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Identifying organic residue on ground stone tools from Mesolithic sandy sites: selected examples from Paliwodzizna 29 in Central Poland

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IDENTIFYING COLLAGEN RESIDUES ON GROUND STONE TOOLS FROM MESOLITHIC SANDY SITES: SELECTED EXAMPLES FROM PALIWODZIZNA 29 IN CENTRAL POLAND

ABSTRACT

Cristiani E., Orłowska J. and Grzegorz Osipowicz. 2024. Identifying collagen residues on ground stone tools from Mesolithic sandy sites: selected examples from Paliwodzizna 29 in Central Poland, *Sprawozdania Archeologiczne* 76/1, 237-250.

During excavations at the Mesolithic site Paliwodzizna 29 (central Poland), in addition to the flint artefacts typical for this period and region, a collection of 175 non-flint stone artefacts was discovered. This collection included various "macro-lithic tools" displaying visible intentional modifications and usage traces. This observation prompted a preliminary traceological analysis of a selection of tools. The primary goal of the presented research was to assess the possibility of analysing and interpreting use-wear traces and potential residues on these tools. The ultimate aim was to investigate the feasibility of conducting such analyses on macro-lithic tools from the sandy sites of the Polish Lowland dating back to the Mesolithic.

Keywords: use-wear; residues; collagen; macro-lithic tools; Polish Lowland; Mesolithic
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1. INTRODUCTION

Stone artefacts constitute one of the sole sources identified at sites from the early Holocene periods in Central Europe. The territory of Poland is rich in flint resources, making it the primary material used for stone tool production. Other types of rocks, especially those with larger crystals, were considered less significant in this regard. However, it is widely believed that they were used for creating various types of macro-lithic forms. The term “macro-lithic tools”, including ground stone tools and other non-flaked artefacts, encompasses a diverse category of non-flint artefacts utilised for percussion, abrasion, polishing, cutting, and grinding activities (Adams *et al.* 2009; Adams 2014). Common tool types found in macro-lithic assemblages include hammerstones, abraders, grinding implements (*e.g.*, grinding slabs, querns), as well as pounding (*e.g.*, mortars and pestles) and cutting (*e.g.*, axes and adzes) implements. What is worth mentioning is that this category of artefacts is generally characterised by extended life histories (Dubreuil *et al.* 2015; Dubreuil and Savage 2014). In recent years, our knowledge of the function of this category of artefacts and their significance in the daily life of prehistoric European hunter-gatherers has greatly advanced. This progress has been made possible through the application of use-wear and residue analysis to these artefacts (*e.g.*, Aranguren *et al.* 2007; Revedin *et al.* 2010; Lippi *et al.* 2015; Cristiani and Zupancich 2020; Cristiani *et al.* 2012; Cristiani *et al.* 2021).

In the Mesolithic period, ground stone tools played an essential role in the toolkit of hunter-gatherer communities (*e.g.*, Gilabert *et al.* 2016). Tools of this type were often used in the Mesolithic for processing plant materials, although many other activities were also performed with them (Holst 2010; Gilabert *et al.* 2016, 213, 214; Cristiani and Zupancich 2020; Cristiani *et al.* 2021).

Various types of macro-lithic tools are also documented within the stone assemblages of Mesolithic sites in Poland, *e.g.*, Pobiel 10 (Bagniewski 1990), Pomorsko 1 (Kobusiewicz and Kabaciński 1991), Luta 1 (Więckowska-Chmielewska 2007), Bolków 1 (Galiński 2014), Ludowice 6 and Sąsieczno 4 (Osipowicz 2017, 68-71). Usually, studies of these artefacts have focused on describing their morphological and technological attributes, and despite their presence, these tools usually remain poorly understood, as no analyses have been conducted to investigate their use so far. Many researchers, of course, are tempted to interpret the functions of these types of objects. Still, these interpretations are usually based solely on macroscopic or low magnification observations and are not supported by more detailed analyses (Bagniewski 1990).

