Pottery Function and Organic Residue: An Appraisal

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Use-wear analysis is commonly employed in the study of stone tools. Researchers are often able to determine, based on microchips and polish, the material that was cut or scraped by the tool and, in many cases, some activities of stone tool use. Pottery use-wear, or what we call "use-alteration," has not received the same amount of attention. But this is not for lack of need; a clear understanding of pottery function is at the core of many archaeological inferences. Inferences about prehistoric diet, demography, social complexity, exchange, and technological change, all would benefit from a technique to determine how pottery was **actually** used.

Pottery use-alteration traces occur in three general forms: attrition, carbon deposits, and the topic of this paper, organic residues. In this paper we will (1) briefly introduce the ethnoarchaeological and experimental studies of pottery residue performed by the authors, (2) demonstrate how organic residue absorbed into the ceramic body can be linked to vessel contents, and (3) discuss some current research and the direction for further study.

This discussion of organic residue analysis of pottery is from an archaeological perspective. The authors have collaborated with organic chemists and approached the study of pottery residue ethnoarchaeologically, experimentally, and with the analysis of prehistoric Woodland and Paleoeskimo collections [Deal and Skibo 1993].

Kalinga Ethnoarchaeological Project

The senior author's interest in residue analysis stems from his research among the Kalinga, the household potters living in the rugged mountains on the Philippine island of Luzon. The staple crop, rice, is grown on irrigated terraces surrounding their villages. Their diet also consists of a variety of vegetables, grown in swidden plots, and also the occasional pig, chicken, duck, dog, or water buffalo. The Kalinga have been the focus of a long-term ethnoarchaeological study that has researched, for example, pottery use-life [Neupert and Longacre 1994], pottery production and distribution [Stark 1994], formation processes [Tani 1994], and the subject of this paper, pottery use-alteration [Skibo 1992; see Longacre and Skibo 1994 for a general discussion of the Kalinga research].

The use-alteration study [see Skibo 1992 for a complete description] was done in the village of Guina-ang, which consists of slightly over 100 houses on a ridge overlooking the Pasil River Valley. Kalinga cooking is done over an open fire in two types of pots: a rice cooking pot, which is taller and has a more constricted neck, and a vegetable meat pot, which is squatter and has a more open aperture. About 200 used vessels were collected and now serve as the

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study collection.

The objective of the use-alteration study was first to link use traces on the vessels to the activity observed in the field, and then to understand the process involved in trace formation. This type of information provides the prehistorian with the ability to infer vessel use from alteration traces.

Three forms of use-alteration were analysed: carbon deposits, attrition, and organic residue. Carbon is deposited on the vessel exteriors as soot, and on the interior as burnt food. The pattern of these deposits is determined by the intensity of the fire, type of food cooked, and the number of use events [Kobayashi 1994; Skibo 1992:147-173]. Attritional traces are the nicks, chips and abrasions formed as the pot is used. Various activities form different attritional patterns. The rice and vegetable meat cooking pots could be discriminated based on both carbon deposits and attrition. Skibo [1992] formulated a general model of attrition and carbon formation so that the prehistorian can apply these findings directly to their ceramic assemblage. The use-alteration study also made considerable progress toward understanding the linkage between vessel contents and absorbed residue.

North American Woodland Pottery

In 1985, Michael Deal began an exploratory study of absorbed and adhering organic residues from prehistoric Eastern Canadian pottery. Both ethnographic and ethnohistoric information indicated that boiling was the most common food preparation technique used by native groups, and it is also assumed to be a characteristic of the late prehistoric period. The study began with experiments to determine the feasibility of identifying prehistoric organic residues. Initial experiments determined that fats from boiled meats are absorbed into porous ceramic fabrics and that fatty acids could be identified in organic residues derived from prehistoric sherds [Deal and Silk 1988]. Subsequent analyses also included residues found adhering to the interior and exterior walls of prehistoric ceramic vessels and Paleoeskimo soapstone bowls [Deal 1990]. These studies focused on methods of identifying the original compounds from the relative concentrations and ratios of specific fatty acids. For comparative purposes, some samples were divided in half and subjected to both gas chromatography / mass spectroscopy (GC/MS) and stable isotope (SI) analysis [Deal *et al.* 1991] or were sent to two different laboratories for GC/MS analysis.

