

# Raw Material Choices in the Palaeolithic of the Inner Asian Mountain Corridor of Kazakhstan

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*“Археология – это не профессия, а образ жизни”*

*[Archaeology is not a profession, it's a lifestyle]*

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# ZUSAMMENFASSUNG

Die Rekonstruktion von Rohstoffnutzungsmustern bei Hominiden ist eines der grundlegenden Ziele der prähistorischen Archäologie. Lithische Rohstoffe wurden häufig als Marker für das Verhalten von Hominiden verwendet und anschließend zur Untersuchung von Beschaffungsstrategien, Mobilität und sogar kognitiven Entwicklungen verwendet. Auf dieser Grundlage verfolgte mein Promotionsprojekt einen multidisziplinären Ansatz zur Analyse von lithischen Rohstoffen und ihrer potenziellen Beziehung zu gewohnheitsmäßigen Aktivitäten von Hominiden.

In meinem ersten Artikel wurde die erste geoarchäologische Felduntersuchung zur Erforschung der lithischen Rohstoffverteilung innerhalb des innerasiatischen Bergkorridors von Kasachstan durchgeführt. Geologische Proben verschiedener Lithologien wurden makroskopisch mit den archäologischen Gesteinsansammlungen verglichen. Die Ergebnisse zeigen, dass die Rohstoffnutzung zwischen den Untersuchungsregionen, die fast 1000 km voneinander entfernt sind, unterschiedlich war.

Im Rahmen eines laufenden multidisziplinären Projektes analysierte ich die gesammelten Proben aus geologischen und archäologischen Kontexten durch die Anwendung ingenieurwissenschaftlicher Tests, um Fragen zur Qualität des lithischen Rohmaterials zu beantworten (Artikel II). Die ausgewählten Hornstein-, Schiefer- und Porphyproben aus drei verschiedenen Regionen Kasachstans wurden mittels objektiver Tests untersucht. Dies geschah, um den Aspekt der Bruchfestigkeit zu untersuchen, ein Wert, der eng mit der Bruchzähigkeit zusammenhängt. Die Ergebnisse legen nahe, dass Materialien, die zuvor als minderwertig angesehen wurden (z. B. Porphy), mechanische Eigenschaften aufweisen, die mit Hornstein verglichen werden können. Abschließend diskutierte ich den Einfluss der mechanischen Eigenschaften von Porphy im Hinblick auf die Steintechnologie und veranschaulichte seine Eignung für die Herstellung anspruchsvoller Werkzeuge.

Artikel III bietet die erste petrographische Charakterisierung verschiedener Rohstoffe, die in den paläolithischen Komplexen Kasachstans verwendet werden, und diskutiert die Rohstoffbeschaffungsstrategien auf der Grundlage von Feldstudienresultaten und einer umfassenden Literaturrecherche. Die petrographische Analyse deckte strukturelle Variationen innerhalb verschiedener Hornstein-Proben auf, die in der

Region Qaratau gesammelt wurden, und legt eine Grundlage für zukünftige Provenienzstudien dieser Materialien. Darüber hinaus deuten die Ergebnisse der Feldbefragung auf direkte selektive Beschaffungsstrategien bei Maibulaq hin.

Insgesamt hat die aktuelle Dissertation versucht, die technologischen Entscheidungen und Beschaffungsstrategien von Hominidengruppen auf der Grundlage multidisziplinärer methodischer Ansätze zu rekonstruieren, die in drei separaten Artikeln veröffentlicht wurden.

## SUMMARY

Reconstructing hominin raw material utilization patterns comprises one of the fundamental objectives of prehistoric archaeology. Lithic raw materials have been widely used as markers of hominin behaviour and subsequently used to study procurement strategies, mobility, and even cognitive developments. On this basis, my PhD project adopted a multi-disciplinary approach to the analysis of lithic raw materials and their potential relationship with habitual activities of hominins.

In *Paper I*, the first geoarchaeological field survey to study the lithic raw material distribution within the Inner Asian Mountain Corridor of Kazakhstan was conducted. Geological specimens of various lithologies were macroscopically compared to the archaeological lithic assemblages. The results revealed that raw material utilization varied between study regions, which are separated by almost 1000 km.

In the framework of an ongoing multi-disciplinary project, I analyzed the collected samples from geological and archaeological contexts by the application of engineering tests to address questions surrounding the lithic raw material quality (*Paper II*). The selected samples of chert, shale, and porphyry from three different regions of Kazakhstan were studied by means of objective tests. This is done to study one aspect of their mechanical properties, the fracture resistance, a value that is closely related to fracture toughness. The results suggest that materials previously considered of lower quality (e.g., porphyry) have mechanical properties that can be compared to chert. Ultimately, I discussed the effect of mechanical properties of porphyry in regard to the lithic technology illustrating its suitability for the production of sophisticated tools.

*Paper III* provides the first petrographic characterization of various raw materials utilized in the Palaeolithic complexes of Kazakhstan and discusses the raw material procurement strategies based on field survey results and a comprehensive literature review. The petrographic analysis revealed structural variation within various chert samples collected in the Qaratau region and lays a foundation for future provenance studies of these materials. In addition, the field survey results suggest direct selective procurement strategies at Maibulag.

Overall, the current PhD dissertation has attempted to reconstruct the technological choices and procurement strategies of hominins groups based on multi-disciplinary methodological approaches published in three separate papers.



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Obtaining a PhD degree is one of the big tasks in one's life. Without the support of many people, it would have been a task that is difficult to complete.

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Additionally, I would like to highlight that I use the Kazakh transliteration of the site names and geographic entities as opposed to Russian transliteration. The latter adapted the locality names into the Russian pronunciation which resulted in changing the designations (e.g., Karatau to Qaratau, Koshkurgan-Shoktas complex is referred to as Qoshqorgan-Shoqtas).

## PERSONAL CONTRIBUTION

- Publication 1:** I was the first and corresponding author, as well as the person responsible for conceiving the study design and the lead author of writing the manuscript. The coauthors helped to analyze and conduct the field trip to locate sources of raw materials (Patrick Cuthbertson, Aristeidis Varis, Zhaken Taimagambetov, Radu Iovita), and design the sampling strategy (Patrick Schmidt). They also helped in writing and editing the manuscript.
- Publication 2:** I was the first and corresponding author, as well as the person along with Patrick Schmidt responsible for conceiving the study design and lead author of writing the manuscript. The coauthors helped to analyze and conduct the field trip to sample various raw materials (Aristeidis Varis, Zhaken Taimagambetov, Radu Iovita), design the sampling strategy (Patrick Schmidt), and the experimental tests (Klaus G. Nickel). They also helped in writing and editing the manuscript.
- Publication 3:** I was the first and corresponding author and the lead author of writing the manuscript. The coauthors helped to conduct the field trip to locate sources of raw materials, sample them (Aristeidis Varis, Zhaken Taimagambetov, Radu Iovita), and design the sampling strategy (Patrick Schmidt). They also helped in writing and editing the manuscript. Additionally, the coauthors helped in interpreting the petrographic data (Patrick Schmidt) and oversaw the study as supervisors (Patrick Schmidt and Radu Iovita).

# 1 INTRODUCTION

## ***1.1 The importance of raw material studies***

Assemblages of lithic artifacts represent the most stable class of artifacts that can survive various forms of physical damage. Hence, they are a major source of information that provide significant data for the evaluation of hominin habitual activities. In recent years, the detailed investigation of the types and qualities of various rocks that were used to produce Palaeolithic stone assemblages has become one of the proxies to study past hominin settlement systems. In this regard, a number of studies dedicated to investigating raw material differences show an apparent link between lithic tools and raw material types, raising several debates around hominin habitual activities (Webb and Domanski, 2008; Manninen, 2016). This link has been explained in a number of ways including 1) the effect of raw material quality on types of end products that can be knapped from a particular material, 2) the possibility to interpret and examine some aspects of hominin behaviour, and 3) the interaction and use of palaeolandscapes for the provisioning of raw materials. The reconstruction of the aforementioned links is central in understanding the social organization of prehistoric hominin groups. Research investigating lithic raw materials has significantly contributed to the study of hominin-environment interactions through the implementation of systematic field works, scientific analyses, and comprehensive synthesis of the existing archaeological record (Bar-Yosef, 2002; Blinkhorn and Petraglia, 2017; Ghasidian and Heydari-Guran, 2018; Heydari-Guran and Ghasidian, 2020; Ekshtain and Zaidner, 2021). Additionally, population movements are inferred from distances between sites where knappable rocks are found and where their closest source is located (Feblot-Augustins, 1993; 2009; Wiśniewski et al., 2012; 2020; Frahm et al., 2019). The evaluation of behavioural developments has been attempted by the study of innovative technologies such as heat-treatment of lower quality raw materials and production of blade tools (Bar-Yosef and Kuhn, 1999; McBrearty and Brooks, 2000; Wadley, 2013; Schmidt, 2021). Such studies were extensively incorporated in the Palaeolithic of Eurasia, Africa, Australia, and Central Asia including the Russian Altai, adding to the knowledge of territory sizes (Binford, 1980; Bar-Yosef, 2002; Anokin and Postnov, 2005; Feblot-Augustins, 2009; Sano, 2010; Yoshikawa, 2010; Ekshtain et al., 2017; Frahm and Hauck, 2017; Caruana et al., 2019; Frahm and Tryon,

2019; Khatsenovich et al., 2020). Thus, the study of lithic raw materials is regarded as one of the fundamental research objectives in prehistoric archaeology.

These kinds of investigations allow us to establish a solid connection between raw materials and lithics. This, in turn, helps to assess the technological advancement and procurement strategies of prehistoric hunter-gatherer groups (Binford, 1979; Hoffecker, 2005). Thus, the study of lithic raw materials contributes to the wide spectrum of approaches to studying human evolution and behaviour. However, detailed investigations of lithic raw materials utilized in the Palaeolithic of Kazakhstan remain largely understudied. The availability of a wide variety of raw materials used in the Palaeolithic of the Inner Asian Mountain Corridor (IAMC), combined with a lack of targeted research in this field constitutes the main reasons behind this first study of raw material selection choices as revealed by an analysis of the distribution and quality of knappable rocks.

### ***1.2 Implications of raw material studies to behavioural theories***

The properties and sources of various knappable raw materials across the landscape greatly influenced the way how these resources were exploited by hominins. Settlement and procurement patterns as well as the lithic reduction strategies developed by the prehistoric people likewise had influenced in defining the selection criteria of these resources. Therefore, the attempts to answer archaeological questions on determining the raw material selection strategies are, in most cases, complemented by the ethnographic and anthropological data to develop discussion on different aspects of the subject (Binford, 1978; 1979; 1980; Gould and Saggars, 1985; Andrefsky, 1994; Kuhn, 2004; 2020; Yoshikawa, 2010). Consequently, the gathered data as reflected in procurement, selection, and management of lithic resources are used to evaluate the behaviour of prehistoric groups. Several theories suggested by scholars argue that the selection of higher quality raw materials reflects the evolution of lithic technology and hominin knowledge of materials' properties (Hoffecker, 2005; Braun et al., 2009; Goldman-Neuman and Hovers, 2012). In an overview of the patterns of behavioural change in Africa and Eurasia, Conard (2005) reviews patterns of lithic technology as potential indicators of modern behaviour. Additionally, in viewing the accumulation of markers for modern behaviour, several scholars argue that the main human achievement was the long-distance exchange of raw materials (Feblot-

Augustins, 1993; Blegen, 2017; Brooks et al., 2018). Bar-Yosef (2002) considers such exchanges as one of the markers of the Upper Palaeolithic revolution that distinguishes it from the slow cultural changes in the Middle Palaeolithic. Furthermore, innovation in lithic technology and ultimately the appearance of blade production in the Upper Palaeolithic complexes signaled a change in hominin cognition (Clark and Lindly, 1989; Ambrose and Lorenz, 1990; Bar-Yosef and Kuhn, 1999). Although blade technology appeared long before the Upper Palaeolithic, it came to dominate the archaeological record during this period of prehistory (Bar-Yosef and Kuhn, 1999). In addition to blade technology, symbolic use of ochre (Chase and Dibble, 1987), the emergence of earliest art and ornamentation (Barton et al., 1994; Dutkiewicz et al., 2018), ritualistic burials (Chase and Dibble, 1987), and others led to the debate whether such evidence could be one of the signs of modern human behaviour in the archaeological record. It should be noted that all of these traits predate the Upper Palaeolithic in Africa. These debates eventually introduced a 'behavioural modernity' term (Klein, 1995; McBrearty and Brooks, 2000; Henshilwood and Marean, 2003). It implies cognitive and technological traits that distinguish *Homo sapiens* from other species of anatomically modern humans (AMH) (Nowell, 2010; Sterelny, 2011). Sometimes this term is also referred to as cultural modernity (Conard, 2005; 2010). The evolution of behavioural modernity hastened in the mid-Late Pleistocene in parts of Africa, Eurasia, and Australia (Conard, 2008; Davidson, 2010; Gilligan, 2010; Derevianko, 2011). The aforementioned debates resulted in a series of competing models that offer theories on what indicates a behavioural modernity (Clark and Lindly, 1989; Ambrose and Lorenz, 1990; McBrearty and Brooks, 2000; Deacon, 2001; Minichillo, 2006; Nowell, 2010). Some studies even argue that innovative technology (e.g., heat-treatment) is associated with the evolution of complex hominin cognition in the Stone Age of Africa (Wadley, 2013). The argument of cognition complexity is believed to be supported by the evidence of deliberate alteration of rock qualities by processes of heat treatment, which increases the knapping quality of silica-rich rocks (Sealy, 2009; Wadley and Kempson, 2011; Schmidt et al., 2020). Ultimately, the mentioned models indicate a great potential in the study of lithic raw materials for the discussion of different aspects of human behaviour.

### ***1.3 Aspects of raw material quality***

Research that explicitly examines the physical and mechanical qualities of rocks from archaeological contexts has emerged relatively recently. It is generally associated with Goodman's (1944) pioneering research on the physical properties of stone tool materials. Since then, a growing body of research on the quality of raw materials has examined various aspects of fracture mechanics and the effect of quality on hominin tool preference (Cotterell and Kamminga, 1987; Brantingham et al., 2000; Braun et al., 2009; Schmidt et al., 2017a; Loendorf et al., 2018; Mraz et al., 2019). Additionally, mechanical tests are used to answer the question of whether differences in raw material procurement may have been driven by different mechanical properties (Moník and Hadraba, 2016), or whether physical properties have direct implications for use-wear accrual rates (Lerner et al., 2007).

In particular, the fracture predictability of different rocks for knapping has been a major topic of discussion among different scholars arguing that good raw material should be brittle, isotropic, and elastic (Cotterell et al., 1985; Cotterell and Kamminga, 1987). The debates on raw material qualities generally derive from the knowledge of modern experimental knappers that classify rocks with homogenous microstructure or crystallinity as being of 'higher' quality. In this regard, rocks possessing isotropic properties, which exhibit properties with uniform values measured in all directions, are often considered to be of higher quality for the production of stone tools. Controlled experiments have demonstrated that 'higher' quality raw materials directly influence the end products' size and shape (Crabtree, 1967; Cotterell and Kamminga, 1987; Collins, 2008; Dogandžić et al., 2020; Marreiros et al., 2020; Lin et al., 2022). These experiments imply that different rock attributes (e.g., impurities, phenocrysts, etc.) affect reduction sequences because mechanisms of fracture propagation vary among different types of lithologies (Mardon et al., 1990). In most cases, fractures in rocks consist of mainly intergranular cracks which occur when a crack propagates along the grain boundaries (Mardon et al., 1990). Thus, the type of rock is responsible for the detachment of flakes from the core, i.e., impurities affect the length and thickness of the end product. This is due to the impurities that prevent the propagation of fracture over the entire length of the flake. Hence, fine-grained lithologies or rocks rich in silica such as various types of cherts, flints, shale, or obsidian were the favoured rocks among prehistoric knappers. However, the archaeological record also demonstrates that hominins exploited a variety of 'lower' quality lithologies in the absence of 'higher'

quality raw materials in the landscape. This includes rocks exhibiting structural flaws such as large phenocrysts or high porosity. The class of lower quality raw materials, as differentiated by prehistoric archaeologists and referred to as such, are represented by a group of volcanic (e.g., porphyry, trachyte, diorite, basalt, etc.) and metamorphic rocks (e.g., hornfels, quartzite). Despite the use of lower quality rocks, the production evidence of more sophisticated lithic reduction techniques such as Levallois, Levallois-like technologies and radial reduction methods are present at Palaeolithic sites throughout the Russian Far East, Siberia, and Central Asia (e.g., Derbina V, Ikhine I and II, Maibulaq, Rahat, Kattasai) (Kharevich et al., 2010; Fitzsimmons et al., 2017; Ozherelyev et al., 2019; Kot et al., 2020). On the other hand, innovative modifications to improve the quality of rocks are known in the Palaeolithic of Africa and Australia (Akerman, 1979; Schmidt et al., 2013; Schmidt and Hiscock, 2019). For instance, the evidence of heat-treatment of rocks indicates that hominins altered the raw materials of lower quality, adapting them for the production of desired end products. Additionally, a detailed investigation of the mechanical and mineralogical transformations of heat-treated artefacts shows a change in their properties confirming an increase in the knapping qualities of rocks (Schmidt et al., 2017b; 2019). The selection of the so-called lower quality raw materials was driven by a variety of factors including their abundance in the palaeolandscapes, mechanical properties such as resistance to abrasion and durability of edges, and others. Therefore, the contemporary classification of 'higher versus lower' quality raw materials became ambiguous in light of archaeological evidence on the use of lower quality rocks for the production of technologically challenging artefacts. This classification requires a revision and perhaps another terminological approach to distinguishing qualitative parameters of different lithologies. The archaeological record visibly demonstrates that other than considering the homogeneity of selected rocks, hominins also paid attention to other physical properties (e.g., durability and resistance to abrasion) that would meet their needs for various purposes whether it is butchering or other subsistence related activities (Braun et al., 2009). In most cases, homogeneous materials such as obsidian or chert were suitable to easily knap but also had a high susceptibility of edges to abrasion.

This PhD project aims to answer the research questions highlighted below by conducting a geoarchaeological field survey, characterizing raw materials, and most importantly exploring the mechanical properties of various raw materials utilized in Palaeolithic complexes in Kazakhstan. The latter will be attempted through the

implementation of methodological approaches widely used in material sciences, involving experimental analysis of indentation fracture resistance (a value closely related to indentation fracture toughness), elasticity, and hardness (see section 4.2.3).



## 2 OBJECTIVES AND EXPECTED OUTPUT OF THE RESEARCH

This dissertation seeks to investigate the hominin raw material selection choices in the Palaeolithic complexes of the IAMC of Kazakhstan. It is achieved by conducting a systematic survey of raw materials, sample collection, and mechanical experiments (e.g., indentation tests). The application of these multi-disciplinary approaches will address raw material selection and procurement strategies of prehistoric hominins in Kazakhstan.

My dissertation will focus on the following research questions:

- is there a pattern of raw material use in different geographic regions of the IAMC of Kazakhstan? If so, what are the variations in raw material utilization in these regions?
- what are the mechanical properties of raw materials utilized in Kazakhstan? Do the mechanical properties of the lithologies allow us to make inferences about how lithic technology is adapted to raw material properties?
- given the larger number of sites from the Upper Palaeolithic period in the IAMC, what are the lithic procurement strategies in this period?

I approach my research questions through several interrelated steps and set four explicit tasks for a systematic investigation to answer these questions:

- to conduct the first systematic lithic raw material survey in the foothill zones of the IAMC of Kazakhstan;
- to describe the raw material distribution and characterize the regional variation of raw materials;
- to petrographically characterize various lithologies of rocks used in the Palaeolithic;
- to investigate the mechanical properties of raw materials and their influence on lithic technology.

With these questions, I attempt to improve our understanding of the raw material procurement, utilization, and effects of rock quality on knapping techniques in the Palaeolithic of Kazakhstan. A major component of the first research project focused on locating sources of raw materials in primary and secondary positions. I conducted the

first systematic work that specifically targeted locating and investigating the distribution of knappable rocks in different key foothill regions of the IAMC. The field survey was embedded in the PALAEOSILKROAD research project's reconnaissance works to find new Palaeolithic sites. Additionally, a field survey was conducted as part of the 'Lithic technology and raw material variation in the Inner Asian Mountain Corridor of Kazakhstan' dissertation project funded by the Leakey Foundation. By exploring the raw material outcrops, I discuss the patterns of raw material distribution in Kazakhstan.

Given that my preliminary results suggest a heterogeneous distribution of various raw materials in the study regions, the second research project of the current PhD aimed at investigating the qualities of three various lithologies (chert, shale, and porphyry) used in the Upper Palaeolithic industries throughout the IAMC. This has been achieved by means of experimental indentation tests. The purpose of the second project was to evaluate and compare different mechanical properties with the production of lithics.

I also highlight the importance of objective tests such as experimental investigation of the mechanical properties of raw materials. Such studies represent an excellent tool to understand and interpret the archaeological contexts and technological aspects of the hominin groups. I expect that correlating the mechanical properties of rocks with the types of raw materials available at site will help to answer procurement activities. An implementation of the methods used in this thesis project will also assist other scientists to make inferences in regions that offer various lithologies for knapping. Ultimately, I also expect that the results of the dissertation will offer a window to wider potential research to investigate various aspects of hominin behaviour.

## 3 RESEARCH BACKGROUND

### ***3.1 A brief history of the Palaeolithic research in Kazakhstan***

Archaeological data for the Palaeolithic of the IAMC of Kazakhstan are still scarce. This is partially related to the research history, which begins in the second half of the 19<sup>th</sup> century with the discovery of several arrowheads during the excavation of a kurgan in southern Kazakhstan (Chekha, 2017). Such accidental finds were later documented by Russian geologists in eastern Kazakhstan replenishing the collections of stone tools at the regional museums (Alpysbaev, 1970). However, systematic surveys and consequent excavations specifically targeting to locate Palaeolithic sites begin in the 1950s and were associated with the name of Kazakh archaeologist Khasan Alpysbayev (Alpysbaev, 1961; 1970; 1972d; 1972c; 1972b; 1972a; 1978; 1979; Kostenko and Alpysbaev, 1969; Alpysbaev and Kostenko, 1974). His work was largely concentrated on the Palaeolithic of southern Kazakhstan, whereas another pioneer Kazakh stone age archaeologist, Alan Medoev, concentrated his research on the prehistory of the Mangyshlaq peninsula, the Saryarqa region north of Lake Balkhash, and regions adjacent to Lake Balkhash (Medoev, 1964; 1972; 1982). Decades of research led by them resulted in finding numerous localities with scatters of surface lithics. The extent of surface lithic scatters reaches some 40 square km in sites such as Qyzyltau (Zhambyl region), Boriqazgan, Tanirqazgan, and Aqkol sites located in southern Kazakhstan, Semizbugu (north of Lake Balkhash), Ustyurt (western Kazakhstan), and others (Artyukhova, 1990; Artyukhova and Mamirov, 2014; Osipova and Artyukhova, 2019). In addition to numerous surface sites, Alpysbayev discovered and first led systematic archaeological excavations of several stratified sites in loessic (e.g., Valikhanova) and cave (e.g., Qaraungir and Ushbas, both in the Qaratau range, southern Kazakhstan) sedimentary contexts (Alpysbaev, 1979; Taimagambetov and Nokhrina, 1998). His work was continued by Zhaken Taimagambetov, Ol'ga Artyukhova, and Valeriy Voloshin along with many local and international collaborators. Since the end of the 1980s, systematic surveys along the southern piedmont of the Greater Qaratau located a complex of sites named Qoshqorgan-Shoqtas in a travertine context. They were subsequently excavated in the late 1990s (Derevianko et al., 1998). The lithic assemblages are characterized by small flake tools. The distinct Middle Pleistocene faunal assemblage suggested a Lower Palaeolithic age for the site, which

was confirmed by the electron paramagnetic resonance (EPR) dating method which tentatively places the chronology between 500-430 ka BP (Derevianko, 2006).

The Palaeolithic of south-eastern Kazakhstan is currently represented by stratified Upper Palaeolithic sites of Maibulaq and Rahat in the piedmont zone of the Ili Alatau range of the Tien Shan, in Almaty province (Taimagambetov and Ozherelyev, 2008; Taimagambetov, 2009; Dzhasybaev et al., 2018; Ozherelyev et al., 2019). Maibulaq was discovered by a local history teacher (Vladimir Sarayev) in the early 2000s, Rahat was discovered by Talgat Mamirov in the 2010s and is situated approximately 80 km east of Maibulaq. The archaeological excavations at Maibulaq were conducted by joint efforts of Kazakh and German scientific teams. The chronology of the Maibulaq was recently reviewed and re-dated by Fitzsimmons et al. (2017) to the period between 40 and 25 ka. The lithic assemblage is largely characterized by several schemes of reduction, including radial as well as single and double platform reduction techniques. Similar techno-typological features are characterized at the Rahat site (Ozherelyev et al., 2019).

Since 2017, the PALAEOSILKROAD multi-disciplinary archaeological project, funded by the European Research Council (ERC), has been conducting field work to discover Palaeolithic sites in the IAMC (Iovita et al., 2020). It aims to test the hypothesis that Pleistocene dispersals correlated with climatic pulses during the Last Glacial Cycle approximately 110 and 15 kya (Owen and Dortch, 2014; Asmerom et al., 2018). The PALAEOSILKROAD team has since surveyed 105 caves and rockshelter features using a ground-truthing model for the karstic cave prediction model (Cuthbertson et al., 2021). In addition to cave and rockshelter research, the project is contributing to studying the formation processes of sediments in cave contexts (Varis et al., 2022) and laying a foundation for the first systematic raw material studies (Namen et al., 2022a). The latter is the basis of the current doctoral dissertation.

### ***3.2 The Inner Asian Mountain Corridor of Kazakhstan***

The Inner Asian Mountain Corridor (the IAMC) is a term first coined by Michael Frachetti (2012). It is used to describe a continuous chain of mountains that stretches from Uzbekistan in the south-west covering the piedmont zones of northern Tajikistan, Kyrgyzstan, Kazakhstan, through western China, and Mongolia. This is a vast territory that spans approximately 4000 km. Due to its geographic position, the foothills of the

IAMC form a continuous landscape featuring complex topographies that offer various micro-climates and shelters for hominin occupation. Thus, the natural corridor along the foothills of various mountain ranges of the Tien Shan Mountain system has been proposed by Iovita et al. (2020) to be one of the regions for the dispersal routes of prehistoric hominins because it linked areas to the north and east (Siberia, China) to those further to the west and south (Uzbekistan).

The piedmont zones of the IAMC of Kazakhstan are the major study regions of the current dissertation (Figure 1). This region has been chosen due to the location of the majority of the stratified and surface Palaeolithic sites. It is also a particularly interesting venue to investigate the hominin occupation as the Late Pleistocene was the time of population contact and possibly dispersal in Central Asia (Boivin, 2013; Gokcumen, 2019). Within the IAMC our work concentrates on three mountain ranges separated by some 700-1000 km. These are the Qaratau, Ili Alatau, and Altai-Tarbagatai ranges. All of the ranges are part of the larger Tien Shan Mountain system. Below we provide a brief geological setting of each range described from west to east.

### ***3.2.1 The Qaratau range***

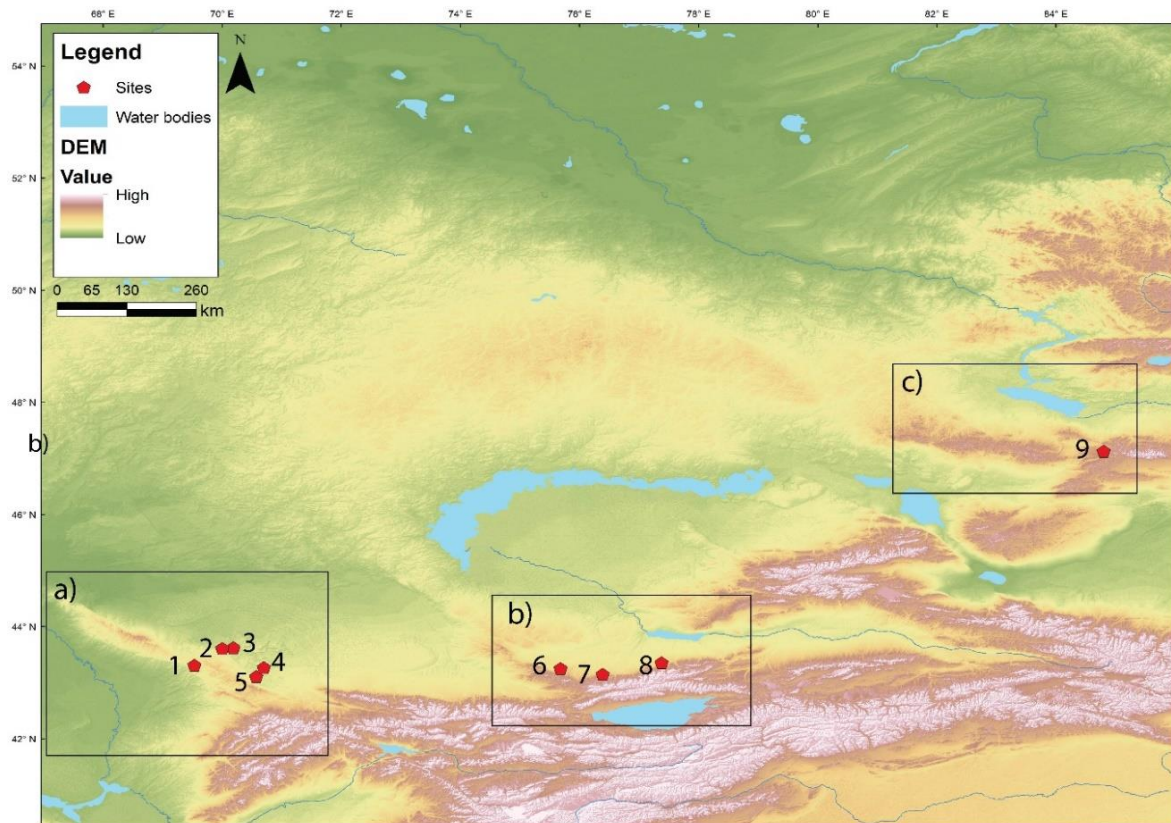
The Qaratau mountain range is a north-western horn of the Tien Shan mountains and is located in southern Kazakhstan. The geology of the mountain has been extensively studied and reported (Allen et al., 2001; Cook et al., 2002; Alexeiev et al., 2009). The range has a rich structural history characterized by multiple deformation events and the presence of slip faults (Alexeiev et al., 2009). Structurally, it is divided into a western 'Greater' and eastern 'Lesser' half which is separated by some 25 km of flat land also known as the 'Leontiev Graben' (Allen et al., 2001). According to Cook et al. (2002), the topographic expressions are characterized by uplifted carbonate beds that formed during the Late Devonian and Carboniferous periods with a wide variety of facies that range from tidal-flat to deep-water deposits. The range contains a large number of rivers, seasonal and perennial water sources flowing towards the plains creating attractive places for hominin occupation. The majority of the known Palaeolithic sites are located within this region and are closely located to notable Pleistocene cave sites containing Neanderthal and anatomically modern human (AMH) remains in Uzbekistan such as Teshik-Tash (Okladnikov, 1940), Obi-Rakhmat (Krivoshapkin et al., 2010), and Selungur in Kyrgyzstan (Krivoshapkin et al., 2020).

### **3.2.2 The Ili Alatau range**

The Ili Alatau range is located in south-eastern Kazakhstan. It is a northern portion of the Tien Shan mountains. The geology of the range is characterized by uplifted volcanic granitoid beds formed during the Late Devonian period. The northern foothills enclose the vast depression of the Ili River and further north borders with the Dzhungarian Alatau range. The topographic expression of the piedmont zone traps wind-blown aeolian sediments. The thickness of the quaternary loess deposits reaches up to 700 m in areas with substantial deposition (Dodonov, 2007). This blanket of loess known as the Central Asian piedmont (CAP) covers a large area extending from the Pamir and Alai to the Ili Alatau (Fitzsimmons et al., 2018). A provenance work led by Li et al. (2020) reports that the unsorted sediments on the piedmont slopes and alluvial-proluvial plains are common sources for loess in northern Tien Shan. Several stratified Upper Palaeolithic sites have been found since the early 2000s calling for systematic loess surveys to find traces of hominin occupation in the region (Taimagambetov and Ozherelyev, 2008; Taimagambetov, 2009; Fitzsimmons et al., 2017; Dzhasybaev et al., 2018; Ozherelyev et al., 2019).

