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FIRING CONDITIONS AND PRODUCTION TECHNIQUES OF ANCIENT CERAMICS

A.S. Ahadova, A.B. Ahadov, A.Z. Abishov, M.A. Gurbanov, S.G. Mammadov

Institute of Radiation Problems, MSE AR
a.ahadova@irp.science.az

Abstract: During the firing process, the mineral components of ceramics experience phase transitions and chemical composition changes, and these processes depend on the temperature at which they are fired. Comprehensive analysis of ancient ceramics often involves the study of their mineralogical, chemical, and thermal characteristics. By analyzing the data through an interdisciplinary approach using thermogravimetry (TG), thermoluminescence (TL), X-ray diffraction (XRD), and X-ray fluorescence (XRF) techniques and considering the archaeological context, researchers can draw important conclusions about the origin of the ceramics and the ancient technologies used. X-ray fluorescence (XRF) analysis provides information on the chemical composition of ancient ceramics and is effective for classifying ceramics based on composition similarities, while thermal analysis and powder X-ray diffraction (PXRD), are commonly utilized to determine the original firing temperature of ancient ceramics.

Keywords: thermal analysis, X-ray diffraction, clays, ancient ceramic, firing temperature.

1. Introduction

TG/DTG, PXRD, and TL methods were used to study four ancient pottery shards obtained from Leletepe archaeological site in Fizuli Province, Azerbaijan. Two modern raw ceramic paste samples were also used to compare the results. The establishment of the firing temperature and composition of these shards suggests the level of development of this settlement as well as the source of raw materials. Therefore, the mineralogical composition of the ancient pottery was analyzed and its thermal properties were studied to establish the temperature at which it was fired.

Leletepe settlement is situated 6 km southeast of Goradiz village in Fizuli district of Azerbaijan. It is assumed that this ancient human settlement appeared in the late Neolithic era, covering an area of about 0.9 hectares. It is one of the three Late Neolithic settlements in the region that were subjected to archaeological research by the “Karabakh Neolithic-Eneolithic Expedition”, which officially documented the site in 2019. The initial excavations, conducted between 2020 and 2021, involved four trenches, each covering an area of 16 square meters. Architectural remains were found to have been built with raw bricks and other objects related to the Neolithic era. These objects somehow resemble materials found at other Late Neolithic sites in the Karabakh plain. Some pottery specimens show a variety of patterns such as chasing and relief ornamentation, as well as fragments decorated with paint with folded edges on the rim and hemispherical handles with remnants of mats on the bottom. According to stratigraphic estimates, Lelepetepe is a Neolithic settlement, which presumably existed in the first half of the sixth millennium BC. This monument is of great importance for the Neolithic-Eneolithic period and is located in the Caucasus region. We chose Leletepe as the central site for our research because of its much larger size compared to other nearby monuments and because it is observed to serve as a central hub for a group of villages.

2. **Experimental procedures and techniques**

Separate fragments of ceramic samples were submitted by the Institute of Archaeology, Ethnography, and Anthropology of ANAS, discovered during the "Karabakh Neolithic-Eneolithic Expedition". The specimens are labeled as N1, N2, N3 and N4. These artifacts presumably belong to the Neolithic Age. Raw ceramic paste samples, labeled CP1 and CP2, were also examined as reference materials. A Bruker D2Phaser diffractometer manufactured by Bruker was used for powder X-ray diffraction (PXRD) analysis. The samples were exposed to Ni-filtered CuKα radiation with a wavelength of 1.5406 A. The orientation of the samples was arbitrary. Scanning was performed at a rate of 1.2 degrees per minute in the region from 5 to 75 degrees (2θ). Semi-quantitative metrics were derived from the PXRD data to assess the prevalence of mineral phases. For this purpose, the intensity of specific reflections, as well as the density and mass absorption coefficients of elements was analyzed using CuKα-radiation.

Ceramic powders were subjected to thermogravimetric and differential thermal analysis using a Perkin Elmer STA6000 synchronous thermal analyzer. The analysis was performed at the following parameters: heating range from room temperature to 950℃, heating rate 5℃, scale sensitivity 0.1 μg, and nitrogen gas flow rate 20 mL/min.

TL measurements were performed using a 3500 TLD at a heating rate of 5℃ .

