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The origins of copper ores at Gre Fıılla (TÜRKİYE): Lead isotopic evidence for multi-source procurement☆

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ABSTRACT

Gre Fılla in southeastern Anatolia is a Pre-Pottery Neolithic site that provides critical insights into the emergence of metallurgy. Excavations at the site have revealed numerous copper ore fragments and copper objects, indicating an early engagement with metal production. Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) and lead isotope analysis (LIA) of copper ore samples from Gre Fılla demonstrates a multi-source procurement strategy. While the majority of samples exhibit isotopic signatures consistent with the Bolkar–Ergani group, defined by overlapping values from southeastern and central Anatolian deposits, some samples closely align with the Alihoca ore deposit. These results indicate that copper used at Gre Fılla came from both nearby and distant sources, reflecting the existence of complex and long-distance networks during the Pre-Pottery Neolithic B (PPNB). Additionally, the presence of various sources both for obsidian and copper highlights the community's integration into extensive systems of "exotic" material exchange. Such networks likely played a key role in fostering cultural connectivity across the Near East, contributing to the shared development of rituals, symbolic practices, and craft traditions observed among early Neolithic societies.

1. Introduction

The Near East plays a pivotal role in understanding the emergence of settled societies and the transition to agriculture around 12,000 BP. This region provides significant evidence regarding the development of food production and its spread to neighboring areas. The Neolithic transformation that began in the 12th millennium BP brought about radical changes in human history. Societies developed new belief systems, social dynamics became more complex, and notable developments in residential architecture took place. During the PPNA and PPNB periods, societies began transitioning from nomadic hunter-gatherer lifestyles to more complex settled communities. Though copper was not fully integrated into daily tool use, its increasing recognition as a valuable resource during this time marks the beginnings of metal experimentation (Rosenberg, 2007).

Archaeological findings from Gre Fılla and other Neolithic settlements like Hallan Çemi (Rosenberg and Redding, 2002), Çayönü, Nevali Çori and Çatalhöyük highlight early metallurgy's emergence. In Hallan Çemi, copper ore was found in semi-subterranean structures, indicating possible control over long-distance trade (Rosenberg, 2011; Rosenberg and Redding, 2002). In Çayönü, malachite and copper were extensively used for beads and tools. Copper objects in various stages of production, along with teardrop-shaped malachite beads with suspension holes, were found inside the buildings (Erim Özdoğan 2007; 2011). Additionally, in the Channeled Building subphase of Çayönü, copper beads began to outnumber malachite beads, and malachite inlays became common. Copper was beaten into flat pieces, and cylindrical beads were produced by twisting copper around a stick. Pins or unpierced needles mounted on wooden handles were also discovered. These were believed to be used for making the fine retouches of the Çayönü tool. Nevali Çori, another significant Neolithic site, produced a winged copper bead during the PPNB period. This bead was not hammered but reduced from copper ore in a manner indicative of early copper smelting techniques (Hauptmann 2007). Beads, ornaments, and textile weights made of native copper, uncovered in Levels VII and VI of the South Area at Çatalhöyük, indicate that copper was already being worked for ornamental purposes in the Near East during the Neolithic period (Birch et al, 2013).

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The identification of procurement methods for exotic raw materials—such as obsidian, native copper, malachite, and salt—and the communication networks shaped by these processes contributes significantly to the interpretation of material culture data and the understanding of the social and cultural structures of Neolithic settlements. The Near East provided raw materials such as minerals, ores, and obsidian, which enabled technological innovations and long-distance trade. These resources, combined with new ideas and technologies, helped establish the cultural and economic foundations of early civilizations. At Gre Fılla, geochemical analyses conducted on 50 obsidian artifacts recovered during the 2019 excavations revealed that the dominant obsidian sources during the PPNB phase were the Bingöl and Nemrut Dağ regions (Muşkara and Konak 2022). Research into the chaîne opératoire of the obsidian artifacts from both PPNB and PPNA layers, as well as raw material procurement strategies, is currently ongoing.

The provenance study to be conducted on copper ore samples recovered from Gre Fılla through lead (Pb) isotope analysis provides crucial data for understanding and interpreting the interactions between the local community and other regions in the acquisition of exotic materials. Communication established through the transfer of raw materials between distinct cultural zones often facilitates the transmission of cultural knowledge as well. In the literature, two primary models are widely accepted to explain raw material procurement: direct acquisition and exchange. Studies focusing particularly on obsidian sources during the Neolithic period suggest the existence of complex distribution networks.

The hypotheses underlying this study are as follows:

- The processing of metal ores during the Pre-Pottery Neolithic (PPN) period—prior to the advent of metallurgical activities—reflects the technological practices of the lithic industry and suggests a similar functional use to that of stone tools.
- Obsidian exchange may have encouraged the development of certain fixed transportation routes that later gained importance in the trade of copper and other exotic materials.
- Interactions and communication between communities are closely related to the settlement's position within exchange networks of strategically traded raw materials.
- The communication networks established through the procurement of exotic raw materials may have led to the emergence of actors with different roles or levels of expertise in the acquisition of obsidian and copper ores.
- The procurement strategies of obsidian at the settlement may have influenced the modes of copper ore acquisition.

The aim of this study is to investigate the origins and distribution pathways of copper during the Neolithic period, with the broader goal of elucidating patterns of technological innovation and cultural transformation in Anatolia. The results offer valuable insights into the emergence of early metallurgical knowledge and the establishment of long-distance exchange networks, highlighting the role of copper utilization in the socio-economic dynamics of early Neolithic communities.

2. The archaeological context

Gre Fılla is located in the Ambar Valley, Upper Tigris Basin, Southeastern Anatolia (Fig. 1). The Ambar Valley served as a natural passage between the Taurus Mountains and Northern Mesopotamia (Ökse, 2020). This location functioned as a crucial link between the raw material sources in the Taurus Mountains and the main trade routes of Northern Mesopotamia. Seasonal nomadic groups used this passage for livestock farming, transportation of goods, and trade.

Rescue excavations conducted between 2018 and 2022 revealed cultural layers, dating from the Pre-Pottery Neolithic A to the Byzantine-Roman periods (Table 1). The Pre-Pottery Neolithic layers of Gre Fılla (ca. 11,300 BP-9,500 BP) provide crucial insights into early human settlement and societal development. The architectural features uncovered include oval and rectangular structures, some of which were used for rituals, showing cultural connections with other significant Neolithic sites such as Göbekli Tepe, Gusir Höyük, Çayönü, and Nevali Çori. These architectural features highlight the region's role in early human civilization socio-cultural and technological advancements.