### **3.2.3 The Tarbagatai range**

The Tarbagatai range lies to the north of the Tien Shan and is located in eastern Kazakhstan and the Xinjiang province of China. It extends approximately 300 km from west to east. The modern topographic expression of the range is largely related to the relatively recent episodes of deformation as a response to the collision of the Indian subcontinent with Eurasia (Dumitru et al., 2001; Gillespie et al., 2021). The lithology of the beds is comprised of volcanic rocks such as granites, diorites, and metasedimentary pelitic rock outcrops distributed in some areas. The range is an important watershed in the region, thus providing an attractive place for human occupation during the Late Pleistocene. Several occurrences of surface scatter of lithics are known along the foothills (Taimagambetov, 2016; Iovita et al., 2020). However, currently, the Upper Palaeolithic site of Ushbulaq is the only stratified site so far discovered in the Tarbagatai region (Anoikin et al., 2019).



**Figure 1.** Study areas (in dark squares) and sites mentioned in the text are shown in relation to the major topographic expression of the IAMC of Kazakhstan. a) the Qaratau, b) Ili Alatau, and c) Tarbagatai ranges. Numbered red marks are the sites mentioned in the text: 1) Valikhanova, 2) Yntaly, 3) Sorkol, 4) Qyzyltau complex, 5) Buirekbastau-Bulaq, 6) Beriktas, 7) Maibulaq, 8) Rahat, and 9) Ushbulaq. Data sources: Global Administrative areas (GADM) (Hijmans, 2012), vector and raster data from Natural Earth ([www.naturalearthdata.com](http://www.naturalearthdata.com)) and Shuttle Radar Topography Mission (SRTM) Version 4 (Jarvis et al., 2008). Figure adapted from Paper III.

### ***3.3 Archaeological and chronological background of the studied sites***

For the current PhD dissertation, I collected and studied samples from both stratified and unstratified sites. So far, the majority of the known Palaeolithic sites are located within the Qaratau range, with two stratified sites (Maibulaq and Rahat) situated in the Ili Alatau, and one site (Ushbulaq) is in the Tarbagatai region. Most of these sites are found in loessic and spring sedimentary contexts. We recently published a comprehensive overview of possible contexts for the preservation of prehistoric occupation (Iovita et al., 2020).

After almost 70 years of systematic research of the Palaeolithic of Kazakhstan, only a handful of stratified sites were located. Such low density of stratified sites could be due to the geography and climate of the region creating deflated surfaces that hinder sediment accumulation. Examples are represented by well-known surface sites in deflated contexts such as Semizbugu, Boriqazgan, and Tanirqazgan in southern Kazakhstan (Alpysbaev, 1979; Osipova and Artyukhova, 2019). On the other hand, a thick aeolian sediment accumulation covering the majority of the piedmont zone could potentially mask hominin occupational layers deep in the loessic sequence (Fitzsimmons et al., 2018; Li et al., 2020). Therefore, surface scatters of lithics dominate the prehistoric archaeological context of Kazakhstan.

The chronology of surface sites involved in this PhD project is determined based on techno-typological features of the assemblages. It tentatively ranges from Upper Palaeolithic to Holocene (e.g., Usiktas and Sorkol), and a complex of assemblages representing all periods of prehistory (e.g., Qyzyltau) (see Table 1). Khasan Alpysbaev (1979) first discovered and studied the sites of Usiktas and Qyzyltau. The Sorkol site was also first located by Alpysbaev (1972d), however, the lithic assemblages were thoroughly investigated by Gani Iskakov (1998). The Sorkol assemblage contains tools with both Holocene and Late Pleistocene techno-typological characteristics.

The stratified sites are dated by a combination of absolute dating methods and chronologically represent the Early Upper Palaeolithic. The chronology of Valikhanova and Maibulaq was determined by means of optically stimulated luminescence dating (OSL). The Valikhanova dates span 43.5 – 9 ka, whereas Maibulaq is dated to 40-25 ka (Fitzsimmons et al., 2017). The lithic assemblage of the Valikhanova is knapped using local cherts and characterized by coarse flakes made from discoid cores (Figure 3) (Taimagambetov, 1990; Fitzsimmons et al., 2017). The Maibulaq assemblage is predominantly knapped on pebbles of local porphyry and some tools are knapped on exogenous shale and chert which are present in much smaller quantities. It is technologically represented by prepared cores with hierarchically-organised surfaces and the production of bladelets (see Figure 2) (Fitzsimmons et al., 2017).

The oldest layer (CH 6.2) of the Ushbulaq site revealed a radiocarbon date of 45-39 ka cal BP as reported by Anoikin et al. (2019). The Ushbulaq lithics were knapped on locally outcropped metasedimentary pelitic rocks (Shunkov et al., 2017; Rybin et al.,

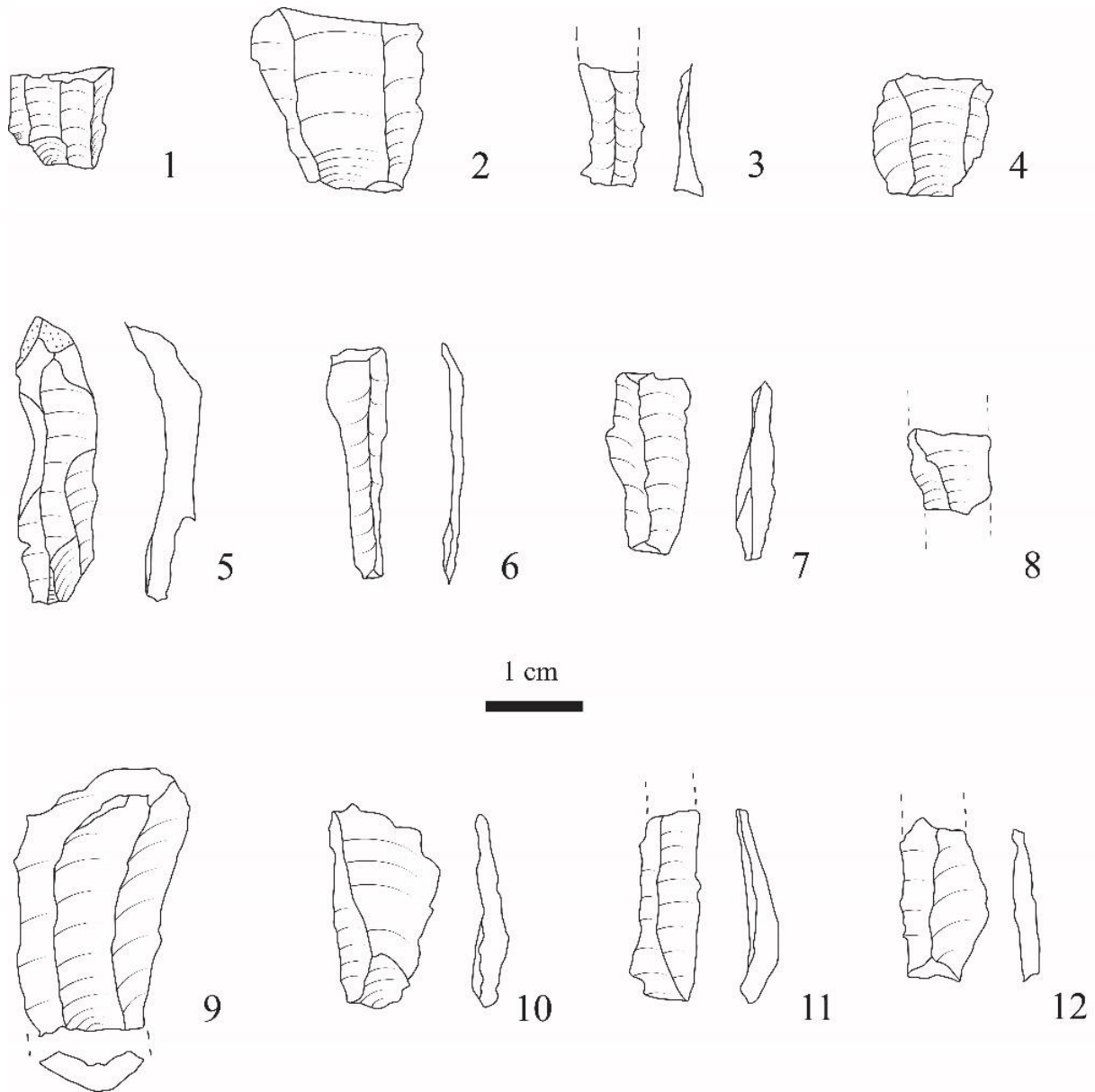


2018). The technology of lithics is characterized by blade production. The length of some of the produced blades sometimes exceeds 10 cm (Anoikin et al., 2017).

The Lower Palaeolithic is represented by the travertine sites of the Qoshqorgan-Shoqtas complex located in the southern foothill of the Greater Qaratau. It has been dated by the electron paramagnetic resonance (EPR) method and revealed a date that spans 500-430 ka (Derevianko et al., 1998; Derevianko, 2006). The lithic assemblage contains a small flake tool industry which is characteristic of most of the Central Asian Lower Palaeolithic (Figure 3) (Derevianko, 2006). On the other hand, the Middle Palaeolithic of Kazakhstan is only represented by scatters of surface lithics (Artyukhova, 1990; 2013; Artyukhova and Mamirov, 2014; Osipova et al., 2020). For more detailed information concerning the background of the studied archaeological sites, readers are referred to Appendix III.

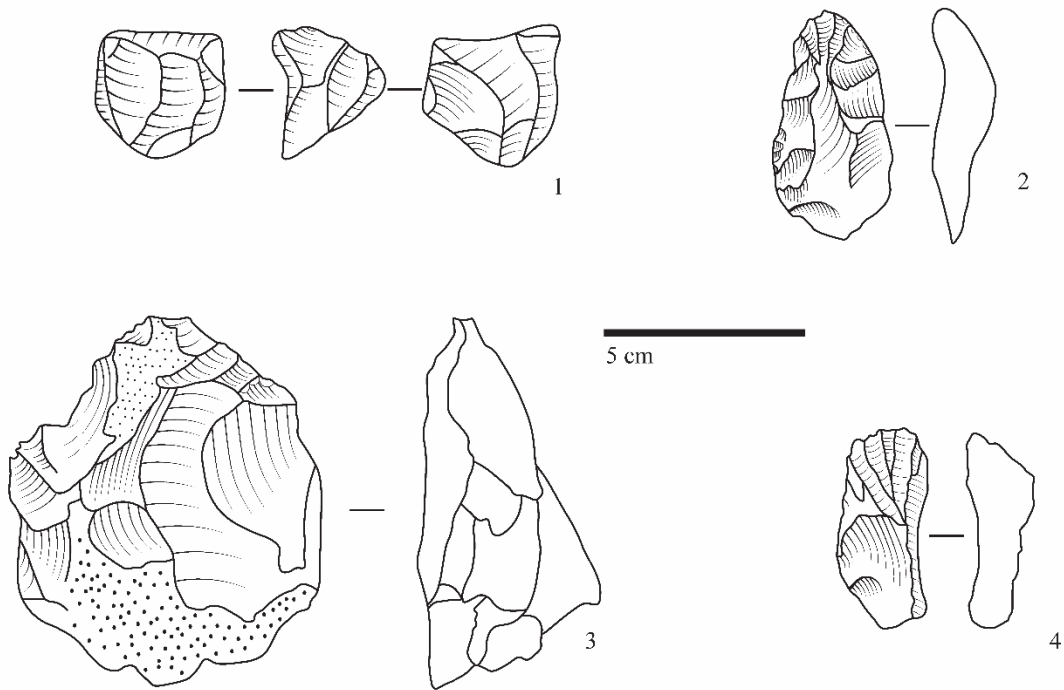
**Table 1. Chronological context of the archaeological sites. Abbreviations: UP – Upper Palaeolithic, EUP – Early Upper Palaeolithic, MP – Middle Palaeolithic, and LP – Lower Palaeolithic.**

Site ID	Context	Chronology	Reference
Qoshqorgan-Shoqtas	Stratified	500-430 ka	(Derevianko, 2006)
Valikhanova	Stratified	43.5-9 ka	(Fitzsimmons et al., 2017)
Sorkol	Surface	UP, surface	(Iskakov, 1998)
Buirekbastau-Bulaq	Stratified	EUP, absolute dates are pending	(Kunitake and Taimagambetov, 2021)
Qyzyltau	Surface	UP-MP-LP	(Derevianko, 2017)
Maibulaq	Stratified	40-25 ka	(Fitzsimmons et al., 2017)
Rahat	Stratified	UP, surface	(Ozherelyev et al., 2019)
Ushbulaq	Stratified	45-39 ka cal BP	(Anoikin et al., 2019)

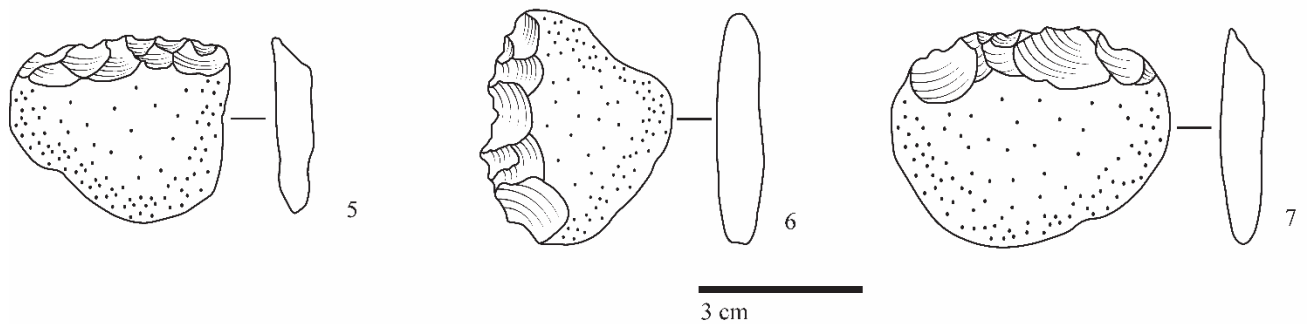


**Figure 2. Illustration of the lithic artefacts from the occupational horizon 3, Maibulaq. 1 – flake-bladelet, 2, 4 – flake, 3, 5, 8-11 – blade, 6, 7, 12 – bladelet. Figure adapted from Paper III.**

## Valikhanova



## Qoshqorgan-Shoqtas



**Figure 3. Illustration of some of the lithic artefacts from Valikhanova and Qoshqorgan-Shoqtas sites. 1 – sub-prismatic core, 2 – endscraper, 3 – core, 4 – scraper, 5, 6, 7 – sidescraper. Valikhanova lithics redrawn from Taimagambetov (1990), Qoshqorgan-Shoqtas lithics redrawn from Derevianko (2006).**

## 4 MATERIALS AND METHODS

### *4.1 Materials*

As the holistic approach of the PhD project is multi-disciplinary, it required a number of samples from archaeological and geological contexts for experimental analysis and direct comparison purposes. All of the samples reported in this work were collected during the PALAEOSILKROAD project's 2019 and 2021 field campaigns (see Iovita et al., 2020; Namen et al., 2020; Cuthbertson et al., 2021). The description of samples indicating the context, location, chronological period, and rock type is shown in Table 2.

The geological samples aided in the direct macroscopic comparison with the raw materials used in the Palaeolithic complexes. Most samples come from the secondary sources found in dried riverbeds in the form of alluvial and colluvial conglomerates. Few samples are found in primary positions (e.g., Yntaly and Valikhanova).

Archaeological samples of lithic artefacts come from stratified sites of Valikhanova, Qoshoqgan-Shoqtas, Buirekbastau-Bulaq, Maibulaq, Rahat, and Ushbulaq, as well as surface sites of Qyzyltau, Usiktas, and Sorkol (see section 3.3). As not to disturb the integrity of the assemblages found in stratified contexts, I only sampled lithics collected from the surface at those sites and that cannot be attributed to a specific cultural layer. The major sampling criterion was the size of lithic pieces. Collecting larger samples with the size of approximately 3-5 cm enabled me to cut the pieces in the middle and then conduct experiments on the polished surface produced from the lithics. The detailed procedure of sample preparation is described in detail in the respective manuscripts below (Appendix II).

**Table 2. Description of the archaeological and geological samples used for the PhD dissertation. Samples from sites indicated with \* sign were petrographically described, whereas samples with \*\* have been tested for the mechanical properties.**

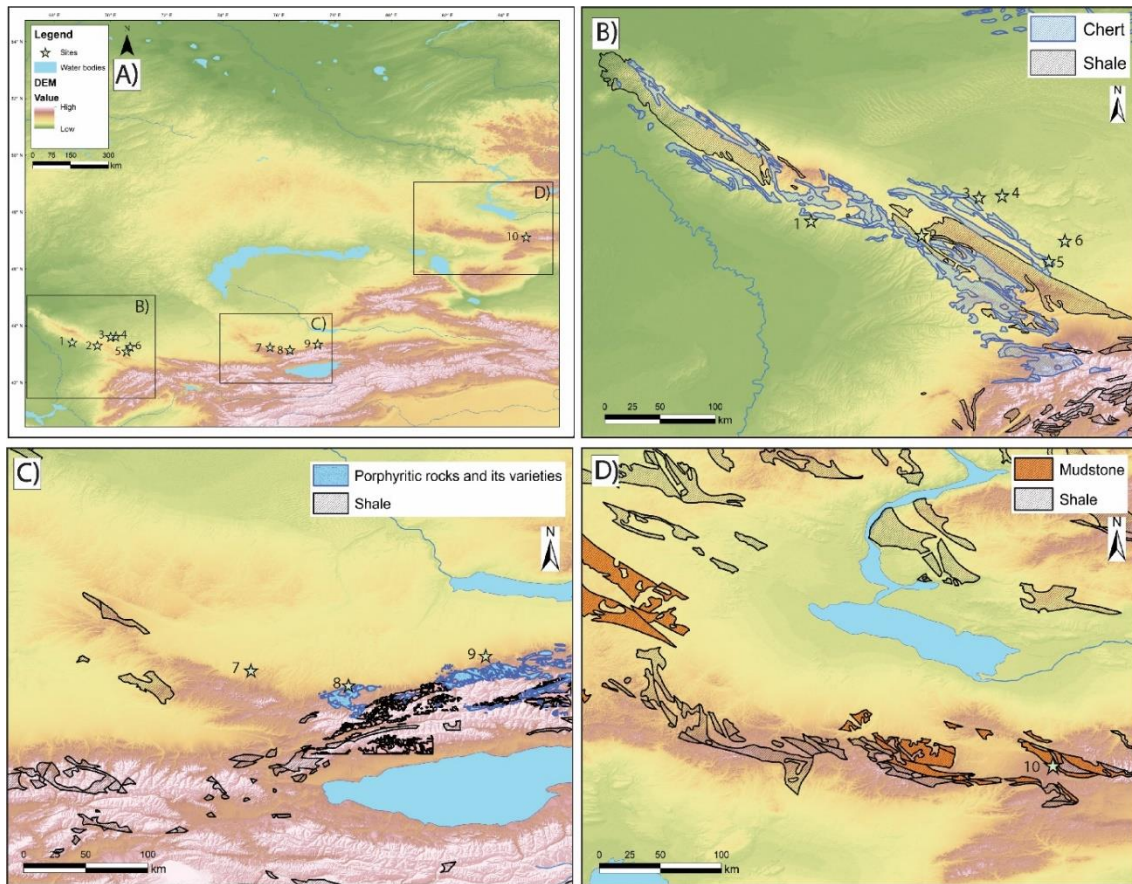
<b><i>Geology</i></b>			
<b>Site ID</b>	<b>Position</b>	<b>Location (province)</b>	<b>Rock type</b>
Yntaly**	Primary	South Kazakhstan	Chert
Valikhanova**	Primary	South Kazakhstan	Chert
Beriktas**	Secondary	Almaty	Siltstone
Maibulaq**	Secondary	Almaty	Porphyry
Rahat*	Secondary	Almaty	Porphyry
Aqtasty	Primary	Almaty	Shale
Ushbulaq**	Secondary	East Kazakhstan	Shale
<b><i>Archaeology</i></b>			
<b>Site ID</b>	<b>Period</b>	<b>Location</b>	<b>Reference</b>
Valikhanova	Upper Palaeolithic	South Kazakhstan	Fitzsimmons et al., 2017
Qyzyltau**	Lower-Upper Palaeolithic	South Kazakhstan	Derevianko et al., 2006
Buirekbastau- Bulaq*	Upper Palaeolithic	South Kazakhstan	Kunitake and Taimagambetov, 2021
Usiktas*	Upper Palaeolithic	South Kazakhstan	Alpysbaev, 1979
Sorkol*	Neolithic	South Kazakhstan	Alpysbaev, 1979
Maibulaq	Upper Palaeolithic	Almaty	Fitzsimmons et al., 2017
Rahat	Upper Palaeolithic	Almaty	Ozherelyev et al., 2019
Ushbulaq	Upper Palaeolithic	East Kazakhstan	Anoikin et al., 2019

## **4.2 Methods**

### **4.2.1 Geoarchaeological survey**

As the basis of the current doctoral dissertation, I conducted a systematic geoarchaeological survey to locate sources of raw materials utilized at Palaeolithic complexes in the IAMC. The field survey continued during the two field seasons in 2019 and 2021. I aimed to concentrate around previously known stratified sites and surface lithic scatters located in the foothill zones of the Qaratau, Ili Alatau, and Tarbagatai ranges. The chief goal was to cover a 20 km radius from the sites to locate sources of knappable raw materials in primary and/or secondary contexts. The exploratory survey was conducted by car and on foot. Similar survey strategies were previously successfully implemented in locating raw materials sources in Eurasian prehistory (Spinapolice, 2012; Ghasidian and Heydari-Guran, 2018; Doronicheva et al., 2019).

In order to predict the location of potential raw material sources, I used digitized and georeferenced geological maps of Kazakhstan. Additionally, lithological data from the 'Mineral Deposits Database and Thematic Maps of Central Asia' ArcGIS platform developed by the Centre for Russian and Central EurAsian Mineral Studies (CERCAMS) was employed (Seltmann et al., 2014). Data on the occurrences of raw materials of interest were retrieved from the CERCAMS database as keyhole markup language (.kml) files and uploaded on field tablets to structure the survey (Figure 4). Lithologies of interest, such as varieties of porphyritic rocks, that were not available on the CERCAMS database, were first digitized then retrieved as shapefiles (.shp) from the geological maps created by the Institute of Geology of the Soviet Academy of Sciences using the open-access platform QGIS (*Geological Map of Kazakhstan and Middle Asia*). Upon finding the sources of lithologies, the GPS locations were recorded, photographed, and samples for further laboratory processing were taken. Furthermore, the knappability of lithologies of interest was tested by means of opportunistic field knapping. This is done to examine the presence of conchoidal fracturing, ease to detach a flake, and overall hardness of the material.



**Figure 4. Distribution of individual types of raw materials in the IAMC of Kazakhstan as extracted from the CERCAMS database and shown in relation to the major topography of the region and sites mentioned in the text. A) Overview of the Kazakh portion of the IAMC illustrating the study regions of the PhD thesis. B) The Qaratau, C) the Ili Alatau, and D) the Tarbagatai ranges, and distribution of raw materials used in the Palaeolithic complexes, 1) Qoshqorgan-Shoqtas, 2) Valikhanova, 3) Ynatly, 4) Sorkol, 5) Buirekbastau-Bulaq, 6) Qyzyltau, 7) Beriktas, 8) Maibulaq, 9) Rahat, and 10) Ushbulaq. Data sources: Global Administrative areas (GADM) (Hijmans, 2012), vector and raster data from Natural Earth ([www.naturelearthdata.com](http://www.naturelearthdata.com)) and Shuttle Radar Topography Mission (SRTM) Version 4 (Jarvis et al., 2008).**

#### **4.2.2 Petrographic analysis**

The geological test samples of potential raw materials and archaeological finds of lithics were cut and prepared following a standard thin section procedure. A total of 80 samples were involved in the study. The thickness of the samples was reduced on a thin section grinder until the thickness of ca. 30  $\mu\text{m}$  was reached. Traditional

petrographic analysis was conducted at the laboratory for Geoarchaeology of the Institute for Archaeological Sciences (INA), University of Tübingen, Germany. Thin sections were analyzed using a Zeiss petrographic microscope and photomicrographs were obtained using an Axio camera coupled to the microscope. Identification and description of minerals were made under plane polarised light (PPL) and cross-polarised light (XPL). A quartz lambda auxiliary plate was used to obtain additional information regarding chalcedony types (i.e., length-fast and length-slow) and the corresponding variety. The identification of chalcedony types is an important parameter to the provenance of chert artefacts. This is because length-slow chalcedony fibres are generally formed under geologic environments different than those for length-fast chalcedony (Hattori, 1989). The length-fast and length-slow term is defined as the relationship between three dimensions of the crystal and the magnitude of refractive indices. The sign of elongation is determined on the basis of whether the slow or fast component is vibrating in the longest direction of the crystal (Miehe et al., 1984). The pelitic rock identification followed the grain size classification by Wentworth (1922).

I used the *'Image J'* software to measure the percentages of inclusions and matrix of thin sections tested for mechanical properties. This is done using the point counting technique suggested by Harwood (1988). I aimed to identify the inclusion and matrix percentages to evaluate whether there is a relationship between these components with the mechanical properties of various lithologies.

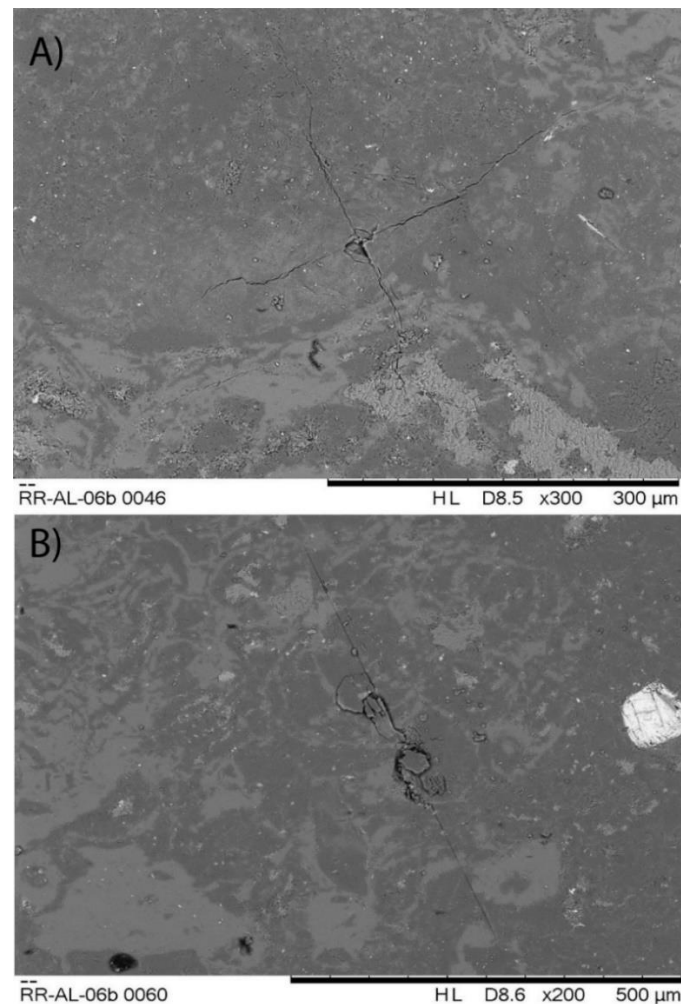
#### **4.2.3 Mechanical experiments**

I tested a total of eight samples (five archaeological and three geological) to determine their mechanical properties (i.e., hardness, elasticity, and indentation fracture resistance) (Table 2). Plane-parallel plates measuring approximately 40 × 30 × 3 mm (thickness of each sample varies) were cut and diamond polished on one side.

I used the Vickers and Knoop indentation methods that are commonly used in material and engineering sciences to measure the hardness of materials. The Vickers diamond creates square-shaped indentations, whereas the Knoop diamond produces elongated diamond-shaped indents. The indentation fracture resistance (a value that is closely related to fracture toughness) values were calculated using the Vickers indentations according to the formula proposed by Niihara et al. (1982). The elasticity (also known as Young's modulus) and hardness values were determined by the Knoop indentations



following the protocol proposed by Ben Ghorbal et al. (2017). Indentation tests were performed using an Instron 4502 universal testing machine at the laboratory for Applied Mineralogy at Tübingen University's Geosciences Department, Germany. The polished surfaces of each sample were indented 20 times for each type of indenter to obtain statistically relevant data. The size and cracks of indentations produced from diamonds was determined using images acquired with Hitachi Tabletop scanning electron microscope (SEM) TM3030. The load of the indenter was set to 100 N (with a pre-load of 10 N), speed of indentation 1 mm/s and a hold time of 20 s. The produced cracks depart from all four corners of the Vickers indentations and from the two acute angle corners of Knoop indentations (Figure 5). The detailed description of the protocol and mathematical formulations used to calculate the mechanical properties are provided in the respective manuscripts below (Appendix II).



**Figure 5. Illustration of representative indentation marks on a porphyry sample. A) Vickers indentation with cracks developed on four apical points, and B) Knoop indentation illustrating the shape of an elongated diamond.**

## 5 RESULTS

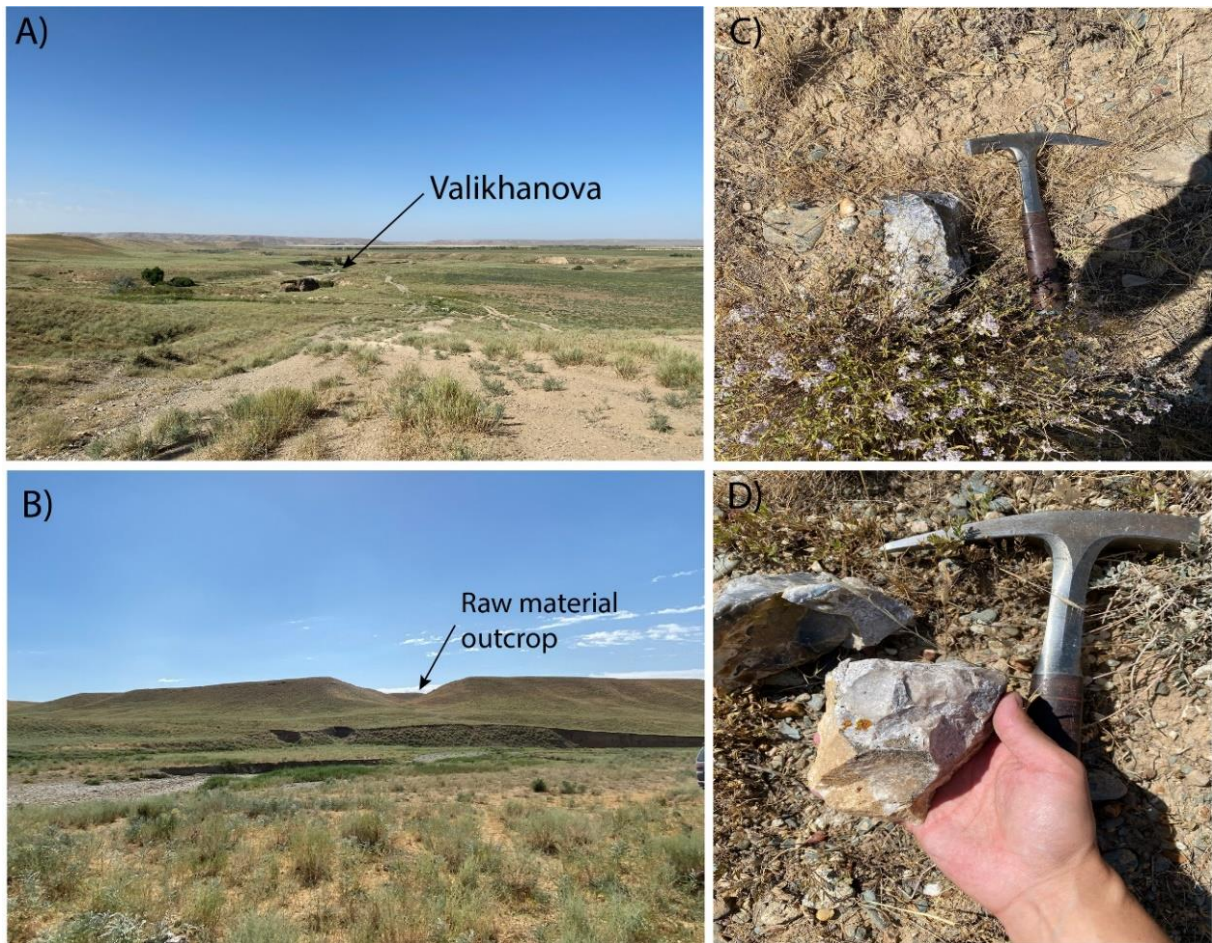
Below, I summarize the results of research papers I and III focusing on the geoarchaeological survey and petrographic analysis, and research paper II on the mechanical properties of various lithologies. These separate research projects address the objectives and research questions outlined in section 2.