In the collection of stone artefacts discovered at Paliwodzizna 29 (central Poland), several items are considered macro-lithic tools. They are dominated by various types of hammerstones, grinders, smoothing stones or pebbles with traces of use (various pebbles bearing visible macroscopically minor traces of processing or use). Most of the described products are made of different types of granite and quartz sandstones. This article aims to present the preliminary results of a functional analysis of selected stone artefacts discovered during excavations at the site Paliwodzizna 29. The primary goal was to address the question of the feasibility of conducting effective and reliable functional analyses of macro-lithic stone tools from this site and other Mesolithic sandy sites in the region in general.

1.1. Site Paliwodzizna 29

The Paliwodzizna 29 archaeological site is located in central Poland in the Drwęca River Valley, which separates the Dobrzyń and Chełmno Lakelands (Fig. 1; *cf.*, Solon *et al.* 2018). It lies on a flat-topped ridge situated in a place where the subglacial valley of Lake Grodno and Lake Plebanka becomes the valley of the Drwęca River (Osipowicz *et al.* 2023). Over five years of archaeological work, an area of 468 m² was explored at the site, 429 m² of which constitute trenches situated in its sandy (dry) part, whereas 39 m2 of excavations

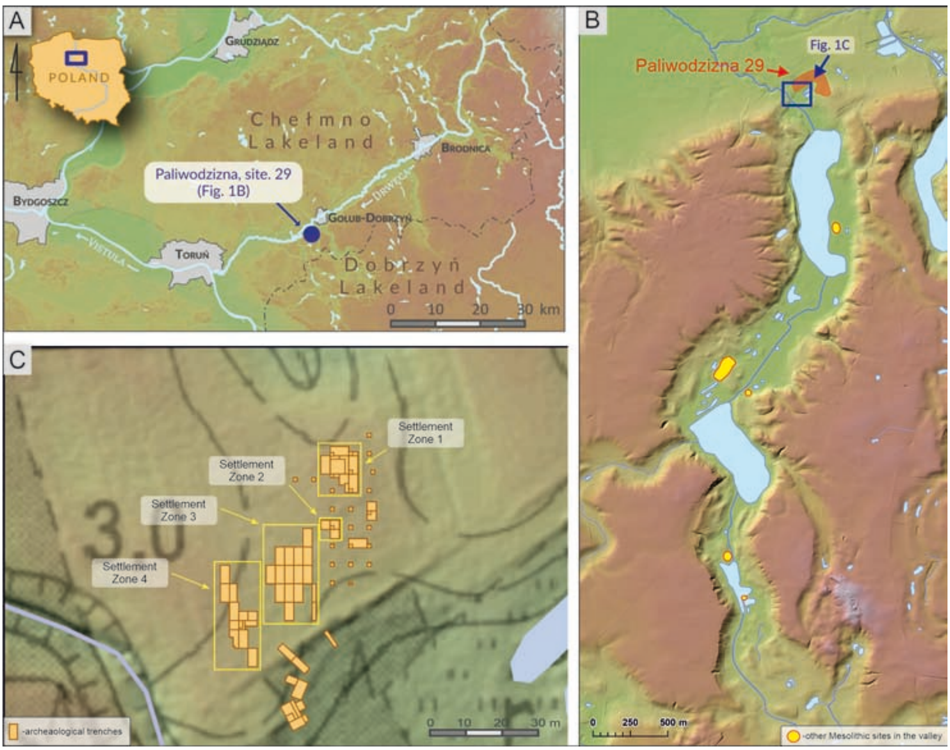

Fig. 1. Location of Site 29 in Paliwodzizna: A – location within the Chełmno-Dobrzyń Lakeland; B – location in the valley of Grodno and Plebanka Lakes; C – situational-altitude plan of the site with the archaeological trenches and settlement zones (based on Osipowicz et al. 2023, Fig. 1)

were made in the wetlands, which are the littoral zone of the former lake. As a result of the work carried out in the dry zone of the site, four clusters of Mesolithic features and artefacts were identified (Fig. 1; Osipowicz *et al.* 2023). Only the archaeological materials from settlement zone 2 have been subjected to more detailed analyses (Osipowicz 2021).