The inhabitants of Gre Fılla practiced agriculture and animal husbandry, supplemented by hunting. Flint and obsidian, transported from Eastern Anatolia (Bingöl and Nemrut Dağı), were used in tool production. The chipped stone industry at Gre Fılla was characterized by a variety of tools, including retouched blades, notched tools, and microliths, indicating a specialized production system. Obsidian and copper

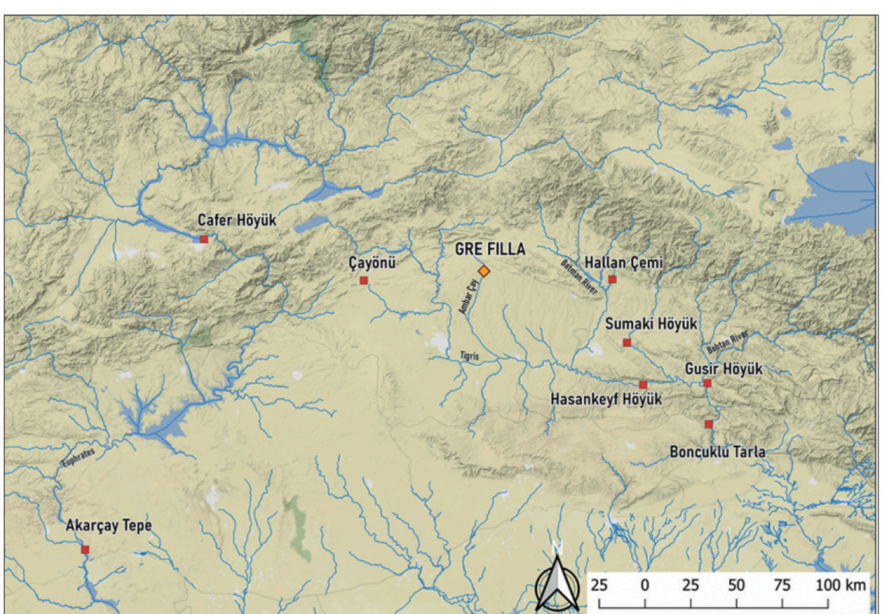


Fig. 1. The Location of Gre Fılla and Contemporary Settlements in the Region.

Table 1
Stratigraphy and archaeological features of Gre Fılla.

Layers	Ages	Periods		Architectural and archaeological features
GFI		BYZANTINE-ROMAN		Cemetery
GFII		MIDDLE AGE		Pottery and small walls
GFIII	6900–5200 BCE	POTTERY NEOLITHIC		Pottery
GFIV	7500–8800 BCE	PRE POTTERY NEOLİTHİC B	GFIV A	Grill buildings, subterranean huts and cell buildings
			GFIV B	Structures with one or two rooms, subterranean huts, cell buildings cobble paved buildings buttressed structures
			GFIV C	Grill buildings and subterranean huts
			GFIV D	Subterranean huts, rectangular buildings with one or two rooms
GFV	8800–9300 BCE	PRE POTTERY NEOLİTHİC A	GFV1	Rectangular and round subterranean huts
			GFV2	Rectangular and round subterranean huts
			GFV3	Round subterranean huts

artifacts found at Gre Fılla were part of a broader distribution system, underscoring the settlement's role in early trade networks.

3. Copper deposits in anatolia

One of the primary objectives of studies on ancient metals is to determine the geological origin of the metal used in the production of artifacts, thereby providing information on trade, social interactions, and the movement of objects. Although the studies initiated in 1934 and later conducted by Otto and Witter (1952) and the Stuttgart group (Junghans et al., 1960, 1968) contributed significantly to our understanding of ancient metallurgy, they did not succeed in identifying the sources of metal ores. As discussed by Gale and Stos-Gale (1982, 2000), due to chemical heterogeneities in ore deposits and the variable fractionation of chemical elements among ore, slag, and metal during smelting processes, elemental analyses alone have been unsuccessful at determining the provenance of metals.

With the widespread acknowledgment that chemical analyses alone are insufficient for identifying the geological origin of metals used in artifact production, two research groups proposed that comparative lead isotope analyses of artifacts and metal ores could be employed for provenance studies (Brill and Wampler, 1965; Grögler et al., 1966). Systematic studies and applications of lead isotope analysis began in the 1970 s through the collaborative work of a group at the Max Planck Institute for Nuclear Physics composed of W. Gentner, G.A. Wagner, and O. Müller with N.H. Gale from the University of Oxford (Gale, 1978; Stos-Gale and Gale, 2009; Wagner et al., 1979).

Lead isotope analysis involves measuring the isotopic ratios of lead in archaeological samples to determine their geological origins and potential sources. Lead has four stable isotopes: ^{204}Pb, ^{206}Pb, ^{207}Pb, and ^{208}Pb. Among these, ^{206}Pb, ^{207}Pb, and ^{208}Pb are the final nuclides in the decay series of uranium, actinium, and thorium, respectively (Rosman and Taylor, 1998).

The relative abundances of these isotopes can vary depending on the geological sources of lead. Different regions and ore deposits possess distinct isotopic signatures due to variations in the isotopic compositions of the local bedrock and minerals. Since Brill and Wampler (1965) proposed comparing lead isotope ratios in artifacts and metal ores to determine the geological origin of the raw materials used in metal object production, numerous studies have been conducted in the field. Although hundreds of samples have been analysed from various parts of the world—particularly from regions such as Bulgaria, Greece, Italy, and Turkey, which hold significant archaeological evidence related to the origins of metallurgy—the existence of a comprehensive database on the lead isotopic compositions of ore deposits remains incomplete (Stos-Gale and Gale, 2009).

In archaeometallurgical investigations of archaeological samples, optical microscopy and scanning electron microscopy (SEM) are employed to identify microstructural phases and their quantities, while energy-dispersive X-ray spectroscopy (EDS) analyses conducted with SEM are used to determine the chemical composition of these phases (Radivojević et al., 2010).