### ***5.1 Geoarchaeological survey***

The geoarchaeological survey was conducted in the Qaratau, Ili Alatau, and Tarbagatai ranges. The 2019 field campaign results were published in Paper I and report the heterogeneous distribution of raw materials in different regions of the IAMC. This work highlights the regional differences in the utilisation of a particular type of raw material (Namen et al., 2022a).

In the 2021 field campaign, I mainly targeted locating sources of chert in the Qaratau region. Additionally, finding the sources of exogenous materials of shale and chert used in Maibulaq and Rahat, as well as shale in the Tarbagatai regions were included in the survey plan. The results of the 2021 raw material survey campaign have been reported in Paper III (Namen et al., 2022b).

In southern Kazakhstan, two localities with primary sources were found in the northern foothills of the Qaratau range. The first is the Valikhanova locality consisting of grey to dark-grey chert with varying degrees of porosity and microfossil inclusions. The outcrop is located close to the site of Valikhanova on the right bank of the Arystandy river (Figure 6). The majority of the chert bearing outcrop is almost completely covered with aeolian loess sediments (Figure 6B). Only small parts of the outcrop remain exposed allowing access for sampling purposes. Yntaly is another locality containing chert in the primary position. It is found in the north-eastern foothills of the Lesser Qaratau. The Yntaly chert is characterized by dark-grey to pink materials which macroscopically differs from the Valikhanova chert.



**Figure 6. Panoramic view of the Valikhanova site (A) and the raw material outcrop located on the right bank of the Arystandy river (B). The outcrop is currently covered by thick loess sediments and illustrated on C) and D). Figure adapted from Paper III.**

In the Ili Alatau range, the survey aimed to cover the area in a radius of 20 km of known stratified Palaeolithic sites. Porphyritic rocks are found in nearby stream beds in the form of pebbles and cobbles. In addition to porphyry, very fine-grained volcanic felsitic rocks are abundantly available up the Maibulaq stream. Some 20 km west of the Maibulaq site, higher quality siltstone was located in a secondary position at a locality of Beriktas (Figure 7). Preliminary, and so far, opportunistic experimental knapping suggests that both felsitic rocks and siltstones produce conchoidal fracture and have relatively better flaking properties compared to porphyry (Figure 7B). Detaching a flake from a porphyry pebble required a harder hammerstone and higher force had to be applied.



**Figure 7. Illustration of secondary position of shale found in a dried streambed at the Beriktas locality (Ili Alatau) (A), and a pebble showing a conchoidal fracturing pattern (B).**

### **5.2 Mechanical properties**

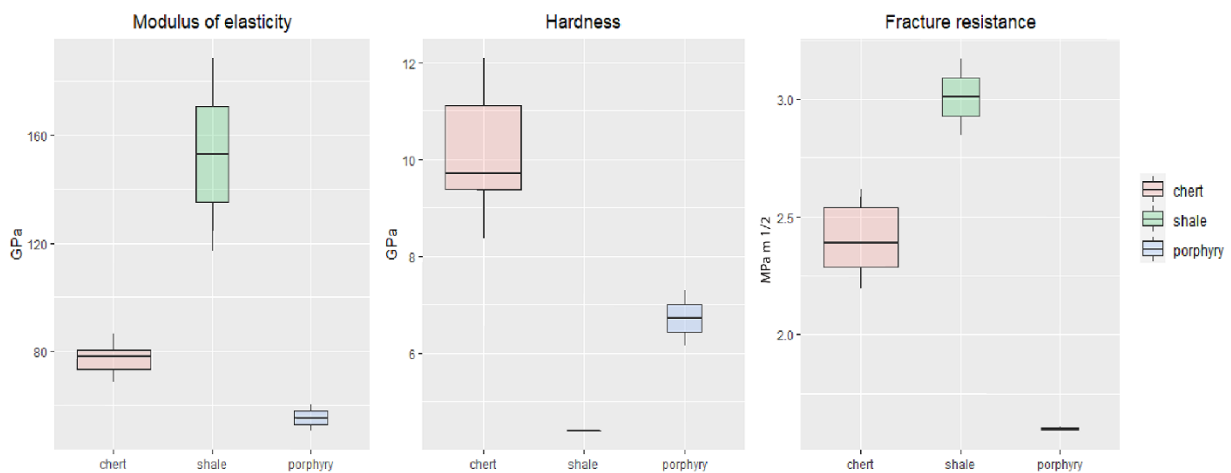
Only three out of eight samples that underwent the indentation tests yielded valuable data. This is due to the flaking off of the margins of the indentations which made the measuring of indentation diagonals and crack lengths impossible. Our results rely on a few successful indentations and must be regarded as preliminary. The values of hardness ( $HK$ ), elasticity ( $E$ ), and fracture resistance ( $K_{Ic}$ ) are illustrated in Table 3. For further details, readers are directed to the Paper II in Appendix II.

**Table 3. Hardness, elasticity, and fracture resistance values as calculated from the indentation tests. Note: the values of  $HK$  and  $E$  are given in GPa, and  $K_{Ic}$  is given in  $MPa\ m^{1/2}$**

<b>Sample ID</b>	<b>Rock type</b>	<b><math>HK</math></b>	<b><math>E</math></b>	<b><math>K_{Ic}</math></b>
UT-22	Chert	10.15	77	2.4
UB-537	Shale	4.39	153	3.01
MB-1-20	Porphyry	6.73	55	1.6

The mechanical experiments reveal that chert is the hardest material with a value of over 10 GPa, followed by porphyry (6.73 GPa) and shale (4.39 GPa) (Figure 8). The

calculated value of the modulus of elasticity suggests that porphyry is a less stiff material with a mean value equal to 55 GPa. Chert yielded a value of 77 GPa. In contrast to these samples, shale exhibits high values of 153 GPa being the stiffest rock among others. Whereas the  $K_{Ic}$  suggest that shale is most resistant to fracture with a mean value of 3.01 MPa m<sup>1/2</sup>. The value of chert is 2.4 MPa m<sup>1/2</sup> and porphyry yielded a value equal to 1.6 MPa m<sup>1/2</sup>.



**Figure 8. Boxplots illustrating the modulus of elasticity, hardness, and fracture resistance values.**

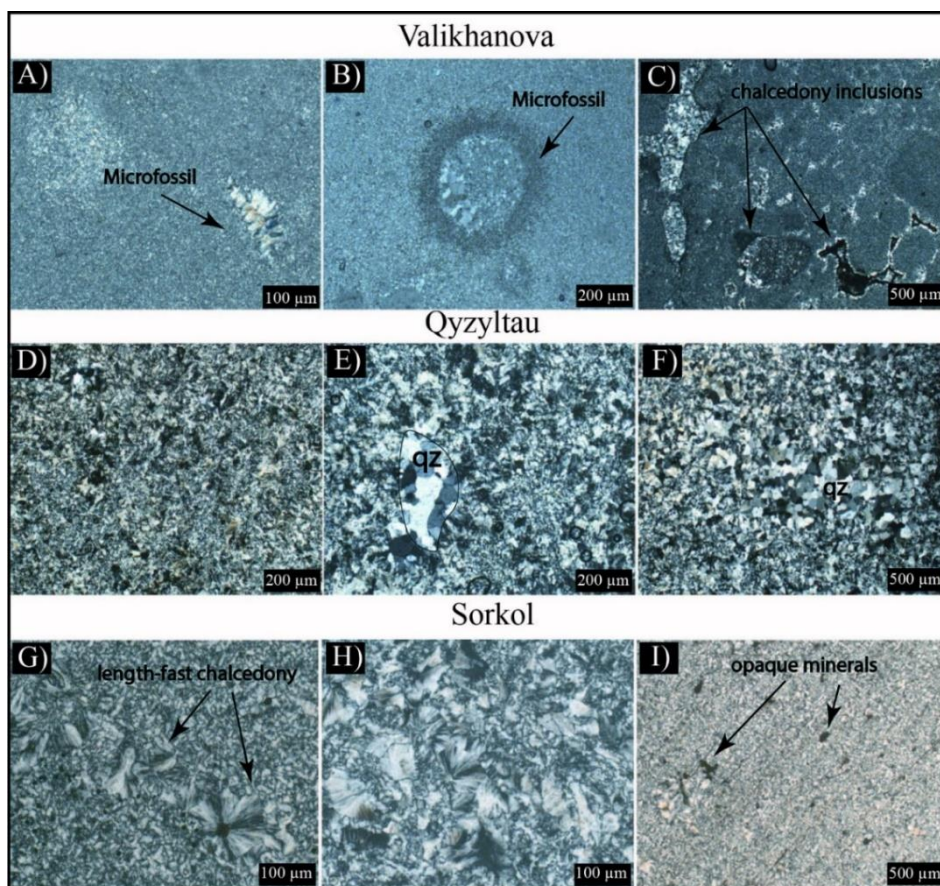
### 5.3 Petrography

The description of the petrographic analysis is presented in detail in paper III, “Insights from petrography into lithic raw material procurement in the Palaeolithic of Kazakhstan”.

The petrographic studies yielded a microstructural variability within the chert samples from the Qaratau range. Based on their petrographic characteristics, I classify them into the Valikhanova, Qyzyltau, and Sorkol cherts (see Figure 9). The Valikhanova chert is characterized by the occurrence of microfossil inclusions (e.g., radiolarian) within the finer-grained fibrous chalcedony grains and quartz veins (Figure 9A, B). In contrast, the Qyzyltau cherts consist of fibrous length-fast chalcedony grains with minor inclusions of secondary mega quartz crystals (Figure 8E). The Sorkol chert is

microscopically similar to the Qyzyltau chert with varying degrees of quartz veins running through the fibrous length-fast chalcedony grains.

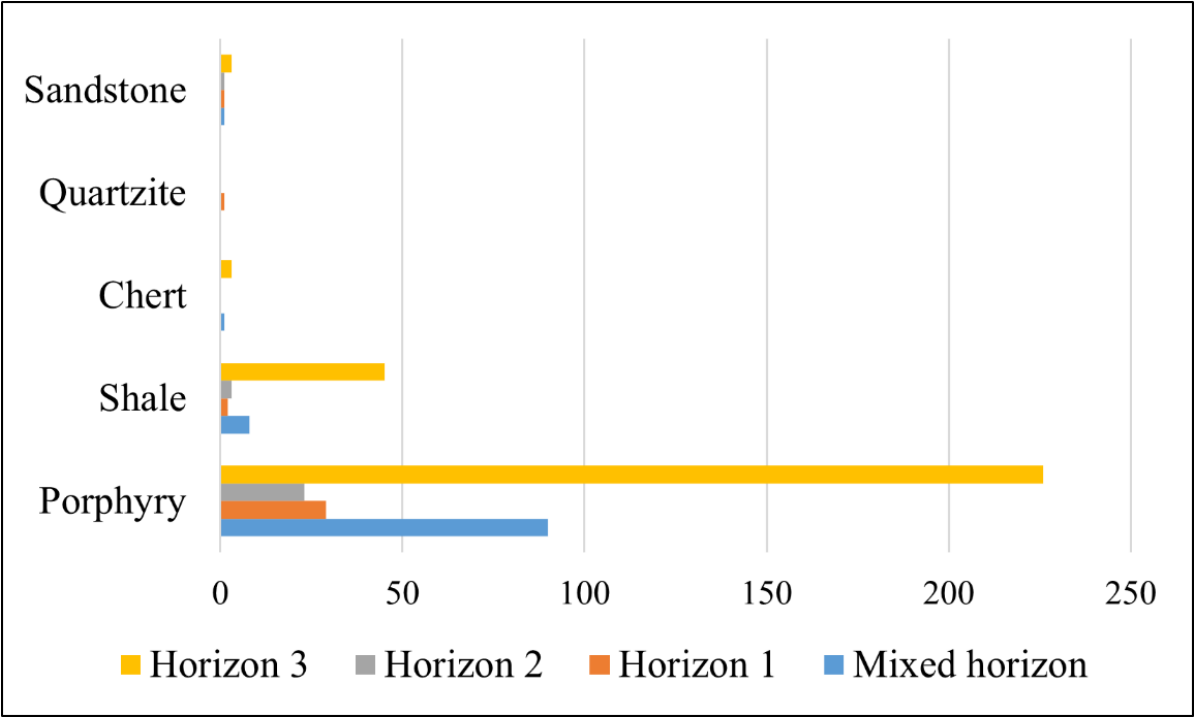
The samples of porphyry collected from the Maibulaq and Rahat sites exhibit petrographic microstructure similar to each other and consist of subangular to angular phenocrysts of quartz, quartz veins, and K-feldspars embedded in a siliceous matrix. The geological reference samples of metasedimentary pelitic rocks from the Beriktas locality consist predominantly of psammitic sandstones, greywacke, and siltstones. The piece counts of exogenous raw materials from Maibulaq are illustrated in Table 4 and graphically shown in Figure 10. It should be noted that the original number of lithics reported by Taimagambetov and Ozherelyev (2008) is larger. For the current thesis, I only studied the Almaty collections from 2004 and 2005 excavations.



**Figure 9. Photomicrographs of the chert types found in the Qaratau range. The Valikhanova chert contains microfossils (A, B) within the cryptocrystalline quartz matrix, and secondary chalcedony inclusions (C). The Qyzyltau chert consists of quartz inclusions within the cryptocrystalline quartz matrix (E, F). The Sorkol chert consists of length-fast chalcedony (G, H), and inclusions of opaque minerals (I).**

**Table 4. The piece counts of local and exogenous raw materials in each horizon at Maibulaq excavated during the 2004 and 2005 excavation seasons.**

Raw material/horizon	2004 and 2005 excavations			
	mixed	1	2	3
porphyry	90	29	23	226
shale	8	2	3	45
chert	1			3
quartzite		1		
sandstone	1	1	1	3



**Figure 10. Bar chart of the Maibulaq raw materials. The 2004 and 2005 excavations were combined.**

The petrographic analysis of metasedimentary pelitic rocks of surface lithics from Ushbulaq shows the use of silica-rich claystone, mudstone, and siltstone. All of these rocks produce a conchoidal fracture scar and should not be confused with fissile shales that consist of platy cleavage.

## 6 DISCUSSION AND CONCLUSIONS

Three methodological approaches applied in this cumulative PhD thesis address different aspects of the research questions outlined in section 2. They are based on the results of field surveys and mechanical experiments on lithic raw material quality as published in separate research papers (attached as Appendices I, II, and III). These results are highly complementary that together form a picture of the raw material choices in the Palaeolithic of the IAMC of Kazakhstan.

### ***6.1 Raw material quality as measured by means of indentation tests***

To answer my research question on the mechanical properties of raw materials utilized in the IAMC of Kazakhstan, I mainly refer to the results of my study attached to the present thesis as **Paper II**. There, I focus on the qualities of the three aforementioned lithologies predominantly used to knap stone tools in the piedmont zones of Kazakhstan (Table 3). It is important to note that this is the first time that such experiments have been conducted to examine the mechanical properties of rocks from archaeological contexts in Kazakhstan. The following is a concise summary of the discussion on raw material quality.

According to the calculations of the method described in section 4.2.3, chert is the hardest material among the other studied lithologies (see Table 3). This is an expected outcome since it is entirely composed of length-fast chalcedony, which in turn is quartz which has an average hardness of  $\approx 10$  GPa. Yet, shale, also containing primarily quartz grains and a siliceous matrix has a lower hardness value. The lower overall hardness of the rock could be due to the presence of clay impurities within the shale microstructure. (Lawn and Wilshaw, 1975). On the whole, Yonekura et al. (2008) observe that tools produced by prehistoric people often demonstrate hardness values that are higher than average values for geological samples of the same lithology. Furthermore, the modulus of elasticity ( $E$ ) also shows significant variation between chert, shale, and porphyry. The latter has the lowest  $E$  values as compared to chert and shale. This may be due to the presence of visible phenocrysts. As previous research indicates, the mechanical properties of heat-treated silcretes with larger pore-space in the samples generally show a strong loss of  $E$  values compared to finer silcrete (Schmidt et al., 2019). This observation is also supported by the relationship of grain size and mechanical properties of the rocks (see Table 5 of Appendix II). Previous



investigations also suggest the impact of petrological characteristics such as grain size influence the fracture toughness of rocks illustrating dependencies between these two parameters (Huang and Wang, 1985).

The indentation fracture toughness ( $K_{Ic}$ ) for porphyry is also relatively lower than that for chert and shale. The overall lower values of  $K_{Ic}$  imply less resistance against fracture, and perhaps also to knap tools. In addition, Wadley and Kempson (2011: 89) state that fracture toughness is a mechanical property combined with hardness and elasticity that affects the fracture of rock. Comparisons can be made between the results of the present study and the results of previous studies on the evolution of mechanical properties of silica rocks during heat treatment. The fracture resistance values of chert and silcrete from a study by Schmidt et al. (2019) vary from 1.3 to 1.85 MPa m<sup>1/2</sup>, whereas the values obtained in this work vary between 1.6 and 2.4 MPa m<sup>1/2</sup>. However, due to the preliminary character and lack of targeted research in mechanical properties of lithic materials the statements on the knappability of rocks based on the indentation fracture toughness values should be considered with some caution. The data obtained in Paper II calls for supplementary analyses of different fracture properties and experimental stone knapping. It is necessary to further bolster the statements on the effect of mechanical properties on the lithic technology.

### ***6.1.1 Implications of mechanical properties for stone tool knapping***

The comparison of the indentation test results with the lithic technology from the sites utilizing porphyry reveals distinct patterns. These patterns are particularly distinguishing for the lithic assemblage of the Maibulaq site that shows complex reduction technology. It is represented by the Levallois and radial reduction sequences. This observation has led me to focus the remainder of the discussion on the implications of the mechanical properties for stone tool knapping on the example of porphyry. In addition, I also compare the major techno-typological similarities between the Palaeolithic industries that used porphyry as a primary raw material.

It was previously considered that volcanic rocks with a porphyritic texture containing visible phenocrysts were of lower quality for knapping. However, some mechanical properties of porphyry, such as fracture toughness values that signify less resistance to fracture, can be compared to those of chert. In spite of the data obtained during this study, additional objective research aimed to investigate the fracture behaviour of

porphyry combined with traditional experimental knapping is required to explicitly discuss its knappability. Additionally, it is necessary to mention that this statement is relevant to porphyry that contains a high content of silica within its microstructure as seen under a microscope.

Nevertheless, a comprehensive literature review permits me to preliminarily compare the techno-typological characteristics of lithic assemblages featuring porphyry as a principal raw material. This step is necessary to compare the typology of knapped tools and the raw materials used. The presence of Levallois or Levallois-like technology at Upper Palaeolithic sites of Maibulaq and Rahat (Fitzsimmons et al., 2017; Ozherelyev et al., 2019), and Kattasai 1 and 2, Uzbekistan (Krajcarz et al., 2015; Kot et al., 2020; 2022), are important in comparing the lithic assemblages. The sites mentioned above have common techno-typological features with several schemes of reduction. They are represented by the radial reduction of cores along with single and double platform parallel reduction (Fitzsimmons et al., 2017; Pavlenok et al., 2021). The Maibulaq assemblage also contains bladelets knapped on porphyry indicating its properties for bladelet detachment (Figure 2). These technological similarities allow me to hypothesize that they could be associated with the mechanical properties of porphyry. Taking into consideration the presence of large phenocrysts within the porphyry matrix and the difficulty to detach a flake, I should emphasize that the radial reduction of cores and production of Levallois tools is, by itself, a technologically challenging task. It requires a combination of knapping skills and relatively good materials. The production of such technologically sophisticated tools using lower quality lithologies is practically not possible. Hence, the suitability of porphyry for knapping sophisticated tools is confirmed by the presence of Levallois and radial reduction technologies. It is further bolstered by the indentation test results demonstrating that its mechanical properties are comparable to those of chert.

## ***6.2 Raw material procurement strategies in the IAMC of Kazakhstan***

In this section, I correlate the results of **Papers I, II, and III** to address different aspects of the research question on the lithic raw material procurement strategies in Kazakhstan. There, I broadly discuss this subject based on the results of the geoarchaeological survey of raw materials (**Paper I and II**). As a consequence, my data suggests that prehistoric hominins used various procurement strategies (Binford,

1979). Earlier investigations of the lithic assemblages from Valikhanova report the procurement of only local, or predominantly local raw materials as in the case of Maibulaq and Ushbulaq (Taimagambetov, 1990; Taimagambetov and Ozherelyev, 2008; Anokin et al., 2019; Ozherelyev et al., 2019). I will concentrate my discussion based on the example of the Maibulaq site. It presents a rather interesting platform to examine the procurement strategies due to the raw material availability and intra-site variation as reported in section 5.1.

### **6.2.1 Patterns of procurement**

As the results indicate, the Maibulaq valley and eponymous stream bed largely offer various volcanic rocks in the form of alluvial conglomerates for the production of stone tools. This includes porphyritic rocks with large quartz and feldspar phenocrysts along with fine-grained felsitic rocks that produce conchoidal scars upon knapping. Within 20 km west of Maibulaq, a secondary source of metasedimentary fine-grained pelitic rocks (predominantly siltstone) is also available as alluvial and colluvial conglomerates (Figure 7) (Namen et al., 2022b). The number of Maibulaq lithic raw materials from 2004 and 2005 excavations including three archaeological horizons shows predominant exploitation of porphyry as compared to locally available felsitic or sub-local pelitic rocks (see Table 4 and Figure 10). Despite the availability of various rocks for knapping, the results imply that the hunter-gatherers favoured the use of porphyry.

In this context, I should note that the Maibulaq site was presumably used as a long-term open-air occupation settlement due to the presence of multiple lithic-bearing layers and fire features in the lowermost cultural horizon three (Fitzsimmons et al., 2017). The aforementioned results indicate that the procurement seems to be largely concentrated in a single sector i.e., provisioning of alluvial conglomerates of porphyry adjacent to the site. This suggests direct procurement of raw materials indicating a restricted procurement area along the stream and valley. However, the presence of easily knappable felsitic rocks in the same stream was disregarded as seen from the lithic assemblage (Table 4). This observation recommends that toolmakers, despite the restricted and direct procurement, were highly selective in their choice of *local* raw materials and that they perhaps optimized something other than the ease of flaking. Additionally, the Maibulaq settlers did not exploit siltstones accessible within a half day's walking distance from the camp site (Binford, 1979; Gould and Saggars, 1985).

The presence of a small fraction of exogenous raw materials most likely was the result of the other activities carried out around the site ('embedded procurement') and subsequently brought by the hunter-gatherers (Binford, 1979; Tomasso and Porraz, 2016; Moncel et al., 2019).

### **6.2.2 The effect of raw material quality on procurement strategies**

The patterns of procurement at Maibulaq suggest that hominins had the knowledge in recognizing physical properties of different raw materials which they incorporated in their selection and acquisition process. The assessment criteria of these properties could be viewed as the flintknapper's knowledge of working with such materials or gained through continuous experiments evaluating the qualities of locally available lithologies. These conjectures are still largely impossible to test. Nonetheless, the preference of porphyry over local felsitic or sub-local pelitic rocks available within 20 km was perhaps driven by its mechanical characteristics (see section 5.2). The preliminary results of the actualistic mechanical experiments demonstrating the quality are also in concert with the aforementioned presumption (Paper II).

Additionally, the selection of explicitly porphyritic rocks could be related to other physical properties that were not investigated in this work, i.e., the ability to resist abrasion. In this context, Braun et al. (2009) suggest that rock types with relatively high abrasion hardness were frequently utilized in Palaeolithic complexes with frequent butchering activities and to process large mammal carcasses. Such subsistence activities may have influenced the selection behaviour of hominins to preferentially choose porphyry that could withstand the abrasiveness. However, because the abrasion rate of porphyry is not yet known, this hypothesis requires laboratory testing to evaluate. Previous experimental works implemented to study the abrasion rate and edge-wear of stone tools from Africa and Eurasia prove the effectiveness of such objectives studies to answer questions of procurement (Lerner et al., 2007; Braun et al., 2009; Iovita, 2011; Abrunhosa et al., 2019; Chen, 2020).

The preliminary, and so far, opportunistic field knapping suggests that a harder hammerstone and much higher force is required to detach a flake from a porphyry pebble. This observation raises additional questions about the selection of this type of rock. Nevertheless, more detailed research investigating the knapping quality and different fracture mechanics (e.g., indentation fracture toughness, fracture strength,

modulus of elasticity etc.) is necessary. This approach combined with experimental knapping will help to understand the factors why the Maibulaq settlers preferred porphyry over other available rocks (see for example Mardon et al., 1990; Domanski et al., 1994; Doelman et al., 2001; Webb and Domanski, 2008; Manninen, 2016; Schmidt et al., 2019).

The direct procurement of local raw materials suggests that the Maibulaq occupants did not have the necessity to travel long distances to acquire knappable materials. In spite of this, the opportunistic provisioning strategy cannot be applied to Maibulaq due to the highly selective acquiring approach of local lithologies. I may, therefore, hypothesize that the quality of porphyry was deemed suitable for the production of end products, or the hominins had to adapt their knapping technology to its quality. Ghasidian and Heydari-Guran (2018) arrived at a similar conclusion in discussing the Upper Palaeolithic settlement patterns. However, they emphasize the opportunistic procurement of the local raw material as a function of decreasing time and energy costs which led to the adaptation of knapping techniques to the available resources (Ghasidian and Heydari-Guran, 2018). Nevertheless, the preference of porphyry over other available rocks discussed above should be viewed as a *selection*, in other words, a *deliberate technological act*. The criteria guiding the selection of explicitly porphyritic rocks were driven by a number of factors including the technological skills and perhaps familiarity of working with these materials. This is expressed in the ability to produce technologically challenging tools (see sections 1.3 and 6.1.1 for discussion). Overall, considering the intra-site variability of raw materials at Maibulaq, I suggest the *direct selective procurement* strategy was a primary setting for the technological and territorial organization.

### **6.3 Concluding remarks and future directions**

This cumulative PhD dissertation examined the raw material selection choices in the Palaeolithic complexes of Kazakhstan by performing three separate studies, which relied on three distinct methodological approaches. The first research project studied the raw material utilization patterns in the piedmont zones of Kazakhstan by conducting systematic field surveys. Subsequently, in the second project, I applied objective tests (see section 4.2.3) to investigate the mechanical properties of various rocks used in the Palaeolithic complexes of Kazakhstan. This approach produced valuable data

concerning the fracture resistance of chert, shale, and porphyry. Finally, the third project combined field survey with petrographic analysis to study the types of sedimentary and volcanic raw materials in the IAMC. The combination of the methodologies proved valuable in the investigation of raw material selection behaviour and procurement patterns.

As a result, I provided objective considerations regarding the raw material selection choices in the Late Pleistocene of Kazakhstan. The following three inter-related aspects have driven me in concluding this work:

- ✦ *The raw material utilization patterns in the IAMC of Kazakhstan.* The documentation of the utilization differences in various geographic areas of the IAMC of Kazakhstan (**Paper I**) provided a basis to determine the types of raw materials used at different Palaeolithic sites. This approach laid the groundwork for further extensive investigations of raw material variability, quality, selection, and procurement patterns by prehistoric hominins. A systematic survey and reconstruction of the raw material distribution (Figure 4) in the study regions further assisted in refining the sampling criteria and selection of representative geological and archaeological samples. This approach was required to objectively examine the raw material qualities.
- ✦ *The raw material quality effect on the production of stone tools.* Based on the data gathered in the first study, I collected samples of chert, shale, and porphyry from each study region. Consequently, the results of the objective analysis on these lithologies indicate that raw materials previously reported as lower quality have some mechanical properties that can be compared to chert (**Paper II**). A number of sites that predominantly utilize porphyry as a principal raw material contain Levallois, Levallois-like, and radial reduction sequences. The presence of these reduction techniques demonstrates its quality for knapping technologically demanding tools (see discussion in section 6.1.1). Such objective tests highlight the importance of the mechanical properties of porphyry and its effect on the production of stone tools. My findings show that materials that are expected to be difficult to knap based on visual criteria objectively may have some good fracture properties. This kind of analysis demonstrates that the conclusions on the quality of raw materials need to be based on objective criteria rather than subjective ones.

- ✦ *The link between raw material quality and procurement strategies.* Considering the qualities of porphyry as shown in the second research project, **Paper III** evaluated the raw material procurement strategies at Maibulaq. There, I focused on the influence of raw material quality on procurement and selection strategies (**Paper II and Paper III**). Based on the mechanical properties and intra-site raw material variation at Maibulaq, I suggested a direct but selective procurement strategy that specifically targeted the provisioning of porphyry. The assumption that porphyry was procured due to its quality has a strong base as shown by the objective tests. Additionally, hominins perhaps chose porphyry due to its hardness and resistance to abrasion. These properties were essential for skinning and butchering of animals, as well as other subsistence-related activities (Braun et al., 2009).

It is important to highlight that this is the first study to examine the mechanical properties of various lithologies and raw material procurement strategies from Kazakhstan. The methodology used for systematically investigating raw material distribution in Kazakhstan is applicable to other vast regions presenting heterogeneous distribution of knappable materials. An objective approach to examine various mechanical properties of raw materials contributes to the theory of quality and what constitutes a better material for knapping. My study calls for a reconsideration of 'higher versus lower' quality terms, based on the results of the actualistic tests and archaeological records. Ultimately, the work initiated in the current PhD thesis laid the groundwork for future studies of raw materials in Kazakhstan and Central Asia.

In the future, it is necessary to conduct further detailed studies to investigate the different fracture behaviour of rocks used by prehistoric hominins. Additionally, objective tests targeted to evaluate the abrasion rate of porphyry would greatly contribute to answering questions on the selection of porphyry.

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# LIST OF PUBLICATIONS FOR CUMULATIVE DISSERTATION

Three publications have been submitted in fulfilment of the requirements of this cumulative dissertation. Numbers in parentheses represent percentages of my own contribution to the articles or manuscripts (original ideas/data analysis/writing/publication).

## *Peer-reviewed publications:*

### **Paper I** (100/100/90/90)

**Namen, A.**, Cuthbertson, P., Varis, A., Schmidt, P., Taimagambetov, Zh., Iovita, R., 2021. Preliminary results of the first lithic raw material survey in the piedmont zones of Kazakhstan. *Asian Archaeology*. doi:10.1007/s41826-022-00051-3

### **Paper II** (90/90/100/90)

**Namen, A.**, Iovita, R., Nickel, K., Varis, A., Taimagambetov, Zh., Schmidt, P., 2022. Mechanical properties of lithic raw materials from Kazakhstan: Comparing chert, shale, and porphyry. *PLOS ONE*. doi:10.1371/journal.pone.0265640

### **Paper III** (100/90/100/90)

**Namen, A.**, Schmidt, P., Varis, A., Taimagambetov, Zh., Iovita, R., 2022. Preference for porphyry: Petrographic insights into lithic raw material procurement from Palaeolithic Kazakhstan. *Journal of Field Archaeology*. doi:10.1080/00934690.2022.2092265

## APPENDIX I

This is the published version of the following article: **“Preliminary results of the first lithic raw material survey in the piedmont zones of Kazakhstan”**. The manuscript has been accepted and published at *Asian Archaeology* journal [doi:10.1007/s41826-022-00051-3.]

