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FIRING CONDITIONS, MINERALOGICAL COMPOSITION AND MICROSTRUCTURAL CHARACTERISTICS OF PREHISTORIC CERAMICS FROM AZERBAIJAN

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The aim of this study is to investigate the firing conditions, mineral assemblage and microstructural characteristics of prehistoric ceramics from Azerbaijan using an integrated archaeometric approach. Twelve ceramic fragments from six Neolithic and Chalcolithic archaeological sites were selected as case-study samples intended to demonstrate the applicability of integrated archaeometric methods rather than to provide a statistically representative characterization of prehistoric pottery in Azerbaijan. The scientific novelty of the study lies in the integrated archaeometric comparison of ceramic materials from multiple archaeological sites through the combined application of thermal analysis (TG/DTG/DTA), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results indicate that the ceramics were fired at relatively moderate temperatures, generally around 700 °C. The ceramic matrix is dominated by quartz and feldspar phases (predominantly albite), whereas the preservation of calcite suggests incomplete thermal decomposition. Thermal analysis revealed characteristic transformation stages, and SEM observations demonstrated the preservation of lamellar structures in the archaeological samples and their modification after experimental heating. The analyzed samples display broadly similar thermal and mineralogical characteristics, although the limited dataset does not permit broader conclusions regarding regional ceramic production traditions.

Keywords: Pottery; Archaeometry; Prehistoric; Archaeology; South Caucasus

УСЛОВИЯ ОБЖИГА, МИНЕРАЛОГИЧЕСКИЙ СОСТАВ И МИКРОСТРУКТУРНЫЕ ХАРАКТЕРИСТИКИ ДОИСТОРИЧЕСКОЙ КЕРАМИКИ ИЗ АЗЕРБАЙДЖАНА

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Целью настоящего исследования является изучение условий обжига, минерального состава и микроструктурных особенностей доисторической керамики Азербайджана с использованием комплексного археометрического подхода. В качестве исследовательских образцов были отобраны двенадцать керамических фрагментов из шести неолитических и энеолитических археологических памятников. Данные образцы рассматриваются как материалы для сравнительного исследования, направленного на демонстрацию возможностей комплексных археометрических методов, а не как статистически репрезентативная выборка неолитической и энеолитической керамики Азербайджана. Научная новизна работы заключается в комплексном сравнительном археометрическом исследовании керамических материалов из нескольких археологических памятников на основе совместного применения методов термического анализа (TG/DTG/DTA), рентгенодифракционного анализа (XRD) и сканирующей электронной микроскопии (SEM). Полученные результаты показывают, что керамика обжигалась при относительно умеренных температурах, преимущественно около 700 °C. Керамическая масса характеризуется преобладанием кварца и полевых шпатов (главным образом альбита), тогда как сохранность кальцита свидетельствует о неполном термическом разложении карбонатной составляющей. Термический анализ выявил характерные стадии минералогических преобразований, а результаты SEM показали сохранение пластинчатых структур в археологических образцах и их изменение после экспериментального нагрева. Исследованные образцы демонстрируют в целом сходные термические и минералогические характеристики, однако ограниченный объем выборки не позволяет делать более широкие выводы о региональных традициях производства керамики.

Ключевые слова: Керамика; Археометрия; Доисторический период; Археология; Южный Кавказ

Introduction

The study of prehistoric ceramics plays a crucial role in evaluating technological traditions, economic practices, and cultural interactions of early agricultural societies. In the South Caucasus (Kohl, Trifonov, 2014, p. 1575-1580),

particularly in Azerbaijan, Neolithic (Maynard et al., 2022) and Eneolithic settlements such as Shomutapa, Hasansu, Goytapa (Alakbarov, 2018), Hajialamkhanli (Taylor, Maynard, 2011), Mentesh tapa (Lyonnet et al., 2016, p. 172-178) and Ismayilbaytapa (Herrscher et al., 2018) represent important archaeological sites associated with early sedentary communities (Nishiaki et al., 2015).

The investigated sites are associated with early farming communities in the Kura River basin (Lyonnet, Guliyev, 2010). These settlements represent important centers of early sedentary life in the South Caucasus (Akhundov et al., 2019, p. 23-29), where ceramic production played a key role in daily activities such as food preparation and storage. These sites provide important archaeological context for interpreting the firing practices and material characteristics of ceramic production in prehistoric Azerbaijan.

Research has mostly concentrated on typology and chronology despite the large-scale archaeological excavations carried out in Azerbaijan; the technological elements of pottery manufacture have not received enough attention. The physicochemical characteristics of ceramics, the choice of raw materials, and fire conditions have all received little consideration. To better understand the characteristics of historical pottery manufacture in this area, an integrated archaeometric approach is therefore necessary. Figure 1 displays the geographic distribution of the locations under study.

The Baku–Tbilisi–Ceyhan (BTC) oil pipeline project played a significant role in advancing archaeological research in Azerbaijan. Between 2001 and 2011, large-scale salvage excavations along the pipeline corridor led to the discovery of numerous prehistoric sites and the recovery of ceramic materials. The samples analyzed in this study were obtained within the framework of these BTC-funded archaeological investigations.

Archaeometry, as an interdisciplinary field combining methods of physics, chemistry, and archaeology, provides powerful tools for investigating ceramic materials. In particular, thermal analysis and X-ray diffraction allow the identification of mineral phases (Maggetti, 1982), evaluation of thermal transformations, and estimation of firing temperatures (Moropoulou, 1995). SEM microstructural analysis, together with thermal analysis and X-ray diffraction, offers valuable information on the structural

development of ceramic materials during firing. SEM analysis allows direct observation of features such as lamellar clay structures, pore development, and sintering effects, which are essential for interpreting thermal transformations and validating results obtained from TG/DTG/DTA and XRD analyses.

Therefore, the integration of thermal analysis (TG/DTG/DTA), X-ray diffraction, and SEM microstructural analysis provides a useful approach for assessing firing conditions and selected technological parameters of prehistoric ceramics.

The purpose of this study is to investigate the thermal, X-ray diffraction, and microstructural properties of twelve ancient pottery fragments from six Azerbaijani archaeological sites. This study contributes additional archaeometric data for the investigation of prehistoric ceramics in the South Caucasus.

Archaeological sites

The *Shomutapa* settlement, situated in the vicinity of the Aghstafa railway station, represents one of the key Neolithic sites of the South Caucasus and is dated to the sixth–fourth millennia BC. The site was extensively investigated between 1960 and 1964 under the direction of the prominent Azerbaijani archaeologist I.G. Narimanov (Narimanov, 1987). Covering an area of approximately 1 ha, the cultural deposit reaches a maximum thickness of 1.5 m.