Eastern Anatolia encompasses a geological structure that includes major copper, lead, zinc, and silver deposits in Türkiye (Fig. 1). It forms the east–west orogenic belt of the Tethyan-Eurasian Metallogenic Belt (TEMB), situated between the continental collision of the Eurasian Plate to the north and the Arabian Plate to the south (Yiğit, 2009; Kuşcu et al., 2019). Between these two belts lies the Taurus-Anatolian Platform, which contains Upper Cretaceous magmatic rocks and a series of ophiolitic formations. The subduction, accretion, and collision events associated with the closure of the Tethys Ocean between the two plates occurred during the Mesozoic period (Moritz and Baker, 2019; Uçurum et al., 2021). To the north, the Eastern Pontides—located near the eastern Black Sea coast—extend toward Transcaucasia. To the south, the ore-rich metallogenic belt is bounded by the Bitlis-Zagros Suture Zone, which forms a geographical boundary with the plains of Upper Mesopotamia (Yiğit, 2009; Kuşcu et al., 2019; Moritz and Baker, 2019).

Numerous volcanogenic massive sulfide (VMS) deposits have formed along the northeastern (Ankara-Erzincan Suture) and southern tectonic zones (Bitlis-Zagros Suture) of Türkiye (Hauptmann et al., 2022; Yazar et al., 2015). In the north, active ore districts are exemplified by the Artvin-Murgul region (Eti Bakır Murgul Mine), while in the south, the Ergani and Siirt-Madenköy deposits exemplify active ore districts (Akıncı, 2009). These deposits are considered among the largest ore bodies in Türkiye. Polymetallic VMS deposits are generally categorized into two types based on their formation characteristics (Yiğit, 2009). In the north, Kuroko-type deposits are hosted within silicified pumice-rich dacitic tuffs, such as those found in the Artvin region (Kraeff, 1963; Çağatay, 1993; Çağatay and Eastoe, 1995). These deposits extend across a wide area from southern Georgia and northern Armenia to Anatolia, Bulgaria, and Romania, and are genetically associated with Late Cretaceous volcanic rocks (Çiftçi, 2019). In the Bitlis-Zagros Suture Zone, Cyprus-type VMS deposits are associated with Early to Middle Eocene mafic ophiolitic rocks, calcareous sandstones, and shales. Today, these ore bodies are generally referred to as copper–gold deposits, with pyrite and chalcopyrite being the dominant ore minerals (Marro and Stöllner, 2021).

Seeliger et al. (1985) and Wagner et al. (1986) published the results of archaeometallurgical investigations, including elemental and lead isotope analyses, conducted in northwestern, northern, and eastern Anatolia. According to their finding correlated with data from the General Directorate of Mineral Research and Exploration (MTA) the copper ore deposits evaluated within the scope of the study are concentrated in the regions of Çankırı, Kastamonu, Amasya, Giresun, Gümüşhane, Keban, and Ergani (Elâzığ).

Upon examining the elemental compositions of Cu, Fe, Zn, Ag, and Pb, it was observed that samples from the central Keban Basin (MTA 133: 79/2-Pb-1) and specifically the Keban–Keban Dere site (sample 175 F8-2), as well as from Ergani (MTA 133: 80/4-Cu-1, sample 176 A5-1), display isotopic similarities to those obtained from Gre Fılla (Muşkara and Aydın, 2022).

Seeliger et al. (1985) noted that the Pb isotope ratios of the samples collected from the Ergani copper ore deposit exhibited surprisingly consistent values ($^{208/206}$Pb ≈ 2.060–2.064; $^{207/206}$Pb ≈ 0.825–0.829). However, two new samples from Ergani analyzed by Wagner et al. (1986) identified as 176A-2 and 176B-2, show a marked deviation from the group previously reported by Seeliger et al. (1985) with isotopic ratios of $^{208/206}$Pb = 2.0697 for sample 176A-2 and $^{208/206}$Pb = 2.0371 for sample 176B-2.

Based on the results of Pb isotope analyses, Yener et al. (1991), Sayre et al. (2001), and Yener et al. (2021) proposed four characteristic isotopic groups for the Central Taurus region. These groups are defined as Taurus 1A (Bolkar Valley), Taurus 1B (east of Niğde and Yahyalı), Taurus 2A (northeast of Yahyalı), and Taurus 2B (Kestel, Çamardı). The isotopic signatures of these groups were compared with those of the Cyprus and Laurion ore sources. According to the findings, the isotopic compositions of the sampled groups from the Central Taurus differ from those of the Cypriot and Greek (Laurion) ore deposits (Yener et al., 1991).

In the study conducted by Hauptmann et al. (2022), artifacts recovered from the Early Bronze Age settlement of Arslantepe (Phase VI B2) were analyzed alongside ore samples from Eastern Anatolia believed to be geologically associated with the site. Additionally, to facilitate archaeological comparison, slag and malachite samples from the site of Çayönü were also examined.

The results of lead isotope analyses indicate that the Arslantepe artifacts likely originated from the ore deposits of Artvin/Murgul, Trabzon, and Ergani. While the slag sample from Çayönü was determined to be of Ergani origin, the isotopic composition of the malachite fragment suggested a potential provenance from sources in Northern Anatolia. However, Hauptmann et al. (2022) argue that the malachite may also have originated from Ergani and propose that this discrepancy is due to unresolved issues related to the isotopic characterization of the Ergani ore field.

The Alihoca Ophiolitic Complex is situated within a tectonic melange unit that includes ultramafic and mafic rocks such as serpentinite, peridotite, gabbro, diabase, and spilite. These lithologies are present in tectonic contact and were emplaced during Late Cretaceous tectonic activity. The oldest overlying stratigraphic unit is the Kalkankaya Formation, dated to the Late Maastrichtian–Early Paleocene (Sünger et al. 2022).

The ophiolitic melange is primarily exposed along the southern margin of the Ulukışla Basin, where it consists of blocks and clasts of ophiolite-derived rocks—commonly serpentinite and gabbro—ranging from several centimeters to over 15 m in size, embedded within a red, sheared, clay-rich matrix (Clark and Robertson 2002).

A mining gallery situated to the west of the village of Alihoca, near Ulukışla at the southern edge of the Bolkardağı Valley, was uncovered by the MTA during surveys conducted between 1975 and 1977 (Ebrahimabadi et al., 2011). While initial assessments identified the site as a lead (galena) mine, further investigations carried out in 1980 revealed that the deposit was in fact polymetallic and tin-rich. The mineral assemblage includes stannite, a naturally occurring tin-copper sulfide, along with associated minerals such as sphalerite, galena, and copper-bearing sulfides like chalcopyrite, pyrite, and arsenopyrite.