The primary period of usage for the site falls within the Mesolithic. Artefacts from this period were discovered in the site's sandy (dry) part and the peat (wetland) trenches. In the "dry" zone, these artefacts were found within distinguishable flint scatters and cloud-like structures associated with numerous hearths, economic and ritual pits (up to 2 metres deep), and other structures. The flint tool typology from this area and radiocarbon dating indicate that this part of the site was used throughout the Mesolithic. In the peat zone, Mesolithic materials were discovered in two layers of sand with a gyttja admixture. These layers contain many large charcoal fragments and seem exclusively associated with Early Mesolithic settlements (Osipowicz *et al.* 2023). The long series of radiocarbon dates hitherto obtained indicate a more than a three-thousand-year period of the development of the Mesolithic settlement in Paliwodzizna 29, covering the period between about 9290 cal BC (Poz-121364: 9700±60 BP) and about 5731 cal BC (Poz-121444: 6780±40 BP; Osipowicz *et al.* 2023).

Various types of non-flint artefacts were identified among the many discovered artefacts at the site. In total, 175 stone items were unearthed. Many of them can be interpreted as an example of the elements of a specific industry from non-flint rocks, represented by flakes, chips, *etc.* (44%), similar to industries identified on other Mesolithic sites in the region (Osipowicz 2013, 2014; Osipowicz *et al.* 2014; Osipowicz and Sobkowiak-Tabaka 2014). However, a significant majority represents a classic set of macro-lithic tools, initially classified by visual inspection and macroscopic use wear as hammers (6%), grinders (25%), smoothing/grinding plates (11%), or pebbles with traces of use (14%).

2. MATERIALS AND METHODS

2.1. Materials

The subject of the analysis reported in this article are two stone artefacts that can be treated as an archaeological sample from Paliwodzizna 29, referred to as Specimen #1 and Specimen #2. Both artefacts were discovered in Early Mesolithic layers of the site.

The first specimen was found in the site's northern part of Settlement Zone 1, in Feature 81 (inv. No. W933; Fig. 2: A), and it is a naturally rounded pebble measuring 15 × 10.4 × 6.4 cm. The weight of the artefact is 1.30 kg. The tool does not present any technological modification on its surface. Based on its morphological features, the artefact was initially identified as a grinder due to the two flatter surfaces with clear signs of use opposite each other. It was made of granite.