3. **Analysis**

Figure 1 shows the results of the TG/DTG studies performed on the raw ceramic paste samples CP1 and CP2. These results show two different stages of mass loss, indicating the absence of calcite in the investigated material. The DTG curve, representing the first derivative of the mass loss rate (dm/dt), gives a clearer picture of the mass loss stages. The mass loss data for both samples are summarized in Table 1. During dehydration, the samples decrease in weight by 8.06 % (CP1) and 15.0 % (CP2). In addition, the samples decrease in weight by 3.16 % (CP1) and 5.45 % (CP2) upon decomposition of hydroxyl groups.

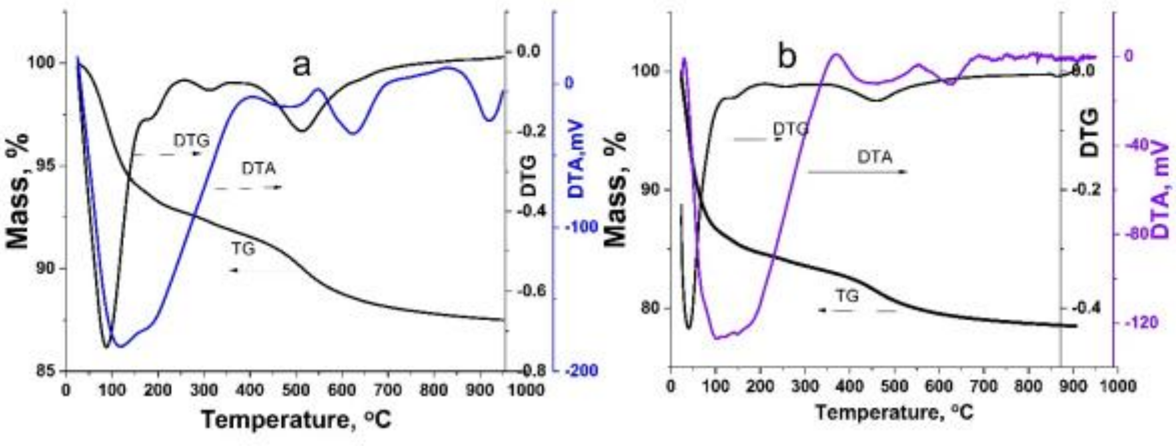

Fig. 1. TG, DTG, and DTA profiles of raw ceramic pastes (a-sample CP1 and b- b-sample CP2).

The ceramic paste consists of smectite and kaolinite, and fillers quartz, feldspar, calcite, and organic fillers. Except for the organics, the fillers are thermally more resistant to temperature compared to clay. Therefore, the changes observed during moderate firing (when the firing temperature is ≤700°C) are only evident in the clay material [15]. The mass reduction when the clay is heated ends with the following processes: dehydration (up to 350°C), hydroxyl decomposition (350-600°C), and carbonate decomposition (600-850°C) if it is present in the starting material [16]. Further modification continues at elevated temperatures. Since, when heated to temperatures above 1000°C, the ceramic paste is transformed into a glassy substance by chemical reactions involving particles of filler ingredients. As a rule, these transformations are irreversible and the composition of the final product changes greatly compared to the original ceramic paste. It is important to note that these temperature ranges are conditional and are only valid if the main clay ingredient is montmorillonite, as it contains both bound water and hydroxyl groups. In contrast to montmorillonite, kaolinite experiences mass reduction starting at temperatures around 400°C and above as a result of dehydroxylation, with a pronounced absence of a dehydration stage [16]. Mass loss also occurs at elevated temperatures, but this depends on the particular clay mineral. The end products of montmorillonite and kaolinite transformation during firing are products in which the clay minerals are transformed into an anhydrous amorphous phase. Despite the fact that the temperature ranges of the changes occurring are conditional, the authors of [15] were able to identify some regularities in the ratio of mass loss in these temperature ranges, which will also be used in the interpretation of the results of these studies.

