

A NEW TECHNIQUE FOR IDENTIFYING PREHISTORIC FEATURES

BY MEANS OF SOIL ANALYSIS

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ABSTRACT

The use of soil chemical analysis can be an invaluable aid in identifying archaeological features. A conventional study undertaken to identify an ash feature by means of soil chemical analysis proved to be inconclusive. The application of a relatively new technique was employed in order to complete the study. This involved taking Energy Dispersive X-Ray (EDX) analysis of the elemental structure of the material. A comparison of the elemental analysis yielded important new data concerning the content of the ash, thereby allowing a positive identification of the feature. This paper will examine the theory, process, goal, and application of the EDX study; it will show how this method evolved from the old methodology.

INTRODUCTION

The identification of archaeological features can be problematic and often inconclusive. Archaeologists rely on intuition and observation to arrive at subjective decisions more often than is admitted. The author has employed soil chemical studies for several years as an aid in the identification of features in seemingly homogeneous soil strata. The uncovering of an ash feature at site CA-ORA-281, which displayed singular aspects, presented an opportunity to utilize an innovative soil chemical approach by identifying the feature in a scientific methodology.

Soil samples were collected off-site, at the site surface, and at arbitrary levels on-site in order to answer the following questions:

- 1) Determine whether the feature was cultural, natural, or some historic disturbance, and
- 2) determine what the feature was.

When humans occupy a site, they alter the existing native

soil environment. This is accomplished by depositing human excreta, plant and animal residues, and by their mere presence, people disturb the plant-soil relationship. Because humans induce these chemical changes in the soil, and since these changes are identifiable in the laboratory, the researcher can analyze soil samples from features and identify past human behavior (Cornwall 1958; Cook and Heizer 1965; Limbrey 1975). This can be accomplished by comparing on-site soil data to the native soil (off-site) data.

The concept for this soil study involved investigating the history of habitation by quantitative means (Cook and Heizer 1965:2). The study by Cook and Heizer (1965) represented a pioneer effort in interpreting aboriginal habitation by means of soil chemical analysis. A study by Halley (1980b:218-246) showed how to utilize a battery of tests in order to evaluate the mode or process in the depositional history. This followed earlier studies of his in which he attempted to identify human burials by soil chemical analysis (Halley 1978, 1979a, 1979b, 1980a). Halley (1980b:218-219) correctly pointed out the importance of looking at the percentage change of each element rather than the amount as the focal point in interpreting human habitation by quantitative means.

Dallas (1988) also attempted to identify human burials and features in a manner similar to Halley's approach. The major difference is that Dallas (1988) compared changes in the vertical profile of each subsequent level excavated. Problems with this technique have been due to a lack of focus of the research or research questions, rather than a weakness of the technique.

The Energy Dispersive X-Ray (EDX) technique used in conjunction with a Scanning Electron Microscope (SEM) can be used to analyze very small samples of any material. This is done by scanning the prepared sample with an electron beam and directly analyzing the X-ray energy. This energy increases proportionally with the atomic number of the element, and each level is unique to that specific element. The results are displayed on a color monitor. A specially adapted Polaroid camera is utilized to capture the results on film for a permanent record. After analyzing known and unknown samples, quantitative results can be compared in an effort to identify the contributing or parent material. Neither age nor physical condition of the samples can significantly affect the EDX's results.

The basic idea behind the test is that most plants, animals, soils, rocks, and minerals have relatively unique chemical structures that can be used to help identify them from other materials. The true importance of this technique is two-fold: its ability to detect trace elements in samples as small as 10-16 g, and it can be utilized equally well on both organic and inorganic materials (Russ 1970:168-171). A word of caution, however, because this test cannot distinguish between organic and

inorganic elements with similar patterns, including contaminants; this analysis should not be used as a sole source in the fingerprinting process, but as part of an overall battery of tests.