Archaeological excavations revealed the remains of circular domestic structures constructed from mudbrick and coated with clay plaster. These dwellings generally range between 2 and 4 m in diameter. In addition, smaller circular utility buildings measuring 1–1.5 m in diameter were identified within the settlement. Hearth installations attached to interior walls, as well as within adjacent open spaces, indicate the organization of domestic activities. The material assemblage recovered from the site includes a wide range of artifacts manufactured from clay, stone, and bone. Ceramic production is represented primarily by simple, flat-based vessels formed from clay tempered predominantly with sand and, to a lesser extent, with vegetal inclusions such as straw. Ground stone implements, including querns, grinders, and mortars, were recovered alongside polished stone axes, blade-like lithic tools, scrapers, perforators, and other utilitarian objects. Particularly noteworthy are



Fig. 1. Location of the studied archaeological sites in Azerbaijan in relation to the Baku–Tbilisi–Ceyhan (BTC) pipeline.

Рис. 1. Расположение исследованных археологических памятников Азербайджана относительно трубопровода Баку–Тбилиси–Джейхан (BTC).

agricultural implements such as primitive sickles composed of bone or wooden handles fitted with flint and obsidian inserts. Bone and antler hoes, numerous awls, spoons, and anthropomorphic figurines further illustrate the technological and cultural practices of the settlement's inhabitants. Archaeobotanical evidence has yielded remains of barley, as well as both free-threshing and hulled wheat varieties, while faunal assemblages include domesticated cattle, sheep and goats, pigs, and various wild animal species.

According to archaeological interpretations, Shomutapa, together with contemporaneous settlements distributed across the Ganja–Gazakh plain and the Marneuli plain of Georgia, represents an early stage in the development of farming and animal husbandry in the South Caucasus. The ceramic assemblage characteristic of the Shomutapa cultural tradition consists predominantly of handmade, brown to greyish vessels with flat bases, simple forms, and an absence of handles. One pottery sherd recovered from the site was selected for the present work (Fig. 2). The analyzed fragment belongs to a ceramic tradition in which some vessels were produced using clay tempered with chopped straw, whereas the majority contain sand, crushed stone, and grog inclusions. Decorative treatment includes applied plastic ornamentation and, in certain cases, painted motifs executed in red pigment. The subsistence economy of the Shomutapa population was based primarily on agriculture

and animal husbandry, supplemented by hunting and fishing activities. Archaeobotanical investigations conducted at Shomutapa cultural sites have identified remains of cultivated or wild barley, wheat, and grape seeds, providing valuable evidence for the economic practices of early agricultural communities in the region.

Menteshtapa is a multilayered archaeological settlement located in the Tovuz region of western Azerbaijan and represents one of the most significant prehistoric sites investigated in recent decades. Between 2008 and 2013, a joint Azerbaijani–French archaeological mission conducted extensive excavations at the site, revealing a long sequence of occupation extending from the Neolithic to the Early Bronze Age (Lyonnet et al., 2016). The investigations identified three major occupational phases, documenting nearly four millennia of cultural development in the region.

Radiocarbon dating indicates that the earliest phase of settlement corresponds to the Late Neolithic period, approximately 5800–5700 BC. This occupation level is characterized by circular architectural structures constructed from mudbrick. The associated ceramic assemblage consists mainly of plant-tempered wares, including both burnished and painted pottery. Although the material culture recovered from this horizon is relatively limited in quantity, several discoveries are of particular archaeological significance. Among these are a copper-based

metal object and fragments of azurite recovered from one of the circular buildings, providing valuable evidence for the early use of mineral resources and metallurgical knowledge in the South Caucasus.

One of the most remarkable discoveries at Mentesh tapa derives from the Late Neolithic cultural deposits, where a collective burial pit containing the remains of 38 individuals was uncovered. Juvenile skeletons constitute a substantial proportion of the assemblage, making this feature particularly important for studies of prehistoric demography and mortuary practices.

For the present archaeometric investigation, a pottery fragment attributed to the Chalcolithic occupation of the site was selected for analysis (Fig. 2). The specimen originates from the collections of the Institute of Archaeology and Anthropology of the Azerbaijan National Academy of Sciences, where the archaeological materials recovered from Mentesh tapa are currently curated.

Ismailbaytapa is a Neolithic settlement located northeast of Khindiristan village in the Aghdam district of Azerbaijan, approximately 250 m west of the Guzanli–Khindiristan highway. The site consists of a mound measuring approximately 100 m in diameter and 4 m in height. Its eastern, southern, and western margins have been partially affected by irrigation channels; however, apart from minor disturbances, the mound remains relatively well preserved.

Archaeological investigations commenced in 2012 with the excavation of a 100 m² area on the eastern slope of the mound. Between 2012 and 2016, the excavation area was expanded to 400 m², encompassing four excavation squares. These investigations led to the identification and study of fifteen architectural features belonging to four successive construction phases, providing important insights into the occupational history of the settlement. Surface surveys and excavations yielded exclusively ceramic materials associated with the Karabakh Neolithic cultural tradition. The pottery assemblage is characterized predominantly by reddish fabrics, with both surfaces exhibiting varying degrees of firing and thermal alteration. Several sherds display red-ochre and chestnut-colored hues. Among the recovered materials are fragments of loop handles, pottery sherds bearing wedge-shaped incisions, and vessels decorated with impressed and incised ornamental motifs. The ceramic

specimen selected for the present archaeometric study belongs to a storage vessel recovered from Ismayilbeytepe (Fig. 2). This sample was chosen as representative of the Neolithic ceramic tradition of the settlement and was subjected to detailed mineralogical and technological analyses.

In 2005, during the construction of the Baku–Tbilisi–Ceyhan oil pipeline, a Neolithic kurgan-type burial monument was discovered on the left bank of the Hasansu River in the Agstafa region of Azerbaijan. The site derives its name, Hasansu, from the nearby river. Archaeological investigations previously conducted at the **Hasansu** settlement were continued in 2011 within Excavation Square VIII, which covered an area located near the central part of the settlement.

A sterile layer separating the cultural deposits into two distinct stratigraphic phases was clearly observed in Square VIII, consistent with earlier excavations. During the investigations, at a depth of approximately 1.5–1.7 m, the remains of a mudbrick structure were uncovered adjacent to the northern profile of the excavation trench. The exposed feature represented approximately one-half of a circular-plan room. Since this structure and several associated architectural fragments were constructed directly above the sterile layer, they can be attributed to the second occupational phase of the settlement. The burial practices documented within the kurgan, together with the ceramic, lithic, and metal artifacts, as well as various ornamental objects recovered during excavation, have been comprehensively analyzed and compared with analogous archaeological materials from other sites in the South Caucasus. These studies have contributed significantly to the understanding of Neolithic cultural traditions in the region. The ceramic fragment (Fig. 2) selected from the Hasansu assemblage for the present archaeometric investigation was identified as belonging to a domestic utilitarian ware used in everyday household activities.