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# Preliminary results of the first lithic raw material survey in the piedmont zones of Kazakhstan

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## Abstract

The study of raw materials was comprehensively studied in European and African Palaeolithic. However, systematic research of raw material sourcing has not been undertaken for the Palaeolithic of Kazakhstan, such surveys being embedded in reconnaissance works aimed at discovering new Palaeolithic sites. Our work presents preliminary results of the first lithic raw material survey in Kazakhstan. This study distinguishes the geographic patterns of land-use and their correlation with the stone tools from stratified sites. We describe primary and secondary sources of raw materials and compare macroscopically with the lithic assemblages. The survey results show a heterogeneous distribution of raw materials throughout the study regions. Macroscopic observations of lithic assemblages, and data extracted from literature suggest that hominins primarily selected local raw materials. Regional differences in the utilisation of a particular type of raw material which can be observed through the macroscopic examination of the lithic collections are confirmed by survey results.

**Keywords** Pleistocene · Palaeolithic archaeology · Geological maps · Stone tools · Central Asia · Raw material survey

## 1 Introduction

As the study of lithic raw materials has grown in importance, this method has begun to reveal new aspects of technological evolution in Prehistoric communities. In the last few decades, the study of lithic raw materials has tended to focus

on hominin mobility strategies, land-use patterns, and raw material transfer. Specifically, scholars have argued that raw material transport over larger distances is a signature of modern behaviour (Bar-Yosef 2002), corresponding to the extent of territories and social networks of human groups. Examples of such transport behaviour are known throughout the Middle and Upper Palaeolithic of Europe, Africa, the Near East, Australia (Binford 1980; Feblot-Augustins 1993, 2009; Bar-Yosef 2002; Sano 2010; Ekshtain et al. 2017; Frahm and Hauck 2017; O’Leary et al. 2017; Caruana et al. 2019; Ditchfield and Ward 2019; Frahm and Tryon 2019) and some evidence is known in Central Asia (Khatsenovich et al. 2020). In addition, the studies of physical and mechanical properties of rocks can reveal the purposeful raw material selection behaviour of hominins (Domanski et al. 1994; Lerner et al. 2007; Schmidt et al. 2017; Schmidt et al. 2019, 2020), which contributes to the debate on what structured raw material sourcing strategies can be considered to constitute modern behaviour.

To date, raw material sourcing has not been attempted for the Palaeolithic of Kazakhstan. Since the 1950s, local and international teams of archaeologists conducted systematic reconnaissance surveys primarily in southern and eastern Kazakhstan, targeting the discovery of new Palaeolithic

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sites (Alpysbaev 1961a, b, 1972a, 1979; Taimagambetov 1997, 2006; Iovita et al. 2020; Anoikin et al. 2019). These surveys revealed a number of surface lithic scatters, reported as open-air sites which are abundant throughout the country, specifically in the foothills of the Qaratau range (Alpysbaev and Kostenko 1974; Derevianko et al. 2002), Mangyshlaq (Derevianko et al. 1999), and Semisbugu (Medoev 1982). Dating such surface sites is currently impossible and, therefore, they remain understudied. Until now, the age of these sites has been estimated on the basis of stone tool typology, tentatively ranging from the Lower to the Upper Palaeolithic periods. In addition, a few stratified sites containing large assemblages of stone tools have been found in different sedimentary contexts (see details in Alpysbaev 1979; Derevianko et al. 1998; Taimagambetov 2009; Shunkov et al. 2017; Dzhasybaev et al. 2018; Anoikin et al. 2019) and the majority of these have been dated to the Late Pleistocene.

In the available literature (Anoikin et al. 2020; Taimagambetov 2012), the raw materials of these lithic collections have only been briefly described, stating the properties and their availability by site. More systematic works are required to further develop raw material sourcing studies in Kazakhstan. In this study, we sketch the raw material distribution in southern, south-eastern, and eastern Kazakhstan, specifically targeting foothill and piedmont zones of mountain ranges. In addition, we distinguish (1) the geographic use patterns of raw materials and (2) their correlation with the lithic assemblages of the stratified sites. The study is based primarily on surveying outcrops, collecting, and sampling of any potential sources of raw materials, and also on a comprehensive review of the literature.

## 2 Study areas

The study areas of the present study have been chosen to complement the goals of the PALAEOSILKROAD (PSR) project, which aims to discover new Palaeolithic sites in different geological and geomorphic contexts in Kazakhstan, and to test the hypothesis that Pleistocene dispersals correlated with climatic pulses during the last Glacial Cycle (ca. 110,000–15,000 years ago). These study areas are located within the Inner Asian Mountain Corridor of Kazakhstan (henceforth, IAMC), the terms introduced by Frachetti (2012) to define a group of mountain ranges that covers most of the Central Asian states. The IAMC stretches from Uzbekistan in the south, through Kazakhstan and Kyrgyzstan to Mongolia to the north-east. The geographical position of the area has played an important role in the hominin occupation and probably further dispersal to Northern and Eastern Asia (Li et al. 2019; Zwyns et al. 2019; Iovita et al. 2020).

Approximately 211,500 km<sup>2</sup> (or 47%) of the IAMC lies in the territory of modern day Kazakhstan (Cuthbertson

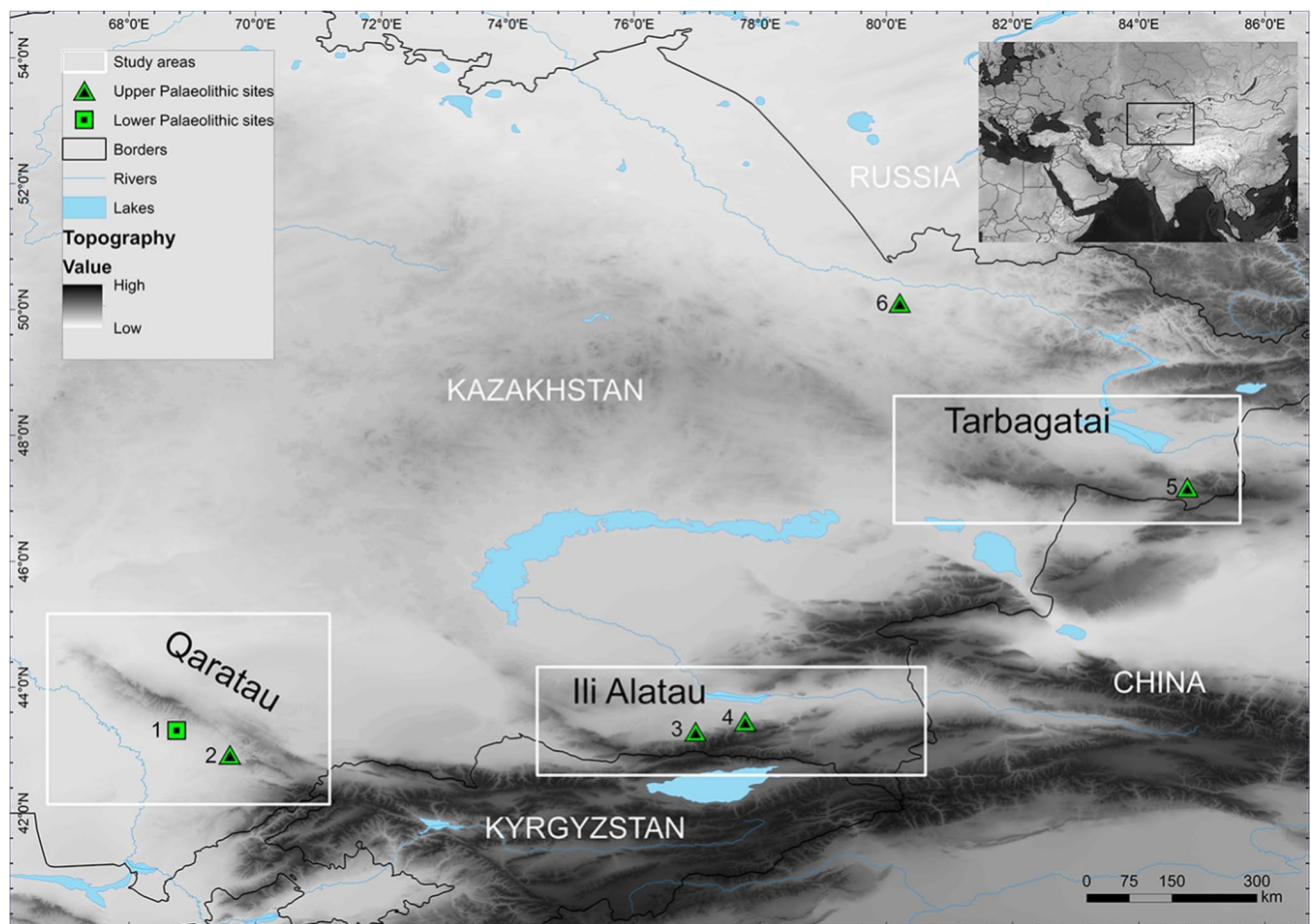
et al. 2021). We primarily focused on the Kazakh portion of the IAMC that is comprised of the Qaratau, Ili Alatau and Tarbagatai mountain ranges (Figs. 1, 2, 3) described below from west to east. These are also the most promising areas to retrieve data concerning raw material distribution, as most of the known stratified Palaeolithic sites are located within them.

*The Qaratau range* is a horn of the Tien Shan mountain system located in the southern part of Kazakhstan and extending about 450 km towards the north-west and south-east. It is sometimes divided into Lesser and Greater Qaratau separated by 25 km of relatively flat lands. In terms of lithology, the Qaratau range consists of carbonate beds and uplifted limestone. There are a number of Palaeolithic finds in the piedmont zones, which have been known since the 1950s (Alpysbaev 1961a, b, 1972a, b, 1979). The most significant ones are Valikhanova (Alpysbaev 1979; Taimagambetov 1990), the chronology of which spans 43.5 – 9 ka (Fitzsimmons et al. 2017), and the Qoshqorgan-Shoqtas complex dated to 500 – 90 ka by ESR method (Derevianko et al. 2000; Derevianko 2006). The abundant evidence for occupation in and around the Qaratau range makes this area especially interesting as a focus of raw material study, which has the potential to shed light on the wider land-use patterns and tool-use decisions of hominins.

*The Ili Alatau* is a portion of the Tien Shan mountains located in the south-eastern part of Kazakhstan. The main bedrock consists of the volcanic granitoid lithology from the Devonian period, and the areas comprised of carbonate beds are located in the south-eastern portion of the range. The foothills contain thick deposits of loess, the thickness of some areas reach up to 700 m (Dodonov 2007). Surveying exposed loess sections is the most promising technique in terms of locating new Palaeolithic sites in this region. Previously, two Upper Palaeolithic stratified loess sites (Maibulaq and Rahat) have been located and studied by local and international teams of archaeologists (Taimagambetov 2009; Fitzsimmons et al. 2017; Ozherelyev et al. 2019).

*The Tarbagatai range* is located on the border of the East Kazakhstan with north-western China and stretches 300 km from west to east. The lithology of the beds is comprised of volcanic rocks, primarily granites and diorites, and fine-grained shale outcrops are distributed in some areas. The stratified Upper Palaeolithic site of Ushbulaq is located in the eastern portion of our study area making it particularly interesting for further systematic survey.

Because these areas cover a large territory (more than 2000 km from west to east), which includes much of the area of the IAMC in Kazakhstan, they provide an opportunity to address several research questions relating to hominin occupation and dispersal through the region.



**Fig. 1** Known stratified Lower and Upper Palaeolithic sites in Kazakhstan, illustrated in relation to the major topography of the study areas. 1) Qoshqorgan-Shoqtas complex. 2) Valikhanova, 3) Maibul'aq, 4) Rahat, 5) Ushbulaq, 6) Shul'binka. Data sources:

Global Administrative areas (GADM) (Hijmans 2012), vector and raster map data from Natural Earth ([www.naturalearthdata.com](http://www.naturalearthdata.com)) and Shuttle Radar Topography Mission (SRTM) Version 4 (Jarvis et al. 2008)

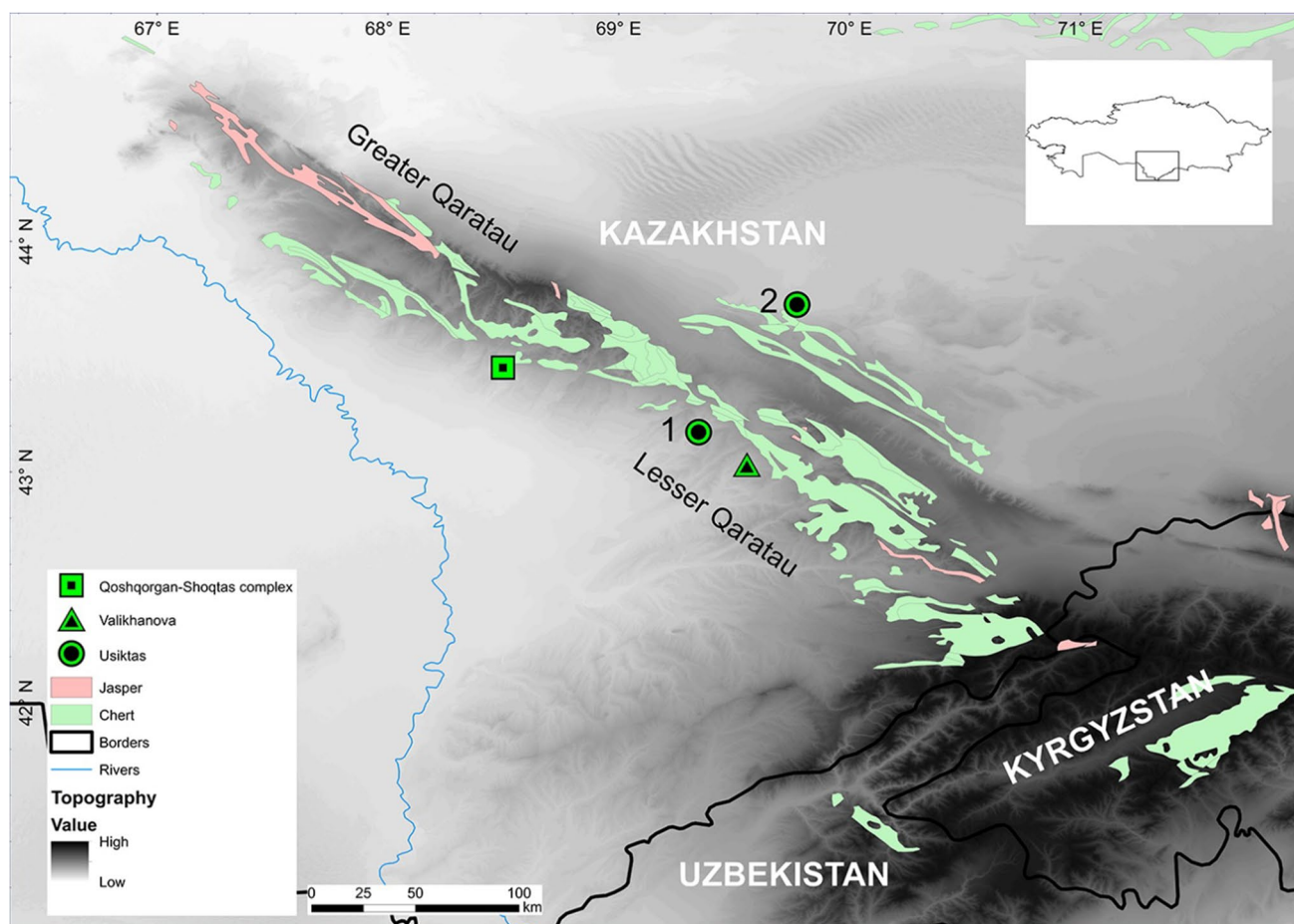
### 3 Survey

A major task for creating any raw material database is having a wide range of samples from potential sources that can be compared with archaeological lithic assemblages. In order to acquire these samples, we undertook a series of field surveys with the aim of locating and characterising raw material sources, and distinguishing regional variations. The exploratory raw material survey, and collection of geological samples were both conducted as a part of the PSR project's fieldwork during the 2018 and 2019 seasons (Iovita et al. 2020; Namen et al. 2020). For the recognition, and locating of potential raw material sources, georeferenced and digitized Soviet geological maps (1:200 k and 1:500 k), as well as lithological data from the ArcGIS platform developed by the Centre for Russian and Central EurAsian Mineral studies (henceforth CERCAMS) 'Mineral Deposits Database and Thematic Maps of Central Asia' (Seltmann et al. 2014) were employed. The latter aided in the retrieval of data on

the occurrences and distribution of individual types of raw materials in our study regions.

During the survey, both primary outcrops and secondary sources from waterways such as river cobbles and pebbles were considered. All the areas of interest such as river banks, outcrops, and valleys were surveyed on foot to locate possible sources of raw materials. The sample collection was based on the quality of the rocks (including cobbles and pebbles), i.e., fracturing properties and macroscopic similarities to the lithologies of the archaeological assemblages (some of the collected samples are illustrated on Fig. 4). We specifically targeted to locate sources of sedimentary rocks such as those of chert, jasper, chalcedony, silica rich shale and mudstone, as well as volcanic rocks such as porphyry. They are the main types of raw materials that were frequently utilised at various Upper Palaeolithic sites in the IAMC of Kazakhstan.

Data on the occurrences of individual types of raw materials were retrieved as KML files from the CERCAMS



**Fig. 2** Distribution of chert and jasper in the Qaratau range shown in relation with stratified Palaeolithic sites and raw material outcrops discovered during the survey. 1) Usiktas and 2) Yntaly raw material outcrops. Data sources: Global Administrative areas (GADM) (Hij-

mans 2012), vector and raster map data from Natural Earth ([www.naturalearthdata.com](http://www.naturalearthdata.com)) and Shuttle Radar Topography Mission (SRTM) Version 4 (Jarvis et al. 2008). Including Mineral deposits database (Seltmann et al. 2014)

database and loaded onto iPads to structure the survey. The data from each survey area were classed and described. The raw material occurrence points were recorded with high accuracy using a GNSS surveyor (Bad Elf GNSS surveyor BE-GPS-3300). Major raw material sources were photographed, and samples were taken for the further lab analyses.

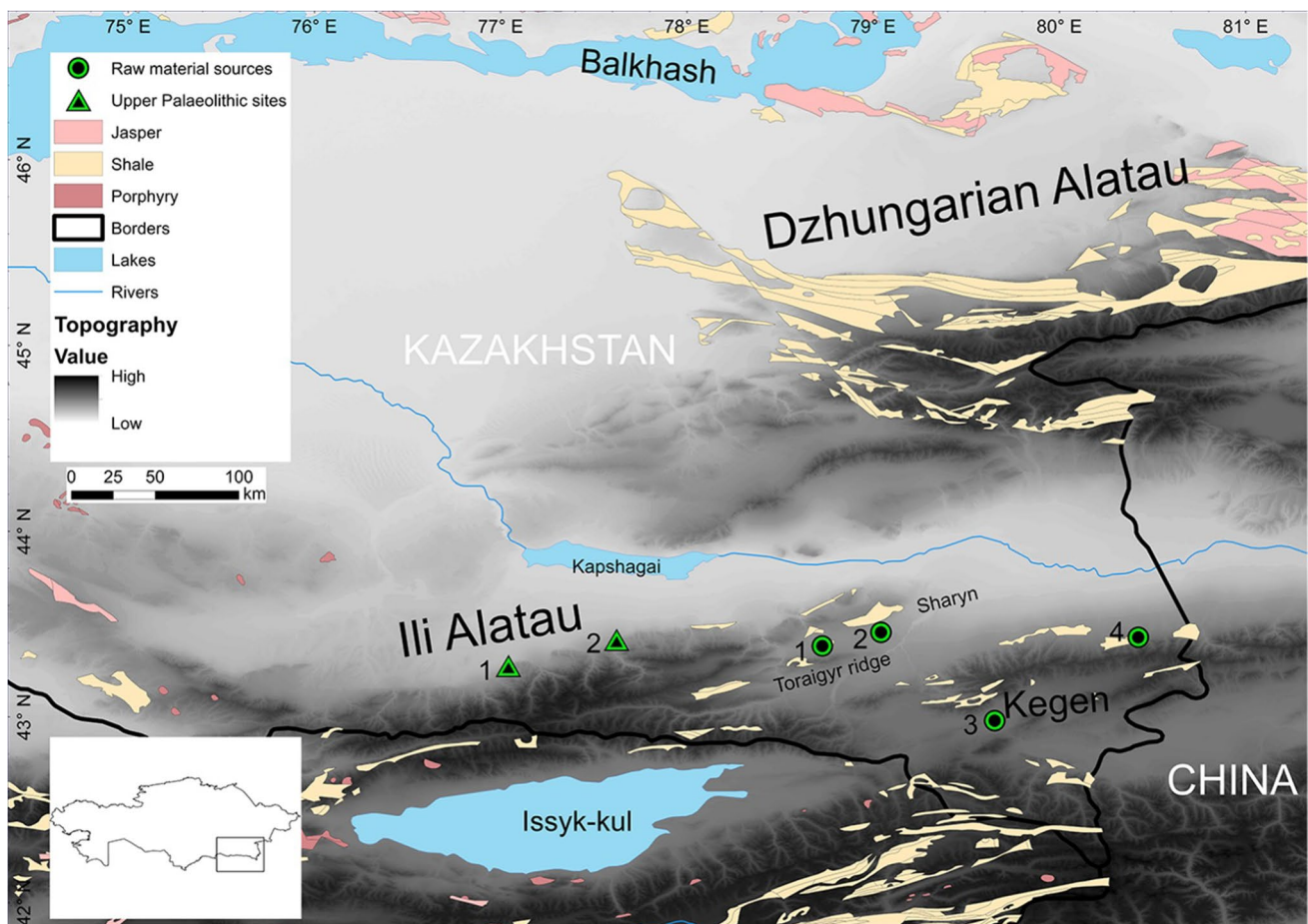
Our survey began in the foothills of the Qaratau range, then continued eastwards into the Ili Alatau and Tarbagatai ranges. The Qaratau range was surveyed as part of a cave prospection goal of the PSR project (Cuthbertson et al. 2021). In this region, we tried to locate sources of chert and jasper in primary and secondary geological contexts as it was suggested in the geological maps (Seltmann et al. 2014). The valleys of the northern and southern slopes of the range, and the adjacent territories, were extensively surveyed on foot.

In the Ili Alatau range, we mostly focused our survey on the piedmont and foothill zones. The survey began in the Kegen district which is located at approx. 2200

masl, followed by the foothills of the Toraigyr ridge, and around the Sharyn national reserve. As the foothills of the range are covered with thick deposits of loess, the primary goal here was to survey loess profiles and spring heads to find new Palaeolithic sites as well as to locate potential sources of raw materials, because these are the areas where erosion of the loess deposits would allow access to outcrops.

The survey in Tarbagatai began from the south-eastern valleys of the Saur range targeting outcrops of silica rich shales and mudstones, as well as jasper as the CERCAMS lithologies suggested the occurrence of these materials in the range. We then moved our survey westward along the northern foothills of the Tarbagatai range.

In addition, areas adjacent to stratified sites were surveyed to determine if the raw materials previously ascribed as ‘local’ were indeed locally sourced and helped decide which raw materials are more likely to have been transported from elsewhere.



**Fig. 3** Distribution of raw materials in the Ili Alatau study area shown in relation with the stratified Palaeolithic sites of 1) Maibulq, and 2) Rahat indicated as triangles, and raw material sources discovered during the survey 1) Tikenekti, 2) Sharyn, 3) Aqtasty and 4) Nazugum indicated as circles. Data sources: Global Administra-

tive areas (GADM) (Hijmans 2012), vector and raster map data from Natural Earth ([www.naturalearthdata.com](http://www.naturalearthdata.com)) and Shuttle Radar Topography Mission (SRTM) Version 4 (Jarvis et al. 2008). Including Mineral deposits database (Seltmann et al. 2014)

Where possible, all of the collected samples were then macroscopically compared with the archaeological materials from surface as well as known stratified sites. In cases where no access to the assemblages was possible the comparative data were extracted from the literature.

## 4 Results

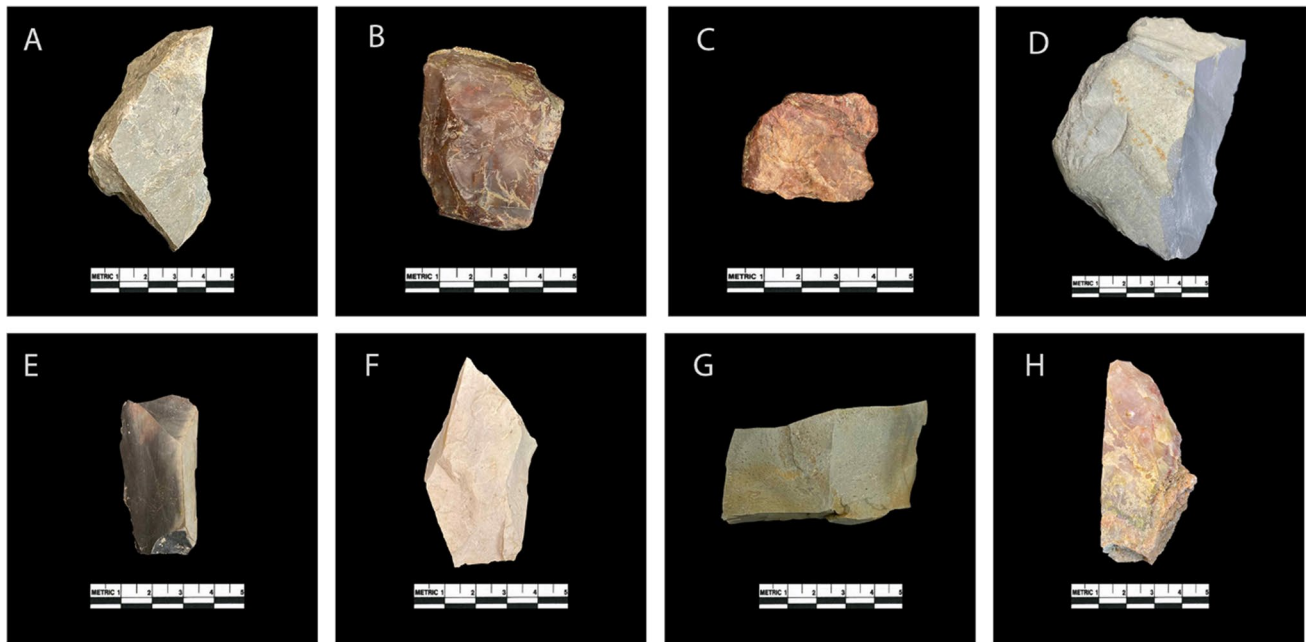
Each study region presents a unique geological evolution providing a variety of types of good quality potential raw materials for use by prehistoric human groups. Below we discuss the preliminary results of raw material occurrences and describe their geographic distribution patterns in each region separately. The size of the landmass and general geological features of study areas presents a series of unusual challenges. As one of such challenges can be considered large distances between study areas as well as thick sediment

deposits, specifically in the southern and south-eastern Kazakhstan, creating difficulties to access outcrops Table 1.

### 4.1 The Qaratau range

In this study area outcrops of chert were discovered approximately 15 km south-east of Saudakent near the village of Yntaly in the north-eastern part of the Lesser Qaratau. The landscape of the valley is characterized by cuesta topography, i.e., a sequence of beds with different degrees of erodibility that dip uniformly in the same direction. The lithology of the beds consists of fossiliferous limestone, marble limestone and conglomerates at the base of the exposed sequence. Between the limestone layers interbedded quartz and chert nodules are common. The Yntaly chert outcrop is found on a small, elongated hill which is oriented north-south. The outcropping chert materials can be described as variously white, grey, pink, and dark colours (for example





**Fig. 4** Illustration of selected raw material samples. **A)** Shale, Tikenekti **B)** Chert, Tikenekti **C)** Chert, Sharyn **D)** Shale, Ushbulaq **E)** Shale, Nazugum **F)** Shale, Aqtasty **G)** Shale, Aqtasty **H)** Chert, Yntaly

**Table 1** Description of major raw material sources discovered during the survey

Study areas	Sites	Context	Rock type
Qaratau	Yntaly	Primary	Chert
	Usiktas	Primary	Vein quartz
Ile Alatau	Aqtasty	Primary	Shale
	Tikenekti	Secondary	Shale
	Sharyn	Secondary	Chert
	Nazugum	Secondary	Shale
Tarbagatai	Ushbulaq	Secondary	Shale
	Chilibastau	Secondary	Shale
	Kokbastau	Secondary	Shale

see Fig. 4H). In a few of the collected samples, intrusive veins of quartz can be observed. Moreover, a number of lithics made of the same material were scattered on the south-western slope of the hill, whereas no lithics were found on the north-eastern slope. The abundance of archaeological lithic materials on the south-western slope of the Yntaly chert outcrop typologically reminiscent to Upper Palaeolithic indicates that the outcrop was likely used by prehistoric human groups for raw material procurement. The presence of Yntaly cave with Neolithic material culture located about 2–3 km to the north-west provides further evidence of prehistoric human occupation in this area.

We also surveyed the area around the locality of Usiktas, located in the southern foothills of the Lesser Qaratau,

10 km north-east of Algabas village. Previously, at this locality Alpysbaev (1979) discovered a cluster of lithics scattered on the surface and exhibiting various techno-typological features. The bedrock geology of the area consists of vein stockwork and limestone. During this survey, we checked the outcrops of carbonates for the presence of replacement chert. However, this survey did not yield any such outcrops. It is important to note that loess accumulation and erosion at different periods may have facilitated or hampered access to raw materials throughout the Pleistocene. It is also possible that the potential raw material outcrops are covered with the thick layers of loess that we observed during our survey.

The majority of the lithic finds in the Qaratau range and adjacent territories, as reported by earlier archaeological surveys (Alpysbaev 1972b, 1979; Taimagambetov 1990), are predominantly made on silica rocks such as chert and jasper. The prevalence of these rock types can be explained by the geological formations of the range being mainly composed of limestone. It creates a precondition for the formation of replacement chert in the carbonate host rocks that may be present as nodules or layers. According to Bushinsky (1966), certain layers of the mountain range are comprised of beds of silica rocks such as quartzite, replacement chert and even silicified shales, which may explain the large number of silica rocks in the lithic collections (see Alpysbaev 1961a; Taimagambetov 1990; Derevianko 2006). This data can also be attested by the data retrieved from the CERCAMS database which illustrates the distribution of individual types of minerals and rocks. In the CERCAMS data, we can observe

an abundant distribution of chert and jasper in the Qaratau range and the adjoining areas (see Fig. 2). Our survey results also corroborate this observation.

## 4.2 Ili Alatau

A vast area stretching approx. 500 km from east to west has been surveyed in this study area. Several localities with surface lithic scatters have been discovered. Such sites were found beside a spring in the locality of Aqtasty in the Kegen district of Kazakhstan. A concentration of lithics, mainly represented by microblades, were identified in the back dirt of a pit dug to accommodate a waterpipe. According to macroscopic analysis of the lithics, various types of raw materials were used, including a few bladelets knapped on chert, but shale being the predominant raw material type. The valley was then extensively surveyed on foot and an outcrop of fine-grained shale was found approx. 1 km north of the surface scatters. The sources of chert were not found during this survey, and so the provenance of these raw materials in the area remains unknown. The distribution of raw materials is illustrated in relation to the known stratified Palaeolithic sites and localities discovered during our survey on Fig. 3.

Additional scatter of lithics was discovered at the locality of Tikenekti located in the north-eastern foothills of the Toraygir ridge previously described in Iovita et al. (2020). Here, we found a cluster of lithics in the slopes of the foothills, which were scattered in a radius of more than 1 km. The raw materials are represented by various types of sedimentary rocks, primarily fine-grained mudstones, and shales. The samples of locally occurring shale and a single find of chert were collected (Fig. 4A and B) and sent for further lab analyses to the University of Tübingen. Other types of potential raw material sources were found around the area of Sharyn. A number of dark-red coloured fine-grained silica rocks that were macroscopically reminiscent of microcrystalline sedimentary rocks were discovered (see Fig. 4C) in a secondary context at the Sharyn district. We were not able to locate the source of this material in the wider landscape. Similar types of rocks were found at the locality of Tikenekti, 20 km south-east of Sharyn. However, we have not found lithics made of the aforementioned material. Considering the vicinity of the petrified wood located in the national park of Sharyn, it could be suggested that these could represent fragments of this material, scattered around the area, which were not suitable for knapping purposes due to its proneness to fragmentation. However, petrographic analysis is necessary to confirm this.