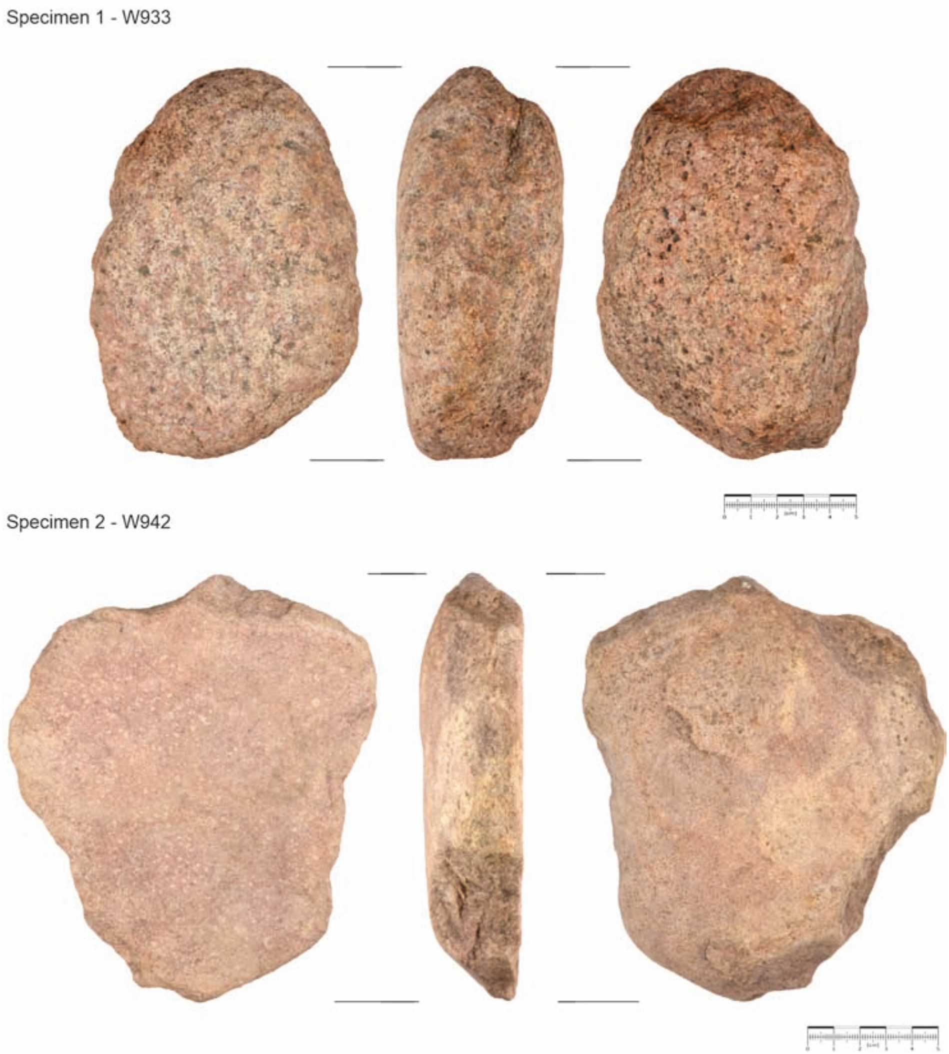

Fig. 2. Ground stone tools analysed in the study from the Paliwodzizna 29 site (photos by Justyna Orłowska)

The second specimen was found in the site's northern part of Settlement Zone 4 (inv. no. W942; Fig. 2: B). The item is characterised by a flat, even surface, which was processed around the circumference, likely to attain the desired tool shape. The dimensions of this tool fall between 16,4 × 14,1 × 4 cm. The weight of the artefact is 1,35 kg. The object can be considered as a type of grinding slab. The tool was made from a block of Jotnian sandstone.

2.2. Methods

The analysed tools were secured directly after their discovery at the site and were not washed.

The macro-lithic tools were sampled and analysed at the DANTE-Diet and Ancient Technology laboratory at the Sapienza University of Rome. Strict protocols were followed to control modern contamination during the residue sampling and functional analysis of GSTs. Before analysing each tool, the bench surfaces were meticulously cleaned with ethanol and hot water and then covered with aluminum foil. Starch-free gloves were used while handling the GSTs. Use-wear and residue analyses were carried out on surfaces unaffected by severe post-depositional modifications and free from concretions. Additionally, the reliability of residues was contingent on their association with use traces.

Use wear study. The functional study examined use-wear traces on the macro-tool surfaces using two magnification levels. At low magnification (0.75×-168×), a Zeiss Axio Zoom V16 stereomicroscope was employed, while high magnification analysis (200×-500×) utilised a Zeiss AxioScope A1 metallographic reflected light microscope (Cristiani and Zupancich 2020; Dubreuil *et al.* 2015). The low magnification facilitated the evaluation of material preservation and the identification of residues still adhering to the surfaces. Factors such as appearance, morphological features, and spatial patterns of macro-residue distribution were taken into consideration (Langejans 2010).