Organic Residue Analysis

Based upon the Kalinga and Woodland Period research, along with a number of other studies, we now know that organic residue recovered from an ancient pot can provide direct evidence of the substance that was once stored or cooked in the vessel. Food residue in pottery refers to the amorphous matter, predominately lipids, proteins, carbohydrates, and other biopolymers that either adhere to the vessel surface or have been absorbed into the vessel body [see Heron and Evershed 1993 for a general discussion]. Various biomolecular techniques are used to identify in the residue what Evershed [1993:78] refers to as "biomarkers." Biomarkers can be individual compounds or combinations of compounds used to link the prehistoric organic residue to a plant or animal.

A whole suite of techniques have been

used in the analysis of ceramic organic residue that either adheres to the surface or has been absorbed into the vessel wall [Heron and Evershed 1993:260-267; Skibo 1992:81-85]. These include elemental analysis [Cackette *et al.* 1987; Fie *et al.* 1990], stable isotope analysis [Deal *et al.* 1991; Morton *et al.* 1991], infrared spectroscopy [Hill and Evens 1989], and pyrolysis [Oudemans and Boon 1991]. Each of these techniques focuses on different components of the food residue and have had varying degrees of success. Most positive results thus far, however, have been accomplished through the analysis of lipids with gas chromatography combined with mass spectroscopy [Deal *et al.* 1991; Evershed 1993; Heron and Evershed 1993; Rottlander 1990]. Gas chromatography is a technique used to separate individual compounds as they elute off a capillary tube. The mass spectrometer records the mass spectra and thus permits the investigator to identify individual compounds. This is a highly sensitive technique that requires very little sample. The remainder of the discussion will focus predominantly on prehistoric lipids analysed by this technique.

Lipids are composed of chains of carbon, hydrogen, and oxygen that bond together in a number of arrangements. There are three main groups of lipids: fats and oils, waxes, and phospholipids. Because different types and different combinations of lipids occur in all plants and animals, they are ideally suited for residue analysis. Moreover, lipids can survive long periods in the depositional environment.

In principle, identifying lipid biomarkers in an ancient residue and linking it to a plant or animal seems like a straight forward exercise, but in practice there are many obstacles. These obstacles include the preservation of the organic compounds, post-use contamination, problems with studying multi-use vessels, and identifying the biomarkers that can be readily attributed to individual plants or animals. Each of these obstacles will be addressed below.

Preservation

Any time that archaeologists work with ancient organic matter, the issue of preservation is paramount. In terms of lipids there are two issues related to their survival. First, lipids within the vessel wall, for cooking pots, must survive cooking temperatures, and second after disposal the deposited lipids are subject to decomposition in the depositional environment.

Many researchers have demonstrated that ancient cooking pot lipids can be identified [Hill and Evans 1989; Marchbanks 1989; Patrick *et al.* 1985; Rottlander 1990], but the question of how lipids are destroyed and /or altered by normal cooking fires had not been addressed. But recent ethnoarchaeological and experimental findings [Deal and Silk 1988; Skibo 1992:81-102] demonstrate that lipids will withstand, relatively unchanged, the heat of a standard open fire. Skibo [1992:88-96] demonstrated that fatty acids were preserved in all 11 of the used Kalinga cooking pots. The vessels were used in numerous cooking episodes directly over a rapidly burning fire. Thermocouple temperatures taken at the base of a cooking pot recorded temperatures between 300° and 600°C.