Raw materials of lithic collections from this region differ from those at the Qaratau sites. The Palaeolithic of the south-eastern region is represented by two stratified open-air loess sites at Maibulaq and Rahat (Taimagambetov 2009; Fitzsimmons et al. 2017; Dzhasybaev et al. 2018;

Ozherelyev et al. 2019), both located in the foothills of the Ili Alatau range. The raw materials of the lithic assemblages are mainly composed of volcanic rocks, such as porphyry, rhyolites, diorites etc., however, tools made of chert and shales are present in limited quantities. The surrounding area of these sites are characterized by uplifted volcanic beds of middle Devonian and late Ordovician granodiorites, granites, granosyenites and their porphyritic varieties. Respectively, a high number of volcanic rocks can be found locally in the form of pebbles in the river and stream beds. Their well-rounded shapes correspond to the medium to long distance transport by water action. However, primary, or secondary sources of chert or other microcrystalline silica rocks were not located in this study region. The absence of chert in the local and sub-local environments around these stratified sites raise questions about the origin of these materials found in archaeological contexts. According to the CERCAMS database and the Soviet geological maps (1:500 k), the nearest outcrops of these rocks are located in the Dzhungarian Alatau and the Qaratau range. If this is true, then we can assume that the chert lithics recovered from the stratified and surface sites, the single find of chert (Fig. 4B) discovered in the Tikenekti locality and, few bladelets from Aqtasty, were possibly transported over a long distance suggesting that hominin groups were highly mobile during the Pleistocene. However, it should also be considered that some smaller outcrops, although not located either in the CERCAMS datasets or during foot survey, might only be visible at a local level and periodically covered or uncovered by loess and colluvium at different periods.

## 4.3 Tarbagatai and adjacent territories

Our survey began in the south-eastern valleys of the Saur range to locate primary and secondary sources of raw materials. Previous discoveries of Palaeolithic sites in the area include Ushublaq (Anoikin et al. 2017; Derevianko et al. 2017; Shunkov et al. 2017), which is an Upper Palaeolithic site located in the southern slopes of the Saur range (Figs. 5, 6). The lithic assemblages are composed of fine-grained shale and mudstone with high silica content (Anoikin et al. 2017). The source of the raw materials is located about 7 km to the south-east of the site. Such raw materials were found in a stream as blocks and pebbles sized around 10–15 cm (Fig. 5). These raw materials are high quality fine-grained, structurally homogeneous and silica rich shales that readily show markers of conchoidal fracture when knapped. The pebbles have fractured and cracked exteriors that resulted from crashing together with other pebbles in the water course over a long period of time. These pebbles are not well rounded, and are coarse in size, which indicates that they were not transported over long distances. Samples were photographed (Fig. 4D) and collected for further lab analysis.

**Fig. 5** The secondary source of raw materials located 7 km east of the Upper Palaeolithic site of Ushbulaq, East Kazakhstan. A) Panoramic view of the spring and the surrounding landscape. B) View of the spring and the pebbles of raw materials brought by the stream (people as scale). C) Fractured pebble from the spring: high quality, fine-grained knappable shale



These observations correspond with the data retrieved from the CERCAMS database, which show that the surrounding area of the site is rich in silica rich shale and mudstone deposits.

Previously, the foothills of the Tarbagatai range were surveyed by Taimagambetov (2016), who found several scatters of lithics around the spring heads. Overall, our Tarbagatai survey also yielded a few localities with surface lithic scatters beside the spring heads of Kokbastau and Chilibastau (see Iovita et al. 2020), as well as secondary sources of raw materials mainly represented by pebbles in river and stream beds. The area is rich in water sources, such as river and perennial springs, and the abundance of knappable pebbles that were found in the riverbeds likely attracted human groups to the area. During this survey we were able to find only secondary sources of raw materials.

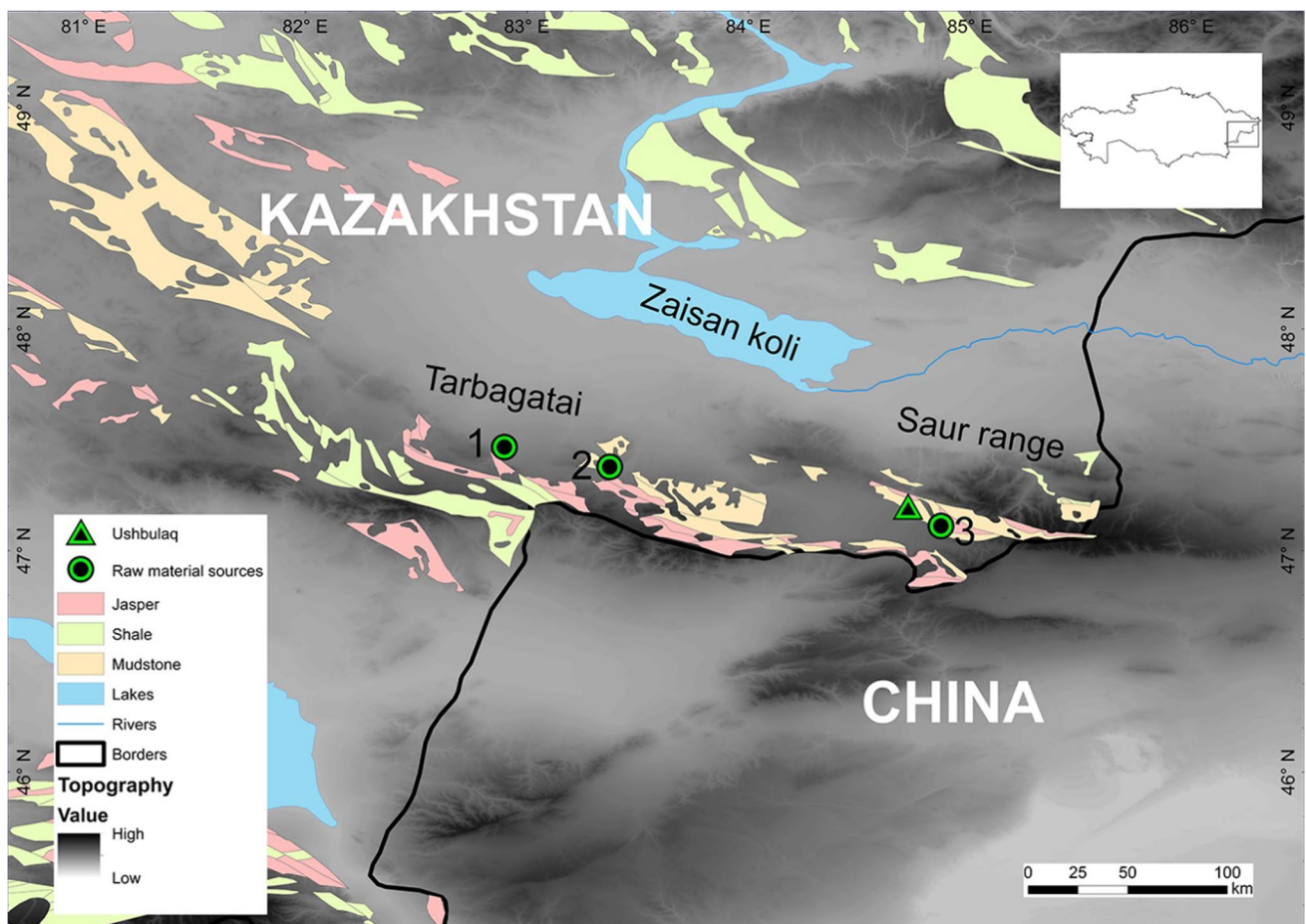
In contrast to other study areas, the stone tool assemblages from the stratified sites in eastern Kazakhstan, such as Ushbulaq, are predominantly knapped using shales and mudstones, which are fine-grained silica rich sedimentary rocks. Our survey results yielded various sources of raw materials in the region, predominantly represented by secondary sources of siliceous shales and mudstones found as pebbles throughout the foothills of the Tarbagatai range. Outcrops of flint, chert, or jasper were not located, although the CERCAMS database indicates the availability of jasper around the site of Ushbulaq, the north-eastern and the south-eastern slopes of the Tarbagatai range. The site of Shul'binka, also

located in the same administrative region around 500 km north-west of our study area, is an Upper Palaeolithic site excavated in the 1980s. The assemblages of stone tools consist of more than 5000 lithics, primarily knapped on flint, jasper, chalcedony and siliceous shale as reported in Aki-shev et al. (1978) and Taimagambetov (1983). These data suggest the availability of high-quality raw materials by the site. However, Taimagambetov (1983) reports that sources of jasper and chalcedony are located 80 km south-west of the site, which is corroborated by the CERCAMS data.

Based on the macroscopic observations of the stone tool assemblages found in both stratified and surface sites, and reviewing the available literature, it is possible to observe that the reported raw materials comprise a variety of types that differ regionally depending on the local specificity of the geological features of each region. However, detailed description of raw materials from specific layers of stratified sites were beyond the scope of the current work.

## 5 Discussions and conclusion

There has been new interest in the Palaeolithic of Central Asia providing original studies on the archaeology of Pleistocene, human occupation, and dispersals through the region (Glantz 2011; Beeton et al. 2014; Fitzsimmons et al. 2017; Khatsenovich et al. 2019; Zwyns et al. 2019; Iovita et al. 2020; Varis et al. 2022; Namen et al. 2022). However, a



**Fig. 6** Raw material distribution in the piedmont and foothill zones of the Tarbagatai range. 1) Chilibastau, 2) Kokbastau, 3) Ushbulaq. Data sources: Global Administrative areas (GADM) (Hijmans 2012), vector and raster map data from Natural Earth ([www.naturalearthdata.com](http://www.naturalearthdata.com)) and Shuttle Radar Topography Mission (SRTM) Version 4 (Jarvis et al. 2008). Including Mineral deposits database (Seltmann et al. 2014)

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lot of the basic data are missing. The current research is the first study of raw material identification in the IAMC of Kazakhstan to be used for examining Palaeolithic raw material economies, transport behaviours, and social network size. Even though preliminary, our results provide new data concerning the types of raw material, their distribution throughout the study areas and observes regional patterns of land-use.

The various raw materials in the different regions within the study area show clearly that the utilisation of raw materials varied between regions, and the hominin groups accessed a variety of landscapes with changing opportunities and limitations. We can clearly see the systematic use of accessible raw materials in both stratified and surface sites. This means that the prehistoric hominin groups predominantly utilised locally available raw materials which were suitable for knapping purposes. However, as discussed above, a few occurrences of higher quality, possibly exogenous raw materials are found at the localities of Aqtasty, Rahat and Tikenekti,

suggesting possible raw material transport over long distances (see subchapter 4.2). If so, this can be considered as an indicator of hominin mobility, and perhaps also an indicator of the value of these raw materials to hominins, in an otherwise expedient and local technology.

Further field survey will provide more details understanding of the distribution of raw material sources. Ongoing experimental analysis into the mechanical properties of various types of raw materials will complement the study of raw material selection behaviour and their effect on knapping technology. Secondly, statistical analyses of lithic assemblages from stratified sites to differentiate the raw material types are required to provide an in-depth study of landscape use, transportation of exogenous materials and hominin mobility.

A number of Palaeolithic sites in the piedmont and foothill zones of Kazakhstan are a major source of evidence for intensive hominin occupation in the region. Understanding the different aspects of raw material utilisation

in these sites still remains to be comprehensively evaluated through more substantial fieldwork and laboratory studies, but this study has provided a necessary first step towards more systematic investigation. In this article, we have described the raw material use patterns observable in a selection of assemblages in the region, and the implications of these patterns for the understanding of hominin behaviour. The results indicate that raw materials are heterogeneously distributed throughout the study regions, revealing regional raw material utilisation patterns. In conclusion, further research into the raw material landscapes of the study areas is desirable to comprehensively examine the factors influencing raw material selection and use, and also to link these factors with technological aspects of hominin behaviour in the region.

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**Data availability** The data that support the findings of this study are openly available in OSF repository via <https://doi.org/10.31235/osf.io/uztq6>

**Code availability** Not applicable.

## Declarations

**Conflict of interest** The authors declare that they have no conflicts of interest.

**Ethics approval** Not applicable.

**Consent to participate** Not applicable.

**Consent for publication** Not applicable.

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## APPENDIX II

This is the published version of the following article: “**Mechanical properties of lithic raw materials from Kazakhstan: Comparing chert, shale, and porphyry**”. The manuscript has been accepted and published at PLoS ONE [DOI:10.1371/journal.pone.0265640].

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## RESEARCH ARTICLE

# Mechanical properties of lithic raw materials from Kazakhstan: Comparing chert, shale, and porphyry

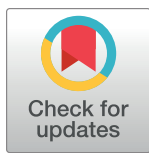
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**Data Availability Statement:** All relevant data are within the manuscript. Map data sources: Global Administrative areas (GADM), vector and raster

## Abstract

The study of lithic raw material quality has become one of the major interpretive tools to investigate the raw material selection behaviour and its influence to the knapping technology. In order to make objective assessments of raw material quality, we need to measure their mechanical properties (e.g., fracture resistance, hardness, modulus of elasticity). However, such comprehensive investigations are lacking for the Palaeolithic of Kazakhstan. In this work, we investigate geological and archaeological lithic raw material samples of chert, porphyry, and shale collected from the Inner Asian Mountain Corridor (henceforth IAMC). Selected samples of aforementioned rocks were tested by means of Vickers and Knoop indentation methods to determine the main aspect of their mechanical properties: their indentation fracture resistance (a value closely related to fracture toughness). These tests were complemented by traditional petrographic studies to characterise the mineralogical composition and evaluate the level of impurities that could have potentially affected the mechanical properties. The results show that materials, such as porphyry possess fracture toughness values that can be compared to those of chert. Previously, porphyry was thought to be of lower quality due to the anisotropic composition and coarse feldspar and quartz phenocrysts embedded in a silica rich matrix. However, our analysis suggests that different raw materials are not different in terms of indentation fracture resistance. This work also offers first insight into the quality of archaeological porphyry that was utilised as a primary raw material at various Upper Palaeolithic sites in the Inner Asian Mountain Corridor from 47–21 ka cal BP.

map data from Natural Earth ([www.naturalearthdata.com](http://www.naturalearthdata.com)) and Shuttle Radar Topography Mission (SRTM) Version 4 (<https://srtm.csi.cgiar.org/>).

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**Competing interests:** The authors have declared that no competing interests exist.

## 1 Introduction

In recent years, the study of lithic raw materials used by prehistoric hunter-gatherers for the production of stone tools has received much attention. People selected different types of rocks to influence the technology and type of tools manufactured [1–6]. Such studies can help to understand the way in which people took advantage of the mechanical attributes of rocks that affect reduction sequences and edge-wear properties of tools [2, 4, 7]. Sedimentary rocks such as chert, flint, silicified shale, and other silica rich rocks were commonly used, and this is presumed to be due to their predictable fracturing properties and good knapping qualities. Raw material quality is commonly related to the mineralogical structure (e.g., grain size and shape) and purity of a given material. In most cases, raw materials that have isotropic mechanical properties are often considered to be of higher quality for tool making [1]. Microstructural characteristics are also thought to potentially affect the size and/or shape of a final knapped product [2]. This is supported by replicative experiments conducted by contemporary knappers which have demonstrated that higher quality raw material has a direct influence on the manufacturing process [8–15].

Some scholars argue that a good raw material that is suitable for knapping should be brittle, elastic, and isotropic [9, 10]. However, only few semi-quantitative and quantitative studies of the mechanical properties of lithic materials have been carried out, and these date to the mid-20<sup>th</sup> century. Goodman's [16] experimental studies on this subject were among the first to analyse the hardness, toughness, and density of archaeological stone tools. Every stone tool can be seen as a unique material that has different raw material structure, morphology, and composition. The study by Moník and Hadraba [17], attempted to answer the question of whether differences in raw material procurement may have been driven by the mechanical properties of selected materials or not. Studies conducted by Lerner et al. [18] suggest that the physical properties of lithic raw materials have direct implications for use-wear accrual rates, meaning that roughness is linked to the rate of wear on stone tools. However, similar works in mechanical characterization of raw materials from archaeological contexts are still scarce. Some of the available studies concern mineralogical, chemical and crystallographic transformations [19]. A few attempted to determine the thermal evolution of fracture mechanics [20–23]. The importance of the studies of mechanical properties become more pronounced due to the growing body of evidence that indicates hominins purposefully altered lithic raw materials to increase the knapping qualities of rocks. This has implications for the evolution of human cognition [20, 22–26]. For instance, deliberate heat treatment of rocks to alter their knapping quality is considered a transformative technique [27] and has in the past been used to make inferences about prehistoric hominin cognition [28].

The current work is based on a systematic investigation of archaeological stone tools and geological samples of raw materials to determine the mechanical properties from different Upper Palaeolithic sites of Kazakhstan (Fig 1). Absolute chronology of Upper Palaeolithic sites is only available for Maibulaq (47–21 ka cal BP) and Ushbulaq (45–39 ka cal BP) [29, 30]. The chronology of the remaining sites was determined by techno-typological characteristics of the lithic assemblage. The primary objectives are (1) to test geological and archaeological samples to determine their mechanical properties, and (2) to preliminarily assess how these properties affect the knapping technology. Previous studies have been primarily concentrated on silica materials such as chert and silcrete [18, 20, 23], but other types of sedimentary and volcanic rocks found in archaeological contexts have received less attention. Here, we attempt to correct this imbalance by testing both sedimentary and volcanic rocks used by prehistoric people.

In this paper, we analyse chert, shale, and porphyry using indentation testing to investigate three mechanical properties: fracture resistance, elasticity (also known as Young's modulus),



**Fig 1.** Palaeolithic sites mentioned in the text and illustrated in relation to the major topography of the Inner Asian Mountain Corridor (Kazakhstan portion). 1) Yntaly, 2) Usiktas (surface site), 3) Valikhanova, 4) Kattasai, Uzbekistan, 5) Maibulq, 6) Rahat, 7) Ushbulaq. Data sources: Global Administrative areas (GADM) [59], vector and raster map data from Natural Earth ([www.naturalearthdata.com](http://www.naturalearthdata.com)) and Shuttle Radar Topography Mission (SRTM) Version 4 [60].

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and hardness (Table 1) [23, 31]. Additionally, we carry out traditional petrographic analysis to assess the mineralogical composition, impurities within the matrix, and individual grain size of minerals that could potentially influence the fracture behaviour of the studied specimens.

## 2 Regional setting

The materials studied here come from archaeological sites located in the piedmont zones of the Qaratau, Ili Alatau, and the Altai-Tarbagatai mountain ranges located in southern, south-eastern, and eastern Kazakhstan, respectively (Fig 1). It includes much of the Inner Asian Mountain Corridor (henceforth IAMC) [32]. The IAMC is a chain of mountain ranges approximately 2000 km in length in the centre of the Eurasian continent and stretching across most of the Central Asian countries, western China, and Mongolia. Although geologically varied, the study areas share common characteristics of geological formation. Topographic expressions originate from the tectonic activity, erosion, and other depositional processes [33–35]. The mountain ranges under study are all affected by a fault system that separates them into different sub-ranges, i.e., the Greater and Lesser Qaratau [33].

Many Palaeolithic sites are found in the Qaratau range [36]. Its structural and geological settings have been previously characterised by a number of geologists [33–35, 37]. The range

Table 1. Description of analysed samples.

Sample ID	Rock type	Location	Sample type	References
UBD-1-20*	Dark shale	Ushbulaq, East Kazakhstan	geological	[58]
UBG-1-20*	Green shale	Ushbulaq, East Kazakhstan	geological	[58]
YNT-1-20*	Chert	Yntaly, South Kazakhstan	geological	[58]
MB-1-20*	Porphyry	Maibulaq, South Kazakhstan	archaeological	[29]
UT-22*	Chert	Usiktas, South Kazakhstan	archaeological	[47]
UT-144*	Chert	Usiktas, South Kazakhstan	archaeological	[47]
UB-526*	Dark shale	Ushbulaq, East Kazakhstan	archaeological	[56]
UB-537*	Green shale	Ushbulaq, East Kazakhstan	archaeological	[56]
UB-571	Green shale	Ushbulaq, East Kazakhstan	archaeological	[56]
UB-532	Dark shale	Ushbulaq, East Kazakhstan	archaeological	[56]
UB-492	Green shale	Ushbulaq, East Kazakhstan	archaeological	[56]
UB-514	Dark shale	Ushbulaq, East Kazakhstan	archaeological	[56]
UB-616	Limestone	Ushbulaq, East Kazakhstan	archaeological	[56]
UT-48	Chert	Usiktas, South Kazakhstan	archaeological	[47]
UT-10	Chert	Usiktas, South Kazakhstan	archaeological	[47]
UT-181	Chert	Usiktas, South Kazakhstan	archaeological	[47]
UT-217	Chert	Usiktas, South Kazakhstan	archaeological	[47]

Localities shown in the table are illustrated on Fig 1. Samples marked with (\*) were tested to determine their mechanical properties, while the rest were characterised petrographically.

<https://doi.org/10.1371/journal.pone.0265640.t001>

mainly consists of Neoproterozoic and Palaeozoic bedrocks, and several carbonate seamounts developed due to thermal subsidence of the newly formed crust. The major carbonate platform formed during the Famennian and early Pennsylvanian [33]. The formation of the carbonate platform affected the structure of the Qaratau range, which is mainly composed of limestone, by creating a precondition to form caves, rockshelters, and silica rich rocks within carbonate beds. Such environmental factors played an important role in the human occupation of the region.

Unlike the Qaratau range, the Ili Alatau mountain range (Kazakh portion of the northern Tian Shan) is characterised by steep slopes and the presence of glaciers at higher elevations [34]. The northern foothills enclose the vast depression of the Ili and Dzungarian Alatau to the north-east. The mountain foothills are blanketed of different types of sediments, of which loess covers most of the area. The loess blanket known as the Central Asian piedmont that extends from the Pamir and Alai to the Ili Alatau was extensively studied and defined by Fitzsimmons et al. [29].

These mountain ranges have an arid to semi-arid climate with high variability in temperature variations between different seasons. The position of the mountain groups has played an important role in the early hominin dispersal across Asia [36, 38–40], and possibly provided a refugium during colder episodes of the Pleistocene [41–43]. Archaeological reports indicate extensive human activity, and earlier studies concerning the Palaeolithic of Kazakhstan already revealed that this region has great potential for studying the patterns of human behaviour and migration throughout Central Asia [29, 36, 44–57].

Despite the limited number of studies on raw material, recent work by Namen et al. [58] describes its distribution in the piedmont zones of Kazakhstan. In that paper, we discussed the geographic use patterns of different raw materials using the Centre for Russian and Central EurAsian Mineral Studies (CERCAMS) database. According to macroscopic observations of lithic assemblages, every stratified site has a distinctive type of raw material, probably

outcropping locally in close vicinity to the sites. Due to complex geological formations of the piedmont and foothill zones of the IAMC, this region offers a large amount of knappable raw materials. Therefore, the Palaeolithic sites across Kazakhstan have assemblages knapped on various lithologies. Our work showcases these differences through its comprehensive investigation of different rocks and offers a detailed insight into their mechanical properties. Without understanding the limitations that each raw material imposes on the knapper, our comparison of sites utilising different lithologies might be limited.

### 3 Materials and methods

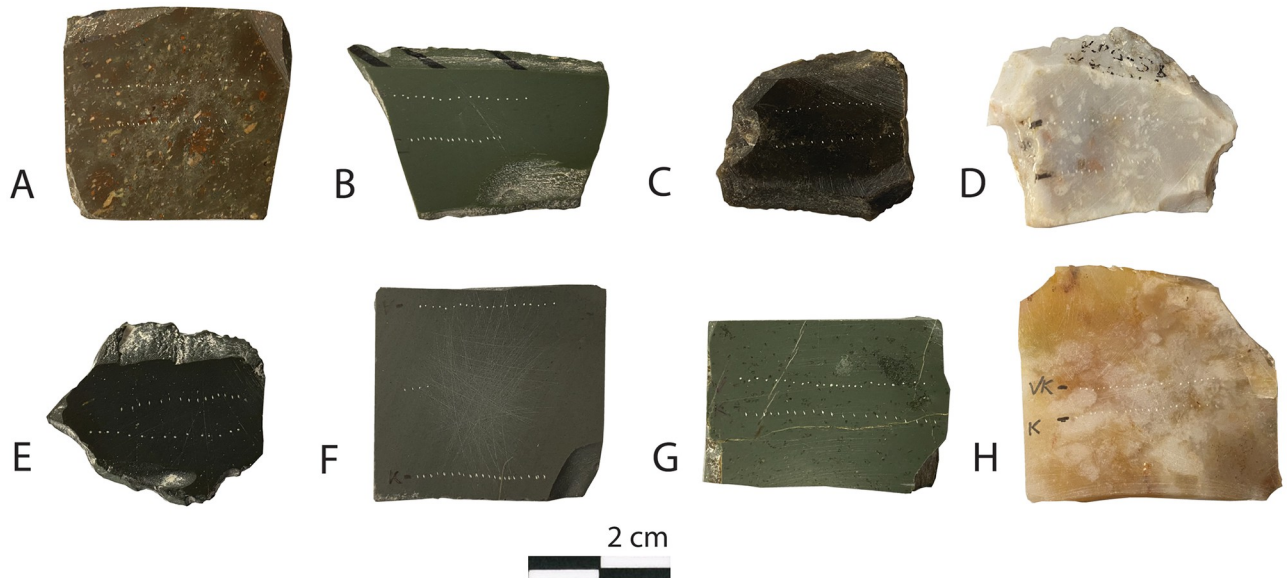
#### 3.1 Samples and sample preparation

For the current study, we selected a total of 17 samples, 14 of which come from archaeological sites, while the remaining three are geological raw materials (Table 1). The geological samples were collected during the PALAEOSILKROAD project's field campaign conducted in 2018 and 2019 [58, 61] under license No. 15008746 (12.05.2015) of the National Museum of the Republic of Kazakhstan based on a collaboration protocol between the Eberhard-Karls University of Tübingen and the National Museum. Primarily, chert and shale outcrops were surveyed and sampled because these materials were commonly used to produce stone tools at different archaeological sites in the piedmont and foothill zones of the IAMC [58]. The archaeological samples come from the stratified sites of Ushbulaq (Eastern Kazakhstan) [30, 56], Maibulaq (Almaty region) [29, 62, 63] and a surface site at Usiktas (South Kazakhstan) [64]. We sampled only lithics from the surface, so as not to disturb the integrity of the assemblages found in stratified contexts. The major sampling criterion was the size of the lithic pieces. Collecting larger samples (approx. 3–5 cm) allowed us to cut the pieces in the middle and then conduct experiments on polished surface produced from the lithics. Chert can be frequently found in stratified and surface sites throughout southern Kazakhstan, and shale is a major raw material of stone tool assemblages from Ushbulaq. Porphyry was commonly utilised in stratified sites at Maibulaq and Rahat [65]. No geological sample of porphyry was included in the current study due to the lack of systematic raw material survey at Maibulaq. We refer to porphyry as a rock of volcanic origin with crystals visible to the naked eye set in a fine grained matrix. The sample descriptions are summarized on Table 1. Location of the sites are shown in Fig 1.

#### 3.2 The indentation tests

A total of eight samples (five archaeological and three geological) were tested to determine their mechanical properties. In order to conduct the analyses, plane-parallel plates measuring approximately  $40 \times 30 \times 3$  mm (thickness of each sample varies) were cut and diamond polished (see Fig 2) on one side. They were then analysed by the Vickers and Knoop indentation [23, 66]. The Vickers and Knoop indentation method is commonly used in material science to measure hardness of materials. The Vickers diamond creates square-shaped indentations, whereas the Knoop diamond produces elongated diamond shaped indents.

Vickers indentations were conducted because it allows to assess data on indentation fracture resistance (according to the method proposed by [31]), Young's modulus (following the protocol proposed by Ben Ghorbal et al. [66]) and hardness of the samples were determined using Knoop indentations. Indentation tests were performed using an Instron 4502 universal testing machine at the laboratory for Applied Mineralogy at Tübingen University's Geosciences department, Germany. Each sample was indented 20 times for each type of indenter to obtain statistically relevant data (see Fig 2). The size of indentations and cracks was determined using images acquired with a HITACHI Tabletop scanning electron microscope (SEM) TM3030. The protocol used was as follows. Load was set to 100 N (with a pre-load of 10 N),



**Fig 2. Illustration of archaeological (A-E) and geological (F-H) samples that undergone mechanical testing by means of indentation tests. The dotted lines are the impressions of the diamond indents.**

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speed of indentation 1 mm/s and a hold time of 20 s. The size of each indentation is proportional to material hardness according to Eq (1). Another consequence of the indentation of brittle materials with Vickers and Knoop indenters is the formation of half penny-shaped cracks that can be observed at the polished surface. Cracks apparently depart from all four corners of Vickers indentations and from the two acute angle corners of Knoop indentations. The length of the cracks forming from Vickers indentations are proportional to the value of indentation fracture resistance (a value closely related to fracture toughness (see [67]).

*Knoop hardness* (HK) was calculated by using the formula suggested by Ben Ghorbal et al. [66]. They proposed a way to calculate Knoop hardness that yields results equivalent to the Vickers hardness (equation 12 in Ben Ghorbal et al. [66]):

$$HK = \frac{P}{\frac{b'}{2} \cdot L'} \quad (1)$$

where,

$P$  is the applied load in kilogram-force (KgF);

$b'$  is the short diagonal of the Knoop indent in mm;

$L'$  is the long diagonal of the Knoop indent in mm.

*Modulus of elasticity* ( $E$ ) is normally obtained by measuring the potential of a material to deform upon applied stress. However, as bending tests lied outside of the possibilities for this study, we used Ben Ghorbal et al. [66]'s formula that relies on comparing the long and short diagonals of the Knoop indentations. The calculation was initially suggested by Marshall et al. [68], but further refined by Ben Ghorbal et al. [66]:

$$E = 0.417 \times HK \div \left( \frac{1}{7.11} - \frac{b'}{L'} \right) \quad (2)$$

where,

$HK$  is the Knoop hardness in gigapascal (GPa);

$b'$  is the short diagonal of the Knoop indent in  $\mu\text{m}$ ;

$L'$  is the long diagonal of the Knoop indent in  $\mu\text{m}$ .

*Indentation fracture resistance* ( $K_{Ic}$ ), as defined by Danzer et al. [67], a value that is closely related to fracture toughness, a measure of a material's resistance to fracture propagation. We used Niihara et al. [31]'s formula to obtain a value for indentation fracture resistance ( $K_{Ic}$ ), which uses the length of the surface cracks that develop from the four apical points of the Vickers indentation:

$$K_{Ic} = 0,067H\sqrt{a} \left(\frac{E}{H}\right)^{0,4} \left(\frac{c}{a}\right)^{-1,5} \quad (3)$$

where,

$E$  is Young's modulus in megapascal (MPa);

$H$  is Vickers hardness in MPa (we used VH here, as calculated from Ben Ghorbal et al. [66], as the differences between HV and HK are negligible when using their calculations);

$c$  is the length of the cracks in  $\mu\text{m}$ ;

$a$  is the diagonal of the Vickers indentation in  $\mu\text{m}$ .

For this calculation, we admitted that the Knoop hardness is equal to the Vickers hardness and use the value obtained by Eq (1). The size of some of our Vickers indentations could not be accurately determined because their edges flaked off during the experiments. We admitted a standard value of quartz that is equal to 11.65 GPa. Diagonals of those indentations with intact margins were measured.

### 3.3 Petrographic study

A total of 16 thin sections, consisting of 13 archaeological samples from surface collections and 3 geological reference samples were microscopically examined (Table 1). We assessed potential impurities, mineralogical composition, the grain size of quartz grains and relative percentages of clasts and matrix. Traditional petrographic analysis was conducted at the laboratory for Geoarchaeology of the Institute for Archaeological Sciences (INA), University of Tübingen, Germany. The geological and archaeological (surface lithics) samples were prepared following a standard thin section preparation procedure. The thickness of the samples was reduced on a thin section grinder until the thickness of ca. 3  $\mu\text{m}$  reached. Thin sections were analysed using a Zeiss petrographic microscope and photomicrographs were obtained using Axio camera coupled to the microscope. Identification and description of minerals were made under plane polarized light (PPL) and cross-polarized light (XPL) following the terminology outlined by Courty et al. [70] and Stoops [71].