Residues. Subsequently, 20µl of ultra-pure water was applied to various areas of the tool surface for extraction. After pipetting out, each sample was mounted on a glass slide with a 50% solution of purified water and glycerol. The observation was conducted in transmitted light (TL) using a Zeiss Imager2 with magnification up to 1000x, 10x eyepiece, and cross-polarizing (CP) filters. Following this, samples underwent staining using Picro-Sirius Red (PSR) according to established protocols (Cristiani and Zupancich 2020; Stephenson 2015) and were analysed in transmitted cross-polarized light. PSR, a biochemical stain known for its ability to react with Type I and Type III collagen fibres, transforming them into a vivid red and enhancing their birefringence in cross-polarized light, was employed (see Stephenson 2015, pp. 236 for details). Additionally, PSR was chosen for its capability to provide a contrast-enhanced view of plant structures, including starch grains or phytoliths (Stephenson 2015).

Photogrammetry

3D Models of analysed artefacts have been created through the application of photogrammetry. The tools were placed on an automatic turntable in order to produce 360° sets of each of the tool's surfaces. Images of the tools were captured using a Nikon D7100 DSLR camera fitted with a Nikon Nikkor AF Micro 60mm f/2.8D lens. A total of 151 images per object were captured and then imported into RealityCapture 1.3 to create high quality, dense point clouds and meshes.

3. RESULTS

Technological analysis

Only in the case of Specimen #2 were traces identified that indicate intentional shaping. In a few places around the circumference of the tool, there are visible traces of deliberately removed flakes (Fig. 4: a). All negatives visible on the surface of the artefact were knapped by hitting the edges of the upper, flat surface of the object, and they vary in shape and size. Their morphology suggests that they were removed using the hard hammer technique.

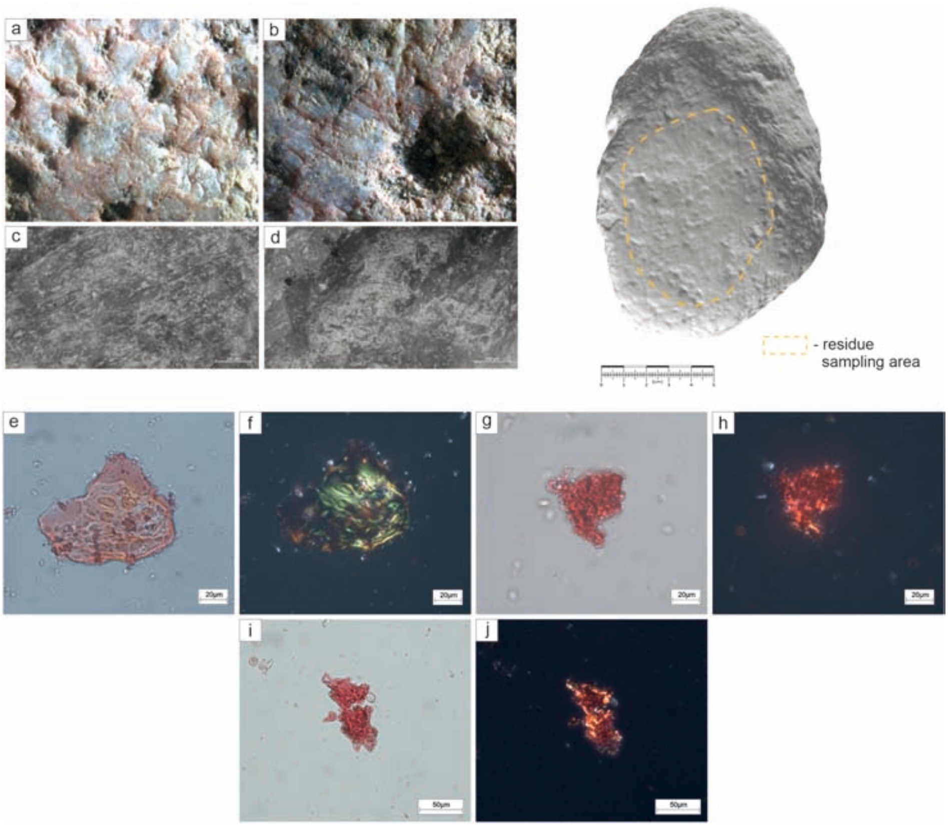

Fig. 3. The 3D model of Specimen #1 with marked residue sampling area (W933) and the examples of the use-wear and organic residues observed on its surface: a, b) sinuous micro-relief of the used surface and grain extractions; c, d) a smooth, domed, and occasionally striated micro-polish; e-j) PSR stained residues documented in extracted solutions under the TL (photos by Emanuela Cristiani and Justyna Orłowska)

Use-wear analysis

The macroscopic involved the analysis of various usage traces, including flattened surfaces, pitted areas, and rounding of single grains, among other things.