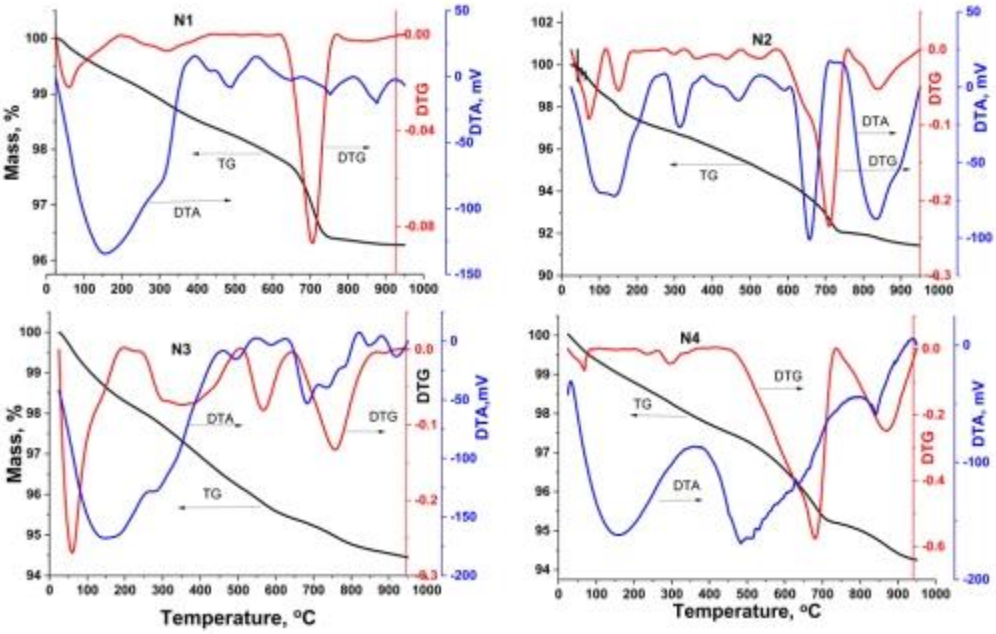

Fig. 2. TG, DTG, and DTA profiles of four (N1, N2, N3, and N4) ancient ceramic shards from Leletepe archeological site.

As shown in Figure 2, the peaks corresponding to dehydration and dehydroxylation in thermogravimetric studies are also characteristic of ancient ceramics samples. TG/DTG data of four samples of ancient ceramics, shown in Fig. 2, indicates that during heating water loss occurs in these samples, but has a smaller value.

The results obtained in the TG studies on four ceramic wares found during the archaeological excavations at Leletepe and in two samples of raw ceramic mass were summarized in Table 1. It is shown that mass loss occurs mainly in three temperature ranges: ≤350°C (m1-dehydration), 350÷600°C (m2-dehydroxylation), and 600÷850°C (m3-

decomposition of carbonates, mica, etc.). The mass losses during the heating of these samples differ from each other and from the raw ceramic paste samples.

Various methods are used to determine the temperature at which antique ceramics were fired [2]. The concept behind the thermogravimetric approach is that some irreversible process occurs during the manufacture of ceramics, and reheating the sample reveals only those thermal changes that can be reversed. In re-firing, any changes that did not occur during the initial heating will only become apparent at temperatures higher than the maximum limit of the first heating. Irreversible transformations in clay include chemical reactions that produce a) gaseous substances, b) new minerals, or c) glass formation in the clay structure.

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℃	**≤600℃**	**≤850℃**				
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

The pronounced peaks observed at TG or DTG can be used to determine the range of temperatures at which the loss of chemically bonded hydroxyl groups in clay minerals occurs. Clearly, if a peak corresponding to dehydrogenation or decomposition of some component of the raw ceramic mass is observed upon reheating, this indicates that the ceramic has not been exposed to temperatures higher than this in the past. Or conversely, the absence of such a peak indicates that the ceramic was previously fired at temperatures above this threshold. In the case of dehydroxylation, this statement is only confirmed for ceramics that have been recently fabricated. Differential thermal analysis (DTA) of wares that have been fabricated in the recent past does not reveal peaks in the temperature range from 400 to 600°C [17]. However, numerous experimental data indicate that in the TG analysis of most ancient ceramic materials, a peak in this temperature region is consistently observed, indicating that the studied samples undergo dehydration, namely the removal of the hydroxyl group when exposed to a temperature of 550°C [16]. Under these conditions, it can be assumed that either the firing temperature of the ancient ceramics was below 600°C or the process of decomposition of hydroxyl groups in ceramics is a reversible process, but proceeds at a very slow rate. In support of this, [20] demonstrated that the process of removing hydroxyl groups from clay by heating at temperatures between 700 and 800°C can be reversible. But, other works [1], [18], and [19] indicate that the process of water release due to the decomposition of hydroxyl groups from clay by firing at temperatures up to 700°C (so-called moderate firing) is only partially reversible. Knowing the patterns of reverse hydroxylation in ceramics we can determine the age of ancient ceramics [2]. Thus, the presence of hydroxyl groups in the process of reheating ancient ceramics does not allow us to speak with certainty about its age, but only that it was fired at a relatively low temperature or for a short time. In order to clarify the age and firing temperature of the pottery, other physical methods of investigation are required.

