Journal of Archaeological Science: Reports 17 (2018) 640–649

Contents lists available at ScienceDirect

Journal of Archaeological Science: Reports

journal homepage: www.elsevier.com/locate/jasrep

Residue analysis of smoking pipe fragments from the Feltus archaeological site, Southeastern North America

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ARTICLE INFO

Keywords:
Tobacco
Nicotine
GC/MS
Residue analysis
Feltus
Smoking pipe

ABSTRACT

The practice of pipe smoking was commonplace among indigenous cultures of the Eastern Woodlands of North America. However, many questions remain concerning what materials were smoked and when tobacco first became a part of this smoking tradition. Chemical analysis of organic residues extracted from archaeological smoking pipes is an encouraging avenue of research into answering questions regarding the development of a smoking complex within indigenous cultures of the Eastern Woodlands. In the right environmental conditions, absorbed organic compounds within artifacts can remain structurally stable for millennia, allowing analyses of organic matter to be performed on relics of advanced age. In this study, organic matter from six pipe fragments derived from the prehistoric Feltus site in Mississippi was extracted and analyzed via GC-MS, a process that allows for the identification of compounds in a complex mixture. Preliminary experiments tested the effects of pH on the efficacy of our extraction solvent to maximize the detectability of alkaloids such as nicotine. Several notable compounds were identified, including nicotine, which serves as a biomarker for tobacco.

1. Introduction

Tobacco (*Nicotiana* spp.) is widely recognized as the most sacred plant used by native North American groups, both past and present. At the time of European contact, Native Americans living in the Eastern Woodlands reportedly cultivated tobacco as widely as maize (Asch 1994; Dunavan and Jones 2011; Ford 1981; Winter 2000). However, despite ethnohistorical accounts of widespread tobacco use, archaeological evidence is seriously lacking, leaving these claims largely unsubstantiated. Tobacco seeds are infrequently recovered from archaeological sites while pipes are generally deemed to be tobacco pipes too frequently without physical evidence. Recently, chemical analyses of pipe residues have provided a new path for archaeologists to explore tobacco use from sites where tobacco seeds are absent (Eerkens et al. 2012; Rafferty 2002, 2006; Rafferty et al. 2012; Tushingham and Eerkens 2016; Tushingham et al. 2013). Here, we briefly discuss factors that may limit the identification of tobacco in archaeological contexts. We then report new data from Feltus (22Je500), a prehistoric mound site dating to the Late Woodland period (AD 400–1100) in Jefferson County, Mississippi (Fig. 1). We analyzed fragments from six ceramic pipes from Feltus using gas chromatography – mass spectrometry (GC-MS) to determine whether they contained nicotine, a biomarker for tobacco.

1.1. Site and samples

Feltus is a prehistoric Native American mound site located in the Lower Mississippi River Valley (see Fig. 1). The site sits on the edge of the aeolian bluffs overlooking the Mississippi alluvial plain and originally consisted of four mounds surrounding an open plaza (Fig. 2). The site was constructed and used during the Late Woodland period. A ring of midden defining a central plaza area indicates that much of the activity at the site took place during Baytown and early Coles Creek times (AD 400–850), before the mounds were built. The mounds were then constructed and used during middle Coles Creek times (AD 850–1100) before the eventual abandonment of the site. Three mounds stand today (A, B, and C), while the fourth (D) is no longer visible. Its location has been reconstructed based on historical documents (Wailes 1852; Steponaitis, 2012). Mounds A and B are flat-topped platform mounds that served either as foundations for structures or stages for activity. Mounds C and D were burial mounds.

Data from excavations undertaken by the Feltus Archaeological

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https://doi.org/10.1016/j.jasrep.2017.12.011
Received 20 August 2017; Received in revised form 29 November 2017; Accepted 8 December 2017

Fig. 1. Map showing the location of the Feltus site.