CA-ORA-281 is a large bluff-top village site overlooking a sheltered Pacific Ocean cove. It was extensively excavated in 1938 by a crew under the direction of John Winterbourne. The site was dated from extant drawings to the Late Prehistoric. In 1984 the Department of Parks and Recreation excavated two test units near the edge of the bluff where there was an erosion problem. The upper levels of Unit 2, with the exposed ash feature, also dated to the Late Prehistoric period by artifact typology and obsidian hydration band analysis. A fragment of polished bone and a few flakes of chert were the only artifacts recovered from the ash feature. C-14 measurements from Mytilus shell resulted in an uncorrected date of 4450 ± 100 years B.P. (U.C. Riverside).

METHODS

For the original soils study, a sampling method was employed that tested off-site (for control), from each strata, and from the feature. Samples were extracted from the sidewalls and placed in clean plastic zip-lock bags. The samples were analyzed with a La Motte GS-01 soil test kit. The soil texture study used a soil separation column technique, reflecting the varying rates of settling of the respective soil particles. This is a relatively simple test (excellent for field use) yielding reliable quantitative results.

A measured amount of the sample was mixed with distilled water and then filtered. The filtrate was then divided to perform each test. The results are depicted in Table 1.

The components tested for in the soils study were Nitrogen (N), pH, Calcium (Ca), Phosphorus (P), Potassium (K), and Humus. The rationale for this battery of tests involved having one or more tests address a specific research question. The interested reader is encouraged to examine some of the primary sources cited earlier.

Basically, calcium and potassium were tested for to confirm the presence or absence of wood ashes (Halley 1980b:223-226). Phosphorus was used to document the presence of human fecal matter and urine, plus further substantiate the presence of shell (being a contributing factor for calcium) by means of the CA-P ratio (Halley 1980b:225; Mathews 1973:188). Nitrogen, in the form of nitrates, was tested to determine the presence or absence of fecal, plant, or animal flesh residues. pH was tested to understand the formation of ions in stable or unstable states (Halley 1980b:228-231). Humus further documents the presence of organic remains.

The second phase of this study was the Energy Dispersive X-Ray (EDX) analysis which entailed an even more stringent sampling technique. The preparation procedures were relatively simple. Samples should be collected with as little contamination as possible. This can be accomplished by using clean tools, eliminating human contact, rinsing material with distilled or deionized water, air drying, then incinerating the samples if necessary, and storing them in sterile plastic containers. The rationale for using plastic containers is that they are manufactured from petroleum by-products that consist of organic carbon. Inorganic carbon chains are easily detected by the EDX, and show up clearly on the monitor because most organic hydrocarbons predominantly form carbon rings that are difficult for the EDX and other analytical equipment to detect. The equipment can be programmed to ignore the inorganic carbon, if this is a contaminant from the incineration process.

Table 1. Soil Analysis.

Sample	Depth	pH	Calcium	Nitrates	Potassium	Phosphorus
Ora-281 Unit 2	surface	6.8	6 ppm	1280 ppm	< 300 ppm	< 1.5 ppm
	23-30 cm (B Horizon)	7.33	1 ppm	20 ppm	< 300 ppm	< 1.0 ppm
	30-35 cm	7.1	12 ppm	60 ppm	< 300 ppm	1.5 ppm
	40-50 cm	---	--	30 ppm	< 300 ppm	negligible
	50-55 cm	7.3	--	20 ppm	< 300 ppm	negligible
	60-70 cm (sand)	7.2	2 ppm	< 20 ppm	< 300 ppm	< 1.0 ppm
Off Site	surface	7.0	1 ppm	100 ppm	< 300 ppm	1.0 ppm

Ideally, incineration should be done in a spectrographically pure graphite crucible with a cover of the same material. Incineration can be done in any kiln that has the ability to reach temperatures of approximately 800 degrees C. To prevent reduction of the graphite crucible, the kiln must be purged of oxygen (O₂) by having a constant flow of Nitrogen (N₂) or most any other inert gas passing through the kiln. Incineration should be performed in the recommended manner in order to avoid the introduction of any foreign elements in the sample. Since this is a rather sensitive test, the more care taken in the preparation, the better the results.