In the case of both analysed specimens, the identified use-wear consists of sinuous micro-relief of the used surface, grain extractions and smooth domed micro polish affecting the low and high micro topographies. In many places the surfaces appear slightly abraded (Fig. 3: a,b; Fig. 4: b-d). In the case of Specimen #1, at high magnification, both high and low micro topographies on the macro-tool surfaces exhibit a smooth, domed, and occasionally striated micro-polish (Fig. 3: c, d), frequently related to material processed of medium hardness (see *e.g.*, Cristiani and Zupancich 2020). A high degree of micro rounding of the edges of the grains is also visible (Fig. 3: d).

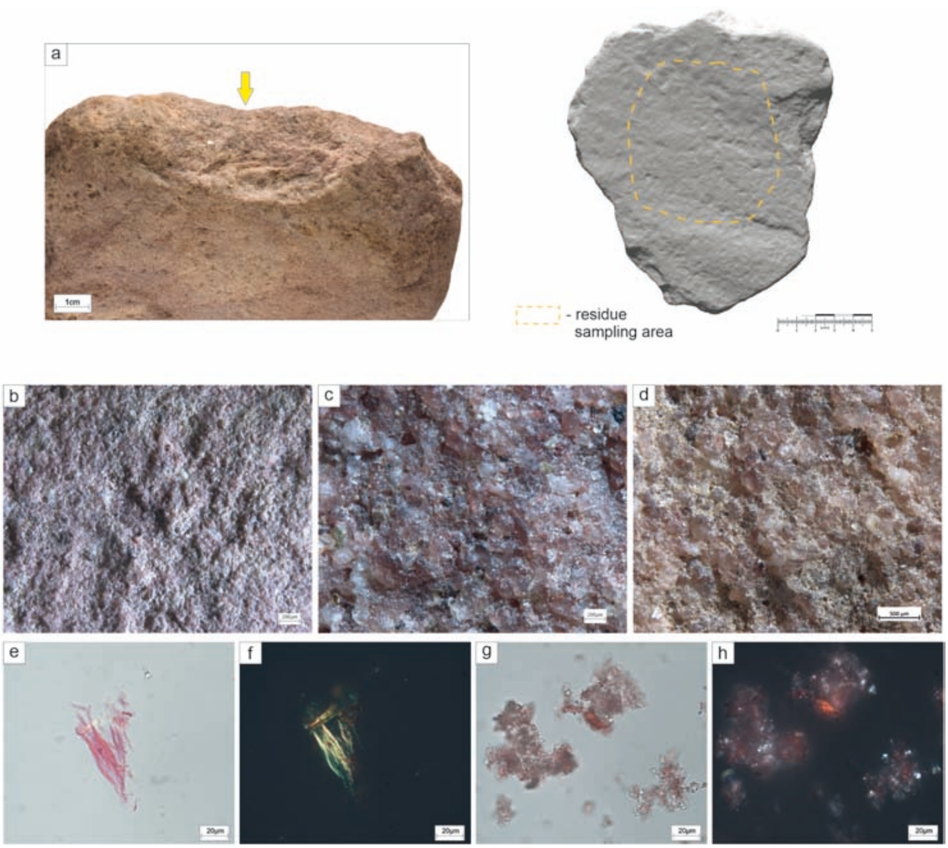

Fig. 4. The 3D model of Specimen #2 (W942) and the examples of the technological, use-wear and organic residues observed on its surface: a) negatives of deliberately removed flakes with the direction of impact marked (yellow arrow); b-d) sinuous micro-relief of the used surface and grain rounding and extractions; e-h) PSR stained residues documented in extracted solutions under the TL (photos by Emanuela Cristiani and Justyna Orłowska)