X-ray diffraction can be used to detect the presence of mineral components as well as the presence of amorphous phases in the sample. These parameters, in turn, provide objective data for building a real picture of the firing of ancient ceramics, and in some cases even the sources of raw materials for its preparation. The formation of an amorphous phase in ceramics can be detected by analyzing the diffraction pattern created by CuKα-radiation in the range from 10 to 50°. These phases appear as broad background humps, and the width of the range depends on their chemical composition, whereas amorphous phases usually occur in inorganic materials in the temperature range of 10 to 18°C [3].

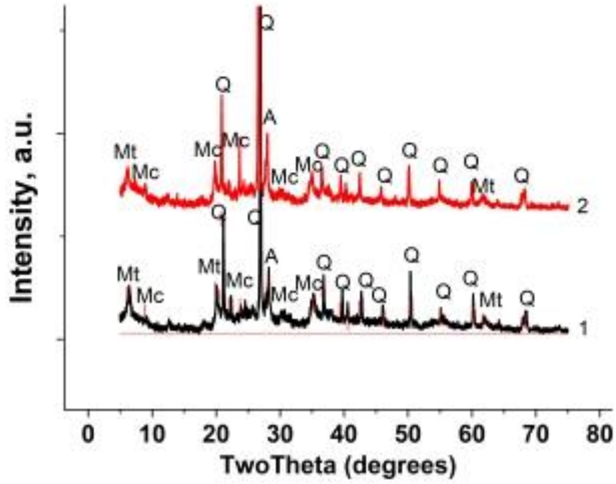

Fig. 3. XRD patterns of two raw ceramic pastes; 1-sample CP1 and 2-sample CP2; Mt-montmorillonite; Mc-muscovite; Q-quartz; A-albite (feldspar).

XRD of the raw ceramic paste reveals the presence of clay minerals such as montmorillonite (PDF 00-060-0318), as well as muscovite, quartz, and feldspars, which serve as hardening materials (Figure 3). At the same time, none of the raw ceramic paste samples contain calcite. For comparison, Figure 4 shows X-ray diffraction images of ceramic fragments where the presence of muscovite (PDF 00-007-0025), quartz (PDF 01-070-7344) and albite (a type of feldspar, PDF 00-041-1480) can be seen. In contrast to the raw pottery, all of the ancient pottery samples examined contain the minerals quartz and feldspar, albeit in different proportions (Table 2). The presence of calcite (PDF 01-089-1304) also varies; it is found in samples N2, N3, and N4, but is absent in sample N1. The results of XRD analysis also showed that samples N1 and N4 contain diopside, while samples N2 and N3 contain maghemite. The absence of these minerals in the composition of the unprocessed ceramic material indicates a possible difference between the sources of ancient ceramic fragments and modern samples.

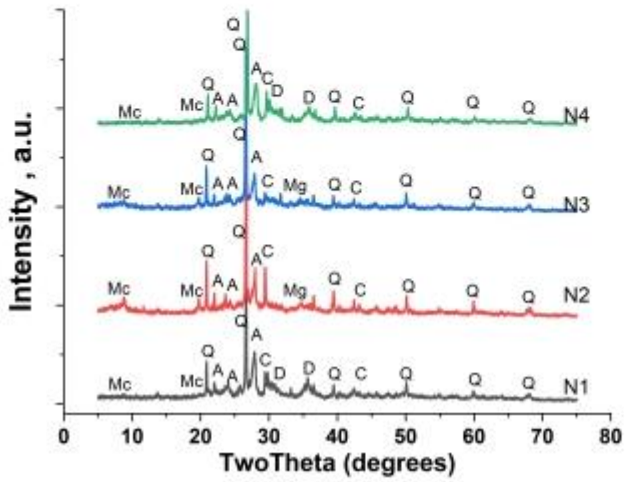

Fig. 4. XRD patterns of four ceramic shreds (samples N1, N2, N3, and N4). Mc-muscovite; Q-quartz; A-albite (feldspar), Mg- Maghemite, D- diopside.