Similarly, Deal and Silk [1988]

simulated the preservation of absorbed lipids in cooking vessels by experimentally impregnating ceramic tiles with fats from a white-tailed deer, beaver, and yellow perch. Forty-nine standardized ceramic tiles were made using different combinations of local clays and quartz temper, and then fired at varying temperatures (i.e., 500°-1000°C) to simulate the fabrics of prehistoric pot sherds from southwestern New Brunswick, Canada. The three tiles most closely characterising the prehistoric sherds, in terms of temper content and water absorption properties, were each immersed in a flask of water (500 ml), along with a small sample of raw mammal or fish meat, and boiled for 30 minutes on a Lindberg Heating Unit. Significant amounts of fatty acids were identified in the extracts derived from the dried crushed tiles, indicating that porous ceramic fabrics will absorb and retain lipids from animal meats prepared by normal aboriginal cooking techniques. By contrast, two unaltered control tiles revealed only minute traces of fatty acids and a high background profile of phthalates, originating from plastic sheeting used to protect the sample during drying and transportation.

Organic residues do not char within the wall of cooking pots used to boil foods because the interior ceramic body never reaches a high enough temperature. Although a normal cooking fire can easily attain a temperature of 600°C, which is well beyond the ignition temperature of organic matter, the interior body of a cooking pot never approaches that temperature because of the presence of moisture. In most low-fired earthenware, water placed in a vessel for boiling is quickly absorbed into the ceramic body. Even in cases where a seemingly impermeable interior treatment is applied, water will soon pass into the vessel wall. For example, the Kalinga water pots are coated with a pine resin while the vessel is still hot immediately after firing. This interior coating will slow but not stop water permeation [Skibo 1994]. Because of the transpiration of water during the boiling process, the interior of the vessel will not, we suspect, greatly exceed 100°C and will certainly be maintained below 300°C. The latter temperature can be inferred from firing experiments [Johnson, et al. 1988; Skibo et al. 1989] in which organic matter added as temper ignites and carbonizes beginning at about 300°C. Substantiating this claim is that there is no evidence for the carbonization of the lipids within the body of the Kalinga cooking pots. In cooking that involves water, as in boiling, the organic compounds should be affected little by the heat.

Organic compounds that become deposited within the vessel wall, however, are not immune to processes of decay. Lipids, once entombed in sherds, certainly can survive long periods in the depositional environment [Nakano 1989a], and lipids are more resistant to deterioration that other compounds [Evershed 1993:77], but they are, nonetheless, subject to a number of degradation processes. The preservation of lipids, however, depends largely on the chemical conditions of the soil (e.g., pH) and the degree to which the organic compounds have become entrapped within the clay matrix [Heron and Evershed 1993:253]. Decay of lipids can occur as a result of three processes: hydrolysis, oxidation, and microbiological degradation [see Evershed et al. 1992 for a more complete discussion].

Unsaturated fats are particularly susceptible to oxidation, a process that can transform fats into a variety of compounds by a direct attack upon double bonds. The under representation of many polyunsaturated fats, although common in many foods, is likely the result of oxidation. Under anaerobic conditions fatty acids can be altered by hydrolysis, which can cause considerable shifts in the proportions of fatty acids. For example, the Kalinga study also looked at a sample of sherds excavated from a village midden. The analysis of the residue demonstrated an over abundance of palmitic acid, which suggests that the residues had undergone considerable hydrolysis or microbiological decay, a process common in adipocere formation [Evershed 1992; Evershed *et al.* 1992; Morgan *et al.* 1992]. Adipocere is a waxy material composed mainly of fatty carboxylic acids and is the principal matter of the bog bodies found in northern Europe [Evershed 1990, 1992]. Morgan *et al.* [1983] suggest that the degree of decomposition, in this case hydrolysis, can be evaluated by looking at the ratio of palmitic to oleic [for a discussion of this and other ratios see Deal *et al.* 1991:199-202].