Thin sections of samples tested for mechanical properties were analysed employing the Image J software to calculate the percentages of inclusions and matrix which were estimated using the point counting technique [72]. We aimed to identify these components to evaluate whether there is a relationship with mechanical properties or not.

## 4 Results

Three out of eight samples yielded mechanical values because of the flaking off of all margins which made it impossible to measure indentation diagonals or crack lengths for the other samples. Since our results rely on few successful indentations, the results of the whole scheme must be regarded with some caution. Here, we present the results obtained from chert, shale, and porphyry as well as describe their petrographic features. Values of hardness, elastic modulus, and indentation fracture resistance are summarised below in Tables 2 and 3 and graphically shown in Figs 3–5. Photomicrographs of studied thin sections are shown in Figs 6 and 7.

**Table 2. Hardness (HK), elasticity (E), and fracture resistance (K<sub>Ic</sub>) values as calculated from the indentation tests.**

Sample ID	Rock type	HK	E	K <sub>Ic</sub>
UT-22	Chert	10.153	77	2.4
UB-537	Shale	4.392	153	3.01
MB-1-20	Porphyry	6.730	55	1.6

Note: the values of *HV* and *HK*, and *E* are given in GPa, and *K<sub>Ic</sub>* is given in MPa m<sup>1/2</sup>.

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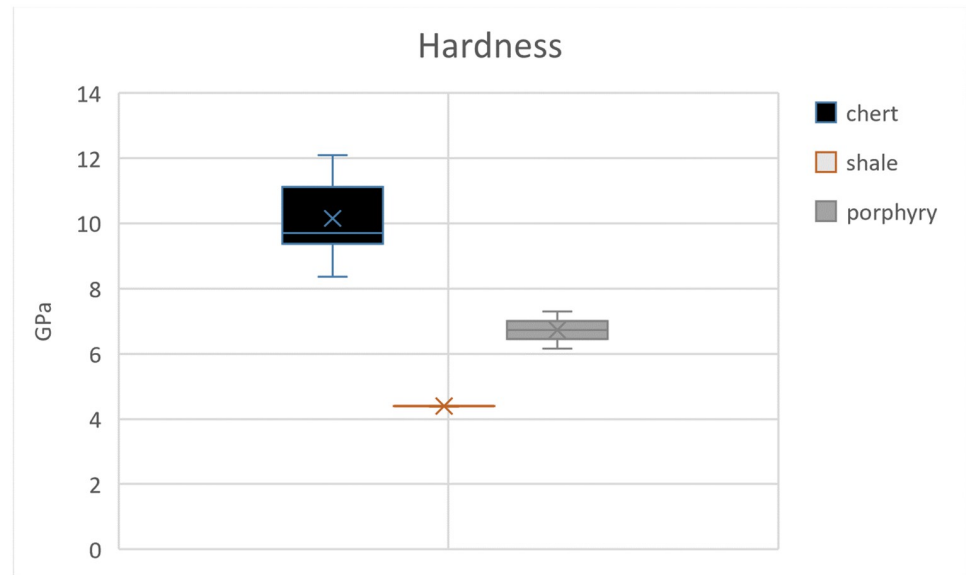
**Table 3. The values of hardness (HK), elastic modulus (E), fracture resistance (K<sub>Ic</sub>), crack lengths of Vickers and Knoop diamonds as calculated from the Vickers and Knoop indentation.**

Sample name	Crack length of Vickers diamond		Crack length of Knoop diamond		HK	E	K <sub>Ic</sub>
	<i>c</i> [μm]	<i>a</i> [μm]	<i>L'</i> [μm]	<i>b'</i> [μm]	[GPa]	[MPa]	[MPa m <sup>1/2</sup> ]
UT-22 (Chert)	188	125	495	36	11.179	68724	<b>2.38</b>
UT-22 (Chert)	188	125	483	44	9.283	80041	<b>2.27</b>
UT-22 (Chert)	188	125	487	37	10.984	71581	<b>2.40</b>
UT-22 (Chert)	188	125	480	34	12.093	73124	<b>2.56</b>
UT-22 (Chert)	188	125	489	41	9.786	73606	<b>2.26</b>
UT-22 (Chert)	188	125	470	37	11.411	77487	<b>2.53</b>
UT-22 (Chert)	188	125	488	48	8.368	86450	<b>2.20</b>
UT-22 (Chert)	188	125	480	44	9.418	80573	<b>2.29</b>
UT-22 (Chert)			476	43	9.644	81872	
UT-22 (Chert)			483	44	9.363	79105	
UB-537 (Shale)	139	125	590	77	4.386	188498	<b>3.17</b>
UB-537 (Shale)	132	125	603	75	4.397	117266	<b>2.84</b>
UB-537 (Shale)	122	125					
UB-537 (Shale)	136	125					
UB-537 (Shale)	122	125					
UB-537 (Shale)	137	125					
UB-537 (Shale)	137	125					
UB-537 (Shale)	156	125					
MB-1-20 (Porphyry)	200	125	551	49	7.298	60380	<b>1.59</b>
MB-1-20 (Porphyry)	177	125	601	53	6.162	50410	<b>1.61</b>
MB-1-20 (Porphyry)	204	125					
MB-1-20 (Porphyry)	182	125					
MB-1-20 (Porphyry)	199	125					
MB-1-20 (Porphyry)	195	125					
MB-1-20 (Porphyry)	102	125					
MB-1-20 (Porphyry)	182	125					
MB-1-20 (Porphyry)	155	125					
MB-1-20 (Porphyry)	143	125					
MB-1-20 (Porphyry)	147	125					
MB-1-20 (Porphyry)	176	125					

Note that the diagonal value (*a*) was admitted from the standard quartz diagonal due to the flaking off of the edges. It was also admitted as a standard diagonal for the rest of the indented samples.

<https://doi.org/10.1371/journal.pone.0265640.t003>



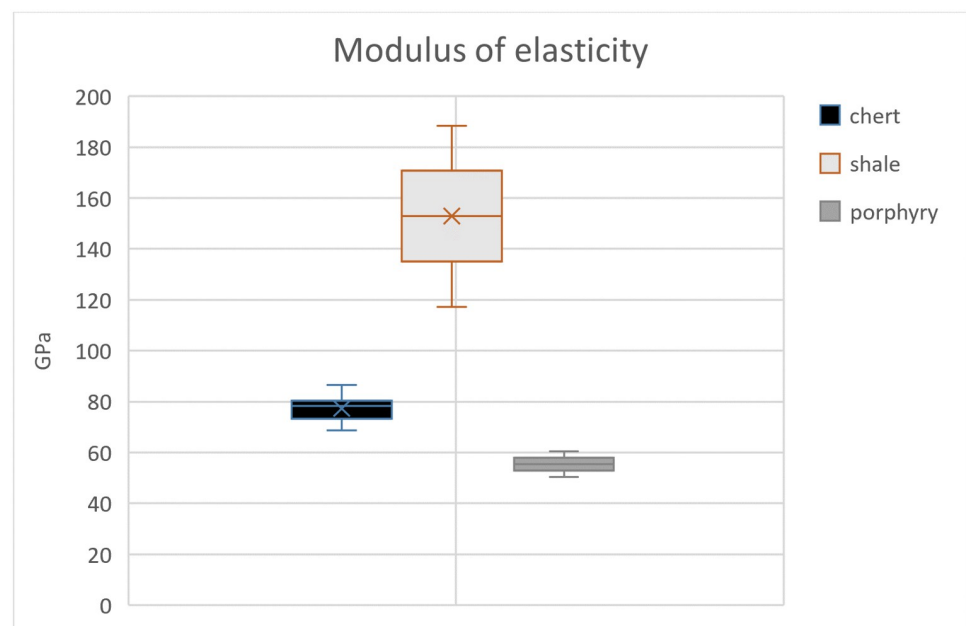


**Fig 3. The hardness values.** The box plot illustrates the highest hardness value for chert and lowest value for shale samples.

<https://doi.org/10.1371/journal.pone.0265640.g003>

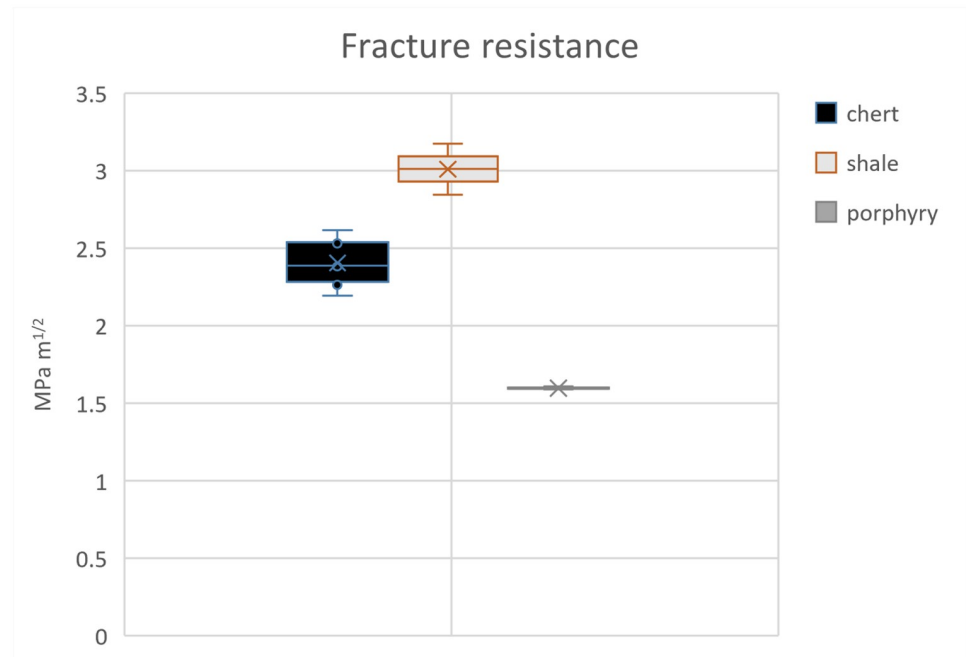
#### 4.1 Indentation tests

Among the three different types of lithologies studied, chert (UT-22) shows the highest hardness value of over 10 GPa compared to 6.730 GPa for porphyry (MB-1-20) and 4.392 GPa for shale (UB-537). Based on these experiment results, the hardness of shale is the lowest as compared to the other two rocks.



**Fig 4. The boxplot illustrates the modulus of elasticity; values show that shale is less stiff than porphyry and chert.**

<https://doi.org/10.1371/journal.pone.0265640.g004>

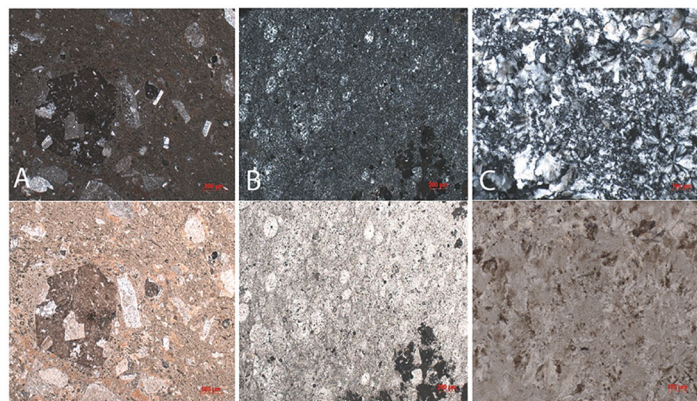


**Fig 5. The fracture resistance of studied samples shown in  $\text{MPa m}^{1/2}$ .** The porphyry sample shows the lowest value ( $1.5 \text{ MPa m}^{1/2}$ ) as compared to chert samples.

<https://doi.org/10.1371/journal.pone.0265640.g005>

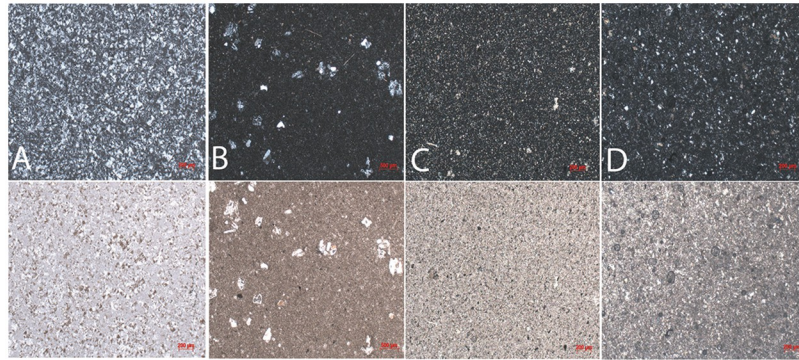
The modulus of elasticity, calculated using Eq (2), suggests that porphyry is the least stiff material with a mean value equal to 55 GPa. While chert yielded a value of 77 GPa, and shale is the stiffest rock among the studied lithologies with an  $E$  value of 153 GPa. Although porphyry is a volcanic rock that contains relatively large phenocrysts of quartz and feldspar, our results suggest that it is less stiff than the other two analysed samples.

The  $K_{Ic}$  values suggest that shale is most resistant to fracture with a mean value of  $3.01 \text{ MPa m}^{1/2}$ . The samples of chert and porphyry have a value equal to  $2.4$  and  $1.6 \text{ MPa m}^{1/2}$ , respectively. These data may suggest that among three lithologies fracture propagates consuming the least amount of energy in porphyry as compared to shale and chert. However, more detailed



**Fig 6. Photomicrographs of A) porphyry, B) shale, and C) chert under XPL (upper) and PPL (lower) lights that were tested by means of indentation.**

<https://doi.org/10.1371/journal.pone.0265640.g006>



**Fig 7. Photomicrographs of A) chert, B) calcareous shale, C-D) and siliceous shale under XPL (upper) and PPL (lower) lights.**

<https://doi.org/10.1371/journal.pone.0265640.g007>

investigation of rock strength is required to determine whether lower  $K_{Ic}$  value equals easier flake detachment.

## 4.2 Petrography

Table 4 summarizes the petrographic characteristics of the studied rock samples. During petrographic analysis, two types of shales were distinguished: calcareous shale (e.g., UBG-1-20) and siliceous shale (e.g., UBD-1-20). The calcareous shale is mainly comprised of well-sorted, subrounded to rounded crystals of calcite and quartz supported by a calcareous matrix. Calcite grains within shale are of secondary origin, which were recrystallised during later rock diagenesis. The siliceous shale consists of subrounded grains of feldspar and quartz. Thin sections

**Table 4. Basic petrographic description of archaeological and geological thin sections.**

Sample ID	Rock type	Matrix	Description
MB-1-20	Porphyry (Rhyolite?)	Siliceous	Coarse and angular grains of quartz, feldspar, and sericite, a product of hydrothermal alteration of feldspar, minerals embedded in a fine-grained siliceous matrix. Large xenolith is observed.
UBD-1-20	Dark shale	Calcareous	Fine-grained, rounded and subrounded grains of quartz and calcite are supported by a calcareous matrix.
UBG-1-20	Green shale	Siliceous	Moderately sorted, subrounded grains of quartz and feldspar, and fine-grained calcite minerals are supported by a siliceous matrix.
YNT-1-20	Chert	Siliceous	Entirely composed of length-fast chalcedony
UB-537	Dark shale	Siliceous	Rounded, microcrystalline quartz grains embedded in a siliceous matrix.
UB-492	Green shale	Siliceous	Subrounded grains of quartz and calcite supported by a siliceous matrix.
UT-48	Chert	Siliceous	Entirely composed of length-fast chalcedony.
UT-10	Chert	Siliceous	Entirely composed of length-fast chalcedony.
UT-181	Chert	Siliceous	Entirely composed of length-fast chalcedony.
UT-144	Chert	Siliceous	Entirely composed of length-fast chalcedony.
UT-22	Chert	Siliceous	Entirely composed of length-fast chalcedony.
UT-217	Chert	Siliceous	Entirely composed of length-fast chalcedony.
UB-571	Green shale	Siliceous	Primarily composed of well-rounded, well-sorted quartz grains, few inclusions of angular feldspar can be observed.
UB-532	Dark shale	Siliceous	Composed of primarily well-sorted calcite and few quartz minerals, several internal cracks are filled with calcite.
UB-514	Dark shale	Siliceous	Subrounded grains of clay sized quartz supported on a siliceous matrix.
UB-616	Silicified limestone (?)	Calcareous	Silicified limestone.

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**Table 5. Grain size of quartz minerals and percentage of inclusions and matrix.** CC stands for cryptocrystalline grain size (<1  $\mu\text{m}$ ). Note: samples marked with (\*) were tested to determine their mechanical properties.

Sample ID	Rock type	% inclusions	%matrix	Quartz		
				Min ( $\mu\text{m}$ )	Max ( $\mu\text{m}$ )	Mean ( $\mu\text{m}$ )
UBD-1-20	Dark shale	34.7	55.1	146.6	566.2	299.5
UBG-1-20	Green shale	10.2	89.8	77.2	740.8	386.9
MB-1-20*	Porphyry	32.9	60.6	409.5	2241.9	1125.4
UB-537*	Shale	55.1	61.3	25.1	89.3	41.9
UT-22*	Chert	CC	CC	CC	CC	CC

<https://doi.org/10.1371/journal.pone.0265640.t005>

prepared from archaeological lithics from Ushbulaq ( $n = 6$ ) show petrographic characteristics similar to the geological samples with varying degrees of quartz, calcite, and feldspar inclusions with the exception of sample UB-616 (Fig 7).

The thin sections of archaeological lithics from Usiktas ( $n = 6$ ) are composed entirely of length-fast chalcedony, similar to the geological sample YNT-1-20 (Yntaly chert). Microscopically, these samples have similar microstructure. However, we can not safely affirm the transport of these lithic raw materials from Yntaly, we only note their structural similarity. The presence of impurities and other minerals within chert samples is not observed. A thin section of porphyry from Maibulaq (MB-1-20) is comprised of coarse and angular grains of quartz and feldspar supported by a siliceous matrix. An inclusion of xenoliths, possibly broken off from the magma chamber or conduit walls around the time of eruption was observed (Fig 6A).

In addition, individual grain sizes of quartz, percentage of inclusions, and matrix of indented samples (see subchapter 4.1 and Table 1) has been measured and shown in Table 5. This is done to examine whether petrographic characteristics (grain size, number of inclusions, and matrix) affects the fracture resistance of tested rocks. As expected, porphyry contains the largest grain size among other studied samples. These clasts are also visible to the naked eye. The geological samples of shale contain relatively large mineral grains with a mean size equal to 299.5  $\mu\text{m}$ , whereas the grain size of archaeological lithic is much smaller and equals to 41.9  $\mu\text{m}$ . Average grain size of chert could not be measured due to the cryptocrystalline nature of quartz grains in chalcedony.

## 5 Discussion

This is the first experimental work to investigate the mechanical properties of chert, shale, and porphyry from the archaeological context of Kazakhstan. However, our discussion relies on few successful indentations (Table 3) and, therefore, the whole scheme must be regarded with some caution. Despite the small size of samples studied, this work offers insights into some of the mechanical properties of chert, shale, and porphyry which add to the knowledge of knapping technology and the formation of assemblages of stone tools. The results suggest that the raw materials considered to be of lower quality (i.e., porphyry) due to the presence of large phenocrysts have, in fact, mechanical properties (e.g., fracture resistance and modulus of elasticity) that can be compared to materials considered to be of higher quality, i.e., chert and silcrete (studied by Schmidt et al. [23]).

### 5.1 Raw material quality

The studied mechanical properties allow us to have a preliminary discussion on the *meaning of quality* with respect to lithic raw materials. Our results suggest that chert is the hardest

material, harder than porphyry and shale. This is expected since it is entirely composed of length-fast chalcedony, which in turn is quartz (with an average hardness of  $\sim 12$  GPa). On the other hand, shale has a lower hardness value despite its siliceous matrix and composition primarily of quartz, as seen under microscope. Although impurities within the shale were not observed during our petrographic study, the lower overall hardness of the rock could be due to the presence of clay impurities within the shale microstructure which were not detectable under 40x magnification [69]. The  $E$  values obtained from our experiments likewise show significant differences among chert, shale, and porphyry. Even though chert is composed of chalcedony and shale consists primarily of microcrystalline quartz, the elasticity values of chert and shale differ greatly, too. On the other hand, the  $E$  values of porphyry are lower than those of chert and shale. In the analysis of mechanical properties of heat-treated silcrete, Schmidt et al. [23] observed that silcretes with more (abundant) clasts generally had a strong loss of  $E$  values upon heat treatment compared to finer silcrete. This means that structural differences developed during the process of heat treatment may affect the elasticity value [23]. This is also supported by our observations on the relationship of grain size and mechanical properties of the rocks. For instance, our investigation shows that samples with larger grain size such as porphyry have a lower modulus of elasticity. Also, based on the measurements using the point counting technique of grain size, inclusions, and matrix, samples with lower percentages of each component demonstrate lower values of indentation fracture resistance (Table 5). An experimental investigation by Huang and Wang [73] on the impact of petrological characteristics, particularly of grain size to fracture toughness, suggests the dependence of these two parameters. However, due to the small size and unheated nature of the studied samples compared to the thermally altered silcretes, our results should be considered with some caution. Further investigations with a larger number of samples are needed to assess whether the large grain size affected the mechanical properties of porphyry or not.

Generally, the modulus of elasticity is proportional to the stiffness of the material. Domanski et al. [20] state that the modulus of elasticity is an important measure of the suitability of materials for blade manufacture because stiffness-controlled fracture propagation is largely responsible for blade detachment [10].

The mean values of  $K_{Ic}$  have a linear correlation with the  $E$  values, meaning that samples with the lowest  $E$  values also present low  $K_{Ic}$  values and vice versa (see Figs 3 and 4). The lower values of  $K_{Ic}$  imply less resistance against fracture, suggesting that the material is easier to fracture, and perhaps also to knap tools. Thus, the obtained values of  $K_{Ic}$  suggests good knapping quality of porphyry, despite the presence of large phenocrysts (see section 5.2 for discussion). Also, the modulus of elasticity and fracture resistance values of porphyry (see Table 2) can be compared to those of chert and silcrete obtained by Schmidt et al. [23]. However, the meaning of  $K_{Ic}$  for the actual knapping properties observed during experimental knapping should be investigated in the future.

This is the first time that such experiments were conducted to investigate the mechanical properties of *archaeological* porphyry. Our results offer a preliminary insight into the knapping quality of this rock. Further experimental lithic knapping and research on other mechanical properties (e.g., fracture strength) is necessary to examine the factors that affect the flaking of rock samples. Similar research specifically targeting the evolution of mechanical properties of silica rocks upon heat treatment allow us to compare the  $K_{Ic}$  values with previously published data and verify the results. For instance, the fracture resistance values of chert and silcrete from a study by Schmidt et al. [23] vary from 1.3 to 1.85 MPa m<sup>1/2</sup>. The resistance values of our samples vary between 1.6 and 2.4 MPa m<sup>1/2</sup> and therefore support previous work.

## 5.2 Implications for stone tool knapping

When we compare the results of the experimental works with the stone tool knapping of the Upper Palaeolithic sites where our samples were collected, we see a distinct pattern. Generally, homogeneous, isotropic, fine-grained, and silica rich rocks were normally preferred for the manufacture of stone tools. However, evidence for the utilisation of locally available materials such as porphyry, which is usually considered as “lower” quality, is known from the Central Asian stratified Palaeolithic sites of Rahat and Maibulaq in Kazakhstan [62, 65], as well as Kattasai-1 and Kattasai-2 in Uzbekistan [5, 74]. We concentrate our discussion on the quality of porphyry and the major techno-typological similarities between Palaeolithic industries that utilised this rock as a principal raw material.

Since porphyry was locally available as river pebbles, a large proportion of lithics knapped at the aforementioned sites were made on this material. The use of higher quality exogenous raw materials such as chert is common, but occurs in much smaller quantities. Based on a comprehensive literature review, we observed the presence of Levallois or Levallois-like technologies in all sites that utilise porphyry [5, 62, 65, 74]. Given that Levallois is considered to involve a combination of knapping skill *and* good quality materials, its presence in porphyry-rich assemblages is important to consider. Furthermore, the analysis of the Kattasai-1 assemblage (Uzbekistan) revealed several schemes of reduction, including radial, as well as single and double platform parallel reduction [75]. A similar knapping technology was documented at both Maibulaq and Rahat. The successful production of Levallois blades and radial reduction of cores at these sites clearly demonstrates the link between the quality of raw material and strategies of reduction. We hypothesise that these similarities could be linked to the mechanical properties of porphyry. Additionally, our results suggest that porphyry has at least some properties that can be compared with those of chert. However, compared to chert, porphyry is a coarse grained volcanic rock with a siliceous matrix (see Table 4). In coarser grained rocks the fracture propagates as both transgranular and intergranular cracks, thus consuming less energy to detach a flake of a desired morphology [76]. Moreover, the presence of bladelets knapped on both porphyry and shale from Maibulaq demonstrates its suitability for the production of smaller tools. The fracture resistance, elasticity, and hardness values obtained from our mechanical tests also attest to its suitability for the knapping of blades and bladelets. This data allows us to hypothesise that hominins who once occupied these sites had to adapt the stone knapping technology to the quality and availability of resources. In addition to the grain size of porphyry, we observed that cobbles currently available in the Maibulaq stream bed today tend to have higher incidence of large phenocrysts than those encountered in the archaeological collection. Exploring the pattern of grain size with a sample of both raw material and archaeological stone tools could potentially indicate that prehistoric communities at Maibulaq deliberately selected rocks with smaller phenocrysts for knapping. However, this must be tested with further analyses of raw materials and archaeological stone tools.

## 6 Conclusions

This is the first time that mechanical tests including fracture resistance, hardness, and the modulus of elasticity have been conducted on unheated samples of archaeological lithics and unmodified rocks at several Upper Palaeolithic sites in Kazakhstan. Our study offers a first insight into the quality of archaeological porphyry, which was used as a principal raw material at Upper Palaeolithic sites in the piedmont zones of the IAMC. We conclude that raw materials that were previously thought of as lower quality (e.g., porphyry) have some mechanical properties similar to those of chert. The prehistoric knappers that inhabited the northern foothills of the IAMC adapted their lithic reduction schemes to the quality of available raw material such

as porphyry. The presence of Levallois or Levallois-like technology that can be observed from several Upper Palaeolithic sites utilising porphyry as a main raw material further support this hypothesis.

This work highlights the importance of the mechanical properties of chert, shale, and porphyry and their effect on the production of stone tools. Our findings show that materials which we expect to be difficult to knap based on visual inspection may have good fracture properties when evaluated objectively. Porphyry comes as a surprise because it is rarely used for knapping, and mainly unfamiliar to archaeologists [5, 65]. However, these kinds of counterintuitive results are also likely for some materials that *are* familiar to archaeologists, such as different types of cherts, basalts, and other materials.

Further investigations involving larger numbers and various types of raw materials and archaeological lithics are necessary to study and eventually build a library including the mechanical properties of the whole corpus of raw materials available in Kazakhstan. Our preliminary data can serve as a baseline for future quantitative and experimental investigations.

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## APPENDIX III

This is the accepted version of the following manuscript: “**Preference for porphyry: Petrographic insights into lithic raw material procurement from Palaeolithic Kazakhstan**”. The manuscript has been accepted for publication at *Journal of Field Archaeology*.

## **Preference for Porphyry: Petrographic Insights into Lithic Raw Material Procurement from Palaeolithic Kazakhstan**

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## **Abstract**

Only a handful of stratified sites are known in loess, spring, and river contexts in the northern piedmonts of the Tian Shan, and the majority are dated to the Upper Palaeolithic. These sites have been studied from a geoarchaeological perspective, however, lithic procurement activities remain unknown. To address this deficiency, we present the results of the extensive field surveys aimed at locating prehistoric raw material sources in the Inner Asian Mountain Corridor of Kazakhstan. We also provide a detailed petrographic description of the lithologies exploited during the Palaeolithic of Kazakhstan. Based on the field survey results, combined with petrographic data, we conclude that the direct procurement strategy was the most common at the stratified sites. However, evidence of both direct and embedded procurement is found in the northern piedmonts of the Ili Alatau range at the site of Maibulaq. Additionally, we highlight the variation of chert lithologies within the larger Qaratau region, laying a foundation for future provenance studies.

**Keywords:** geoarchaeological survey; chert; PALAEO-SILKROAD; petrography; Inner Asian Mountain Corridor

## **Introduction**

In recent years, most of the research in central Asia, and particularly in Kazakhstan, has been focused on finding new Palaeolithic sites (Iovita et al. 2020; Namen et al. 2020; Cuthbertson et al. 2021; Leloch et al. 2021; Kot et al. 2022). The majority of the finds are surface scatters of lithics discovered in the piedmont and foothill zones of the Inner Asian Mountain Corridor (henceforth, IAMC) of Kazakhstan, which includes the Qaratau, Ili Alatau, Dzhungarian Alatau, and the Tarbagatai ranges (Frachetti 2012). Known stratified Palaeolithic sites are located in the piedmont and foothill zones of these ranges, and the majority are chronologically attributed to the Upper Palaeolithic. Currently, all known Palaeolithic localities in Kazakhstan are found in karst/pseudokarst, loess, spring, and river settings, which constitute an important setting for the preservation of archaeological horizons (Iovita et al. 2020; Varis et al. 2022; Namen et al. 2022a). Most stratified Palaeolithic sites (e.g., Maibulaq, Rahat, Valikhanova, and Ushbulaq) are found by water bodies such as rivers and springs. The water sources are certainly of great importance for settling in the landscape and for the subsistence of humans. However, this could possibly be a research bias in the archaeological record, since archaeologists look for evidence of human occupation in the aforementioned settings.

Since 2018, field campaigns within the framework of the bigger PALAEOSILKROAD project have been attempting to determine the adaptation strategies of prehistoric human populations in regard to the local raw materials in the IAMC of Kazakhstan. The major aim has been to locate and study lithic raw materials and their variability in the techno-complexes of the Palaeolithic sites of the region. The first preliminary field observations show that hominins predominantly exploited local raw materials, and due to the diverse formation history of the mountain ranges within the IAMC, raw materials of the lithic assemblages differed depending on the geographic location of the sites (Namen et al. 2022b). Despite the utilization of predominantly locally available raw

materials in the form of pebbles or from outcrops at most of the sites, the use of exogenous types of raw materials was previously described at the Upper Palaeolithic sites of Maibulaq, Rahat, and Ushbulaq (Taimagambetov 2009; Fitzsimmons et al. 2017; Anoikin et al. 2019; Ozherelyev, Dzhasybaev, and Mamirov 2019). Chert and microcrystalline varieties of metasedimentary pelitic rocks (e.g., shale, claystone, siltstone, etc.) are typically considered to be higher quality because they are fine-grained and flake easily and are found as exogenous raw materials at the aforementioned sites. However, in the piedmont of the Ili Alatau range, the number of tools knapped on these rocks is small compared to the local porphyritic rocks. Recent experiments conducted to determine the mechanical properties of these rocks demonstrate that some mechanical properties of porphyry can be compared to chert (Namen et al. 2022c), making the latter an unexpectedly high-quality raw material.

Nevertheless, the question of the characterization, provenance, and transport distance of the exogenous raw materials from these sites remains open and requires a comprehensive multidisciplinary approach employing a variety of analytical tools. Examples of long-distance transport of higher quality raw materials such as micro- and cryptocrystalline varieties of silica rocks are known in the Palaeolithic industries of Europe, Africa, and across the Asian continent (Binford 1980; Feblot-Augustins 1993, 2009; Bar-Yosef 2002; Sano 2010; Arrizabalaga et al. 2014; Tomasso and Porraz 2016; Ekshtain et al. 2017; Frahm and Hauck 2017; Caruana, Tasker, and Stratford 2019; Khatsenovich et al. 2020; Koch and Schmidt 2022). This led to the growth of raw material provenance studies (Brandl 2016), which, in turn, became an interpretive tool to investigate hominin raw material selection behaviors (McBrearty and Brooks 2000; Wadley 2013), population movements (Feblot-Augustins 2009), and human-environment interactions (Bar-Yosef 2002; Blinkhorn and Petraglia 2017; Ghasidian and Heydari-Guran 2018; Heydari-Guran and Ghasidian 2020). Many of the models applied to archaeological sites are derived from ethnoarchaeological research examining the procurement strategies of indigenous people in the Arctic (Binford 1979, 1980)



and Australia (Gould and Saggers 1985). In central Asia, Ghasidian and Heydari-Guran (2018) addressed raw material acquisition in the Iranian Palaeolithic, suggesting an opportunistic use of local raw materials among highly mobile hunter-gatherer groups. The authors explain that the opportunistic use of raw materials is driven by decreased time and energy costs and eventually the adaptation of knapping techniques with available resources (Ghasidian and Heydari-Guran 2018).

