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The function of prehistoric lithic tools: A combined study of use-wear analysis and FTIR microspectroscopy

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ABSTRACT

The application of combined use-wear analysis and FTIR micro spectroscopy for the investigation of the flint and obsidian tools from the archaeological sites of Masseria Candelaro (Foggia, Italy) and Sant'Anna di Oria (Brindisi, Italy) aiming to clarify their functional use is described. The tools excavated in the former site showed in a very high percentage spectroscopically detectable residues on their working edges. The identification of micro deposits is based on comparison with a great number of replicas studied in the same experimental conditions.

FTIR data confirmed in almost all cases the use-wear analysis suggestions and added details about the material processed and about the working procedures.

1. Introduction

The study of prehistoric stone tools aiming to infer their use gives an important contribution to the knowledge of economic, social and symbolic aspects of ancient communities life.

The interest towards prehistoric activities induced archaeologists, at the end of the 19th century, to analyze the polishes induced by the worked material on prehistoric stone tools and compare them to traces observed on replicas [1,2] or on ethnographic items whose function was known [2]. The interest engendered by the translation of the Semenov's book "Prehistoric Technology" [3] encouraged many western scholars to deal with use-wear analysis. Lithic tools were usually analyzed by means of low powered microscopes in reflected light looking principally at the micro-fractures developed on the working edge-profiles [4–6].

The use of high powered microscopes was introduced by Keeley [7] and allowed to gain information on polishes defined as visible alteration of the stone tool surface influencing its reflectivity when viewed through the microscope.

The capability of the low or high powered microscopic analysis was controversial until the methodological settlement reached only in 1989 at the Conference "*The interpretative possibilities of micro-wear studies*" held in Uppsala (Sweden) where the validity and complementarity of both methods was recognized [8]. Nowadays, the microscopic observation of a variety of traces, hereafter referred as use-wear analysis, based on the evidence that each worked material induces distinctive alteration on lithic artefacts, is a well established method.

The presence of microscopic amounts of the worked material entrapped in the micro-cavities of the irregular surface of lithic tools edges was ascertained in the late 70s and reported in a number of papers sometimes aiming to understand the formation process of use-wear whose genesis is still an open problem [9 and refs. therein]. In this view the pioneer morphological and/or chemical analysis of residues [10,11] of the material worked opened new perspectives of research alongside the more traditional use-wear analysis. Although residues can suffer morphological and chemical degradation in the archaeological deposits still the detection of surviving residues with a variety of experimental procedures gave encouraging results in the last decades [9,12].

Scanning electron microscopy (SEM), electron dispersive X-ray (EDX) and ion beam analysis (IBA) techniques, for example, were extensively applied to determine the elemental composition of residues and allowed to distinguish between bone or wood deposits from the relative abundance of calcium and phosphor [13–15]. In addition, it was possible to estimate for many residues a surface density of few $\mu g/cm^2$ and a thickness ranging from few $\mu g/cm^2$ up to 1 mg/cm^2. This means that thick deposits are not simple overlayers but diffuse into the stone and this fact explains their survival since prehistoric periods [15].

More recently, organic residues of animal and vegetable origin were identified by means of gas chromatography–mass spectrometry (GC–MS) after extraction via Fatty Acid methyl Ester (FAME)

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technique [16, 17 and refs. therein] or studied adopting the cross-over immune electrophoresis (CIEF) method [18].

Up till now FTIR micro-spectroscopy was never adopted for non-invasive identification of residues on archaeological lithic tools although this technique does not require chemical or mechanical pre-treatment of the sample investigated and can distinguish between organic and inorganic samples.

In order to test the advantages and the limits of this technique and to combine two independent methods we carried out a systematic use-wear analysis of lithic assemblages from well preserved prehistoric sites. All items showing micro-traces were therefore spectroscopically analyzed to ascertain the presence of residues. These were identified by comparison with a reference collection of replicas that worked a wide selection of animal, plants and mineral materials.

The procedure here proposed can introduce a new perspective in the functional study of lithic industries. Since stone tools were used for a variety of activities including butchering, hide, bone and wood-working, harvesting, etc., we are providing a database where inferences drawn by the mentioned approaches are reported and critically compared. In this paper results related to the activities involving contact with animal materials are presented.

2. Experimental

2.1. Samples

699 archaeological flint tools and 53 obsidian tools were selected for use-wear analysis. They represent the knapped lithic assemblage of two Neolithic sites from Southern Italy dating from VII to VI millennium BP.

