not be in the original material; therefore, in this case, its absence in ceramics cannot be an indicator of the firing temperature. At the same time, the presence of calcite cannot be used to determine the origin of ceramics, suggesting that it can accumulate over time under natural storage conditions.

X-ray phase analysis did not detect mullite in the raw ceramic paste or shreds. The absence of this mineral is also a criterion for assessing the firing temperature of ancient ceramics since mullite has yet to be introduced into the composition of the clay fraction of ancient ceramics as a tempering mineral. Since it occurs during the high-temperature firing of kaolinite, it can be a signature for the upper limit of the firing temperature of ancient ceramics.

Montmorillonite can also serve as a guideline for determining the lower limit of the firing temperature of ceramics since, at 600 °C, this mineral turns into an amorphous phase (Drebushchak *et al.*, 2005).

The presence of the iron-containing mineral is also confirmed by XRD (Figure 4) and TGA analysis. Differential thermal curves of maghemite show a peak at 815 °C, and XRD analysis attributed this peak to the recrystallization of maghemite (PDF 01-076-4113) or hematite (Taylor & Schwertmann, 1974).

To reveal the differences between the thermogravimetric data for various samples of ancient ceramics, the authors of (Drebushchak *et al.*, 2011) developed a mass-loss diagram. The mass-loss diagram is an alternative way to visualize the variations in the tempering and degree of thermal transformations of ancient ceramics (Drebushchak *et al.*, 2011). This method is not a description of experiments on the reconstruction of the firing of ancient pottery but rather a way to identify principal components for group differences among the studied samples. Figure 5 shows the mass-loss diagram for the investigated samples based on the data given in Table 1. It can be seen from the Figure 5 that the mass loss ratios of samples of ancient ceramics lie almost in the same line as for raw ceramic pastes. Although the authors of (Drebushchak *et al.*, 2011) warned about the consequences of misuse of the diagram, we still allow the possibility of asserting that, according to the diagram, the samples under study restored the original hydroxyl cover over time while in the ground. And according to stratigraphic data, this time was long since the archaeological site belonged to the Neolithic period. Another conclusion is that the initial firing conditions were relatively mild; otherwise, rehydroxylation would not have been possible.

CONCLUSION

The complex characterization of ancient ceramics, which includes a description of its mineralogical, chemical, and thermal properties, was provided in an interdisciplinary approach using thermogravimetric (TG), thermoluminescence (TL), and X-ray diffraction (XRD). XRD analysis of ceramic paste reveals that all investigated samples contain similar minerals: quartz, feldspar, and clay minerals, and three ancient ceramic samples out of four contain calcite. Based on the traditional approach, it has been assumed that the firing process in these samples stopped before 700 °C. The mass loss ratios of samples of ancient ceramics also indicate that irreversible dehydroxylation took place in all four samples, thus indicating the initial mild firing conditions. The summary of all the applied methods indicates that the ceramic samples were made using a similar manufacturing technology. XRD analyses reveal the presence of accompanying minerals in the composition of ceramics. The difference in the concentration of calcite and accompanying minerals (diopside and maghemite) indicates that the sources of the investigated ceramic samples were somewhat different.

The current research represents the initial all-inclusive examination of the ceramic artifacts recovered by archaeologists within the borders of the Azerbaijan Republic. Consequently, it has been asserted that the study in question has added original perspectives to the current corpus of literature regarding the subject matter.

CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The authors did not receive financial support from any organization for the submitted work.

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