A small piece of the sample is then mounted individually on a spectrographically pure SEM specimen stub, using any two-sided adhesive tape. However, prepared samples are not sputter coated with gold-palladium, as is done with other SEM samples, because it would show up in the EDX analysis. Again, care must be taken not to touch either the sample or the specimen stub, as this would introduce contaminants--possibly affecting the results.

The sample is now ready to be analyzed by the EDX, and is placed into the specimen chamber of the SEM, vacuum drawn, equipment activated, and the analysis begun. The results are then displayed on a color monitor in a histogram-type format by emission levels and relative stages. A Polaroid photograph is then taken for a permanent record, which can be compared to others in order to determine varieties and relative amounts of elements in each sample.

DISCUSSION

In order to explain the need for this newly applied technique (EDX), an interpretation of the results of the soil chemical study is necessary.

Results of the physical tests show how the soil ranged from clay-sandy soil at the surface, to sandy-clay soil at the feature, and back to the clay-sandy soils at the lower levels (Table 2). This indicates only one major episodic change in the particle size, indicating that the natural forces have been consistent for at least several hundred years. The only change is at the feature level and is, therefore, localized in nature. On and off-site samples showed a variation of no more than 8%.

The rationale for the physical tests are that human occupation alters the native soil profile by the compaction of the soil particles, removal of the soil, or additions to the matrix. Loss of the A-horizon could occur. A recovery would occur if the site was only temporarily occupied. Of course, if erosion occurs, horizons would disappear. During the recovery stage, plants will grow and die, leading to the production of humus (from the decomposition of plants) which will form dark layers (Halley 1980b:232). If we test for humus in these buried horizons, we will be able to understand whether the profile was disturbed by human or natural forces.

In general, while the potassium and phosphorus levels were consistent on and off-site and throughout the vertical profile, the calcium, nitrogen, and humus results varied dramatically, as can be seen in Table 1.

The results showed that the nitrogen levels varied greatly with depth. Since nitrogen tends to pool at the surface, surface

Table 2. Fraction of Soil Particles in Matrix.

Sample	Depth	Sand	Silt	Clay	Humus
Ora-281 Unit 2	surface	75%	6%	19%	5%
	23-30 cm (B Horizon)	42%	16%	42%	3%
	30-35 cm (Feature)	41%	9%	50%	1%
	40-50 cm	67%	13%	20%	--
	50-55 cm	75%	6%	19%	1%
	53-55 cm	80%	13%	7%	--
	60-70 cm (sand)	80%	7%	13%	--
Off Site	surface	67%	11%	22%	5%

quantities usually are appreciably higher than subsurface quantities. The feature level (30-35 cm showed a dramatic 300% increase from the previous level (which is significant), as any change of 300% or more is noteworthy (Halley 1980b:218-219). This indicates that either fecal matter or plant remains were a contributing factor at the feature level (cf., Cornwall 1958; Halley 1980b).

However, as nitrogen in the soluble nitrate form tends to deplete by leaching, an even higher amount could have been present, but leached through to the lower levels. Looking at the physical results indicate that the fairly high proportion of clay at the feature level prevented any further leaching in the vertical profile.

The results of the phosphorus tests were informative. The low amounts of phosphorus indicate that no significant amounts of urine, bone, and fecal matter were present; they were not deposited in this area of the site. No significant increase occurred on-site as compared to off-site values. This indicates that urine and feces contributed very little to the nitrogen and phosphorus levels at this area of the site. This infers that plant remains were the contributing factor for nitrogen at the feature level.