628 flint tools come from the three layers of the Middle Neolithic deposit of Masseria Candelaro (Foggia), one of the few entrenched villages characterizing the Neolithic period of the wide plain bordering eastwards the Adriatic Sea called “Tavoliere” [19].

267 lithic tools had use-wear; out of them 72 showed traces attributable to animal materials processing [20].

71 flint tools and 53 obsidian tools come from the Neolithic settlement of Sant’Anna di Oria (Brindisi) consisting of two huts, the more recent built over the remains of the previous one [21].

According to use-wear analysis, 8 flint tools and 13 obsidian tools were used to process animal tissues [22].

The reference collection for FTIR analysis consists of 64 flint and 22 obsidian tools (Table 1) that were used to reproduce prehistoric activities as hunting, butchering, hide processing and production of hard animal material implements and ornaments.

2.2. Cleaning procedures

Following the most diffused protocol carried out by use-wear analysts, the archaeological tools were washed with water to remove the soil deposit from the surface. A further washing with de-ionised water in ultrasonic tank for 5–10′ concluded the procedure.

Before FTIR analysis, the second step of the washing procedure was repeated in order to eliminate all the residues not firmly entrapped in the micro-cavities of the surface.

Replicas expressly made for infrared observation were not washed at all. Conversely, experimental samples to be submitted to use-wear study only, were washed in threes steps: water and soap, chemical washing with a diluted acid followed by diluted base and finally with de-mineralized water in ultrasonic tank, in order to maximize the removal of residues while preserving the traces.

Table 1
List of the lithic replicas and material worked.

Replicas	Raw material	Material worked	Replicas with detectable residues
2	Obsidian	Antler	2
3	Flint	Antler	1
2	Obsidian	Bone	0
7	Flint	Bone	6
6	Obsidian	Fleshy tissues	5
1	Flint	Fleshy tissues	1
1	Obsidian	Fleshy tissues + bone	0
7	Flint	Fleshy tissues + bone	4
11	Flint arrowhead	Fleshy tissues + bone	6
1	Flint	Fleshy tissues + bone boiled + marrow	1
9	Obsidian	Hide	3
20	Flint	Hide	18
6	Flint	Hide + brain	4
1	Flint	Hide + salt + brain	1
1	Flint	Hide + fleshy tissues	1
4	Flint	Tendons	4
2	Obsidian	Shell	0
1	Flint	Shell	1
1	Flint	Teeth	1

2.3. Optical analysis of archaeological use-wear

Use-wear analysis was performed with both low and high-power approach using respectively a stereomicroscope SMZ (Nikon) with objective 0.5×, oculars 10× and magnification range 0.75×–7.5× and a metallographic microscope Eclypse (Nikon) with oculars 10× and objectives 10× and 20×. Both microscopes were used in reflected light.

2.4. FTIR spectroscopy

Reflectance spectra were obtained using the last generation infrared microscope Hyperion (Bruker) in the frequency range 4000–600 cm^{-1} at a resolution of 2 cm^{-1} or better cumulating at least 200 scans to achieve an optimal signal-to-noise ratio. Spots of $100 \times 100\ \mu$ were normally selected. All archaeological and experimental items were analyzed both on regions not showing use-wear, in order to have a suitable reference (‘blank’ spectra) and on many points of the used edges in order to individuate the micro-residues and check the reproducibility of their spectral patterns. The samples housing was kept under continuous flow of dry nitrogen to eliminate atmospheric water and carbon dioxide.

3. Results and discussion

FTIR analysis singled out detectable residues on 49 flint replicas out of 64 (77%) and 10 obsidian replicas out of 22 (45%) (Table 1). Among the artefacts showing use-wear attributed to animal material contact, the presence of residues was spectroscopically ascertained on 52 archaeological flint implements out of 79 (66%) and on 3 archaeological obsidian implements out of 13 (23%). This are collected in Table 2 where use-wear analysis suggestions are compared to the proposed nature of micro-residues spectroscopically individuated. In the same table, 9 flint tools from Masseria Candelaro showing use-wear interpreted as stone, minerals, abrasive and not defined medium hard material are also reported since FTIR analysis detected residues of animal tissues in contrast with use-wear suggestions.

The higher percentage of residues observed on both prehistoric and experimental flint tools with respect to obsidian ones seems compatible with the greater roughness of the former material. The stones, in fact, have identical chemical composition (mainly silicon oxide) but flint is microcrystalline while obsidian is a volcanic glass