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GAS CHROMATOGRAPHY AND PREHISTORIC TOOL USE RESIDUES: A PRELIMINARY STUDY

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When people occupy a locality for any length of time, they leave in the soil and on their discarded implements traces of the organic materials they utilised at that site. These materials can include substances such as bone, skin, feathers, vegetable fibres and wood. In situations where the effects of decay are minimised, such as dry cave or wetland sites, organic material may remain structually intact for hundreds or even thousands of years (cf Carbone and Keel 1985). Under these conditions organic remains can be visually detected and identified using relatively straightforward microscopic techniques (Clement and others 1981; Fredricksen 1985; Heuse and Adolf 1982; Shafer and Holloway 1979).

More often than not however, enviromental conditions at archaeological sites are not conducive to the complete preservation of organic materials. In most archaeological sites organic remains break down to their chemical constituents within a relatively short time. This of course precludes the identification of these materials using microscopic techniques.

In an attempt to overcome this limitation, archaeologists have begun to explore chemical alternatives for detecting and identifying organic materials. A number of chemical and related techniques have been developed in various scientific fields for identifying organic remains (Fredericksen 1987:57ff). One of the most promising of these techniques, in terms of its application to archaeological analysis, is chromatography.

Chromatography and fatty acid identification

Chromatography is basically a chemical technique for separating fats into component fatty acids, which are present in all organic substances. All plant and animal species have distinct ratios of fatty acids, which provides every species with a unique chemical signature. Given this fact, chromatography offers archaeologists the possibility of identifying prehistoric organic residues to specific plant and animal species.

One major problem with the chromatographic analysis of prehistoric organic residues is the absence of a reference collection. The identification of prehistoric residues is usually attempted by comparing the fatty acid ratios for archaeological specimens with a reference collection of known ratios for moderns species (see Hilditch and Williams 1964 for an example of one such reference collection). However, very little is known of the effects of prolonged burial on organic substances and the possible changes this induces in the fatty acid composition of these materials (cf Morgan and others 1973; Morgan and others 1984:46; Thornton and others 1970). Ideally therefore, archaeologists attempting this form of study should possess a reference collection of fatty acid ratios from known plant and animal species recovered from an archaeological context. For obvious reasons this is not practicable in most cases. Archaeologists have therefore resorted to making tentative or broad identifications of organic residues.

Despite this limitation, the potential for the identification of archaeological residues through chromatography has been demonstrated by a number of studies (Campbell and Munro 1983; Hill and Evans 1987; Hill and others 1985; Patrick and others 1985). This research provided the impetus for the present preliminary study, which was designed to test the feasibility of using chromatography to detect and identify organic residues on stone tools from New Zealand open-air archaeological sites.

Stone tools from N77/588-6

The stone tools chosen for analysis were excavated in 1982 from Site N77/588-6, Kawerau (Lawlor 1984). This site constituted a habitation, food storage and stone tool activity area, and was probably occupied during the sixteenth century (Fredericksen 1987:23ff).

The stone assemblage from N77/588-6 included 275 items of flaked obsidian. All the obsidian artefacts were recovered from within Layer III, which consisted of a "charcoal rich sandy loam" attaining a maximum depth of 200 mm. Thirty two of these artefacts possessed identifiable use-wear. Variation in this wear demonstrated different modes of tool use had been undertaken at the site (Fredericksen 1987:66ff). However, it was not known which types of materials had been worked using these tools. A study of organic residues adhering to the edges of obsidian tools was thought to be one possible method for identifying these materials.

The obsidian artefacts recovered from N77/588-6 were amenable to organic residue analysis. These items had been collected with such an analysis in mind and had been bagged separately in clean plastic bags. None of the artefacts had been cleaned in any way. Contamination was, however, still a possibility as the artefacts had been handled extensively during excavation and preliminary analysis.

Residue on Tool 817

The first step in the study involved detecting likely looking organic residues on the obsidian tools. All 32 tools were examined under a binocular microscope at magnifications of x 20 to x 120. A Large number of the tools possessed substances which resembled a variety of organic residues.

A number of these possible residues were examined under a scanning electron microscope in an attempt to verify if they were related to tool using activities. Although this revealed a wide variety of strucutures within the residues, the absence of an adequate reference collection precluded their correlation with prehistoric tool use.

The next step in the analysis involved subjecting a number of these residues to chromatographic analysis. The residues obtained from two obsidian tools were chosen for study. This small sample size was due to the financial constraints imposed by using a commercial laboratory for the analysis. The residues chosen were considered to be of sufficient quantity for gas chromatography. In the event however, only the residue obtained from the working edge of Tool 817 was found to be of sufficient quantity to facilitate analysis.

The residue from Tool 817 was extracted and broken down to be analysed as fatty acid methylesters in a gas chromatograph. Details of the method of extraction and analysis may be found in Fredericksen (1987: Appendix B). The results of the analysis are presented in Figure 1.

The chromatograph was unfortunately not well attentuated so smaller peaks, which differentiate animal-based from vegetable-based fats, could not be identified (Fig. 1). This problem might be resolved by analysing more and/or larger samples of residue. In terms of the present study however, this was not possible and the fats detected on Tool 817 remain unidentified.