The copper deposits in the Artvin-Murgul region are located approximately 575 km from Gre Fılla. The most comprehensive archaeological evidence of prehistoric mining and metallurgy has been recovered from the Murgul area (Wagner et al., 1989; Lutz, 1990). Murgul comprises two major ore bodies, Anayatak and Çakmakkaya, with several smaller ore occurrences known to exist. In contrast, the Ergani Mine is situated about 80 km west of Gre Fılla. Nevali Çori lies 140 km away, while Norşuntepe is approximately 70 km distant.

From a geographical perspective, Ergani's location near the Tigris River provides accessible connectivity between southeastern Anatolia and northern Mesopotamia. Although modern mining operations have largely obliterated the remnants of ancient ore extraction, Ergani is recognized as one of the most significant prehistoric copper sources in Anatolia (Wagner et al., 1989). Malachite, azurite, and other secondary copper minerals are now only present in trace amounts in the Ergani deposit (Griffitts et al., 1972). However, it is assumed that these minerals occurred in much greater quantities at the surface during antiquity (Hauptmann et al., 2022).

Two principal ore bodies have been identified at Ergani: Anayatak and Mihrap Dağ (Weiss deposit). The presence of green secondary copper ores and native copper in Anayatak suggests their possible use in bead production during the Pre-Pottery Neolithic periods (PPNA and PPNB) (Özdoğan and Özdoğan, 1999). Based on current knowledge, copper use in Murgul dates back to the Chalcolithic period (Lutz et al, 1994; Wagner et al., 1989).

Two metal artifacts (AH.91-343/A and AH.91-194) unearthed from Aşıklı Höyük, an Early Neolithic settlement located in Central Anatolia, were analyzed at the Max Planck Institute using Neutron Activation Analysis (NAA) for elemental composition, as well as lead (Pb) isotope analysis (Esin, 1995). Another artifact (AH.92.ISK.13) was subjected to trace element analysis using Atomic Absorption Spectroscopy (AAS) at Boğaziçi University. Based on the NAA results, one of the analyzed samples was identified as native copper and the other as malachite. Based on the trace element concentrations, researchers concluded that the Aşıklı samples and the copper ore artifacts from Çayönü originated from different sources (Maddin et al., 1991). Although the study refers to Pb isotope analyses, the results of these analyses were not provided.

4. Materials and methods

4.1. Archaeological samples

During the 2019–2022 excavation seasons, remains related to copper production—including unworked samples and tools—were discovered. Various copper ores, with a total weight of 142.5 g, were most frequently found in the rubble deposits of the oval building K8 (Fig. 2). The quantity of copper ore finds recovered from public and private structures dated to the PPNB suggests that copper production was carried out at the site. This interpretation is further supported by the variety of artifacts found, including beads, pendants, chisel-axes, labrets, and fishhooks, which indicate local production. A recent study has also revealed signs of melting and possible ore processing at the site, predating known traditions of metallurgy—an important discovery (Muşkara et al., 2025). A total of 22 samples were collected from various trenches representing the site's spatial distribution, including contexts from both the PPNA and PPNB layers (Table 2). Particularly noteworthy is Structure K2, which yielded a composite copper chisel-axe (Muşkara and Aydın, 2022).

4.2. Methods

Lead isotope ratio determination of copper ore samples was carried out at the Radiogenic Isotope Laboratory, Central Laboratory, Middle East Technical University (METU). Chemical preparation and column chromatography were conducted in a class-100 clean laboratory using ultrapure reagents. Drilled samples (~80 mg) were dissolved overnight in 4 mL of 14 M HNO_3, followed by 4 mL of 6 M HCl on a hot plate. After drying, the residues were re-dissolved in 1 mL of 2 M HCl for lead separation. Pb was isolated using Teflon columns packed with BioRad AG1-X8 anion exchange resin. A modified HBr–HCl ion exchange procedure was applied after Romer (2001). Lead fractions were collected through successive HCl and HBr elutions. The purified Pb was loaded onto single filaments with silica gel and 0.005 M H_3PO_4 and measured under static conditions at 1250–1350 °C. NIST SRM 981 was used as the reference standard, and appropriate corrections were applied. Isotopic measurements were conducted using a Thermo Fisher Triton thermal ionization mass spectrometer (TIMS), and errors are reported at the 2σ level.

Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) analyses were carried out by an Agilent 7500A model Inductively Coupled Plasma-Mass Spectrometer for the major elements Cu, Fe and Zn and for trace elements As, Pb, Sb, Sn, Zn, Ag, Au, Ni, Cr, Co. The powdered samples were taken from GRE-C010, C029, C043, C044 by a diamond drill. The drillings obtained from the patina were discarded during the sampling to prevent possible contamination. Then they were ground using an agate mortar. 10 mg of drillings was weighed, and 5 mL mixture

Fig. 2. Unworked copper ore pieces in recovered from Gre Fılla.

of hydrochloric acid (HCl) and nitric acid (HNO_3) was added to the samples. This mixture was placed in a PTFE vessel and digested by microwave heating. The distilled deionized Ultra High-Quality water obtained from a Milli-Q Plus water system (Millipore, Bedford, MA, USA) was used throughout the experiments. The accuracy of the analysis was checked using a secondary standard, which was analyzed previously (Muşkara and Aydın 2022).

5. Results

In this study, we have analysed a total of 22 copper ore samples from Gre Fılla. The elemental compositions of the samples analysed with ICP-OES are compiled in Table 3. The samples GRE-C001-004, 008, 010, 012, and 013 were previously measured and the results are used here for comparison. Bulk analysis of the copper ore samples revealed copper ores with varying amounts of iron, zinc, and other trace elements.

Previous studies on the characterization of regional copper deposits have identified distinct groups based on LIA, including those located southwest of the Bolkar Mountain Valley (Taurus 1A, 1B), the north-eastern part of Yahyalı and Aladağ mountains (Taurus 2A and 2B), as well as deposits in Trabzon and Artvin (Sayre et al., 2001). Volcanogenic massive sulfide (VMS) deposits in Anatolia serve as major copper sources and are distributed across three distinct tectonic settings: the Eastern Pontides, the Central Pontides, and the Bitlis-Zagros suture zone (Çiftçi, 2019). Among these, Ergani Maden in the Bitlis-Zagros zone is particularly significant in both geological and archaeological contexts and holds relevance for Gre Fılla due to its proximity to the site (Bamba 1976, Akıncı 2009; Wagner et al. 1985). The isotopic ratios obtained from samples were compared with the ore deposits from Ergani-Maden, Siirt-Madenköy, Elâzığ, Taurus (Taurus 1A, 1B, 2A, and 2B), North Central Anatolia, Artvin-Murgul.