Regarding the presence of other minerals such as feldspars, in particular albite and microcline (PDF 01-076-0830), it can be said that they can be added to the ceramic mixture to increase its hardness. Similarly, they may also be present in the original clay composition as a natural component. As is known, in nature, clays are formed by the weathering of feldspar minerals. Feldspars can be an indicator in determining the annealing temperature of ceramics, because they demonstrate thermal stability up to 950°C [21], and feldspar feldspars retain a vitreous state when melting, while anorthite undergoes relatively rapid crystallization. However, the presence of feldspars provides limited information on the firing temperature of ceramics, since their chemical changes depend significantly on the presence of other components.

Quartz plays a crucial role in regulating the thermal and mechanical properties of ceramics and in most cases, is naturally present in raw clay as a natural mixture. A characteristic peak with high intensity corresponding to quartz in the XRD spectra of ceramics was observed in the region of 27.19° in all the studied samples. The characteristic feature of quartz is that it experiences a reversible phase shift under heat at about 573°C, with no evidence of preheating after cooling. This characteristic peak was also found in [7] and identified as a reversible phase transition of quartz. However, as mentioned above, this signal cannot in any way identify the upper heating limit of ceramics during production.

Another mineral component, calcite, is often used as "hard evidence" to determine the origin of ceramics. Like quartz, it can be intentionally added to the original ceramic paste or naturally present in the original clay as an impurity. The detection of calcite in a thermogram or XRD analysis is considered evidence of the firing of ancient pottery at a relatively low temperature of about 700°C [7]. As shown in Table 2, the calcite content in samples N2, N3, and N4 is 3.7%, 1.9%, and 8.6%, respectively, and according to a common interpretation, the maximum annealing temperature of these samples can be defined as around 700°C [24]. XRD analysis did not reveal the presence of calcite in sample N1, suggesting that this sample was annealed at temperatures above 800°C. However, examination of the raw ceramic paste CP1 and CP2 indicated that calcite may not have been present in the starting material. Therefore, in this particular case, the absence of calcite in the ceramic cannot be an indicator of the maximum temperature at which it was fired and the sample needs further investigation.

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	

Table 2 shows that mullite was not detected in the samples of drenei ceramics by XRD analysis. The absence of this mineral is also one of the factors used to determine the firing temperature of ancient ceramics since it is formed during the high-temperature firing of kaolinite. It can serve as an indicator of the maximum firing temperature reached during the production of ancient ceramics. The phase transformation of kaolinite into mullite was investigated in [23][4][5] and it was shown that kaolinite transforms into mullite at a temperature of about 940°C. During firing, mullite, cristobalite, and hematite are formed, associated with helenite and anorthite at temperatures above 1150°C. Further increase in temperature leads to the formation of a vitreous phase along with the development of hematite, resulting in decreased porosity and water adsorption [18]. Consequently, we can say with a high degree of certainty that the samples of ancient ceramics were made at temperatures below 950°C.

At temperatures above 600°C, montmorillonite transforms into an amorphous phase [17] and can be used as a reference for determining the minimum temperature at which ceramics can be fired.

The iron-bearing material was also confirmed by X-ray diffraction (Fig. 4) and TGA analysis. Differential thermal curves of maghemite show a prominent peak at 815°C. An X-ray diffraction (XRD) study identified the maghemite peak (PDF 01-076-4113) [25], however, this fact means little to determine the maximum firing temperature of the ancient pottery.

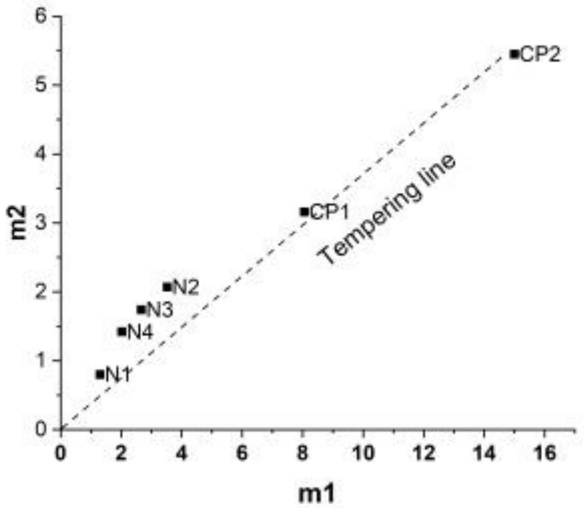

Fig. 5. Mass-loss diagram for the ancient ceramic samples and raw ceramic pastes.

