
Investigating "black stains" on Paleolithic artefacts: looking for hafting adhesives with OLM, SEM-EDX, Raman and FTIR

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Abstract

Although the debate regarding whether the organic material on ancient lithic artefacts may survive and be microscopically identified is still on-going, the study of residues is in fact already one of the key components of functional analysis. Indeed, the study of different kind of organic residues (e.g. bone, wood, plants, blood cells, starch granules etc.) on the archaeological lithic artefacts is currently being carried out by many authors.

Here we focus on a particular type of residue, which appears in the form of black stains on some lithic artefacts from different archaeological sites. These have been most commonly interpreted by researchers as being residues of adhesives (bitumen, tar or bark birch pitch, pine or other plants resin) used for hafting purposes.

To get insights into the nature of these black stains, we offer here our first systematic results obtained through a multi-technique approach. This starts with the application of optical and scanning electron microscopy to determine morphological features. A preliminary chemical characterisation is obtained then by means of energy dispersive spectrometry (EDX). Finally, molecular composition and structure of the sample is provided by means of vibrational spectroscopies such as FTIR and Raman.

This combination of non-invasive and non-destructive techniques has been used to analyse a reference collection including different substances which, alone or mixed with other products, can be used as hafting adhesives: tars and pitches, bitumen, beeswax, conifer resins, *Pistacia* resin, etc. Such a combination provides a detailed morphological and chemical characterisation of these substances, which is strong enough to identify preserved archaeological residues.

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As archaeological case studies, we present here some examples of different Palaeolithic sites showing such black stains apparently related to tool hafting: Azokh Cave, in Nagorno Karabakh, Gilvaran and Kaldar, in Iran, and Cova Eirós, in Spain.

Although the reference collection is still in construction, the combination of techniques demonstrated the feasibility of differentiating and identifying some of the analysed residues as adhesive bituminous substances. On the other hand, the methodology used enabled to rule out some of the stains as hafting residues.

Keywords: organic residues, adhesives, Palaeolithic tool hafting, multi, technique analysis