When plotted on a $^{208}Pb/^{206}Pb$ vs. $^{207}Pb/^{206}Pb$ diagram, several Gre Fılla copper ore samples—particularly GRE-C034 and C044—cluster closely with reference signatures from Ergani Maden, with additional overlap with the Taurus 1A field observed. This alignment strongly suggests that these samples likely originated from southeastern Anatolian sources (Fig. 3a). Stos-Gale (2016) defined this isotopic grouping as the "Bolkar and Ergani Maden group", refering to the overlapping Pb isotope signatures of the Ergani and Bolkar ore fields. The majority of Gre Fılla samples exhibit $^{208}Pb/^{206}Pb$ and $^{207}Pb/^{206}Pb$ ratios ranging between 2.03204 and 2.05598 and 0.81874–0.83023, respectively (Table 4).

The Ergani Maden copper district in eastern Anatolia contains several mineralized zones, with the Ana Yatak (TG 176A) and Mihrap Dağ (Weiss-deposit, TG 176B) being the most significant. These deposits exhibit distinct vertical zonation, consisting of an oxidized zone rich in malachite, azurite, and native copper (2–3 % Cu), a secondary enrichment zone with copper grades up to 25 %, and a primary sulfide zone dominated by pyrite and chalcopyrite. Hauptmann et al. (2022), based on findings from Arslantepe, noted that Ergani Maden typically forms a narrow isotopic cluster according to the data published by Seeliger et al. (1985). However, subsequent analyses—such as that of sample TG 176A-2, later published by Wagner et al. (1986)—indicate that the lead isotope composition of Ergani ores may show greater variability. In particular, the variation observed in TG 176B-1, a chalcopyrite sample from Ergani Maden, has been attributed to differences in Pb concentration and a high U/Pb ratio, which may have promoted the insitu accumulation of radiogenic lead. Seeliger et al. (1985) reported that the earlier measurement, which appeared highly radiogenic, was likely affected by this process, whereas a subsequent analysis with higher Pb content (~10 μg/g) yielded more typical values consistent with the Ergani ore field.

Samples GRE-C029 and C043 show higher $^{208}Pb/^{206}Pb$ and $^{207}Pb/^{206}Pb$ ratios similar to those of TG-176A-2 from Ergani Maden, and Alihoca samples. Therefore, the copper ore samples from Gre Fılla seem to originate from the Bolkar and Ergani Maden region. Additionally, GRE-C010 exhibits distinctly low $^{208}Pb/^{206}Pb$ and $^{207}Pb/^{206}Pb$ ratios. Its isotopic signature does not correspond to the primary Ergani Maden – Bolkar clusters but instead aligns closely with the sample 287F from Alihoca ore field in Bolkar district (Begemann et al., 2003).

The pattern of Gre Fılla copper ores becomes more nuanced in the diagrams of $^{208}Pb/^{204}Pb$ vs. $^{206}Pb/^{204}Pb$ and $^{207}Pb/^{204}Pb$ (Fig. 3b, c). While GRE-C034 and GRE-C044 remain tightly associated with the Bolkar–Ergani group, the majority of Gre Fılla samples form a homogeneous cluster that does not directly match any known reference source. In contrast, GRE-C010, C029, and C043 are clearly scattered and in proximity to the compositional fields of Alihoca and Ergani sample TG 176A-2. These results suggest that while most Gre Fılla copper exhibits internal isotopic consistency, only a few samples show definitive links to known ore fields—specifically Bolkar–Ergani or Alihoca—indicating complex or multi-source procurement strategies.

Table 2
The list of samples.

Sample Nr.	Period	Trench	Building	Context	Material
GRE-C001	GF IV	PPNB	K8 (Oval Building)	O8/064/M	Ore
GRE-C002	GF IV	PPNB	K8 (Oval Building)	O8/064/M	Ore
GRE-C003	GF IV	PPNB	K8 (Oval Building)	O8/064/M	Ore
GRE-C004	GF IV	PPNB	K8 (Oval Building)	O8/064/M	Ore
GRE-C008	GF IV	PPNB	K8 (Oval Building)	O8/064/M	Ore
.GRE-C010	GF IV	PPNB	K8 (Oval Building)	O8/064/M	Ore
GRE-C012	GF IV	PPNB	K8 (Oval Building)	O8/064/M	Ore
GRE-C013	GF IV	PPNB	K8 (Oval Building)	O8/064/M	Ore
GRE-C029	GF IV	PPNB	K31 (Rectangular Building)	N9/579/V	Ore
GRE-C030	GF IV	PPNB	Cultural Fill	L9/051/V	Ore
GRE-C034	GF IV	PPNB	K13 (Rectangular Building)	O8/343/A	Ore
GRE-C035	GF IV	PPNB	K7 (Rectangular Building)	P9/474/V	Ore
GRE-C036	GF V	PPNA	K60 (Oval Building)	O9/896/V	Ore
GRE-C037	GF IV	PPNB	Alluvial fill	O8/817/V	Ore
GRE-C038	GF IV	PPNB	K7 (Oval Building)	P9/302/V	Ore
GRE-C039	GF IV	PPNB	K7 (Oval Building)	P9/314/V	Ore
GRE-C040	GF V	PPNA	K60 (Oval Building)	O9/824/V	Ore
GRE-C041	GF V	PPNA	K43 (Oval Building)	O8/789/V	Ore
GRE-C042	GF IV	PPNB	Cultural Fill	N9/095/A	Ore
GRE-C043	GF IV	PPNB	K42 (Oval Building)	O9/552/A	Ore
GRE-C044	GF IV	PPNB	K7 (Oval Building)	P10/041/V	Ore
GRE-C045	GF IV	PPNB	K2 (Rectangular Building)	O8/210/A	Ore