Residue analysis

Residues in the form of the organic film and possibly animal fibres were identified on both analysed tools (Fig. 3: e-j and Fig. 4: e-h). These residues were sampled and stained using PSR for observation in cross-polarized light, highlighting the presence of collagen. In cross-polarized light, certain fibres transformed into a yellow-green colour (Fig. 3: f and Fig. 4: f), typical of Type III collagen, characterising the reticular fibres present in flat collagen tissues. Others observed in cross-polarized light, displayed an enhanced yellow-orange birefringence, typical of Type I collagen (Fig.3: h, j; Fig. 4: h).

4. DISCUSSION

Building on prior research into the composition and frequency of Fennoscandian erratics found in the Polish Lowlands, it is established that the primary source of raw materials for stone processing in prehistory was local post-glacial rock resources. Specifically, Fennoscandian erratic boulders and pebbles found in sediments shaping various forms of the young glacial landscape in the Polish Lowland played a crucial role (*e.g.*, Chachlikowski 2013). These raw materials were likely acquired by Mesolithic people through gathering from the ground surface and also extracted using the open-pit method to obtain suitable stone raw materials in terms of their lithology and size from the surrounding Pleistocene deposits (Chachlikowski 2013, 40-59, 97-106). It is important to note that the boulder pavement (lag deposits) found at the site Paliwodzizna 29 served as a resource for the Mesolithic people, who utilised it to extract stones and, potentially, flint. Pits excavated for this purpose reached depths of up to approximately 2 m and diameters of about 5 m. Engaging in such extensive earthmoving not only indicates the extraction of resources but also suggests the possibility of a more permanent settlement at the site (Osipowicz *et al.* 2023).

As mentioned in the introduction, various collections of Mesolithic stone macro-lithic tools were discovered at many sandy sites in the Polish Lowland, *e.g.*, Pobiel 10 (Bagniewski 1990), Pomorsko 1 (Kobusiewicz and Kabaciński 1991), Luta 1 (Wieckowska-Chmielewska 2007), Bolków 1 (Galiński 2014), Ludowice 6 and Sąsieczno 4 (Osipowicz 2017, 68-71). However, the limited archaeological visibility of certain types of tools, especially those presenting minimal or no technological modifications, may have contributed to many tools being overlooked or interpreted as ordinary stones. However, as the example of stone tools discovered at the Pobiel 10 site shows, the first attempts to analyse their surface using low magnifications were also made (Bagniewski 1990). Nevertheless, these tools remain insufficiently understood, as no specialised analyses have been conducted to investigate their function.

A preliminary traceological and residue analysis of macro-lithic tools from site Paliwodzizna 29 has revealed the presence of technological and functional modifications as well as well-preserved residues on their surfaces. The patterns of distribution and orientation observed in both macro and micro wear indicate the use of analysed non-flaked stones in activities involving thrusting and resting ("scraping") percussion including longitudinal movements. Similar gestures are used to soften dry hides, for example (compare Cristiani and Zupancich, 2020). The nature of the residues observed on the tools – fibres typical of Type I and Type III collagen- confirms functional interpretations based on the use-wear traces. Collagen is the most abundant mammalian protein and is commonly found in tendons, skin, ligaments, bone, the gut, and blood vessels. It is a crucial component of connective tissues, such as tendons, ligaments, skin, and bones. It provides structure, strength, and support. In tissues like skin and tendons, collagen fibres are arranged in bundles, providing tensile strength (Di Lullo *et al.* 2002; Stephenson 2015). More importantly, collagen can endure for extended periods in archaeological contexts, particularly within the thick-crystalline morphology of macro-lithic stone tool surfaces (Buonasera 2005; Field *et al.* 2009, 228). Different types of collagens can be distinguished, two of which have been identified on tools from Paliwodzizna 29. Type I collagen is the most abundant collagen and is found in virtually all connective tissues. It is a component of the interstitial matrix and the major structural protein of bone, skin, tendon, ligament, sclera, cornea, blood vessels, as well as being an important component of other tissues. Of these tissues, bone and skin are the organs with the most prominent functional role for type I collagen (Henriksen and Karsdal 2016). In turn, Type III collagen is a fibrillar collagen and, unlike most other collagens, consists of only one collagen *alpha* chain. This type of collagen is abundant in connective tissues such as skin, lung, liver, intestine and the vascular system, and is often associated with type I collagen, except in bone. Together with type I collagen, type III collagen is the major component of the interstitial matrix (Nielsen and Karsdal 2016). The observed tools from Paliwodzizna often exhibit a combination of use-wear and residues, which characteristic is frequently associated with sandstone macro-tools used for working hides (Cristiani and Zupancich 2020).