Hajialamkhanli is a Neolithic settlement located approximately 8 km east of Tovuz city and 1.5 km northeast of the well-known Neolithic site of Goytepe in western Azerbaijan. The settlement is represented by a mound measuring approximately 60 m in width, 80 m in length, and 1.5 m in height. Archaeological investigations commenced in 2012 and were conducted in parallel with the excavations at Goytepe. Fieldwork continued during the 2013 season, providing important data on the Neolithic

occupation of the region. Excavations were carried out within a 10×10 m trench located near the center of the mound. These investigations revealed a Neolithic cultural layer approximately 1.5 m thick. Within this deposit, four architectural horizons containing mudbrick building remains were identified, indicating successive phases of occupation. Among the most notable discoveries was an architectural feature resembling the numeral “8”, uncovered in the central part of the settlement during investigations led by archaeologist Farhad Guliyev. Similar figure-eight-shaped structures were subsequently identified in other areas of the mound and within different construction horizons, suggesting that this architectural form may have been a recurring element of the settlement’s building tradition. One of the excavated ovens was associated with a mudbrick partition wall. Its interior contained substantial ash deposits, numerous charcoal fragments, and burned stone and bone remains. Archaeological evidence demonstrated that this oven had been constructed directly above an earlier oven, indicating continuity in the use of domestic space. Another oven identified in the central sector of the settlement was surrounded by four small circular postholes of similar diameter (13–17 cm). These features have been interpreted as post supports associated with the roof structure of a dwelling. To the west of the oven, two pits and a storage installation were discovered adjacent to a wall. These clay-lined pits, measuring approximately 15–20 cm in depth, are thought to have functioned as storage facilities. In addition to the central oven, two further hearths were documented within the excavation area. Unlike the storage pits and architectural features, these hearths did not exhibit a direct structural association with surrounding buildings. Nevertheless, animal bones and river pebbles were recovered from their vicinity. The artifact assemblage includes several noteworthy finds, particularly an obsidian core, numerous bone awls, and sickle elements secured with bitumen. Collectively, the archaeological evidence demonstrates a long-term and structured pattern of habitation at Hajialamkhanli. The architectural remains exposed during excavation are of considerable chronological significance, representing some of the earliest stages of Neolithic settlement development in the South Caucasus. The buildings constructed at the beginning of the sixth millennium BC provide important

evidence for understanding the emergence and development of early farming communities in the region. A ceramic sherd recovered from the site was selected for laboratory-based archaeometric analysis in the present study (Fig. 2).

Goytapa, dated to the sixth millennium BC, is one of the largest and most significant Neolithic archaeological complexes in the South Caucasus (Nishiaki et al., 2015). The site is located in the Tovuz district of western Azerbaijan, approximately 10 km east of the district center and 340 m from the Baku–Gazakh highway. Since 2008, extensive archaeological investigations have been carried out through an international collaboration involving Azerbaijani, Japanese, and French researchers. The settlement mound measures approximately 145 m in diameter and 9 m in height. Under the direction of archaeologist Farhad Guliyev, the Goytapa Archaeological Expedition conducted systematic excavations over an eight-year period, investigating an area of nearly 1000 m². During the 2013–2014 field seasons, excavations in the northeastern sector of the mound reached the natural subsoil, allowing the complete stratigraphic sequence to be documented. As a result of these investigations, fourteen architectural horizons were identified, providing an exceptionally detailed record of Neolithic occupation and settlement development.

Archaeological evidence demonstrates that Goytapa represents an advanced stage in the development of Neolithic communities in the South Caucasus. Excavations revealed numerous mudbrick architectural complexes, including both large and small circular structures, interconnected residential units, projecting walls, hearth installations, and associated domestic features. Smaller circular buildings were connected to larger structures by substantial walls, forming enclosed courtyard spaces. The arrangement of these architectural units into small neighborhood-like clusters reflects important social and economic transformations within Neolithic society. The architectural layout, spatial organization, and stratigraphic continuity documented at Goytapa provide valuable insights into patterns of social organization and community development during the Neolithic period. Detailed analysis of the stratigraphic sequence has also contributed significantly to the understanding of Neolithic architectural traditions and settlement dynamics in the South Caucasus. The excavations yielded a rich assemblage of