Microbiological degradation is the final general process that affects the survival of food residues. Unless protected, lipids will be subject to attack by the same microorganisms that break down all buried organic materials. That lipids survive at all demonstrates that the ceramic matrix often provides an environment that protects the material from chemical or microbial decay. Clearly, the survival of lipids depends upon micro environmental conditions and must be evaluated on a case-by-case basis.

Contamination

Because all living things contain lipids, it is quite possible that new organic residue could be deposited between the time the vessel was last used and then finally investigated by the archaeologist. Because adhering residue would be particularly susceptible to contamination from lipids in the soil or even from the archaeologist's hands [Evershed 1993:87-90; Heron *et al.* 1991], most researchers have focused on the residue that has become entrapped within the porous vessel wall. Marchbanks [1989] and others have been employing a technique to avoid possibly contaminated residue in which the exterior millimeter or so of ceramic is discarded and the sample removed only from the interior of the vessel wall.

The two most likely possible sources for contamination are the burial soil and post-excavation procedures [Evershed *et al.* 1992:193]. Soils contain organic matter and of course lipids. Heron *et al.* [1991] have recently considered if lipids in soil can migrate into the sherd material and contaminate the food residue. They performed GC/MS on soil adhering to sherds as well on the sherd itself. Heron *et al.* [1991] found that the soil and sherds had very distinct lipids and they argue that little or no migration occurred between the soil and ceramic. But lipid migration may be affected greatly by local conditions, so a standard procedure for lipid analysis should be the analysis of burial soil.

Handling of sherds and the laboratory procedures can also introduce contaminates. Because lipid analysis deals with extremely small amounts of material, lipids can be added by unclean glassware, human hands, or storage containers. The ideal situation for

lipid analysis is to obtain freshly excavated and unwashed sherds so that the post-excavation history of the material can be carefully monitored.

Multi-use Vessels

The identification of a vessel's contents from the residue has the highest possibility of success if the pot was used to cook or store a single item. For example, cocao residue was identified in a specialized Mayan vessel [Hall *et al.* 1990] and tartaric acid found within Egyptian amphora provided unambiguous evidence of wine storage [Badler *et al.* 1990]. Single use vessels, however, are the exception; the vast majority of prehistoric vessels were certainly used to store or cook a variety of items. The Kalinga study illustrates the contrast between single and multi-use vessels by simply looking at the ratio of oleic to palmitic acid [Skibo 1992:92-96]. Based on this ratio, the rice cooking pots form a distinct cluster whereas the vegetable/meat cooking pot ratios are highly variable. Only rice is cooked in the rice cooking pots whereas many kinds of vegetables and also pig, dog, chicken, and duck are commonly boiled in the vegetable/meat pots. Organic residue from the latter pots appears to be an accumulation of the many cooking events. Carbonized exterior encrustations, however, may be more useful for identifying individual items from multi-use pots because they represent food charring from a single cooking event [Deal *et al.* 1991:177-178]. The possibility of making a food group identification from the absorbed residue depends upon the investigators ability to isolate biomarkers.

Identifying Biomarkers

In organic residue analysis it is rare to find a case where the lipids from the prehistoric vessel can be linked directly to the food source. This would be the residue analysis equivalent of finding Pompeii. Most archaeologists never excavate a site like Pompeii and so are left with trying to infer the prehistoric behaviour from bits of pottery, broken tools, and crumbled buildings. Residue analysts must make similar types of inferences when trying to connect the lipids to their food source—rarely is there a one-to-one correlation. consequently, one must search for biomarkers, which are the combinations or ratios of various compounds that one can use to infer a food source [see Evershed 1993:76-87]. Residue analysts compare biomarkers to contemporary foods or in some cases to ancient foods [Deal *et al.* 1991]. Unfortunately, the lipid makeup of many contemporary foods, not to mention prehistoric foods, is not known. Ironically, archaeologists studying pottery residues may ultimately contribute a great deal to the basic lipid chemistry of foods. It is still common procedure to perform GC/MS on a variety of foods as well as the pottery sample because one cannot turn to the chemical literature for very fine distinctions in plant or animal lipid makeup. Four types of lipids have been used most successfully as biomarkers, sterols, diterpenoids, fatty acids, and acyclic compounds [Evershed 1993:80-87].