Results of the calcium test showed variability, and were problematic. Some factor is affecting the amount of calcium present at the site. The amounts vary from on to off-site. Significant quantities of shell are present at the surface, suggesting that increases in calcium could be a result of shell decomposing. As the surface pH is slightly acidic, it could be causing the decomposition of shell. The off-site value of calcium is low and, as the calcium values increase dramatically at the surface (600% change) and at the feature level (1,200% increase) from off-site, it suggests that something at this level is contributing to the increase in the amount of calcium. The ratio of Ca:P varies greatly but at times is 1:1; this would infer that shell calcium might be a contributing factor. This is not conclusive because the ratio of calcium to phosphorus in shell is 10:1 (Halley 1980b:223-225). However, this is only true when the pH is above 7.0, as it is at CA-ORA-281. The ratio of calcium to phosphorus in bone is 2:15 (Mathews et al. 1973:188; Halley 1980b:224) which does not correspond at this site. This suggests that shell rather than bone might be contributing to the calcium levels at this site, but it is not conclusive. Even though these results were inconclusive, they are consonant with the fact that very little bone was recovered from this site; also, since the pH is relatively neutral to alkaline, the bone would not have decomposed (Dallas 1988; Hole and Heizer 1973; Halley 1980b).

While amounts of humus varied, the most significant result is at the feature level (30-35 cm) where only 1% humus is shown. This suggests that either very little organic matter was present at that level or that very little oxygen was available to decompose the organics. As the physical tests showed a dominant amount of clay at this level (50%), the latter thought is more likely the case.

Potassium levels showed the most consistency. Since the levels of potassium did not change at the feature level, this suggests that wood ashes were not a contributing factor at the feature level.

Examination of the feature sediment revealed that the sediment was composed of ash, charcoal, and sand particles. Since the soil results indicated that wood ashes were not a contributing factor as hypothesized, then what kind of ash was the feature composed of? This called for a new step (test) in the methodology.

The second phase of the study was the EDX analysis. A partially completed study by the author had indicated that seaweed was harvested at coastal sites (Dallas 1988; Lyneis 1981). Six samples of surf-grass (Phylospadix torreyi), kelp (Macrocystis integrifolia), and the feature ash were submitted to the Stanford Research Institute (SRI) for the EDX analysis. The results are intriguing and suggest that the ash of the feature

and the surf-grass are nearly identical. It was initially thought that if possible seawater contamination (Sodium-NA, Magnesium-Mg, Sulphur-S, and Chlorine-Cl) and organic Silica (Si) were eliminated from the kelp sample, it would be similar to the feature ash. However, the obvious difference in the Ca quantities from the samples cannot be accounted for with this explanation. So, very clearly, the feature ash is not kelp.

One question arose during the EDX analysis: If the samples were contaminated with seawater, what happened to the Iodine (I) that is only found in seawater? Even after all the samples were run, Iodine was still a missing factor; possibly it is not stable outside of a soluble state. The difference in the Silica (Si) content between the samples was initially thought to be a condition from marine organisms. However, after consultation with a floral expert, Dr. W. Savage of San Jose State University, it was discovered that surf-grass contains no Silica in its internal structure, while kelp does. It was also the opinion of the EDX operator that these were not from the same parent material.

Samples of the surf-grass and kelp were submitted in two forms, air-dried and incinerated. Because of the consistent patterns between surf-grass and kelp, it is now thought that the modern kelps normally contain Na, Si, S, Cl, K, and Ca, and that these elements are not contamination. However, Mg, Al, and Ti are possible seawater contaminants or trace elements. The surf-grass normally contains only Ca, with minute amounts of Si, K, and Mg, that are possible trace elements or contaminants.

Because most organic materials, like other things, are not homogeneous, it is expected that any two samples, even if from the same source, will not have the exact same EDX elemental content; therefore, the Si, K, and may not be contaminants.

CONCLUSIONS

This new technique shows great promise in identifying all particles of plant material or ash whose identity in an archaeological feature is a mystery. The ability of this technique to identify an ash sample of even minute quantities can be an invaluable aid to the researcher. Today there is no reason to assume that all ash features that appear to be hearths, are actually hearths. Now we have a technique that allows us to analyze the remains, and determine what human behavior could be attributed to a particular feature.

This technique is not limited to botanical samples. It can be used to differentiate metal, ceramics, shell, bone, ash, paint, and hair; the potential is virtually limitless (Russ 1970:168-171).

The findings from this study indicate that either the feature was a shellfish cooking feature, or a surf-grass food processing area. In order to obtain positive identification, a larger sample of the feature would need to be excavated.

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