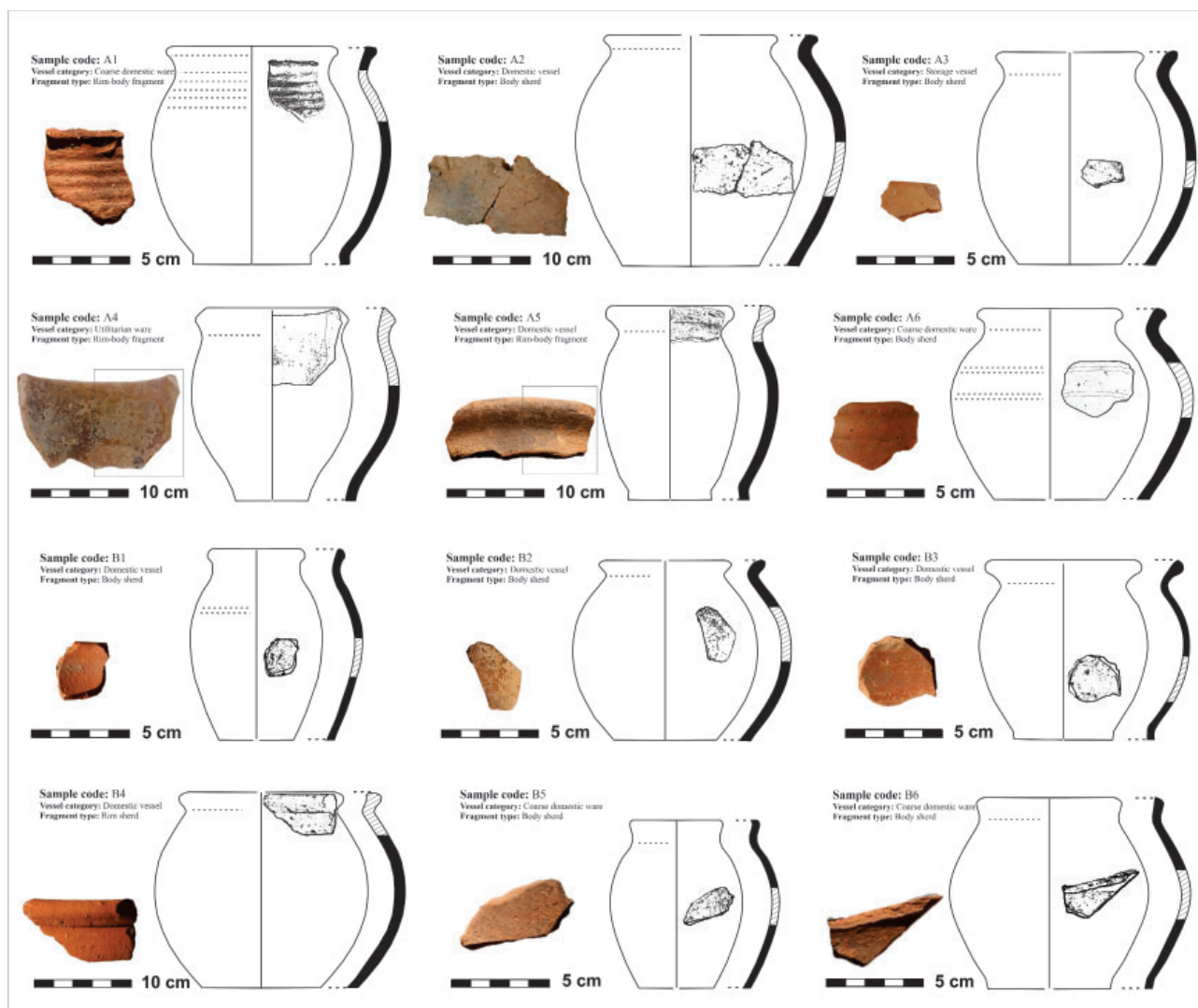


Fig. 2. Archaeological ceramic fragments selected for archaeometric analyses and their schematic evaluation (the highlighted areas indicate the approximate position of the analyzed fragments within the reconstructed vessel profiles). Shomutapa – A1, B1; Menteshapa – A2, B2; Ismailbaytapa – A3, B3; Hasansu – A4, B4; Hajialamkhanli – A5, B5 and Goytapa – A6, B6.

Рис. 2. Археологические фрагменты керамики, отобранные для археометрического анализа, и их схематическая оценка (выделенные области указывают приблизительное положение анализируемых фрагментов в пределах реконструированных профилей сосудов). Шомутапа – A1, B1; Ментештапа – A2, B2; Исмаилбайтапа – A3, B3; Хасансу – A4, B4; Хаджияламханли – A5, B5 и Гойтапа – A6, B6.

material culture, including finely crafted stone and bone tools, implements, and weapons, all of which have considerable scientific importance for evaluating technological and economic practices of early farming communities. The exceptional preservation and diversity of archaeological remains have established Goytapa as a key reference site for the study of the Neolithic in the region. In April 2012, the President of the Republic of Azerbaijan, Ilham Aliyev, visited the site and reviewed the ongoing archaeological investigations. Subsequently, a special decree was issued concerning the establishment of the

Goytapa Archaeological Park, recognizing the outstanding cultural and scientific significance of the monument. Numerous ceramic fragments recovered during the excavations are currently preserved in the collections of the Institute of Archaeology and Anthropology. The pottery sherd selected for the present archaeometric study was obtained from this collection (Fig. 2).

Literature Overview

In recent decades, archaeometric approaches have become an essential tool for investigating prehistoric ceramic production technologies. To ascertain the mineralogical composition,

detect phase transitions, and calculate the firing temperatures of ancient ceramics, analytical methods like thermal analysis and XRD are frequently employed (Cultrone et al, 2001).

According to earlier research, mineralogical markers like muscovite and calcite are crucial for recreating fire conditions. While the presence of muscovite indicates insufficient thermal transformation of clay minerals, the survival of calcite in ceramic matrices is frequently linked to relatively low firing temperatures, usually below 750–800 °C. Quartz and feldspar are common constituents of ceramic fabrics and may derive from either the natural phase composition of clays or from added inclusions; however, distinguishing between these possibilities requires additional petrographic investigation.

Early ceramic production was frequently characterized by moderate firing temperatures and variable mineralogical compositions reflecting different geological environments, according to research done in areas like Anatolia (Sagona, 2004), the Near East (Kingery, 1988; Maniatis, 1981), and Europe (Tite, 1975) (Korolev, 2024; Gusentsova, 2024; Mosin, 2024; Kolpakov et al., 2024; Kirichenko, 2022; Kirichenko, 2023). These investigations highlight how crucial it is to combine several analytical techniques in order to produce accurate interpretations of ceramic mineral assemblage (Quinn, 2013).

Although archaeometric studies of prehistoric ceramics have been conducted in Anatolia, the Near East and Eastern Europe (Rathossi, Pontikes, 2010), comparable integrated TG/DTG/DTA, XRD and SEM investigations remain extremely limited for Neolithic and Chalcolithic ceramics of Azerbaijan. Therefore, the present study provides a comparative dataset linking thermal behaviour, mineralogical composition and microstructural observations for prehistoric ceramics from several Azerbaijani archaeological sites.

Archaeological studies in the South Caucasus have yielded important details on the distribution, type, and dating of Neolithic and Eneolithic pottery. Nevertheless, there are still very few in-depth archaeometric studies that concentrate on the physicochemical characteristics and manufacturing processes of ceramics in Azerbaijan. Instead of focusing on material composition and firing circumstances, the majority of current research has focused on cultural and stylistic elements.

To better understand ancient pottery manufacture in this area, integrated research combining thermal and mineralogical data are obviously needed (Bakhmatova, 2018). By using integrated TG/DTG/DTA, XRD, and SEM techniques on ceramic materials from many archaeological sites in Azerbaijan, the current work contributes new data on firing conditions, mineral assemblage and microstructural characteristics of prehistoric ceramics from Azerbaijan.

The significance of integrating microstructural analysis with thermal and mineralogical techniques has been highlighted by recent archaeometric research. SEM studies have shown that vitrification characteristics, pore morphology, and the preservation or collapse of lamellar clay structures may be used as accurate markers of firing temperature and thermal behaviour procedures. When it comes to finding incomplete mineral transformations and differentiating between low-fired and high-fired ceramics, the combination of SEM with TG/DTG/DTA and XRD investigations has been very successful. However, such combined approaches remain relatively limited in studies focused on prehistoric ceramics of the South Caucasus region (Ashley, 2022), highlighting the significance of the present work (Agalarov, Guliyev, 2025).

The current work is not typical since it applies these complimentary techniques to ceramic samples from many archaeological sites, offering a comparative dataset on microstructural features, XRD-derived mineral phases, and fire circumstances.

Methodology

The analyzed ceramic samples were obtained from several well-known Neolithic and Eneolithic archaeological sites in Azerbaijan and were provided for analysis by Dr. Farhad Guliyev, Director of the Institute of Archaeology and Anthropology of the Azerbaijan National Academy of Sciences. The analyses were carried out by the author at the Institute of Physics of the Ministry of Science and Education of the Republic of Azerbaijan.