Sterols, though found in small amounts in lipids, have been used as biomarkers to successfully discriminate plant and animal residue [Evershed 1993; Marchbanks 1989]. Campesterol and sistosterol are found in plants and cholesterol in animals. Diterpenoids and also triterpenoid have been used to identify ancient pitch and tar [Evershed *et al.* 1985], and acyclic compounds (waxes) served as biomarkers

in the identification of leafy vegetables [Evershed *et al.* 1991]. Finally, the proportions and combinations of various fatty acids have been employed as biomarkers. Rarely have fatty acids alone been able to identify specific plant or animal species, but there has been some success in assigning the residue to general food groups: marine, freshwater, plants, and terrestrial animals [Deal *et al.* 1991; Patrick *et al.* 1985]. But certainly, fatty acid ratios can be combined with other biomarkers to make finer distinctions.

In collaboration with geologist June Morton, Deal [1990; Deal *et al.* 1991; Morton 1989] has found some success in combining fatty acid data with stable isotope ratios [see also DeNiro 1987; Hastorf and DeNiro 1985]. These studies have shown that the ratios of stable carbon and nitrogen are maintained in carbonized organic residue. Although siotope analysis requires that the researcher have a general idea of the potential food groups, Deal [1990:9] has shown that isotope ratios can be combined with lipid analysis to make finer food group distinctions.

Future Directions

We are well on our way to solve some of these obstacles and further advance the study of organic residue in pottery. To conclude, we offer suggestions for continued work on some of the obstacles, described above, as well as new directions for residue analysis.

Biomarkers

Residue analysis of pottery is a relatively new area of study and researchers are still struggling with the basic problem of linking biomarkers to foods. We feel that a clear distinction should be made between "pure"

and "impure" biomarkers. Pure biomarkers would include the individual compounds from specific foods and impure biomarkers would include mixtures of compounds, such as residues from multi-use vessels, prepared foods (e.g., stews and soup) or processed substances (gums, resins, etc.). Further, Evershed only considers identification of biomarkers based on comparisons with modern plants and animals, yet comparison with preserved prehistoric samples is also possible (e.g., from charred fauna or flora and waterlogged materials; Evans and Heron 1993; Deal *et al.* 1991).

The identification problems can also be discussed in terms of pure and impure biomarkers. We start off by identifying single compounds (pure biomarkers) that degrade at different rates and we use ratios of different compounds to look at decomposition and to point to specific impure substances (foods, resins, etc.). We sometimes get a long list of compounds from a single residue, but we can only identify a few of them (e.g., many papers list the proportion of unidentified compounds along with the fatty acids).

Impure biomarkers represent the remnants of the original stored, cooked or otherwise processed organic substance. For example, adipocere is the result of the general degradation of the original individual (pure) compounds. This point is graphically illustrated by Morgan *et al.* [1992:132]. They compare the cumulative percentage compositions of adipocere from butter fat, an archaeological sample, and fresh whale oil, in order to determine the degree of decomposition of the archaeological substance. Future study on impure biomarkers should include work on the characterization of interior and exterior

residues, the effects of contamination from other substances in soils, and establishing a library of comparative samples for individual species and mixtures.

Provenience

When exterior carbonized encrustations are present they certainly should be analysed along with the absorbed residues. Deal's [1990] work on exterior encrustations suggests that many compounds can be preserved within this charred material [see also Oudemans and Boon 1991]. But charred residues have the added problem, usually not affecting interior residue, of degradation of the organic compounds from the heat of the cooking fire. Oudemans and Boon [1991] did some preliminary experiments to assess the modification and preservation of exterior residue and found that heating temperature and time are key variables. Certainly, further such experiments are necessary because exterior charred residue may provide unique insights. For example, charred encrustations may represent a single cooking event, which would be advantageous for identifying biomarkers in stew pots or multi-use vessels. Moreover, some of the compounds that rapidity degrade when absorbed within the vessel wall may be preserved in the charred state [see Oudemans and Boon 1991].