6. Discussion

In interpreting our data, we follow the established observation that Pb isotope ratios in ores with low Pb concentrations and elevated U/Th contents may undergo significant post-formational modification (Pernicka et al. 1993; cf. Seeliger et al. 1985; Wagner et al. 1986). This effect, noted for the Selevac malachites, results in wide scatter in $^{206}Pb/^{204}Pb$ and $^{207}Pb/^{204}Pb$ values that do not reflect primary ore signatures but rather variable U/Pb ratios. The same concern applies to the Gre Fılla copper ores, which contain lower Pb contents, making them similarly susceptible to radiogenic ingrowth. As demonstrated for Selevac, the recalculation of isotope ratios relative to ^{206}Pb yields a more robust framework: in a $^{207}Pb/^{206}Pb$ vs. $^{208}Pb/^{206}Pb$ diagram, radiogenically modified samples fall on a straight-line array, permitting clearer distinction between genetic fields. For this reason, we consider the $^{208}Pb/^{206}Pb$ vs. $^{207}Pb/^{206}Pb$ space the most diagnostic in our study. In this framework, Gre Fılla samples such as **GRE-**C034 and C044 cluster closely with the "Bolkar–Ergani group" (Stos-Gale 2016), while GRE-C010, C029, and C043 overlap with Alihoca and Ergani sample TG 176A-2. The generally consistent isotopic range observed ($^{208}Pb/^{206}Pb$ = 2.032–2.056; $^{207}Pb/^{206}Pb$ = 0.819–0.830) suggests internal coherence for most Gre Fılla ores, although the few scattered samples point to more complex or multi-source procurement strategies.

While the majority of Gre Fılla copper samples fall within the Bolkar–Ergani lead isotope group defined by Stos-Gale (2016), the identification of one sample (GRE-C010) with a lead isotopic signature matching Alihoca, a central Anatolian deposit, suggests a more localized pattern of copper acquisition. Given that Alihoca and Bolkar are geographically and geologically closer to each other than to Ergani, and that Bolkar and Ergani are isotopically indistinct, it is plausible to interpret the entire isotopic group as reflecting a central Anatolian source region, rather than eastern Anatolia. This interpretation is further supported by the lack of uniquely Ergani-type trace element markers or archaeological evidence of contact with eastern Anatolia at Gre Fılla.

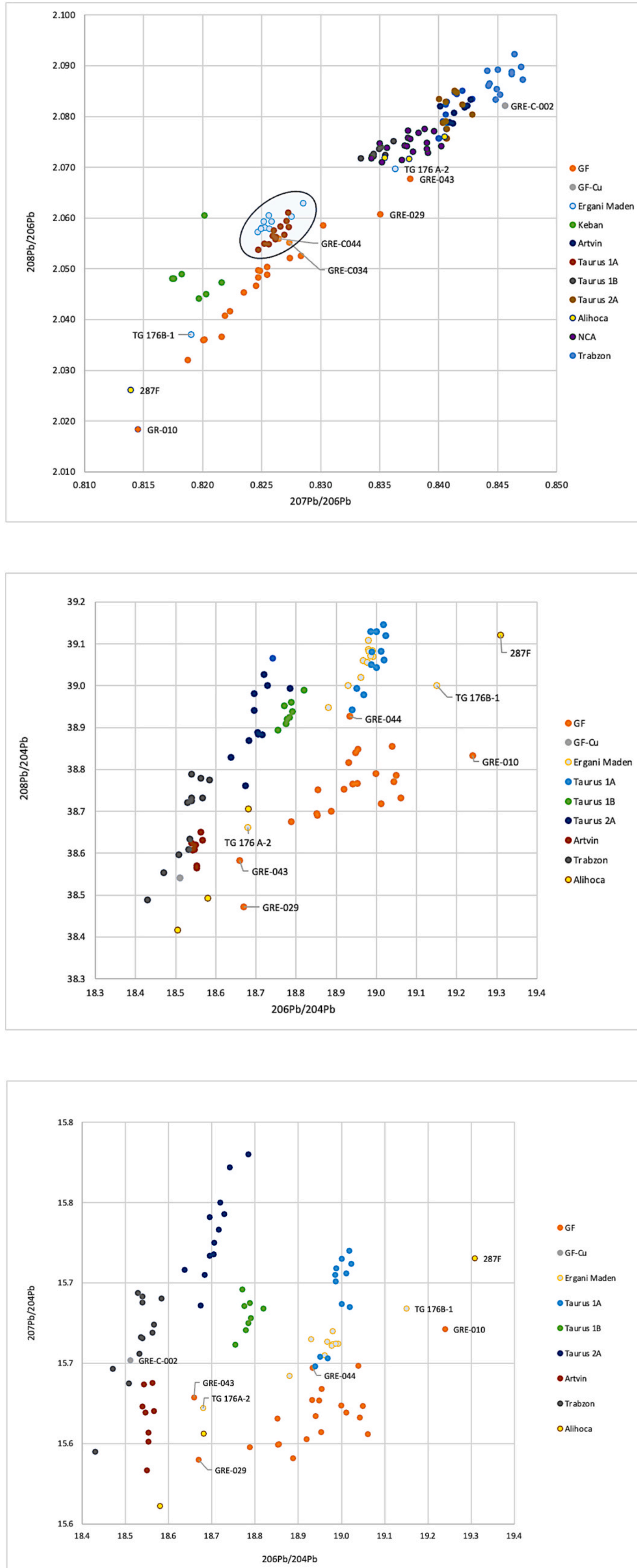
Lead isotope analysis shows that copper ores were brought to Gre Fılla from multiple sources, including both local and distant regions. These sources include:

- Ergani-Maden/Bolkar Mountains: Gre Fılla's proximity to the copper deposits at Ergani-Maden highlights the important role of the Ambar Çayı Valley in early metal trade. This closeness suggests that local sources played a significant role in supplying copper to Gre Fılla.
- Alihoca-Niğde (Central Anatolia) and Trabzon (Black Sea Region): Despite the proximity to Southeast and Eastern Anatolian deposits, the presence of copper from Central Anatolia and the Black Sea Region emphasizes the complexity of trade networks during this period. Copper from these distant sources indicates far-reaching exchange systems that connected various regions.

In conclusion, the analysis of lead isotopic ratios indicates that Gre Fılla possesses a unique isotopic fingerprint. The copper used at Gre Fılla likely came from multiple sources, including the Ergani and Trabzon deposits, and possibly from the Niğde-Alihoca deposit, suggesting the presence of a complex supply network. The use of nearby deposits such as Ergani-Maden highlights the community's access to local resources. However, the isotopic composition of the Ergani deposits has been minimally studied, emphasizing the need for more detailed research on this source.