The analyzed ceramic fragments originate from six archaeological sites located within the Kura River basin and investigated during the Baku–Tbilisi–Ceyhan (BTC) pipeline archaeological project. Shomutapa, Hasansu, Goytapa and Hajialamkhanli are generally attributed to the Early Neolithic period and are

Table 1. Archaeological characteristics of analyzed ceramic samples
Таблица 1. Археологические характеристики проанализированных образцов керамики.

Sample	Site	Period	Cultural affiliation	Context	Vessel category	Fragment type
A1	Shomutapa	Early Neo-lithic	Shomutapa-Shulaveri	habitation layer	Coarse domestic ware	Rim-body fragment
B1	Shomutapa	Early Neo-lithic	Shomutapa-Shulaveri	habitation layer	Domestic vessel	Body sherd
A2	Menteshtapa	Chalcolithic	Chalcolithic horizon	habitation layer	Domestic vessel	Body sherd
B2	Menteshtapa	Chalcolithic	Chalcolithic horizon	habitation layer	Domestic vessel	Body sherd
A3	Ismailbaytapa	Neolithic	Karabakh Neolithic	habitation layer	Storage vessel	Body sherd
B3	Ismailbaytapa	Neolithic	Karabakh Neolithic	habitation layer	Domestic vessel	Body sherd
A4	Hasansu	Neolithic	Shomutepe-Shulaveri	habitation layer	Utilitarian ware	Rim-body fragment
B4	Hasansu	Neolithic	Shomutepe-Shulaveri	habitation layer	Domestic vessel	Rim sherd
A5	Hajialamkhanli	Neolithic	Shomutepe-Shulaveri	habitation layer	Domestic vessel	Rim-body fragment
B5	Hajialamkhan	Neolithic	Shomutepe-Shulaveri	habitation layer	Coarse domestic ware	Body sherd
A6	Goytapa	Early Neo-lithic	Shomutepe-Shulaveri	habitation layer	Coarse domestic ware	Body sherd
B6	Goytapa	Early Neo-lithic	Shomutepe-Shulaveri	habitation layer	Coarse domestic ware	Body sherd

associated with the Shomutapa-Shulaveri cultural tradition, while Menteshtapa and Ismailbaytapa belong to the Late Neolithic–Eneolithic horizon. The selected samples represent ordinary domestic pottery and consist mainly of body sherds and small vessel fragments recovered from habitation contexts (Table 1). Due to the fragmentary state of preservation, complete vessel reconstruction was not possible; however, the samples were selected as representative ceramic fabrics from each archaeological site. Rather than offering a statistically representative description of Neolithic and Chalcolithic pottery in Azerbaijan, twelve ceramic fragments (Fig. 2) were chosen as case-study samples to illustrate the usefulness of integrated archaeometric techniques.

The purpose of the investigation was not to establish a comprehensive technological classification of Neolithic and Chalcolithic pottery in the region, but rather to evaluate the applicability of integrated archaeometric methods (TG/DTG/DTA, XRD and SEM) for the characterization of selected ceramic materials

from different archaeological contexts. Therefore, the results should be considered as a preliminary comparative dataset rather than a statistically representative assessment of prehistoric ceramic production in Azerbaijan.

Prior to examination, the samples were air-dried overnight at 50°C and ground into a fine powder using an agate mortar.

A D2Phaser (Bruker) diffractometer with Ni-filtered CuK α radiation ($\lambda = 1.5406 \text{ \AA}$) was used for XRD/PXRD (Zhou, 2018) on randomly oriented samples. Diffraction data were collected in the 2θ range of 5°–75° with a scanning rate of 1.2°·min⁻¹. Based on the XRD data, semi-quantitative estimations of the mineral phases were obtained (Rice, 2015). To evaluate the amount of amorphous material present in the samples, the area of the broad background hump representing the amorphous phase was compared with the corresponding areas obtained from standard mixtures containing known proportions of natural amorphous materials. These standards were analyzed under identical scanning conditions. Using this approach, semi-

quantitative estimates of the total amorphous content were calculated.

The semi-quantitative evaluation considered the intensity of characteristic diffraction peaks, sample density, and the mass absorption coefficients of the elements for CuK α radiation. Mineral phase identification was performed using DIFFRAC.SUITE software, together with the TOPAS program for non-standard quantitative and structural analysis, and the ICDD Powder Diffraction Database.

The relative abundance of mineral phases was estimated using the Reference Intensity Ratio (RIR) method, a widely applied approach for quantitative phase analysis in powder X-ray diffraction. In this study, experimentally determined laboratory RIR values were used for the calculations.

Thermogravimetric (TG) (Mammadov, Ahadova, 2023) and differential thermal analyses (DTA) of the ceramic powders were carried out using a PerkinElmer STA 6000 Simultaneous Thermal Analyzer under the following conditions: heating from ambient temperature to 950°C at a heating rate of 5 °C·min⁻¹, with a balance sensitivity of 0.1 µg and a nitrogen gas flow rate (Ahadova et al., 2024; Mammadov et al., 2023) of 20 mL·min⁻¹.

Micrographs were obtained under high-vacuum conditions using an accelerating voltage of 15 kV. Both secondary electron (SE) and backscattered electron (BSE) imaging modes were employed to observe surface morphology and compositional contrast. To evaluate thermal transformations, selected samples were additionally subjected to controlled laboratory heating at 900 °C for 1 hour. The microstructure of these experimentally heated samples was compared with that of the original archaeological specimens to assess changes related to dehydroxylation, carbonate decomposition, and sintering processes.

Results

XRD analysis.

The mineralogical composition and microstructural characteristics (Ahadov et al., 2024) of the studied ceramic samples provide important information on the mineral composition and firing conditions of the ceramics. The dominance of quartz (27.4–56.3%) reflects the mineral composition of the ceramic bodies (Table 2). For analytical purposes (Karmanov, 2024), the archaeological ceramic samples were assigned laboratory codes A1–A6 and B1–B6 corresponding to their respective archaeological sites.

Table 2. Mineral phase composition of ceramic samples based on XRD analysis
Таблица 2. Минеральный фазовый состав керамических образцов по данным XRD-анализа.

Sample Code	Sample Site	Quartz (%)	Albite (%)	Calcite (%)	Muscovite (%)	Other minerals (%)
A1	Shomutapa	42.4	28.9	16.2	11.5	1.0
B1	Shomutapa	40.8	30.1	15.8	11.9	1.4
A2	Menteshtapa	42.9	12.1	8.8	35.0	1.2
B2	Menteshtapa	41.3	13.4	8.1	35.8	1.4
A3	Ismailbaytapa	35.2	21.3	17.7	23.0	2.8
B3	Ismailbaytapa	33.9	22.8	16.2	24.3	2.8
A4	Hasansu	37.4	41.7	6.1	12.8	2.0
B4	Hasansu	38.6	40.2	6.8	12.4	2.0
A5	Hajialamkhanli	27.4	42.4	5.6	22.6	2.0
B5	Hajialamkhan	28.8	40.9	6.4	21.7	2.2
A6	Goytapa	56.3	40.7	1.9	0.0	1.1
B6	Goytapa	54.9	42.1	1.7	0.1	1.2

SEM was performed to investigate the microstructural characteristics of the ceramic samples. The analyses were carried out using a field-emission scanning electron microscope (FE-SEM). Prior to analysis, the samples were mounted on aluminum stubs and coated with a thin conductive layer to prevent surface charging.