Mass loss is one of the main parameters of TG studies and the authors of (15) developed a diagram to clarify the differences in thermogravimetric data for ancient ceramic samples. This method allows us to graphically represent the dependence of mass loss on the degree of heat treatment. Figure 5 presents the mass loss diagram is presented in Figure 5 for the samples given in Table 1. The figure shows that the mass loss coefficients of ancient ceramic samples correlate with the mass loss coefficients of raw ceramic pastes. Based on the diagram, it can be assumed that the studied samples gradually recovered their original hydroxyl layer while in the ground.

4. Conclusions

Using thermogravimetry (TG) and X-ray diffraction (XRD) techniques, comprehensive assessments of ancient ceramics were conducted, including their mineralogical, chemical, and thermal properties. X-ray diffraction analysis of raw ceramic paste and ancient ceramics revealed that all examined samples consisted of comparable minerals, including quartz, feldspar, and clay minerals. Although only three of the four ancient pottery samples contain calcite, it is assumed that the firing process in all four of these samples ceased when the temperature reached 700°C.

TG analysis shows that reversible hydroxylation occurred with time in all four samples, indicating initial gentle firing conditions. The use of different interdisciplinary methods and the results of the analysis suggest that the ceramic samples were manufactured using a comparable production process.

X-ray diffraction (XRD) studies indicate the presence of minerals such as diopside and maghemite in the ceramics, suggesting that the origin of the ceramic samples studied was quite different.

This research represents the first comprehensive study of ceramic objects found by archaeologists in the territory of the Republic of Azerbaijan. Therefore, it can be asserted that this study introduces a novel perspective to the existing literature on the subject.

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УСЛОВИЯ ОБЖИГА И ТЕХНОЛОГИЯ ИЗГОТОВЛЕНИЯ ДРЕВНЕЙ КЕРАМИКИ

А.С. Ахадова, А.Б. Ахадов, А.З. Абышов, М.А. Гурбанов, С.Г. Мамедов

Резюме: В процессе обжига минеральные компоненты керамики испытывают фазовые переходы и изменения химического состава, и эти процессы зависят от температуры, при которой они обжигаются. Комплексный анализ древней керамики часто включает изучение ее минералогических, химических и термических характеристик. Анализируя данные с помощью междисциплинарного подхода с использованием методов термогравиметрии (ТГ), термолюминесценции (ТЛ), рентгеновской дифракции (РДА) и рентгеновской флуоресценции (РФ) и учитывая археологический контекст, исследователи могут сделать важные выводы о происхождении керамики и использованных древних технологиях. Анализ рентгеновской флуоресценции (РФ) дает информацию о химическом составе древней керамики и эффективен для классификации керамики на основе схожего состава, в то время как термический анализ и порошковая рентгеновская дифракция (ПРД) обычно используются для определения первоначальной температуры обжига древней керамики.

Ключевые слова: термический анализ, рентгеновская дифракция, глины, древняя керамика, температура обжига.

QƏDİM KERAMİKA MƏMULATLARININ BİŞİRİLMƏ ŞƏRAİTİ VƏ İSTEHSAL TEXNOLOGİYASI

A.S. Əhədova, Ə.B. Əhədov, Ə.Z. Abışov, M.A. Qurbanov, S.Q. Məmmədov

Xülasə: Yandırma prosesi zamanı keramikanın mineral komponentləri faza keçidlərinə və kimyəvi tərkibində dəyişikliklərə məruz qalır və bu proseslər onların bişirildiyi temperaturdan asılıdır. Qədim keramikaların hərtərəfli təhlili çox vaxt onların mineraloji, kimyəvi və termiki xüsusiyyətlərinin öyrənilməsini nəzərdə tutur. Termoqravimetriya (TG) termolüminesans (TL) rentgen difraksiyası (XRD) və rentgen flüoresans (XRF) üsullarından istifadə edərək məlumatları fənlərarası yanaşma vasitəsilə təhlil edərək və arxeoloji konteksti nəzərə alaraq tədqiqatçılar keramika və istifadə edilən qədim texnologiyaların mənşəyi haqqında mühüm nəticələr çıxara bilərlər. rentgen-flüoressens (XRF) analizi qədim keramikaların kimyəvi tərkibi haqqında məlumat verir və keramikaları oxşar tərkibə əsasən təsnif etmək üçün effektivdir, termal analiz və rentgen difraksiyası (PXRD) isə qədim keramikaların orijinal yanma temperaturunu təyin etmək üçün adətən istifadə olunur.

Açar sözlər: termal analiz, rentgen şüalarının difraksiyası, gillər, qədim dulusçuluq, yanma temperaturu.