Table 3
Bulk chemical analysis obtained by ICP-MS from powder samples. * wt. % major oxides, others in mg/kg; n.d: not detected. (for GRE-C001-013 see Muşkara and Aydın 2022).

Sample	Cu*	Fe*	Zn*	Cr	Co	Ni	As	Ag	Sn	Sb	Pb
GRE-C001	54	0.40 ± 0.01	0.24 ± 0.01	24 ± 2	12.3 ± 0.5	21 ± 1	n.d.	n.d.	226 ± 5	1.56 ± 0.04	32 ± 1
GRE-C002	55	0.36 ± 0.01	0.30 ± 0.01	22 ± 2	48 ± 1	16 ± 1	n.d.	1.9 ± 0.1	0.59 ± 0.05	n.d.	69 ± 2
GRE-C003	38	0.63 ± 0.01	0.17 ± 0.01	47 ± 3	6.4 ± 0.6	47 ± 4	n.d.	34 ± 1	n.d.	n.d.	13.9 ± 0.6
GRE-C004	49	1.89 ± 0.01	0.26 ± 0.01	110 ± 3	17 ± 1	64 ± 1	56 ± 5	63 ± 2	n.d.	0.85 ± 0.05	195 ± 3
GRE-C008	45	2.9 ± 0.01	0.82 ± 0.01	74 ± 3	675 ± 20	217 ± 12	197 ± 16	6.7 ± 0.3	0.51 ± 0.04	6.4 ± 0.4	136 ± 2
GRE-C010	37	1.18 ± 0.01	0.17 ± 0.01	52 ± 1	n.d.	29 ± 1	10.1 ± 0.4	n.d.	n.d.	0.36 ± 0.02	68 ± 0.4
GRE-C012	45	0.77 ± 0.01	0.43 ± 0.01	143 ± 1	475 ± 22	0.16 ± 0.01*	n.d.	n.d.	1.4 ± 0.1	0.69 ± 0.02	117 ± 2
GRE-C013	55	0.38 ± 0.01	0.24 ± 0.01	22 ± 2	n.d.	125 ± 5	n.d.	18 ± 1	2.3 ± 0.1	0.44 ± 0.04	24 ± 1
GRE-C029	50	0.94 ± 0.01	0.22 ± 0.01	30 ± 3	n.d.	n.d	n.d	n.d	n.d.	n.d.	13 ± 0.3
GRE-C043	34	0.76 ± 0.01	0.18 ± 0.01	n.d.	n.d.	n.d	n.d	n.d.	n.d	n.d	90 ± 0.4
GRE-C044	51	0.33 ± 0.01	0.19 ± 0.01	n.d.	25 ± 1	n.d	n.d	n.d	n.d.	n.d	12 ± 0.1



a

b

c

Fig. 3. Lead isotope ratios of Gre Filla samples in comparison to copper ores of Anatolia. These data are taken from the OXALID database (http://oxalid.arch.ox.ac.uk). The classification of ore deposits by Yener, et al. (1991) and Sayre, et al. (2001).

Table 4
Lead isotope data of copper ores found at Gre Fılla.

Sample Name	$^{208}Pb/^{206}Pb$	2σ	$^{207}Pb/^{206}Pb$	2σ	$^{206}Pb/^{204}Pb$	2σ	$^{207}Pb/^{204}Pb$	2σ	$^{208}Pb/^{204}Pb$	2σ
GRE-C-002	2.08217	0.00005	0.84560	0.00001	18.51098	0.00536	15.65196	0.00448	38.54057	0.01106
GRE-C001	2.04664	0.00002	0.82454	0.00001	18.94059	0.00063	15.61722	0.00055	38.76497	0.00043
GRE-C002	2.03595	0.00001	0.82006	0.00001	19.04275	0.00019	15.61627	0.00023	38.77056	0.00041
GRE-C003	2.04167	0.00004	0.82233	0.00001	18.99928	0.00176	15.62370	0.00150	38.79062	0.00370
GRE-C004	2.04833	0.00002	0.82471	0.00001	18.91878	0.00044	15.60265	0.00049	38.75260	0.00096
GRE-C008	2.05216	0.00002	0.82740	0.00001	18.85329	0.00055	15.59918	0.00048	38.69045	0.00119
GRE-C010	2.01836	0.00011	0.81453	0.00002	19.23963	0.00248	15.67133	0.00221	38.83295	0.00615
GRE-C012	2.04888	0.00001	0.82544	0.00001	18.88800	0.00016	15.59091	0.00014	38.69963	0.00034
GRE-C013	2.03661	0.00003	0.82159	0.00001	19.01103	0.00051	15.61935	0.00043	38.71849	0.00135
GRE-C029	2.06069	0.00001	0.83507	0.00001	18.66902	0.00061	15.58985	0.00071	38.47156	0.00132
GRE-C030	2.04966	0.00006	0.82486	0.00001	18.95437	0.00132	15.63393	0.00115	38.84865	0.00326
GRE-C034	2.05522	0.00001	0.82736	0.00001	18.85496	0.00071	15.59976	0.00059	38.75152	0.00149
GRE-C035	2.05856	0.00003	0.83023	0.00001	18.78739	0.00062	15.59782	0.00059	38.67536	0.00160
GRE-C036	2.05254	0.00001	0.82835	0.00001	18.85163	0.00041	15.61569	0.00035	38.69425	0.00092
GRE-C037	2.04081	0.00003	0.82191	0.00001	19.03891	0.00228	15.64831	0.00187	38.85518	0.00472
GRE-C038	2.04539	0.00001	0.82348	0.00001	18.95274	0.00105	15.60720	0.00085	38.76642	0.00209
GRE-C039	2.04972	0.00001	0.82472	0.00001	18.94821	0.00069	15.62691	0.00058	38.83954	0.00141
GRE-C040	2.03204	0.00001	0.81874	0.00001	19.06049	0.00081	15.60588	0.00071	38.73209	0.00162
GRE-C041	2.05037	0.00002	0.82546	0.00001	18.93149	0.00126	15.62722	0.00108	38.81692	0.00264
GRE-C042	2.03607	0.00003	0.82015	0.00002	19.04950	0.00277	15.62351	0.00229	38.78651	0.00566
GRE-C043	2.06774	0.00004	0.83758	0.00003	18.65943	0.00109	15.62875	0.00074	38.58316	0.00191
GRE-C044	2.05598	0.00008	0.82643	0.00003	18.93330	0.00277	15.64709	0.00237	38.92684	0.00602
SRM981 (found) n = 12	2.16719		0.91461		16.94033		15.49384		36.71246	
SRM981 (certified value)	2.16810		0.91464		16.93709		15.49135		36.72132	