Paired samples obtained from the same archaeological sites (A and B series) exhibited very similar mineralogical compositions and thermal characteristics.

TG/DTG/DTA analysis.

Because paired samples from the same archaeological sites exhibited nearly identical

thermal behaviour, only representative TG/DTG/DTA curves of samples A1–A6 are presented in Figure 3. Therefore, the detailed discussion focuses primarily on the representative A-series samples (whereas the B-series samples are presented in Table 2 to demonstrate intra-site consistency). According to the TG curves, the sample mass gradually decreases with increasing temperature. (Mammadov et al., 2025). The primary transformations take place in three distinct temperature ranges: 50–350 °C, 350–650 °C, and 650–850 °C.

A mass loss of 1.5–4.5% was observed in the first period (50–350 °C). Dehydration processes, including the removal of physically adsorbed and weakly bound water, are associated with this stage. A prominent DTG peak at around 120–150 °C and a substantial endothermic impact (Mammadov et al., 2025a) in the DTA curves, which show energy absorption connected to water release, support this.

The dehydroxylation of clay minerals can be attributed to for the lesser mass loss of 0.2–2.8% seen in the next interval (350–650 °C). This process, which corresponds to the structural disintegration of hydroxyl-bearing phases like muscovite, is reflected by a moderate DTG peak that is focused about 450–550 °C. The very small mass loss in this range indicates that the first fire process did not fully complete dehydroxylation.

A mass loss of 0.4 to 4.5% is seen in the next interval (650–850 °C), which is linked to the partial breakdown of carbonate phases, mainly calcite. The most noticeable DTG peak, which occurs between 680 and 720 °C, amply illustrates this. Samples differ in this peak's strength; A3 has the greatest reaction, which is consistent with its greater calcite concentration, while A2 has a somewhat weaker peak, which is indicative of its lower carbonate content in comparison to A1 and A3.

The overall mass loss for the archaeological samples (A1–A6) varies from 2.9 to 11.7%, which is indicative of variations in thermal stability and mineral composition. These findings are further supported by the DTA curves, which display additional thermal effects at higher temperatures that correspond to mineral transformations and a prominent endothermic peak at low temperatures. Minimal thermal changes are seen beyond around 750 °C, suggesting that most processes are finished below this temperature range.

SEM analysis.

SEM microstructural investigation provides independent evidence supporting these results for these temperature data (Fig. 4). The lamellar and layered morphology characteristic of clay minerals is well-preserved in the original archeological samples (a–f). In samples A1 (a), A2 (b), A4 (d), and A5 (e), stacked plate-like structures are clearly visible, indicating incomplete transformation of phyllosilicate phases such as muscovite. This microstructural preservation is consistent with the TG results, particularly the moderate mass loss in the 350–650 °C range, confirming that dehydroxylation was not fully achieved.

Sample A3 (c) displays a more heterogeneous and porous structure compared to the other samples, which correlates with its higher total mass loss (11.7%) and the strongest DTG peak near 700 °C. This reflects a higher content of carbonate phases and their partial decomposition (Ahadova et al., 2026). Sample A6 (f) shows localized structural disruption but still retains identifiable lamellar features, indicating limited thermal alteration.

In contrast, the samples heated experimentally at 900 °C for 1 hour (g–l) exhibit a significantly altered microstructure. The lamellar organization observed in the original samples is completely destroyed, and the structure becomes highly porous and disordered. Round pores and partially fused grain surfaces are observed in samples g (A1) and h (A2), suggesting advanced heat transformation and the beginning of sintering. The original samples lack these characteristics.

Due to the breakdown of carbonate phases and the release of CO₂, samples I (A3) and L (A6) exhibit significant pore formation and structural collapse. The significant DTG peak in the 650–850 °C range is closely correlated with this result. Samples j (A4) and k (A5) show partial densification and surface smoothing, indicating the start of vitrification processes at high temperatures.

The archaeological ceramics did not experience such advanced thermal alteration, as seen by the stark difference between the original and experimentally heated samples. The initial firing temperature did not exceed about 700°C, as evidenced by the retention of lamellar structures, the lack of considerable sintering or vitrification, and the absence of widespread pore development (Mammadov et al., 2024). The studied Neolithic pottery pieces were burned at a moderate

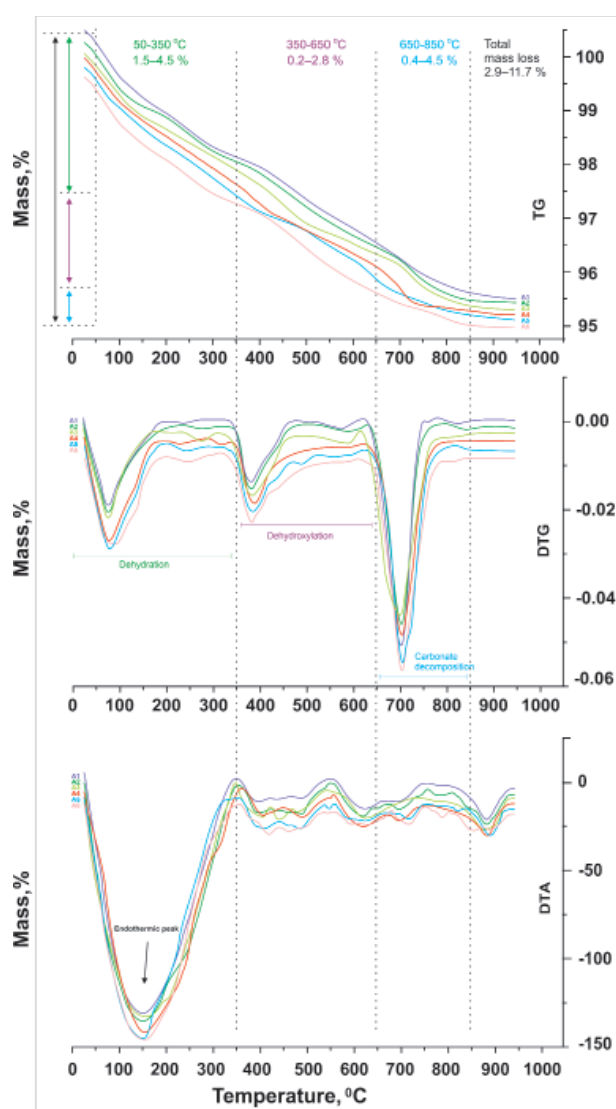


Fig. 3. TG/DTG/DTA curves of the archaeological ceramic samples (A1–A6)

Рис. 3. Кривые TG/DTG/DTA археологических керамических образцов (A1–A6).

temperature, most likely below 700°C, according to consistent and complimentary data from the combined TG/DTG/DTA and SEM studies. XRD data, which verify the existence of partly altered phases like calcite and muscovite, lend additional credence to this theory.