In this study, we characterize a variety of lithologies (from both archaeological and geological contexts) that were available to the hominins in the IAMC of Kazakhstan employing a traditional petrographic microscope. The primary objectives are to characterize the mineralogical composition of geological samples of rocks and archaeological lithics and to attempt to describe specific mineralogical components that could aid future provenance analysis. Previous investigations demonstrated the feasibility of petrographic analysis to discriminate characteristic mineral components for the sourcing of stone tools (Soto, Gómez de Soler, and Vallverdú 2018; Soto et al. 2020; Favreau et al. 2020; Prieto, Yusta, and Arrizabalaga 2020). Our results contribute to the in-depth study of raw material variability in the IAMC of Kazakhstan and will serve as a referential framework for research on both raw material provenance and on ancient hominin economic strategies.

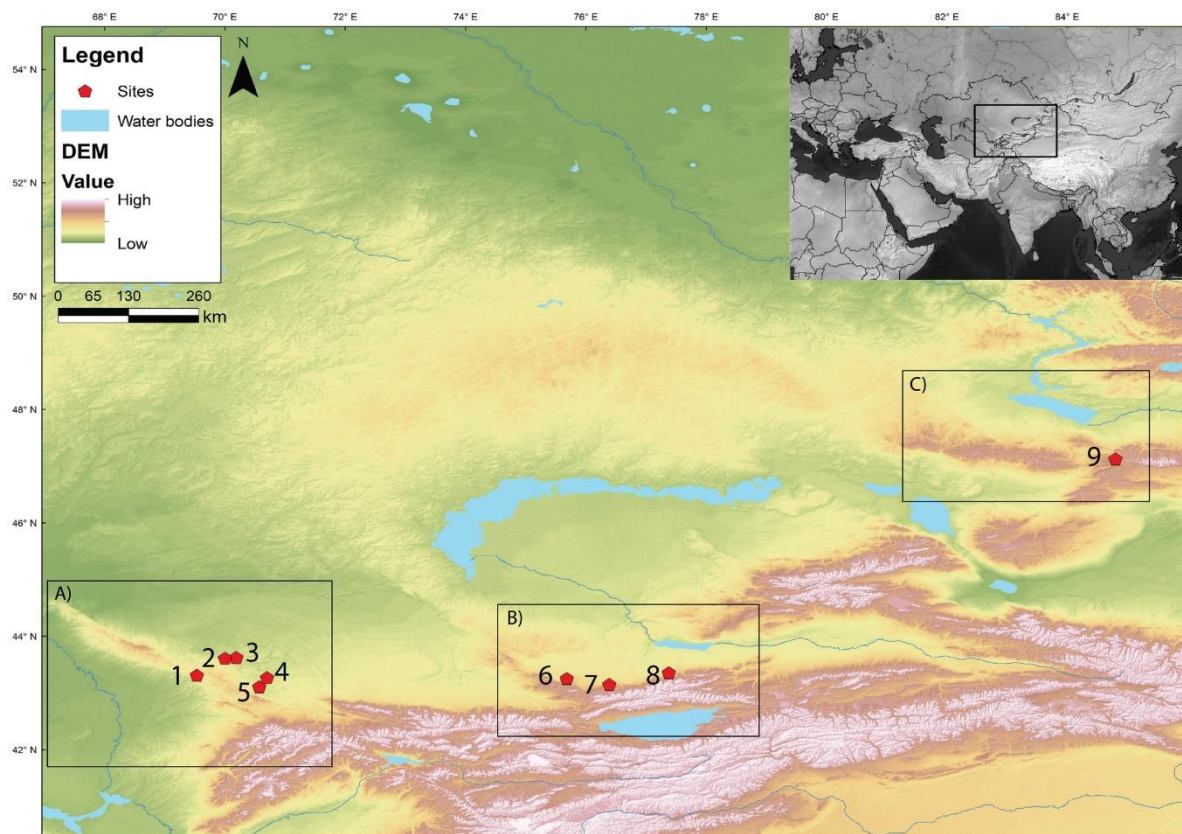


Figure 1. Topographic map of the Kazakh portion of the IAMC that is comprised of A) the Qaratau, B) Ili Alatau, and C) Tarbagatai mountain ranges (highlighted in squares). Numbered red marks are the sites mentioned in the text: 1) Valikhanova, 2) Yntaly, 3) Sorkol, 4) Qyzyltau complex, 5) Buirekbastau-Bulaq, 6) Beriktas, 7) Maibulaq, 8) Rahat, and 9) Ushbulaq. Data sources: Global Administrative areas (GADM) (Hijmans 2012), vector and raster data from Natural Earth ([www.naturalearthdata.com](http://www.naturalearthdata.com)) and Shuttle Radar Topography Mission (SRTM) Version 4 (Jarvis et al. 2008).

### **Archaeological Setting of the Inner Asian Mountain Corridor of Kazakhstan**

The IAMC is a chain of different mountain ranges (the Qaratau, Ili Alatau, Dzhungarian Alatau, Tarbagatai, and Altai) that are affected by fault systems separating them into different ranges, and the study areas are separated by major geological boundaries. However, they share a common geological formation with modern topographic expressions originating from tectonic activity, erosion, and other surface processes. The foothill and piedmont zones of the IAMC were probably some of the regions that provided shelter during the climatic instabilities in the Late Pleistocene (Beeton et al. 2014; Iovita et al. 2020) and perhaps played

an important role in human dispersals across the Eurasian continent (Khatsenovich et al. 2019; Li et al. 2019).

The Palaeolithic record of this vast region is largely represented by well-dated Lower (Qoshqorgan-Shoqtas site complex, 500–430 kya; Derevianko 2006) and Upper Palaeolithic sites (i.e., Ushbulaq [45–39 kya CAL B.P.], Maibulaq [40–25 kya], and Valikhanova [43.5–9 kya]), but surface finds at localities such as Qyzyltau, Boriqazgan, Tanirqazgan, and Semizbugu contain lithics that can be attributed to all periods of the Palaeolithic, based on techno-typological characteristics (Alpysbaev 1979; Artyukhova 1990; Artyukhova and Mamirov 2014; Osipova and Artyukhova 2019). Our work concentrates on identifying and characterizing lithic raw materials from the Upper Palaeolithic sites of Ushbulaq, Maibulaq, Rahat, Buirekbastabulaq, and Valikhanova, as well as the Lower Palaeolithic Qoshqorgan-Shoqtas site complex (Figure 1). The stratigraphic setting and archaeology of the sites are well-documented and described (see Alpysbaev 1979; Taimagambetov 1990, 2009; Derevianko 2006; Fitzsimmons et al. 2017; Shunkov et al. 2017; Anoikin et al. 2019; Ozherelyev, Dzhasybaev, and Mamirov 2019; Kunitake and Taimagambetov 2021). Below, we provide a brief background to each site studied, organized by region.

### *The Qaratau sites*

So far, the majority of the known Palaeolithic sites are located within the Qaratau range. They cover the periods spanning from the Lower Palaeolithic to the Neolithic. The Lower Palaeolithic is represented by the travertine sites of Qoshqorgan-Shoqtas. The complex of sites is located on a piedmont plain on the southwestern slopes of the Greater Qaratau. Due to the high salinity of the water, the springs are encircled by travertine rings. Subsequent excavations revealed several stages of travertine formation and distinct archaeological horizons attesting to human presence (Derevianko 2006). The assemblage is mainly represented by small flake tools and dated by electron paramagnetic resonance (EPR) to  $500 \pm$

430 kya (Derevianko et al. 1998; Derevianko 2006). This is the only stratified Lower Palaeolithic complex discovered in Kazakhstan, the rest being surface finds that are typologically ascribed to the Lower Palaeolithic.

The Upper Palaeolithic is represented by the site of Valikhanova, which was discovered by Khasan Alpysbaev (1979) in the 1960s. It is located on the left bank of the Arystandy river in the southern foothills of the Lesser Qaratau. The original Alpysbaev publications describe three archaeological horizons preserved in a loessic sedimentary context, with each horizon separated by sterile layers of varying thicknesses. He erroneously attributed the lowermost horizon to the late Mousterian period (Alpysbaev 1979). Archaeological excavations were resumed in the 1980s (Taimagambetov 1990). The main sequence was re-dated in 2013, attributing the age of the earliest archaeological horizon (CH6) to ca. 43.5–35.5 kya (see Fitzsimmons et al. 2017). New excavations in 2018 revealed three additional horizons under the previously recorded three (Z. Taimagambetov, personal communication 2020). Although these additional horizons have not yet been dated, currently the archaeological sequence of the Valikhanova site spans at least ca. 43.5–9 ka and so far represents the Early Upper Palaeolithic rather than the final Mousterian as suggested earlier by Alpysbaev (1979).

Buirekbastau-Bulaq was discovered in 2017. The site is located in the northeastern foothills of the Lesser Qaratau and is found in a spring geomorphological context. The archaeological assemblage is characterized by a blade industry and is chronologically attributed to the Early Upper Palaeolithic. However, the periodization of the site is based on a techno-typological analysis of the unearthed lithic artifacts, with absolute dates pending (Kunitake and Taimagambetov 2021).

Several surface lithics were collected from the large surface Palaeolithic complex of Qyzyltau. It is located in the northeastern foothills of the Lesser Qaratau, and lithic artifacts are scattered in a territory of over 40 km<sup>2</sup>. The site was discovered by Khasan Alpysbaev

(1979) in the 1960s, and a Kazakh-Russian archaeological expedition continued the fieldwork in the 1990s (Derevianko 2017). The lithic collection is attributed to all periods of the Palaeolithic. This signifies intense human interaction with the environment for long periods of time. The authors explain the formation of the complex by the regression of the paleolake surface and landscape evolution (Derevianko et al. 2002).

### *The Ili Alatau sites*

The piedmont zone of the Ili Alatau range has recently become a promising region to search for and locate new Palaeolithic sites. This is because thickly deposited loess accumulations preserve traces of human occupation (Li et al. 2020). Currently, Palaeolithic horizons are known at the sites of Maibulaq and Rahat (Taimagambetov 2009; Ozherelyev, Dzhasybaev, and Mamirov 2019). The Maibulaq site was discovered in 2004 and excavated in the following years, which revealed three cultural horizons divided by sterile loess sediments (Taimagambetov 2009; Fitzsimmons et al. 2017). All three cultural horizons have been dated, and, so far, the chronology of the site spans ca. 40–25 kya. Currently, new detailed chronology for Maibulaq is in preparation. Cultural horizon 1 is reported to be partially disturbed by Holocene activity, possibly an Early Iron Age burial (kurgan), whereas the lowermost cultural horizons 2 and 3 revealed an abundant number of lithic artifacts. The assemblages are characterized by prepared cores with two hierarchically-organized surfaces, their flake products, and bladelet and Levallois technologies (Figure 2) (Fitzsimmons et al. 2017). Thus, the chronology and technological features containing Levallois technology and bladelet production suggest an Early Upper Palaeolithic character for the site, although not enough is currently known about the cultural sequences in this region to make such characterizations. In particular, the total absence of dated Middle Palaeolithic sequences makes such discussions counterproductive.

Rahat is another Upper Palaeolithic site, located approximately 80 km east of Maibulaq. It has been excavated since 2018 (Dzhasybaev, Ozherelyev, and Mamirov 2018). The archaeological assemblage is techno-typologically analogous to the Maibulaq materials and knapped on local volcanic rock pebbles (Ozherelyev, Dzhasybaev, and Mamirov 2019). The chronology of Rahat is preliminarily attributed to the Upper Palaeolithic based on techno-typological features and site stratigraphy.

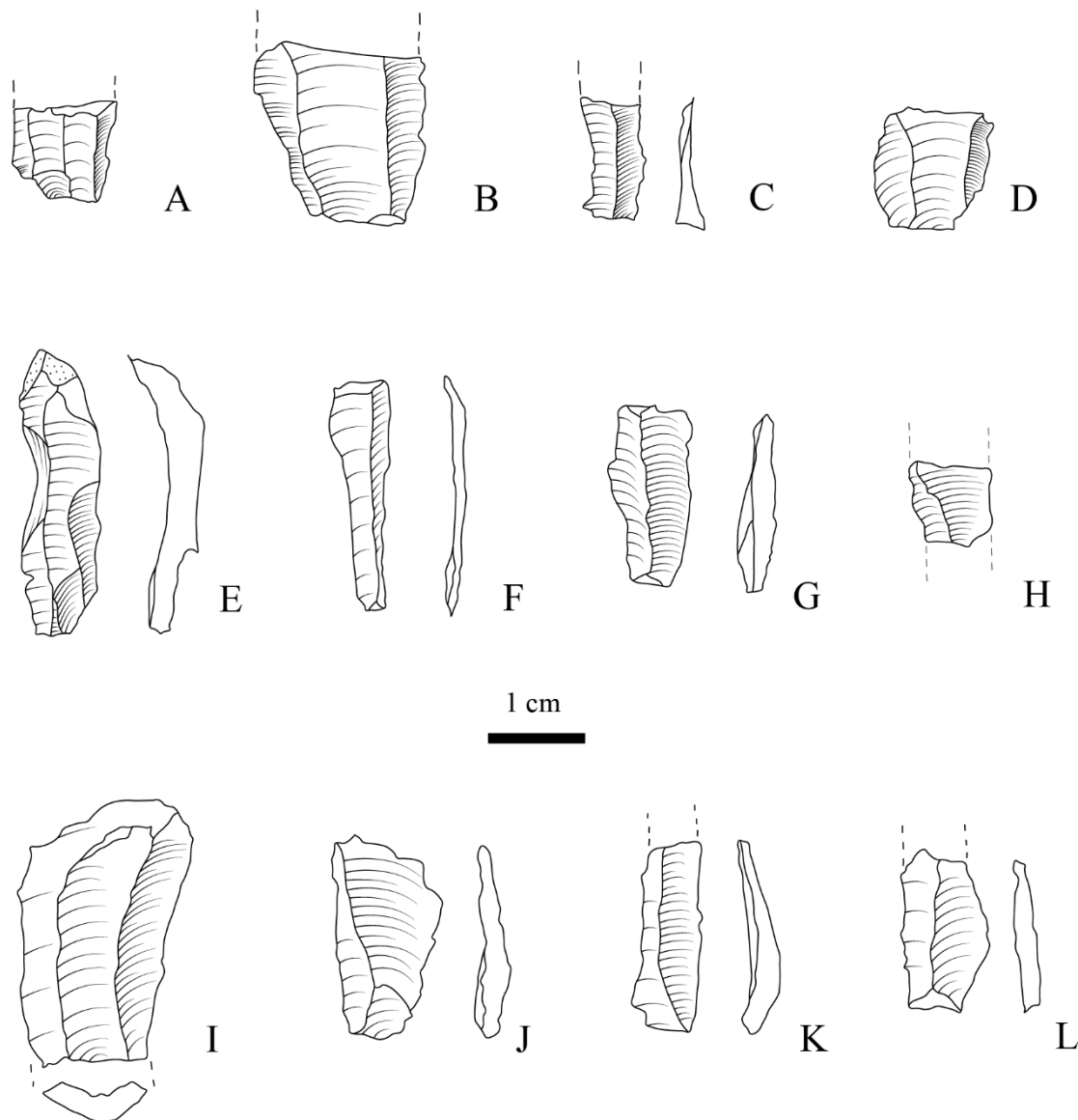


Figure 2. Illustration of the lithic artefacts from the occupational horizon 3, Maibulaq. A, F, G, L –bladelet; B, D – flake; C, E, H-K – blade.

### *The Tarbagatai sites*

The Tarbagatai range is located in eastern Kazakhstan, south of the Kazakh Altai. It stretches in an east-west direction and spans over 200 km. We located several occurrences of surface lithics in the northern foothills of the range. These are described by Iovita and colleagues (2020). However, Ushbulaq is the only currently known stratified Upper Palaeolithic site in the region. It was discovered in 2016 by a Kazakh-Russian archaeological expedition (Derevianko et al. 2017). Excavations carried out in the following years revealed several horizons with lithics. However, the lowermost two horizons contained a high density of lithic artifacts. The assemblages are dominated by prismatic and double-platform bidirectional cores knapped to detach long blades. The radiocarbon dates were taken from Stratum 6 and date the site to 42,100–39,364 CAL B.P. (AA-111921) and 45,249–44,012 CAL B.P. (NSKA-01811) (Anoikin et al. 2019). Based on the presence of characteristic features of double-platform blade cores and blades exceeding 20 cm, the authors associate the lower horizons with the Initial Upper Palaeolithic (Shunkov et al. 2017; Anoikin et al. 2019).

## **Materials and Methods**

### *Geoarchaeological survey*

The geoarchaeological surveys were conducted in the 2019 and 2021 field seasons. The survey primarily concentrated around previously known stratified sites and unstratified surface lithic scatters located in the foothill zones of the Ili Alatau (Almaty region) and the Qaratau mountain ranges (southern Kazakhstan). Preliminary results of this work were previously reported by Namen and colleagues (2022b). We aimed to cover a 20 km radius from the sites to locate primary and/or secondary sources of knappable raw materials. The

exploratory raw material survey was conducted by car and on foot where necessary. Given that porphyry constitutes the majority of the local raw materials, we focused on locating sources of exogenous materials (e.g., shale and chert) exploited at the sites of Maibulaq and Rahat.

For this reason, we surveyed the piedmont zone stretching approximately 20 km west and 5 km east of the site of Maibulaq. The eastern part of the area is located on private land within the densely populated Almaty metropolitan area and could not be systematically surveyed. The southern part is constrained by the steep slopes of the Ili Alatau range (Tian Shan Mountain system), and the northern part is primarily represented by eroded debris of rocks outcropping from the mountain range and the accumulation of aeolian sediments. The Rahat site is situated in a similar geomorphological context, and therefore our survey focused on the piedmont zone along the mountain front. A similar survey strategy was also applied in the Qaratau range, where the uplifted carbonate beds create a precondition for the formation of specific types of silica rocks, such as cherts and siliceous shales.

In order to predict the location of potential raw material sources, we used georeferenced geological maps of Kazakhstan, as well as lithological data from the Mineral Deposits Database and Thematic Maps of Central Asia ArcGIS platform developed by the Centre for Russian and Central EurAsian Mineral studies (CERCAMS; Seltmann, Armstrong, and Dolgoplova 2004). We successfully used the CERCAMS database in previous raw material surveys to locate sources of individual types of raw materials (Namen et al. 2022b). Data on the occurrences of raw materials of interest were retrieved from the CERCAMS database as keyhole markup language (.kml) files and uploaded onto field tablets to structure the survey. Lithologies of interest, such as varieties of porphyritic rocks, that were not available on the CERCAMS database, were retrieved as shapefiles (.shp) from the geological maps created by the Institute of Geology of the Soviet Academy of Sciences using the open-access platform QGIS (Geological Map of Kazakhstan and Middle Asia). The GPS locations



of sources of interest were recorded and photographed, and samples for further laboratory processing were taken.



Figure 3. Secondary position of shale found in a dried riverbed at the Beriktas locality (Trans-Ili Alatau) (A), and a pebble showing a conchoidal fracturing pattern (B).

### *Samples*

The types of samples collected during the fieldwork are summarized in Table 1. The archaeological samples of surface lithics were collected from the Upper Palaeolithic sites of Ushbulaq, Rahat, Maibulaq, Buirekbastaubulaq, and Valikhanova and the Lower Palaeolithic complex of Qoshqorgan-Shoqtas (see Table 1, Figure 1). So as not to disturb the integrity of assemblages found in stratified contexts, we only sampled lithics collected from the surface that could not be attributed to a specific cultural layer. The geological samples were collected from primary positions such as outcrops or secondary positions such as river pebbles located close to the sites ( $\leq 20$  km). The sample collection criteria were based on macroscopic similarity to the archaeological assemblage and other macroscopic attributes such as color, texture, translucence, and surface roughness as identified by hand lenses and visual inspection. Such macroscopic analysis provides a fast, non-destructive, and inexpensive means of comparing large collections. Similar survey strategies have also been used to

investigate raw material distribution in other parts of the world (Spinapolice 2012; Suga et al. 2022; Ghasidian and Heydari-Guran 2018).

### ***Sample preparation and petrographic analysis***

Macroscopic and microscopic observations were used to identify different types of lithologies. Firstly, we classified each material by color and texture. A total of 64 thin sections, consisting of 23 geological reference samples and 41 surface lithics, were microscopically investigated. The thin sections were prepared at the Geological Laboratory of Satbayev University (Almaty, Kazakhstan). Traditional petrographic analysis was conducted at the Laboratory for Geoarchaeology of the Institute for Archaeological Sciences (INA), University of Tübingen (Germany). Thin sections were analyzed using a Zeiss petrographic microscope, and photomicrographs were obtained using an Axio camera coupled to the microscope.

The geological reference samples were studied for direct comparison with the archaeological lithic artifacts (see for example Rybin et al. 2018; Prieto, Yusta, and Arrizabalaga 2020; Favreau et al. 2020; Abrunhosa et al. 2020). Thin sections were investigated under plain parallel and cross-polarized light to characterize their mineralogical composition, matrix, crystal size, and shape. Due to the homogeneity of chert samples under the optical microscopy, a quartz lambda auxiliary plate was used to obtain additional information regarding chalcedony types (i.e., length-fast and length-slow) and the corresponding variety. The length-fast and the length-slow terms are defined as the relationship between the crystallographic axes and the magnitude of refractive indices. The sign of elongation is determined on the basis of whether the slow and fast component is vibrating in the longest direction of the crystal (Miehe, Graetsch, and Flörke 1984). A recent paper by Koch and Schmidt (2022) graphically represents the length-fast and length-slow chalcedony types. The identification of chalcedony types is an important parameter that helps

to provenance chert. This is because length-slow chalcedony fibers generally form under geological environments different from those for length-fast chalcedony (Hattori 1989). The identification of pelitic rocks followed the grain size classification by Wentworth (1922).

Table 1. Description of the archaeological sites and geological localities where the samples of raw materials were collected.

Site ID	Period	Absolute Dates	Raw Material Source	Type of Raw Material	References
Maibulaq	Upper Palaeolithic	40–25 kya	Secondary	Porphyry, rhyolite	Fitzsimmons et al. 2017
Rahat	Upper Palaeolithic	Pending	Secondary	Porphyry	Ozherelyev, Dzhasybaev, and Mamirov 2019
Beriktas	Riverbed	N/A	Secondary	Siltstone	Current work
Buirekbastaubulaq	Upper Palaeolithic	Pending	Secondary	Chert, claystone	Kunitake and Taimagambetov 2021
Qyzyltau	Middle–Upper Palaeolithic	N/A	-	Chert	Derevianko et al. 2002
Sorkol	Neolithic	N/A	-	Chert	Iskakov 1998
Valikhanova	Upper Palaeolithic	43.5–9 kya	Primary	Chert	Fitzsimmons et al. 2017
Valikhanova outcrop	Outcrop	N/A	Primary	Chert	Current work
Ushbulaq	Upper Palaeolithic	45–39 kya CAL B.P.	Secondary	Siltstone	Anoikin et al. 2019
Qoshqorgan-Shoqtas complex	Lower Palaeolithic	500–430 kya	-	Chert, quartzite	Derevianko et al. 1998

## Results

Here we provide petrographic characteristics for each geological and archaeological raw material sample collected during surveys, organized by the different mountain ranges within the IAMC. The piece counts of exogenous raw materials are shown in Table 2, and the detailed petrographic description is provided in Table 3.

### *Geoarchaeological survey*

In the Ili Alatau range, the survey yielded secondary and primary sources of igneous lithologies which originate from the Devonian volcanic granitoid base rock. Even though cobbles and pebbles of different volcanic rocks are visible in fluvially eroded valleys, the thick accumulation of wind-blown aeolian loess sediments hinders locating the outcrops of various lithologies in the piedmont and foothill zones. There, we observed a variety of porphyritic rocks available in riverbeds and channels. The geological samples for further laboratory investigation were collected based on macroscopic similarities to the lithic assemblage from the mentioned sites. Our survey failed to locate chert or flint in primary or secondary contexts. According to geological map data retrieved from the CERCAMS database, the nearest sources of chert are located to the north in the Dzhungarian Alatau and to the west in the Chu-Ili Alatau (previously reported by Namen and colleagues [2022b]). We mainly located sources of knappable porphyritic and felsitic rocks in the vicinity of Maibulaq. However, a secondary source of higher quality exogenous metasedimentary pelitic rocks was located in the piedmont zone of the Ili Alatau range some 20 km west of Maibulaq and 70 km west of Rahat at a locality named Beriktas (see Figures 1, 3). The Beriktas locality is thus far the only known location in the Ili Alatau foothills that has siltstone, a material that is macroscopically similar to the exogenous materials knapped at the aforementioned sites. Both geological and archaeological samples from Maibulaq and Rahat, and geological samples from Beriktas, were collected for the petrographic studies and are described below.

We located primary and secondary sources of chert, mainly in the northern foothills of the Qaratau (see Figure 1). In the 2019 field season, we recorded a primary source of chert at the Yntaly locality (Namen et al. 2022b) in the northeastern foothills of the Lesser Qaratau range. Archaeological and geological samples of lithic raw materials were collected at the sites of Buirekbastau-Bulaq, Qyzyltau, Sorkol, Qoshqorgan-Shoqtas complex, and

Valikhanova. Hominins at all of the mentioned sites used locally occurring varieties of chert and other silica-rich rocks. The raw material survey was primarily concentrated near the stratified site of Valikhanova. A primary outcrop of chert was located approximately 200–300 m from the site on the right bank of the Arystandy River. The majority of the chert-bearing outcrop is currently almost completely covered by aeolian loess sediments; however, small parts of the outcrop remain exposed, allowing access for sampling purposes (Figure 4).

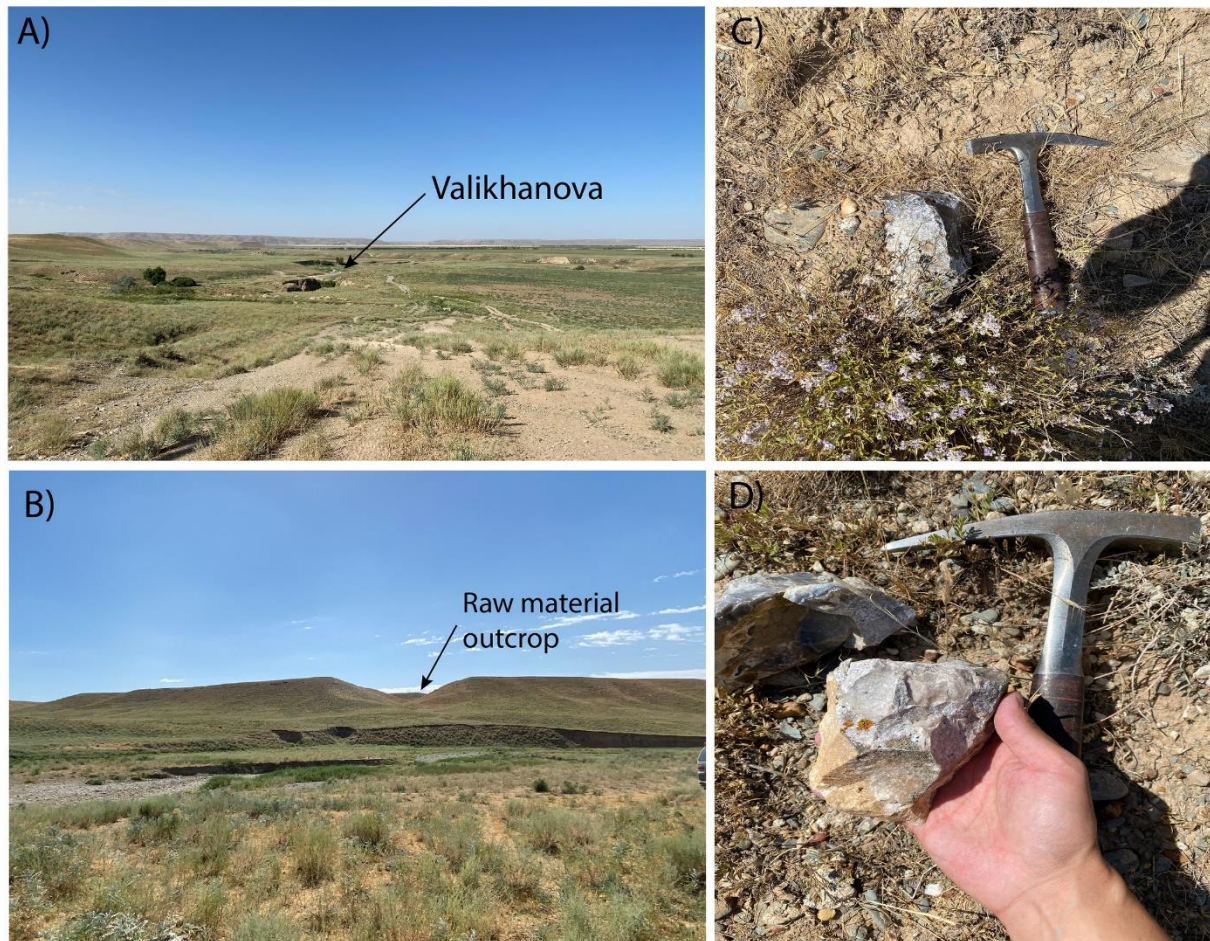


Figure 4. Panoramic view to the Valikhanova site (A) and the raw material outcrop located on the right bank of the Arystandy river (B). The outcrop is currently covered by thick loess sediments and illustrated on C) and D).

### ***Petrographic analysis***

#### ***The Qaratau sites***

From the Qaratau range, raw materials from five Palaeolithic sites were sampled (Table 2).

The Valikhanova assemblage is entirely knapped on a locally outcropping grey to dark grey chert with a varying degree of fossil inclusions. It is located on the right bank of the

Arystandy River, some 200 m away from the Valikhanova site. A total of eight geological and one archaeological surface lithic samples were thin sectioned and studied using the petrographic microscope. They are primarily composed of cryptocrystalline fibrous chalcedony grains and partly quartz veins. Some samples contain diagnostic microfossils of radiolarian filled with secondary quartz grains (Figure 5A–C). All of the studied samples are microscopically similar.

The Qyzyltau assemblage also contains lithic artifacts exclusively knapped on a locally outcropping dark grey to black variety of chert. A total of nine surface lithics were thin sectioned. The microstructural difference between Valikhanova and Qyzyltau chert is easily visible under the microscope (Figure 5D–F). The latter is entirely composed of length-fast fibrous chalcedony grains with minor inclusions of secondary mega quartz crystals. Similarly, the Sorkol samples were macroscopically identified as a grey and light pink variety of chert. The outcrop of the materials was not located in the nearby vicinity. However, a macroscopically similar light pink variety of chert outcrop is known some 3 km southwest of the site at a locality named Yntaly. As with the Qyzyltau chert, the Sorkol thin sections are composed of fibrous length-fast chalcedony.

Based on the petrographic characteristics of each chert, we can distinguish three types: Valikhanova, Qyzyltau, and Sorkol chert (shown in Figure 5). They possess characteristic inclusions such as microfossils, as well as mega quartz and quartz veins within the chalcedony fibers. These structural differences (microfacies) may assist future provenance analyses in distinguishing different sources of chert artifacts in the Qaratau region.

Despite the abundance of chert in the Qaratau region, the lithic assemblage from Buirekbastau-Bulaq is knapped on pelitic rocks such as siltstones and claystone (see Table 2, Figure 6 A–B). The preference for pelitic rocks over chert raises questions about hominin raw material selection choices in the region. The Qoshqorgan-Shoqtas complex assemblage was

also knapped on various types of available raw materials such as quartz, quartzite, limestone, chert, and sandstone (Derevianko et al. 2000).