Decomposition

The general processes of lipid degradation and modification are known, but we need many more behaviourally relevant experiments to understand the process. Realistic cooking experiments should be done in which both exterior encrustation residues as well as those absorbed within the ceramic can be examined immediately after cooking and after periods of burial in simulated depositional environments. It appears that most lipid degradation occurs within months of being deposited [Nakano 1989b], so realistic results could be expected in an experiment performed in a matter of months not years.

Beyond lipids

One reason that archaeologists have focused on lipids is that they are less susceptible than other compounds to structural modification and degradation [Evershed 1993:77]. But this has yet to be explored comprehensively. We recommend that pottery residue analysis move beyond lipids to proteins, carbohydrates, and nucleic acids [see also Heron and Everhsed 1993:272]. Because these compounds could provide more information to pinpoint food sources, one should not preclude the possibility that many more compounds could be preserved under some conditions. We need to determine if and when proteins, carbohydrates, and even DNA can survive.

Although tremendous progress has been made, organic residue analysis of prehistoric pottery is not yet at the stage of generalized application. Each researcher who attempts organic residue analysis must know that there are still many pitfalls and dead ends; this is an area of study still in its infancy.

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陶器用途與有機殘渣

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【摘要】

根據史前陶器碳含量的堆積及磨損程度，固然可以推斷其原來用途及使用情況，但除此之外，陶器上的殘留有機物亦能發揮同樣功能。近年以氣體層析(gas chromatography)及質量分光(mass spectroscopy)等科學方法成功找出古代各種脂類與某些食物的化學關係。由於每種動物及植物都有其獨特的脂類混合模式，而這些化合物在堆積層中經歷千萬年仍變化不大，故往往能為陶器的用途提供可靠資料。本文作者就其在菲律賓呂宋地區及加拿大東部研究所得資料為本，對脂類分析作出評價，並為未來研究提出意見。

然而，利用有機殘渣鑑定陶器的用途亦存在不少困難。脂類在堆積層中雖可保存一段長時間，但期間或多或少總有變化，或有腐朽的情況。此外，土壤本身及發掘過程亦可能使脂類受到相當程度的污染，而同一器皿又往往曾用作烹調及貯存不同的食物。如此種種，都為研究帶來不少障礙，使研究人員鮮能找出殘留有機物和某種食物的直接關係。

近年來研究工作屢見進展，已漸漸克服部份障礙。為使未來的研究更見成績，本文作者提出四項建議。首先，考古學者對生物標記一定要倍加認識，才能透徹理解這些化合物及化合物組合是如何將殘渣與某種食物拉上關係。由於一般化學著錄未能提供這方面的資料，因此考古學者除鑽研古陶器上的脂類外，還要研究食物中所含的脂類。研究人員應成立一個比較樣本貯藏庫，供日後研究者參考之用。第二點建議是將陶器表面燒焦的食物殘渣和滲入陶土內的殘渣多作比較和對照。據初步比較，兩種殘渣的形成過程和化合物的內容都有分別，能反映出多方面不同而重要的資料。第三項建議是必須通過實驗方式研究有機殘渣的分解情況：以陶器盛載食物進行烹煮後，隨即檢查器表及滲入陶土內殘渣的變化，模擬埋葬一段時間後又再監察其變化情況。最後，殘渣的分析不應只局限於脂類，應將蛋白質、碳水化合物，甚至DNA等列入分析範圍。

若能認真實行以上四項建議，脂類分析對陶瓷用途和古飲食的研究，應該給予很大的幫助。