Project from 2006 to 2012 support the conclusion that Coles Creek people inhabited Feltus episodically for some 400 years (Fig. 3), with little evidence of permanent habitation (Steponaitis et al. 2012, 2014). More specifically, analysis of the ceramic, floral, and faunal assemblages suggest that Feltus provided a location for periodic ritual events that included feasting, the setting and use of ceremonial posts, mound building, and burial of the dead (Kassabaum 2014). Kassabaum (2014, in press b) analyzed over 40,000 sherds from these excavations. Vessel form was identifiable on only a subset of these (n = 1131), and included beakers, bowls, jars, restricted bowls, and pipes. While pipes are quite rare compared to other vessel forms, they are more common at Feltus than at other Coles Creek sites, making up about 1.5% of the identifiable vessel assemblage (n = 18). Their presence has been interpreted as evidence for the ritual use of plants (Kassabaum, in press a; Kassabaum and Nelson 2016; Nelson and Kassabaum 2014); however, the identities of the plant species smoked in the Feltus pipes have remained a mystery. Paleobotanical analysis of plant remains recovered through flotation of feature fill and midden deposits at Feltus has identified potentially important ritual plants (e.g., nightshade, morning glory, sumac, pokeweed, and maygrass), but no tobacco seeds have been recovered (Kassabaum, 2014, in press a).

1.2. Issues with tobacco identification

Several factors potentially affect the identification of tobacco in archaeological contexts. Traditionally, identification of tobacco has relied on the recovery of small (0.5–1.1 mm) carbonized seeds. Carbonization affects plants differentially, based on the temperature of the fire, length of exposure of the plant material to fire, moisture content of the plant tissue, and the plant part's surface area (Braadbaart and Wright 2007; Lentz et al. 2001; Wright 2008, 2010). During the carbonization process, bioorganic compounds are converted to more stable substances, making them more durable and protecting the remains from elemental decay and microbial activity (Lopinot 1984; Miksicek 1987; Hastorf and Popper 1988:57). While this process makes seeds more stable, it can also distort or damage them beyond recognition, and they are still susceptible to post-depositional mechanical damage as well as damage during recovery and processing (Miksicek 1987). The particularly small size of tobacco seeds amplifies these challenges.

In addition to these general issues with the identification of carbonized remains, tobacco poses added challenges because leaves and flowers are the mostly commonly used portions of the plant. Aside from being smoked, leaves and flowers were chewed, turned into a drink, snuffed as a powder, and burned as incense. In some ceremonies, tobacco was buried or cast into the air, rock crevices, water, or onto the ground (Kroeber 1941; McGuire 1897; Paper 1988). Due to these practices, it is likely that tobacco seeds never entered the archaeological record at all, while small bits of leaves and flowers would be more common. However, preservation depends largely upon the physical properties of the plant parts, such as their size and density (Dennell 1976; Popper and Hastorf, 1988), and carbonization tends to favor parts that are denser, for example nutshell and corncobs, and exclude non-dense parts, such as leaves, flowers, tubers, and fruits (Dennell, 1976; Fritz 1994; Miksicek 1987). Because of their high water and/or sugar content, exposure to fire would make the archaeological identification of these parts difficult, further hampering our ability to find evidence of tobacco.

Finally, the recovery of tobacco is further complicated by the plants primary use in rituals and ceremonies as opposed to more typically domestic contexts. Whereas the small seeds of plants that were used for subsistence were usually parched, toasted, or boiled, increasing their chances for carbonization and preservation, tobacco seeds were not. Subsistence activities are more easily recognizable at archaeological sites (hearths and pits) and are more often the focus of collection during excavations. The remains from ritual and ceremonial events are often treated differently during disposal, and may not occur in these traditional midden deposits.

1.3. Alternative methods to identify tobacco

In lieu of paleobotanical evidence of tobacco, another strategy for ascertaining which plants were smoked in pipes – including *Nicotiana* species – is through chemical analyses of organic residues from the artifacts (Tushingham and Eerkens 2016). The realization that organic matter associated with buried archaeological artifacts can persist for thousands of years has revolutionized the study of excavated cultural materials (Evershed 2008a). Most buried organic substances decompose rather quickly, generally in less than a century, with the rate of decomposition governed primarily by environmental (soil type, moisture, temperature, etc.) and biological conditions (Schmidt et al. 2011). However, if there is no influx of fresh bioorganic matter from plant decay, which is vital for microbial communities, buried organic compounds can become structurally stable for millennia (Fontaine et al. 2007; Chaopricha and Marin-Spiotta 2013). This is especially pertinent for bioorganic substances absorbed into ceramics, as organic matter does not readily migrate into ceramic fabrics (Heron et al. 1991); thus, substances can be preserved within the artifact. Such persistent organic substances are providing new sources of information on human activities in the past based on the extraction and analysis of bioorganic residues from excavated artifacts (Evershed 2008a, 2008b; Steele 2013).

There are a variety of strategies for establishing information from archaeological organic residues (Roffet-Salque et al. 2017). For