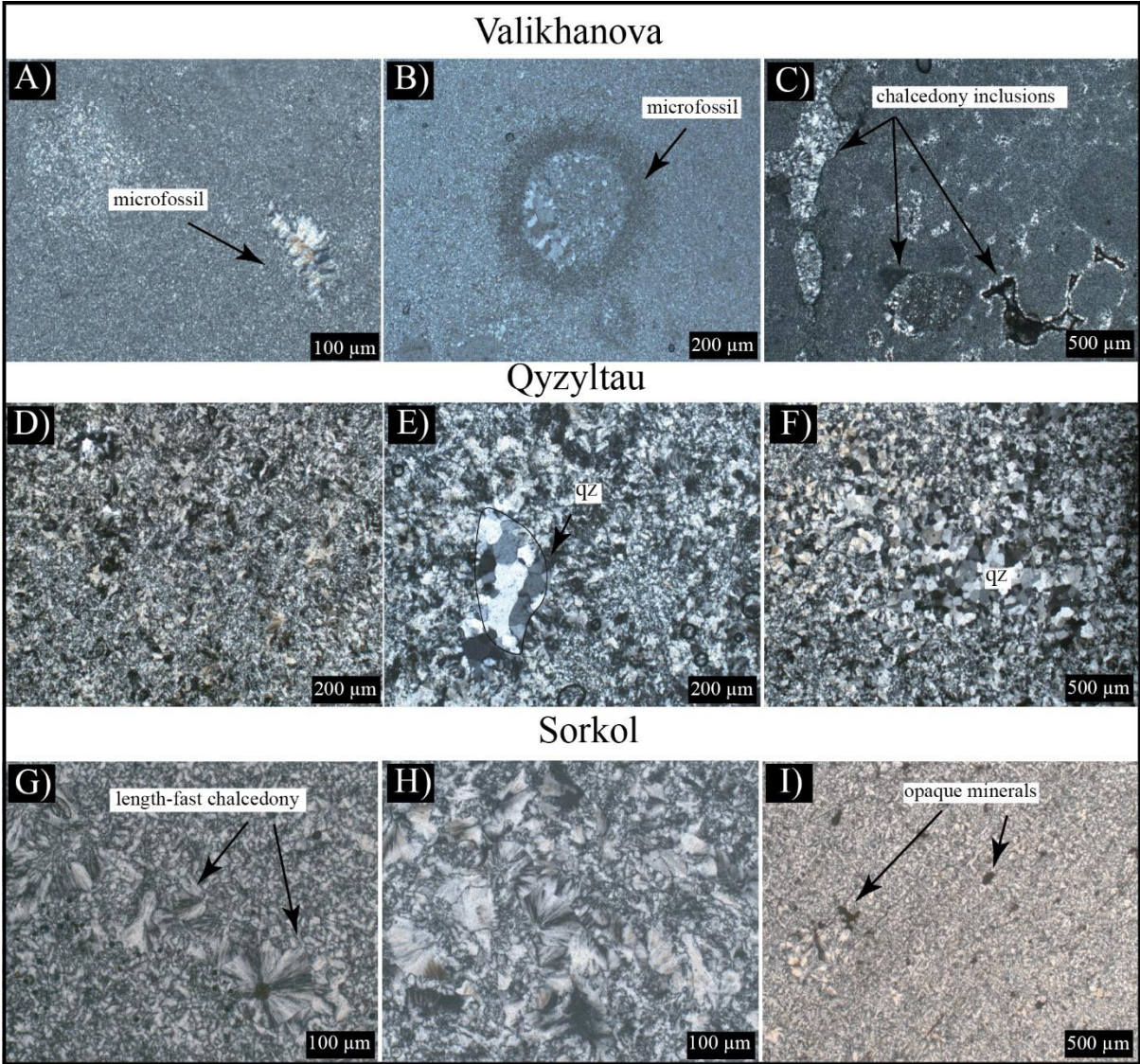


Figure 5. Photomicrograph of the chert types found in the Qaratau range. The Valikhanova chert contains microfossils (A, B) within the cryptocrystalline quartz matrix, and secondary chalcidony inclusions (C). The Qyzyltau chert consists of quartz inclusions within the cryptocrystalline quartz matrix (E, F). The Sorkol chert consists of length-fast chalcidony (G, H), and inclusions of opaque minerals (I).

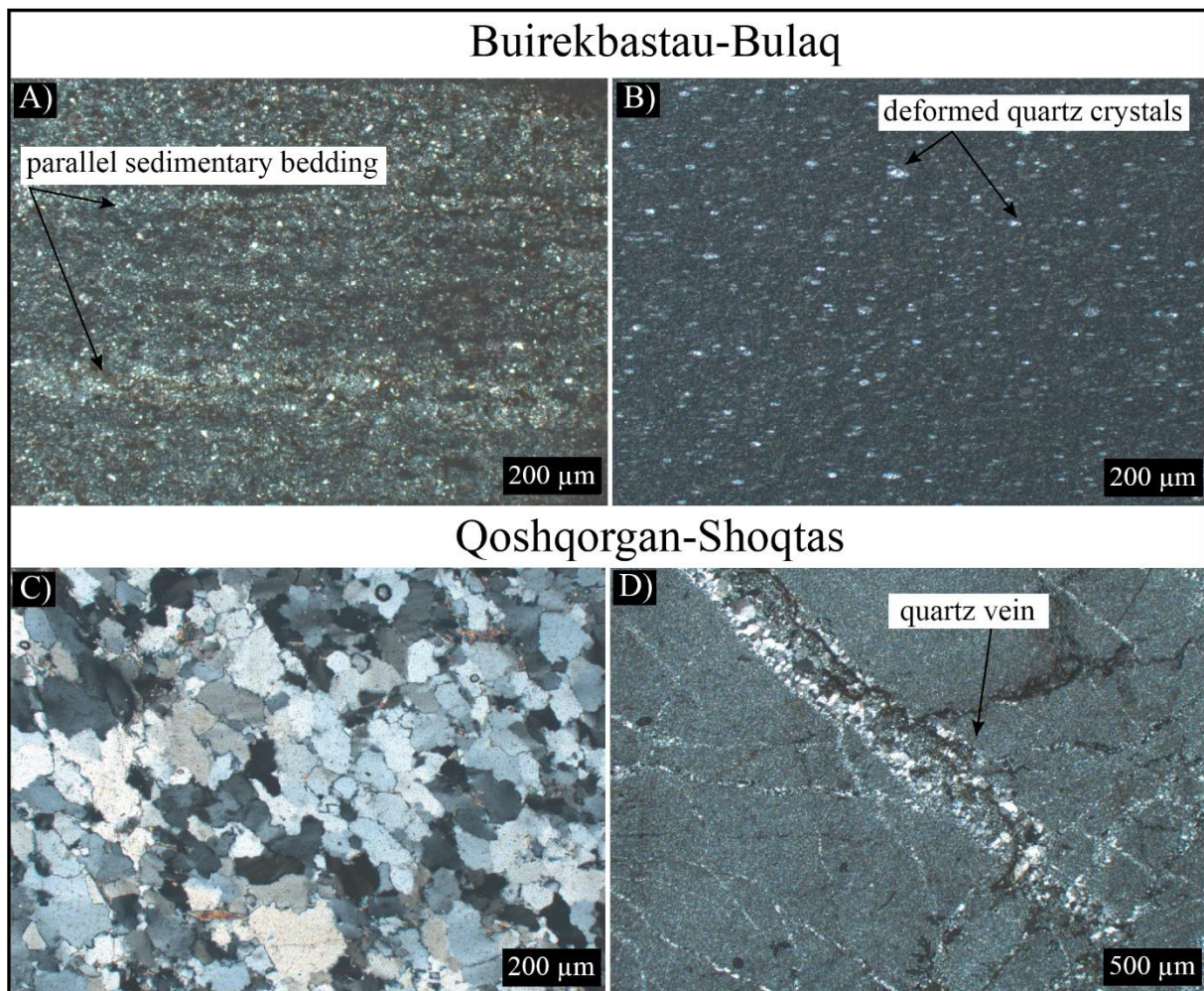


Figure 6. Photomicrograph of the raw materials from Buirekbastau-Bulaq site illustrating parallel sedimentary bedding lines (A) and deformed quartz crystals possibly indicating a process of metamorphism (B), and Qoshqorgan-Shoqtas complex demonstrates quartzite (C) and chert with quartz veins running through the rock (D).

### *The Ili Alatau sites*

The thin sections from Maibulaq porphyry can be petrographically characterized by phenocrysts of quartz and K-feldspars embedded in a siliceous matrix (Namen et al. 2022c).

The proportion of exogenous raw materials (e.g., chert and shale) from Maibulaq is illustrated in Table 2 and graphically shown in Figure 7. Whereas two geological samples and one surface lithic from Rahat were thin sectioned. They are also characterized by a siliceous matrix with randomly oriented subangular to angular crystals of quartz, cracks filled with quartz, and K-feldspars (Figure 8).. Microscopically, all of the samples exhibit similar features. Additionally, the Beriktas samples contain clay minerals of varying sizes. The



microscopic investigation revealed predominantly psammitic sandstones, greywacke, and siltstones.

Table 2. The piece counts of local and exogenous raw materials in each horizon at Maibulaq excavated during the 2004 and 2005 excavation seasons.

Raw Material	2004 and 2005 Excavation Horizon			
	Mixed	1	2	3
Porphyry	90	29	23	226
Shale	8	2	3	45
Chert	1			3
Quartzite		1		
Sandstone	1	1	1	3

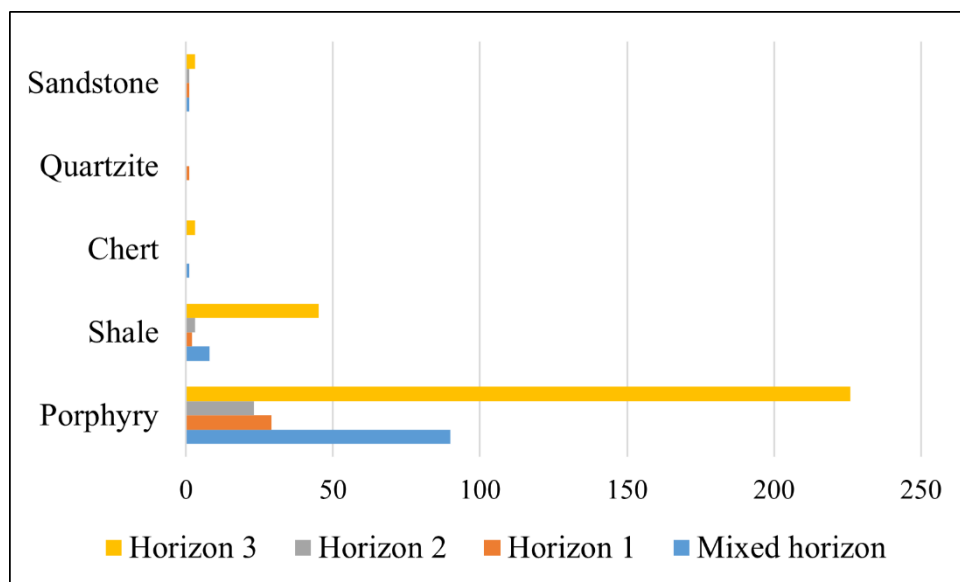


Figure 7. Bar chart of the Maibulaq raw materials. The 2004 and 2005 excavations were combined.

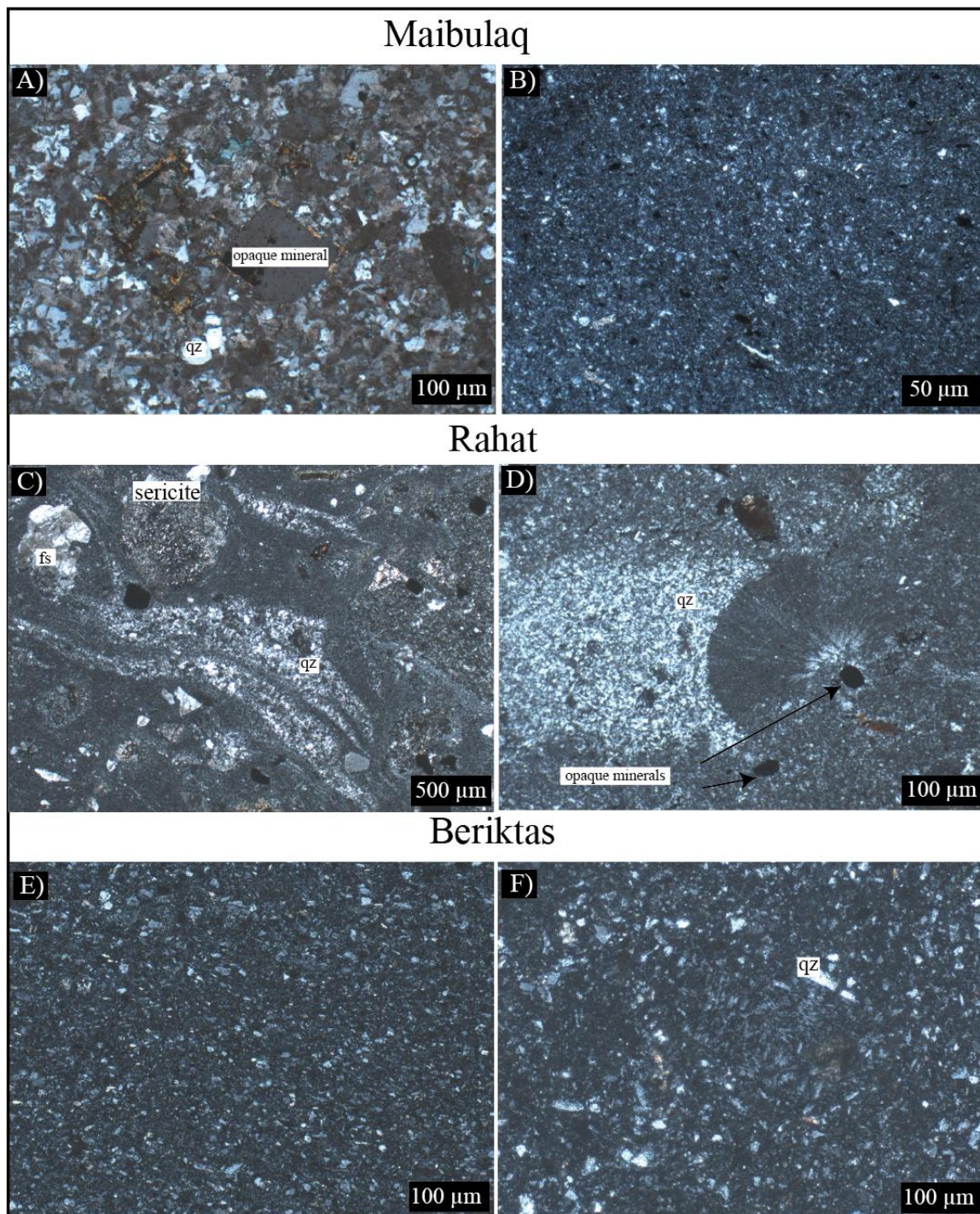


Figure 8. Photomicrograph of porphyry from Maibulaq (A and B), Rahat (C and D), and Beriktas localities (E and F) taken in different magnifications under cross-polarised light (XPL).

### *The Ushbulaq site*

Previously, the exploitation of the sedimentary rocks and petrographic features of the assemblage were investigated and described in detail by Rybin and colleagues (2018). They report the predominance of aleurolithic, fine, and microcrystalline varieties of silicites

(*silicium*), as well as a few siltstones and tuffs (Rybin et al. 2018). The terminology of silicite in Russian is used to describe a sedimentary rock enriched by silica content (Krishtofovich 1978). For the current work, we examined a total of seven surface lithics from the Upper Palaeolithic site of Ushbulaq. The macroscopic investigation of the Ushbulaq assemblage and further petrographic analysis show a predominant utilization of fine-grained rocks from the pelitic group, mainly mudstones, claystone, and siltstone enriched by silica content, as shown in the microphotographs (Figure 9).

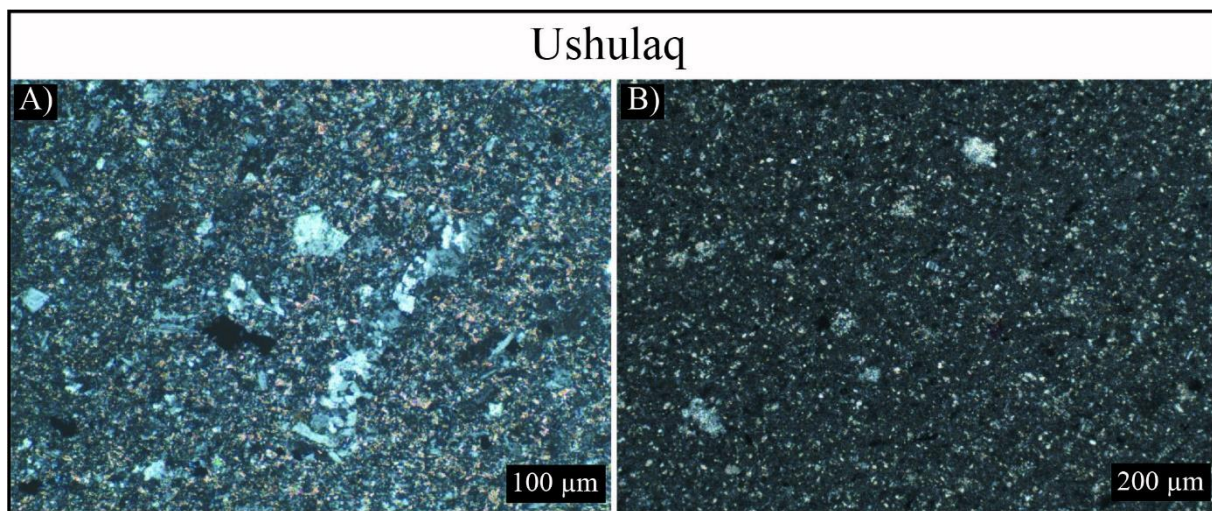


Figure 9. Photomicrograph of claystone from Ushbulaq (A and B) taken in different magnifications under cross-polarised light (XPL).

Table 3. Detailed petrographic description of the studied thin sections. Legend: qz = quartz, fs = feldspar, ca = calcite, Kfs = potassium feldspar, MS = matrix supported, and CS = clast supported.

Locality ID	Sample ID	Context	Matrix	Crystals	Grain Orientation	Grain Shape	Rock Name
	BT-2-21	geological	siliceous, CS	qz, fs	random	rounded, subrounded, subangular, angular	graywacke
Beriktas	BT-1-21	geological	siliceous, MS	qz, fs	random	rounded, subrounded, subangular, angular	fine-grained sandstone
	BT-3-21	geological	siliceous, MS	qz, fs, ca	random	subrounded, subangular, angular	very fine-grained sandstone
	BT-4-21	geological	siliceous, MS	qz, ca	random	subrounded, subangular, angular	siltstone

	BT-5-21	geological	siliceous, MS	qz, ca	random	subrounded, subangular, angular	very fine- grained sandstone
	R-1-21	archaeological	siliceous, MS	qz, Kfs, vein qz	random, poorly sorted	subangular, angular rounded, subrounded, subangular, angular	porphyry
Rahat	R-2-21	geological	siliceous, MS	qz, Kfs, vein qz	random, poorly sorted	subangular, angular	porphyry
	R-3-21	geological	siliceous, MS	qz, Kfs, iron stains chalcedony, vein	random, poorly sorted	subangular, angular	porphyry
	VK-2-21	geological	siliceous	qz, qz	cryptocrystalline	cryptocrystalline	chert
	VK-4-21	geological	siliceous	qz, vein qz, chalcedony	cryptocrystalline	cryptocrystalline	chert
	VK-9-21	archaeological	siliceous	qz, vein qz, chalcedony	cryptocrystalline	cryptocrystalline	chert
Valikhanova	VK-8-21	geological	siliceous	qz, vein qz, chalcedony	cryptocrystalline	cryptocrystalline	chert
	VK-7-21	geological	siliceous	qz, vein qz, chalcedony	cryptocrystalline	cryptocrystalline	chert
	VK-6-21	geological	siliceous	qz, vein qz, chalcedony	cryptocrystalline	cryptocrystalline	chert
	VK-4-21	geological	siliceous	qz, vein qz, radiolarian fossils	cryptocrystalline	cryptocrystalline	chert
	VK-1-21	geological	siliceous	qz, vein qz, radiolarian fossils	cryptocrystalline	cryptocrystalline	chert
	VK-5-21	geological	siliceous	qz, vein qz, radiolarian fossils	cryptocrystalline	cryptocrystalline	chert
	SK-4-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
	SK-1-21	archaeological	siliceous	length-slow chalcedony	cryptocrystalline	cryptocrystalline	chert
	SK-2-21	archaeological	siliceous	vein qz, length- fast chalcedony	cryptocrystalline	cryptocrystalline	chert
	SK-3-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
Sorkol	Sk-5-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
	SK-7-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
	SK-8-21	archaeological	siliceous	length-fast chalcedony, calcite crystals (?)	cryptocrystalline	cryptocrystalline	chert
	SK-6-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
	QT-1-21	archaeological	siliceous	length-fast chalcedony, vein qz	cryptocrystalline	cryptocrystalline	chert
	QT-2-21	archaeological	siliceous	length-fast chalcedony, micritic inclusions	cryptocrystalline	cryptocrystalline	chert
	QT-3-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
Qyzyltau	QT-4-21	archaeological	siliceous	length-fast chalcedony, vein qz	cryptocrystalline	cryptocrystalline	chert
	QT-5-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
	QT-6-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
	QT-7-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
	QT-8-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
	QT-9-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert

Maibulaq	QT-10-21	archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert medium- grained siltstone
	MB-9-21	archaeological	siliceous, MS siliceous, MS	qz, Kfs, hematite, ca vein qz, qz, fs, hematite	random, well sorted	subrounded, rounded	porphyry porphyry porphyry porphyry
	MB-7-21	geological			-	very angular	porphyry
	MB-8-21	geological	siliceous, MS	qz, fs, hematite	-	very angular	porphyry
	MB-4-21	geological	siliceous, MS	qz, fs	-	very angular	porphyry
	MB-5-21	geological	siliceous, MS siliceous, MS	qz, fs	-	very angular	porphyry
	MB-3-21	geological		qz, fs, magnetite, iron inclusions	-	very angular	porphyry
	MB-6-21	geological	siliceous, MS siliceous, MS	qz, fs, magnetite	-	very angular very angular grains of feldspar	porphyry porphyry
	MB-2-21	geological		microcrystalline qz, fs	-		rhyolite? medium- grained sandstone
	MB-1-21	geological		qz, fs, iron inclusions	moderately sorted	angular, subrounded	
	BBB-1-21	archaeological	siliceous, MS	qz, sericite, magnetite	random, well sorted	angular, subrounded	siltstone
	Buirekbastau- Bulaq	BBB-11-21	archaeological	siliceous, MS	microcrystalline qz, magnetite	well sorted	rounded rounded,
BBB-4-21		archaeological	siliceous, MS	qz, iron minerals microcrystalline	well sorted	subrounded	siltstone
BBB-3-21		archaeological	siliceous, MS	qz, biotite, opaque minerals	well sorted	rounded	claystone
BBB-2-21		archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
BBB-5-21		archaeological	siliceous, MS	microcrystalline qz, clay minerals	well sorted	rounded subrounded,	claystone
BBB-6-21		archaeological	siliceous	qz, fs, iron inclusions,	moderately sorted	rounded, subangular	siltstone
BBB-7-21		archaeological	siliceous	length-fast chalcedony	cryptocrystalline	cryptocrystalline	chert
BBB-9-21		archaeological	siliceous	length-fast chalcedony, microcrystalline qz inclusions	cryptocrystalline	cryptocrystalline rounded,	chert
BBB-10-21		archaeological	siliceous	qz, clay minerals, iron inclusions	well sorted	subrounded rounded, subrounded,	siltstone
BBB-8-21		archaeological	siliceous	fs, qz, biotite	well sorted	subangular	siltstone
UB-1-19		archaeological	siliceous	qz, clay minerals microcrystalline	well sorted	rounded	claystone
Ushbulaq		UB-2-19	archaeological	siliceous	qz, fs microcrystalline	well sorted	rounded rounded,
	UB-3-19	archaeological	siliceous	qz	well sorted	subrounded	siltstone
	UB-4-19	archaeological	siliceous	qz, clay minerals microcrystalline	well sorted	rounded	claystone
	UB-5-19	archaeological	siliceous	qz opaque minerals,	well sorted	rounded	claystone
	UB-6-19	archaeological	siliceous	qz, iron inclusions microcrystalline	well sorted	rounded	siltstone
	UB-7-19	archaeological	siliceous	qz	well sorted	rounded	claystone
	Qoshqorgan- Shoqtas complex	raw1	archaeological	siliceous, CS	mega qz	well sorted	rounded
raw4		archaeological	siliceous, CS	mega qz microcrystalline	well sorted	rounded	quartzite
raw3		archaeological	siliceous	qz, vein qz microcrystalline	cryptocrystalline	cryptocrystalline	chert
	raw2	archaeological	siliceous	qz, vein qz	cryptocrystalline	cryptocrystalline	chert

## Discussions and Conclusions

The petrographic analysis revealed the types of sedimentary and volcanic rocks exploited during the Palaeolithic in the IAMC of Kazakhstan. Based on these results, we observe differences between the types of chert utilized in the Palaeolithic assemblages of the Qaratau range. The photomicrographs of the chert thin sections demonstrate microscopic variations between the Valikhanova, Qyzyltau, and Sorkol cherts with varying degrees of quartz, chalcedony, and other opaque mineral inclusions (see Figure 5). It is especially important to point out the presence of different microfossils (mainly radiolarian) within the Valikhanova chert that makes it distinguishable from other types of chert available in the Qaratau region. Such microscopic features will help in the future to provenience chert artifacts in Kazakhstan. It is also necessary to investigate the influence of different inclusions on the knapping quality and the mechanical properties of these lithologies. Such investigations will inform discussions on knappability and technological variations between different types of silica rocks (Manninen 2016; Loendorf et al. 2018; Schmidt et al. 2019; Namen et al. 2022c).

The geoarchaeological survey results around the stratified and surface sites suggest that prehistoric hominins preferred to set their camps by water bodies and in close vicinity to raw material sources. This is a pattern that is characteristic of all of the sites mentioned in the current study. However, this observation could be the result of research bias (because archaeologists typically look for sites near water sources). Nevertheless, earlier investigations of the lithic assemblages report the acquisition of only local (e.g., Valikhanova) or predominantly local raw materials (e.g., Maibulaq, Rahat, and Ushbulaq; Taimagambetov 1990; Taimagambetov and Ozherelyev 2008; Anoikin et al. 2019; Ozherelyev, Dzhasybaev, and Mamirov 2019). In this context, the case of the Maibulaq site is rather interesting in terms of discussing the raw material availability and variation around the site. The eponymous valley where the site is located offers various types of knappable raw materials, including

porphyritic rocks and very fine-grained felsitic rocks that are found up the stream from the Maibulaq. In addition to the locally available rocks, a systematic survey along the Ili Alatau foothills revealed a source of higher quality siltstone that is located some 20 km west of the site. Despite the better knappability of felsitic rocks and siltstone, porphyritic rocks were the main raw material choice in the organization of tool manufacturing. This allows us to conclude that the toolmakers were highly selective in their choice of local raw materials and that they perhaps optimized something other than the ease of flaking. Moreover, the analysis of lithic raw material variation of the Maibulaq assemblage that is available in the collections of Al Farabi Kazakh National University ( $n = 437$ ) shows that a small fraction of tools was knapped on materials transported from elsewhere (see Table 2, Figure 7). The small number of tools knapped on exogenous materials (siltstones and chert) could be an indicator of an embedded procurement strategy (Binford 1979). We suggest that these materials were collected while carrying out other tasks (embedded procurement) and subsequently carried to the site. However, we should note that our assumption is solely based on the presence of tools knapped on exogenous raw materials. Additionally, the preference for porphyritic rocks by human groups at Maibulaq calls for supplementary investigations into its quality for knapping and fracture mechanics (e.g., indentation fracture toughness, fracture strength, elasticity, etc.) (Mardon et al. 1990; Domanski, Webb, and Boland 1994; Doelman, Webb, and Domanski 2001; Schmidt et al. 2019; Namen et al. 2022c).

Based on the above results, we conclude that hominin groups at Maibulaq had both direct and embedded raw material procurement strategies. However, the choice of mainly porphyritic rocks for the organization of knapping activities suggests that direct procurement was predominant. The human groups at Maibulaq were highly selective while acquiring local materials, as evidenced by selection of porphyritic rocks over other locally available materials. Moreover, the cobbles currently available in the Maibulaq stream bed today tend to have a higher incidence of large phenocrysts than those encountered in the archaeological

collection. We may, therefore, hypothesize that the quality of available lithologies was deemed suitable for the production of desired end products or that the hominins had to adapt their knapping technology to the quality of available rocks. Similar conclusions have been made before, but the major emphasis has been on the opportunistic use of the local raw material as a function of decreasing time and energy costs, which led to the adaptation of knapping techniques to the available resources (Ghasidian and Heydari-Guran 2018). However, the opportunistic use of local raw materials should not be considered in the Maibulaq case. The preference of porphyry over felsitic rocks available at the site or siltstones available within a 20 km distance should be viewed as the result of deliberate selection. The evidence of raw material selection patterns has already been discussed for the Palaeolithic of Africa (Goldman-Neuman and Hovers 2012; Harmand 2009; Harmand et al. 2015). Braun and colleagues (2009) argue that the selectivity of lithic raw material in Oldowan technology was driven by raw material quality. The use of so-called lower-quality porphyritic rocks is common in Palaeolithic assemblages of neighboring regions in central Asia (e.g., Kattasai 1 and 2 in Uzbekistan [Kot et al. 2022]), Siberia (Derbina V [Kharevich, Akimova, and Stasyuk 2010]), and the Russian Far East (Ezhantsy I and II [Mochanov 1977]). The exploitation of volcanic porphyritic rocks at these sites could possibly be interpreted as the ability of prehistoric human groups to knap these raw materials. We also believe the criteria guiding the selection of explicitly porphyritic rocks were driven by a number of factors, including the raw material quality, technological skills, and familiarity with working these materials. Despite the presence of exogenous rocks, and considering the inter-site variability of raw materials at Maibulaq, we suggest that a direct selective procurement strategy formed the basis for the technological and territorial organization.



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## **Conflict of Interest**

The authors declare that they have no conflicts of interest.

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## LIST OF PUBLICATIONS

### *Peer-reviewed publications:*

1. Iovita, R., Varis, A., **Namen, A.**, Cuthbertson, P., Taimagambetov, Z., Miller, C.E., 2020. In search of a Paleolithic Silk Road in Kazakhstan. *Quaternary International* 559, 119–132. doi:10.1016/j.quaint.2020.02.023
2. Cuthbertson, P., Ullmann, T., Büdel, C., Varis, A., **Namen, A.**, Seltmann, R., Reed, D., Taimagambetov, Z., Iovita, R., 2021. Finding karstic caves and rockshelters in the Inner Asian mountain corridor using predictive modelling and field survey. *PLOS ONE* 16, e0245170. doi:10.1371/journal.pone.0245170
3. Varis, A., Miller, C., Cuthbertson, P., **Namen, A.**, Taimagambetov, Z., Iovita, R., 2021. The Effect of Formation Processes on The Frequency of Palaeolithic Cave Sites in Semi-Arid Zones: Insights From Kazakhstan. *Geoarchaeology*. doi:10.1002/gea.21909
4. **Namen, A.**, Varis, A., Lindauer, S., Friedrich, R., Taimagambetov, Zh., Iovita, R. Nazugum, a new 4000 year old rockshelter site in the Ili Alatau, Tien Shan. Submitted to *Archaeological Research in Asia*. doi:10.1016/j.ara.2022.100370

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5. **Namen, A.**, Varis, A., Cuthbertson, P., Iovita, R., Taimagambetov, Z., 2020. Predvaritel'nye itogi razvedovatel'nykh rabot proekta PALAEOSILKROAD: mul'tidistsiplinarnyi podkhod v issledovaniyakh [Preliminary survey results of the PALAEOSILKROAD project: multidisciplinary approach]. In: *Proceedings of the International scientific and practical conference "Great Steppe in light of archaeological and interdisciplinary research."* Presented at the Margulan Readings - 2020, A. Kh. Margulan Institute of Archaeology, Almaty, pp. 60–69.