The obtained results are consistent with the situation at the site. Mammal remains constitute nearly 99% of all discovered bones. Among them, bones belonging to various-sized mammals were identified, including deer (0.6%) (Osipowicz *et al.* 2023). The significance of animal resources for the settled hunter-gatherers at Paliwodzizna is evident in the predominance of pike remains among the animal bones dating back to the second settlement phase at the site. Specifically, 52.5% of all bones are from the 'wetland' zone and a remarkable 92.8% of the fish bones belong to the pike species. This finding strongly suggests that an abundance of pike could constitute the main reason for visiting this place in the early Mesolithic (Osipowicz *et al.* 2023). Importantly, in the context of the obtained results, one of the bone accumulations was discovered right next to Feature 81, from which the analysed Specimen 1 originates. This could be indirect evidence that macro-lithic tools may have been important in processing animal materials at the site, including skin or meat.

Unfortunately, it is challenging to relate the obtained results to findings from other Polish Mesolithic sites due to the lack of more detailed analyses conducted on stone macro-lithic tools. Regrettably, products of this type were often overlooked in the detailed characterisation of sites, or their analysis ended with typological classifications or functional interpretations based solely on morphological features. Unfortunately, the situation doesn't look much better in other European regions, where we can also observe a substantial lack of information about these types of artefacts for the Mesolithic period. One of the more detailed studies relating to the function of macro-lithic tools was carried out in the context of materials from the Mesolithic site of Font del Ros (southeastern Pre-Pyrenees, Spain; Gilabert *et al.* 2012; 2016). The analysis revealed specific functions of ground stone tools that can be connected with particular domestic activities, including, among others, processing of fauna materials and hide-working. Analyses of macro-lithic tools from other European Mesolithic sites showed that tools of this type were often used for processing plant materials, for example hazelnuts in northern Germany (Holst 2010) and wild cereals in central Balkans (Cristiani *et al.* 2021). The versatility of ground stone tools at early Holocene hunter-gatherer sites suggests that they played an essential role in the daily lives of these communities, contributing to various aspects of their survival and cultural practices.

CONCLUSIONS

The article presents the preliminary results of the technological and functional analyses conducted on two macro-lithic artefacts from the Mesolithic site at Paliwodzizna 29 in central Poland. The role of macro-lithic tools in Mesolithic Europe remains poorly understood. The collection of macro-lithic tools from site Paliwodzizna 29 appears to be an excellent material for conducting traceological and residue research on Mesolithic stone tools. The obtained results indicate that both use-wear traces and residues on these materials are well-preserved and identifiable. Subsequent stages of the research will involve incorporating additional materials and conducting experimental work. This phase will consider the variability of stone raw materials and the specificity of local plant and animal raw materials that could have been processed using macro-lithic tools. In the future, they can serve as a foundation for comparative analyses and interpretations of similar artefacts in studies encompassing other sites in the region and across Europe. The research on the Paliwodzizna assemblage will be continued.

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