Discussion

The presence of feldspar minerals (albite) in significant amounts, particularly in samples A4–A6 and B4–B6, further is compatible with clay materials containing feldspar minerals or mixed sedimentary deposits. These minerals contribute to the structural stability observed in SEM micrographs and are consistent with the relatively low mass loss at higher temperatures in TG analysis.

Calcite is detected in all samples, with variable content ranging from 1.7% to 17.7%. Calcite may originate either from naturally calcareous clay deposits or from intentionally added carbonate-rich inclusions. The present dataset does not allow discrimination between these possibilities. The variation in calcite content among samples reflects differences in mineralogical composition; (Urbina et al., 1998) however, the available data do not allow determination of whether these differences are related to raw material provenance or ceramic preparation practices. The strong correlation between calcite content and the intensity of DTG (Molodin et al., 2019) peaks in the 650–850 °C range confirms that carbonate phases play a key role in the thermal behavior of the ceramics (Maritan, Mazzoli, Freestone, 2004). Muscovite, present in most samples (up to 35.8%), indicates the use of clay materials rich in phyllosilicates, likely derived from weathered metamorphic or sedimentary rocks. The preservation of muscovite in XRD and the lamellar structures observed in SEM images suggest incomplete thermal transformation, further supporting relatively low firing temperatures.

Mineralogical heterogeneity among the examined ceramic bodies is revealed by the combined mineralogical and thermal data. The variability in mineral composition between samples (e.g., high calcite in A3 versus lower amounts in B6) may reflect differences in ceramic paste composition; however, the available data do not allow determination of whether these differences result from raw material selection or ceramic preparation practices.

Furthermore, the absence of extensive vitrification and the preservation of primary clay structures indicate a relatively low-temperature firing, without controlled high-temperature conditions. This supports the interpretation that Neolithic ceramic production relied on low to moderate firing regimes. The obtained data primarily reflect firing conditions and mineralogical characteristics and do not permit a complete reconstruction of the entire ceramic production sequence.

XRD and SEM alone cannot be used to accurately identify deliberate tempering materials. To differentiate intentionally introduced temper from naturally occurring mineral inclusions, petrographic thin-section studies would be required. For the purpose of determining clay origin or deliberate temper additions,

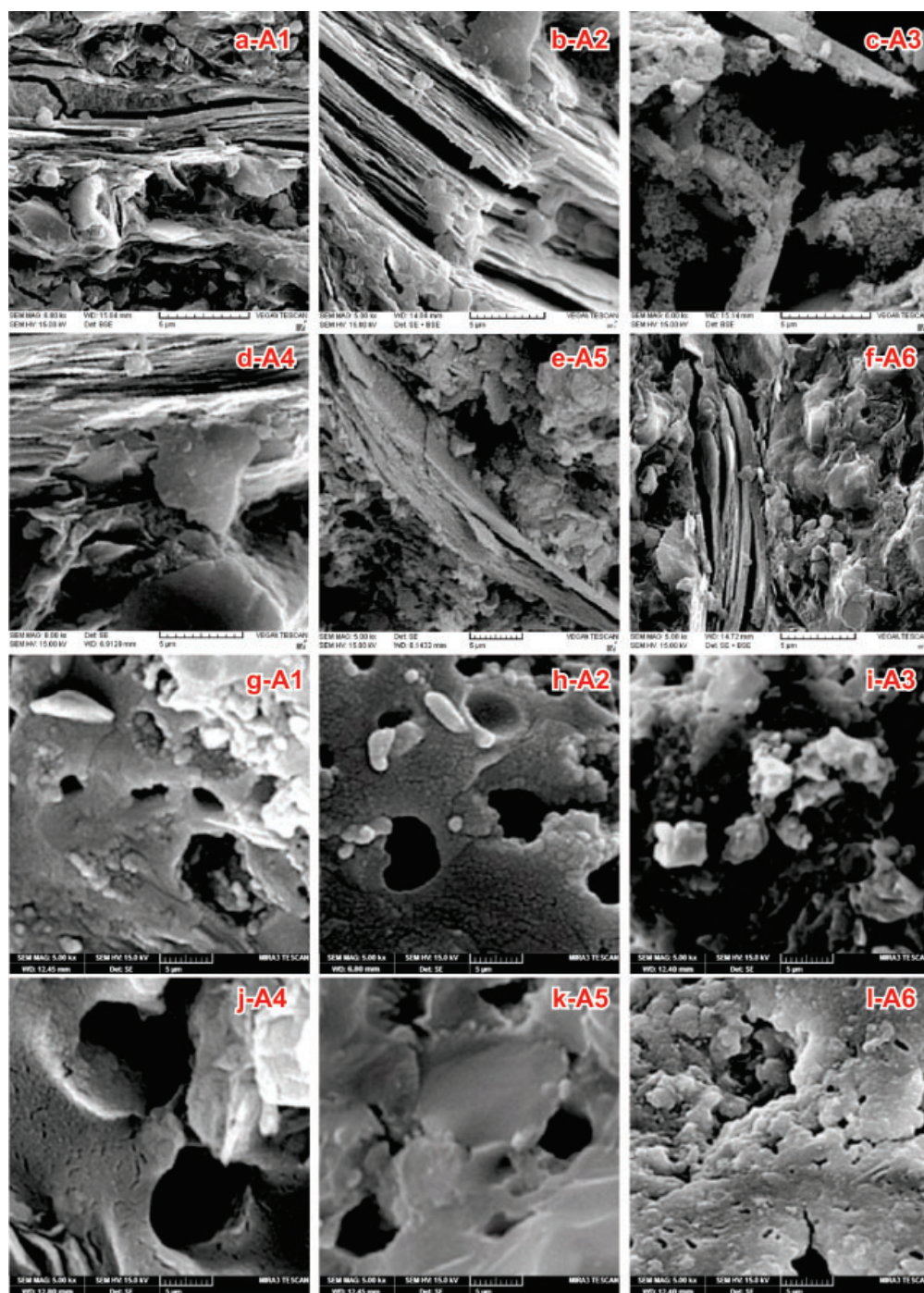


Fig. 4. FE-SEM micrographs of ceramic samples (A1–A6).

(a–f) Original archaeological samples showing layered (lamellar) clay structure;
(g–l) Same samples after controlled laboratory heating at 900 °C, showing collapse of lamellar morphology and development of porous structure.