Problems encountered

The two main problems encountered in this preliminary study were finding an uncontaminated sample for analysis and finding a laboratory able to undertake the analysis.

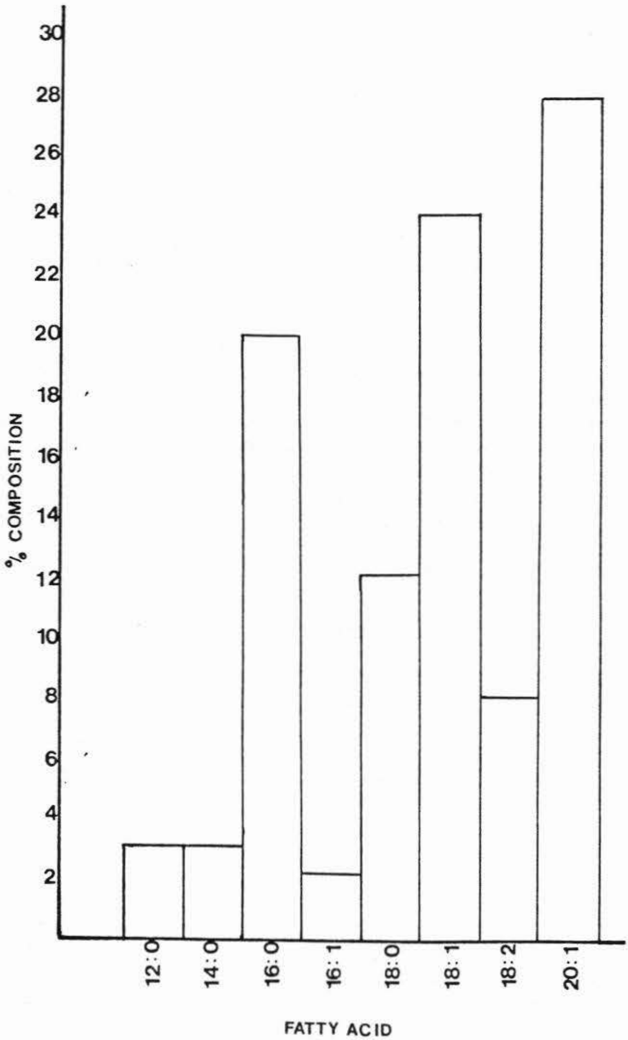

FIGURE 1. Composition of organic residue on Tool 817.

Virtually all archaeological stone tool assemblages are chemically contaminated and this fact, if ignored, can mislead researchers engaged in organic residue analysis. Contamination can occur in all stages of pre- excavation, excavation and post-excavation.

Pre-excavation contamination of residues occurs when microorganisms alter the chemical makeup of those residues. The degree to which this occurs depends on the nature of the residue, the types of microorganism present, the amount of time the residue has been deposited, and local enviromental factors (humidity, soil pH, exposure to sunlight, etc). Little is known of the effect these factors have on archaeological residues and a great deal more research is needed in this area.

Excavation and post-excavation contamination can occur when archaeologists handle or bag together artefacts possessing residues. Such sources of contamination can occur when archaeologists handle or bag together artefacts possessing residues. Such sources of contamination can be minimised by proper handling and storage procedures. These procedures have been outlined elsewhere (Fredericksen 1985: 162: Lampert and Sim 1986). Some of the procedures were not followed in the excavation and analysis of stone tools from Site N77/588-6, and the possibility exists that the residue detected on Tool 817 represents an excavator's fingerprint (Fredericksen 1987: Appendix B).

The second problem encountered during this study involved finding a laboratory system which was not only sensitive enough to permit the identification of small amounts of residue, but would also not conflict with strict budgetry and time constraints within which most archaeologists are forced to work.

In Auckland at least one laboratory deals specifically with the study of fats. This facility possesses a gas chromatograph with a glass column capable of very refined sample quantification. However, the sample yielded by the extraction of residue from Tool 817 was not sufficient for the analytical system of this laboratory, which was designed for minimum samples of 100 mg. Clearly, if a research project involving the comprehensive analysis of organic residues were to be contemplated, the system would require adjustment.

In terms of finance and time, gas chromatography is not cheap. Each sample analysed can cost in excess of $60.00 (1986 prices) and may take weeks to be completed (depending on the laboratory's workload). This is a severe limitation for an archaeologist wishing to identify organic residues on a large number of tools. However, the indivdual cost of samples and the time taken to analyse them would probably decrease if large batch samples were submitted.

Summary and Conclusions

This preliminary study was aimed at testing the potential of gas chromatography for identifying prehistoric tool use residues. A number of obsidian tools from N77/588-6 were analysed and organic residue detected on one of these. Although this residue could not be identified, the study was valuable in pointing out the necessity of careful sample collection and preparation and the need to greatly refine chromatographic techniques for use in archaeological analysis.

In conclusion, gas chromatography and other chemical techniques are becoming more commonly used by archaeologists involved in research on tool use. With the contiued development of such techniques it will not be long before residue analysis becomes, like use-wear analysis, a standard procedure for ascertaining the fuctions of prehistoric stone tools.

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