The sample labeled GF-Cu (GRE-C-002), (Fig. 3) offers a new clue regarding the variety of sources used at Gre Fılla. This sample has slightly higher isotopic values compared to other Gre Fılla ores, and its distinct isotopic signature suggests that it could be associated with a different source or undergone a different processing technique. Furthermore, GF-Cu may be part of a separate trade or supply chain, reinforcing the idea of a diversified resource network at Gre Fılla. It should also be emphasized that GF-Cu is not an ore fragment but a metallic copper object, as previously demonstrated through detailed archaeometallurgical analysis (Muşkara et al., 2025). Its inclusion in the isotopic dataset therefore provides a crucial reference point, allowing us to distinguish between unworked ore samples and processed copper artifacts in the Gre Fılla assemblage.

Numerous copper ores and copper artifacts and ores were found in the PPNB levels of Gre Fılla. The early use of copper ores and copper for tools, ornaments, and symbolic objects highlights their importance in social hierarchies and trade. Lead isotope analysis has shown that the copper ores came from both local sources, such as Ergani-Maden, and distant sources, such as Central Anatolia and the Black Sea Region. Despite the proximity of local deposits, the presence of copper ores and copper from distant regions reveals the complexity of trade and resource use during the Pre-Pottery Neolithic B period. These results indicate that copper at Gre Fılla was not only imported but also locally transformed through small-scale experimental activities. Considering that the inhabitants of Gre Fılla were primarily hunter-gatherers rather than specialized metalworkers, the arrival and processing of copper likely represented an occasional or exploratory practice rather than systematic production.

In addition to copper transported over long distances, obsidian sourced from Bingöl A, Bingöl B, and Nemrut Dağı was also found at Gre Fılla (Muşkara and Konak, 2022). The presence of these materials from distant regions reflects the extensive interactions among PPNB communities. The procurement of valuable raw materials such as copper and obsidian through a complex distribution system, facilitated by specialized groups involved in long-distance trade, likely promoted cultural interaction and technological exchange. Trade networks that spread from the Anatolian center during the PPNB period likely facilitated not only the exchange of goods but also the diffusion of ideas, rituals, belief systems, and cultural practices such as burial traditions. The spread of craftsmanship, such as malachite and copper processing techniques and obsidian tool production, likely occurred through these trade networks.

In conclusion, Gre Fılla's strategic location in the Ambar Çayı Valley, along with its proximity to raw material sources and the presence of copper and obsidian brought from distant regions, illustrates the nature of its connections to trade networks during the PPNB period.

7. Conclusion

This study presents a comprehensive archaeometric and isotopic investigation of copper ores unearthed at Gre Fılla, a Pre-Pottery Neolithic site located in the Upper Tigris Basin of southeastern Anatolia. Through the analysis of 22 copper ore samples collected from multiple architectural contexts dated to both the PPNA and PPNB periods, we sought to trace the provenance of the raw materials and to better understand the community's participation in early metallurgical practices and long-distance exchange networks.

The lead isotope analysis revealed that the copper used at Gre Fılla was sourced from multiple deposits, reflecting a complex procurement system rather than reliance on a single local source. Most samples exhibit isotopic ratios that align with the Bolkar-Ergani group, a cluster of indistinguishable signatures first defined by Stos-Gale (2006), and suggesting a shared metallogenic origin. Some specimens, notably however, notably GRE-C010, show isotopic affinities with the Alihoca deposit in Niğde, central Anatolia, while others (e.g., GRE-C029, C043) plot near the TG-176A-2 sample from Ergani or scatter toward the Alihoca field. These findings indicate that copper at Gre Fılla was not sourced exclusively from the nearby Ergani Maden deposit, but rather obtained from multiple regions including both southeastern and central Anatolia.

Furthermore, the detection of copper ores and other copper-based artifacts such as beads, pendants, chisel-axes, and fishhooks in the PPNB levels of Gre Fılla provides strong archaeological evidence for on-site processing and symbolic use of copper. This aligns with emerging evidence from other contemporary Neolithic sites in the region, suggesting that early experimentation with copper was already underway long before the advent of large-scale metallurgy. Importantly, these results show certain points of convergence as well as divergence with the evidence obtained from obsidian. Obsidian sourced from Bingöl A, Bingöl B, and Nemrut Dağ indicates the operation of long-distance procurement mechanisms. Part of the copper assemblage at Gre Fılla was obtained from the nearby Ergani deposit, which is located directly along the route by which obsidian was transported, while other samples point to more distant sources in Central Anatolia (Bolkar–Alihoca) and the Black Sea region (Trabzon), from which obsidian was not procured. This discrepancy highlights an intriguing pattern: whereas obsidian circulated predominantly from eastern Anatolia, copper could also arrive from regions outside the established obsidian exchange networks. Thus, while copper and obsidian may occasionally have traveled through overlapping systems, their supply chains were not entirely congruent, suggesting both shared and distinct pathways for the circulation of exotic raw materials. The association of obsidian from Bingöl and Nemrut Dağ with imported copper reinforces the notion of Gre Fılla as an active node within expansive exchange networks during the early Holocene.

The results of this study underscore the diversity of raw material sources used by Neolithic communities in southeastern Anatolia and provide compelling evidence for the early development of technological and social systems centered on resource procurement and craft specialization. The combination of elemental and isotopic data offers critical insights into the emergence of metallurgy as a socio-cultural practice, rather than a solely technological innovation. Ultimately, Gre Fılla emerges not only as a site of early copper use but also as a strategic location within broader Neolithic networks that facilitated the circulation of goods, knowledge, and cultural traditions across Anatolia and beyond.

CRediT authorship contribution statement

A. Konak: Writing – original draft, Validation, Supervision, Methodology, Investigation, Conceptualization. **Ü. Muşkara:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Data curation, Conceptualization. **S. Karayünlü-Bozbaş:** Writing – original draft, Methodology, Formal analysis, Data curation. **M.B. Telli:** Writing – original draft, Investigation.

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Declaration of competing 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.

Data availability

No data was used for the research described in the article.

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