Рис. 4. FE-SEM микрофотографии керамических образцов (A1–A6).

(a–f) Исходные археологические образцы, демонстрирующие слоистую (ламеллярную) структуру глины; (g–l) Те же образцы после контролируемого лабораторного нагрева при 900 °C, демонстрирующие разрушение ламеллярной морфологии и формирование пористой структуры.

mineralogical data are typically insufficient; petrographic investigations are typically needed (Quinn, 2013).

The lack of significant vitrification, the conservation of lamellar clay structures shown

by SEM, and the preservation of muscovite and calcite in a number of samples indicate that fire temperatures did not rise above around 700–750 °C (Livingstone, 2001). Similar archaeometric findings have been documented for Neolithic

pottery from the Near East and Anatolia, where similar mineralogical traces were formed by low-efficiency kiln systems or open fire.

Caution should be used when interpreting the presence of calcite. The information at hand does not permit distinguishing between these options since petrographic thin-section examination was not carried out. As a result, the current investigation is unable to ascertain whether carbonate phases are a reflection of ceramic preparation techniques or geological provenance. Similar findings of calcite preservation have been documented in low-fired ancient pottery from different areas (Moropoulou et al., 1995).

Since quartz and feldspar are often found in ceramic textiles, they cannot be used as direct markers of particular sources of raw materials. Rather than providing clear proof of local provenance, their presence in the examined samples mostly reflects the mineralogical makeup of the ceramic bodies.

The estimated firing temperatures obtained for the Azerbaijani samples fall within the range commonly reported for Neolithic ceramics from Anatolia, the Near East, and other regions of the South Caucasus. The present results complement archaeological investigations conducted at Goytepe and Monteshtepe (Nishiaki et al., 2015; Lyonnet et al., 2016). Previous archaeometric investigations have demonstrated that many early ceramic traditions were characterized by firing temperatures between approximately 600 and 800 °C, indicating broadly similar firing practices among early farming communities. Similar firing temperatures (600–800 °C) have been reported for prehistoric ceramics from the Near East and the Mediterranean region (Maniatis and Tite, 1981; Moropoulou et al., 1995; Cultrone et al., 2001).

Comparison between archaeological samples and experimentally heated specimens demonstrates that substantial microstructural reorganization occurs after exposure to 900 °C. The absence of such features in the archaeological ceramics indicates that the original firing process did not reach similar temperatures. The SEM observations therefore provide independent support for the firing temperature estimates derived from thermal analysis and XRD results.

Several limitations should be acknowledged. The study is based on only twelve ceramic fragments originating from different archaeological sites and cultural contexts. Consequently, the dataset should be regarded as exploratory rather than

statistically representative. In addition, the absence of petrographic analysis prevents detailed evaluation of temper composition, raw material provenance, and paste preparation techniques. Future studies involving larger sample sets and petrographic investigations will be necessary to address these questions.

Conclusions

The combined use of TG/DTG/DTA, XRD and SEM provides valuable information on firing conditions, mineral composition and microstructural characteristics of prehistoric ceramics. The consistency between thermal, X-ray diffraction, and microstructural data allows for a more reliable interpretation of firing conditions and transformation processes.

According to the thermal analysis, the primary physicochemical changes take place in three temperature ranges: carbonate breakdown (650–850 °C), dehydroxylation (350–650 °C), and dehydration (50–350 °C). When compared to contemporary ceramic paste, the relatively low total mass loss of the archaeological samples (2.9–11.7%) suggests that the materials had previously experienced some thermal alteration during their initial fire. Further evidence that these processes were not finished comes from the modest strength of DTG peaks in the carbonate decomposition and dehydroxylation ranges.

XRD data, which demonstrate the survival of mineral phases including muscovite and calcite in several samples, corroborate these conclusions. While the existence of calcite shows only partial decarbonation, the retention of muscovite indicates incomplete dehydroxylation of clay minerals. When taken as a whole, these characteristics indicate that the fire conditions were not high enough to fully convert the material.

These observations are further supported by direct microstructural data from SEM research. Limited structural rearrangement is indicated by the original samples' well-preserved lamellar and layered morphologies, which are typical of phyllosilicate minerals. On the other hand, samples that have been heated experimentally to 900 °C exhibit a collapse of the lamellar structure, increased porosity, and the beginning of sintering, which includes partial grain fusion and surface smoothing. The absence of such features in the archaeological specimens further supports the interpretation of relatively low firing temperatures.

Taken together, the evidence indicates that the ceramics were probably fired at moderate temperatures, usually not going over around 700 °C. Rather than controlled high-temperature firing, such firing conditions may reflect low-efficiency firing systems, perhaps employing open firing or low-efficiency kiln systems.

From the standpoint of raw materials, the analyzed ceramics display variability in mineralogical composition, particularly in the proportions of quartz, feldspar, calcite and muscovite. These differences may reflect variations in ceramic paste composition and raw material preparation. However, the available XRD and SEM data do not permit the identification of specific clay sources or the distinction between natural inclusions and intentionally added temper.

Despite these variations, the samples' microstructural traits, thermal behavior, and mineral makeup are generally consistent. The similarities observed among the analyzed samples may indicate comparable firing behaviour and mineralogical characteristics, but the small sample size prevents drawing more general conclusions about Azerbaijan's Neolithic and Chalcolithic pottery manufacture.

This study provides a comparative archaeometric dataset for prehistoric ceramics from Azerbaijan and demonstrates the applicability of integrated TG/DTG/DTA, XRD and SEM analyses for investigating firing technology in the South Caucasus.

The present study should be regarded as a preliminary archaeometric investigation based

on a limited number of ceramic fragments from different archaeological contexts. Future studies involving larger sample sets and additional analytical methods, including petrographic analysis, will be necessary to evaluate regional technological variability in greater detail.

Use of AI: During the preparation of the manuscript, the ChatGPT tool (developed by OpenAI; last accessed on [09 May 2026]) was used for specific tasks related to the linguistic processing of the scientific text. In particular, the tool was employed to correct grammatical errors, refine the syntactic structure of complex sentences, and adapt certain sections of the manuscript to a more standard academic writing style (primarily in the sections “Literature Overview,” descriptive parts of “Results and Discussion,” and “Conclusions”).

In several cases, the tool was also used to reformulate sentences originally written by the authors in order to reduce repetitive phrasing and improve the logical coherence of the text, without altering its scientific meaning. All original formulations, interpretations of the results of X-ray diffraction, X-ray fluorescence, and thermal analyses, as well as the final conclusions, were independently developed by the authors based on the conducted experimental research. Artificial intelligence tools were not used for the generation of scientific data, data analysis, or formulation of scientific conclusions. The authors confirm that the scientific content, data interpretation, and conclusions were produced independently.

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