Pre-Columbian lead pollution from Native American galena processing and land use in the midcontinental United States

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ABSTRACT

The presence and sources of pre-Columbian (before 1492 CE) lead (Pb) pollution in the midcontinental United States were investigated using geochemical and Pb isotope analyses on sediment cores recovered from Avery Lake, a floodplain lake located directly adjacent to the Kincaid Mounds archaeological site on the lower Ohio River, Illinois. Geochemical results indicate the presence of Pb pollution during the Baumer (300 BCE to 300 CE) and Mississippian (1150–1450 CE) occupations, and since the 1800s. Pb isotope results link Mississippian Pb pollution to the processing and use of galena primarily from southeastern and/or central Missouri, and to a lesser the upper Mississippi River valley, with ~1.5 t (metric tons) of galena-derived Pb deposited in Avery Lake during this time. Pb pollution during the Baumer phase, equating to ~0.4 t of Pb, was not accompanied by a Pb isotope excursion and most likely originated from local biomass burning. These results provide new information about the environmental impacts associated with pre-Columbian Native Americans' interaction with and utilization of their landscape and its resources.

INTRODUCTION

Archaeological records indicate that galena (PbS, a lead-sulfide mineral and primary lead ore) was utilized by pre-Columbian Native American populations in eastern North America for nearly 10,000 yr (Walthall, 1981). While some evidence has indicated that pre-Columbian societies generated limited lead (Pb) pollution as a biproduct of copper mining (Pompeani et al., 2013) and biomass burning (Bird et al., 2019; Pompeani et al., 2019), there has been no conclusive evidence that the extraction, trade, and use of galena specifically resulted in environmental impacts. This contrasts with other regions with long histories of Pb ore utilization, like Europe and South America, where early Pb pollution signals are widely distributed and well preserved in natural archives (e.g., Bränvall et al., 2001; Cooke et al., 2008). One explanation for this difference is that smelting was not needed to extract Pb from North American Pb ores given extensive surface exposures of high-purity galena deposits throughout the Missouri, Ohio, and upper Mississippi River basins (Walthall, 1981). There is also little indication that pre-Columbian

societies developed smelting techniques prior to European contact. Instead, Pb ores like galena were either utilized in crystal form or ground into a powder. Despite this, it is unlikely that there were no environmental impacts associated with the extraction, processing, and use of galena by Native American populations prior to 1500 CE. Instead, pollution was likely expressed on a local level, which would account for the lack of widespread pre-Columbian Pb pollution in eastern North America. As a consequence, site-specific investigations are required to uncover environmental impacts associated with pre-Columbian galena use.

Here we present new data from Avery Lake, a floodplain lake located directly adjacent to the Kincaid Mounds archaeological site in southern Illinois, USA (Bird et al., 2019; Butler et al., 2011). Specifically, we used geochemical and isotopic methods to quantify Pb pollution and its provenance through time.

STUDY SITE AND BACKGROUND

Kincaid Mounds (hereafter Kincaid) is located in southern Illinois on a portion of the lower

Ohio River's floodplain known as the Black Bottom (Fig. 1). While smaller archaeological sites have been identified across the Black Bottom, Kincaid represents a primary locus of habitation on Avery Lake's northern shore (Muller, 1986). Archaeological evidence indicates that this site was intermittently occupied since ca. 4000 BCE, with the first evidence for intensive settlement and village construction beginning during the Early and Middle Woodland Baumer phase from 300 BCE to 300 CE (Butler and Crow, 2013; Butler and Welch, 2006; Parker and Butler, 2017). Kincaid was subsequently abandoned, or intermittently occupied by a sparse population, from ca. 350 to 650 CE (Butler and Wagner, 2012). After 650 CE, Kincaid was repopulated during the Late Woodland Lewis phase, which transitioned into the Mississippian period sometime between 1000 and 1150 CE (Pursell, 2016). The Mississippian occupation (1150–1450 CE) is the best studied at Kincaid because large earthworks, bastioned fortifications, and an extensive village were constructed during this time (Butler et al., 2011). Between 1400 and 1450 CE, Mississippians abandoned Kincaid, along with much of the central Mississippi and Ohio River valleys (Cobb and Butler, 2002; Milner and Chaplin, 2010), in what has been suggested to be a response to a severe ~100-yr-long drought between 1350 and 1450 CE (Bird et al., 2017). Kincaid and the surrounding Black Bottom remained largely unoccupied until the 1800s, when Euro-American settlers began utilizing the region for river commerce and agriculture (Bird et al., 2019; Muller, 1986).

Recently reported multi-proxy results from Avery Lake complement the archaeological perspective of Kincaid's occupation history and reveal additional information about pre-Columbian land use and environmental impacts
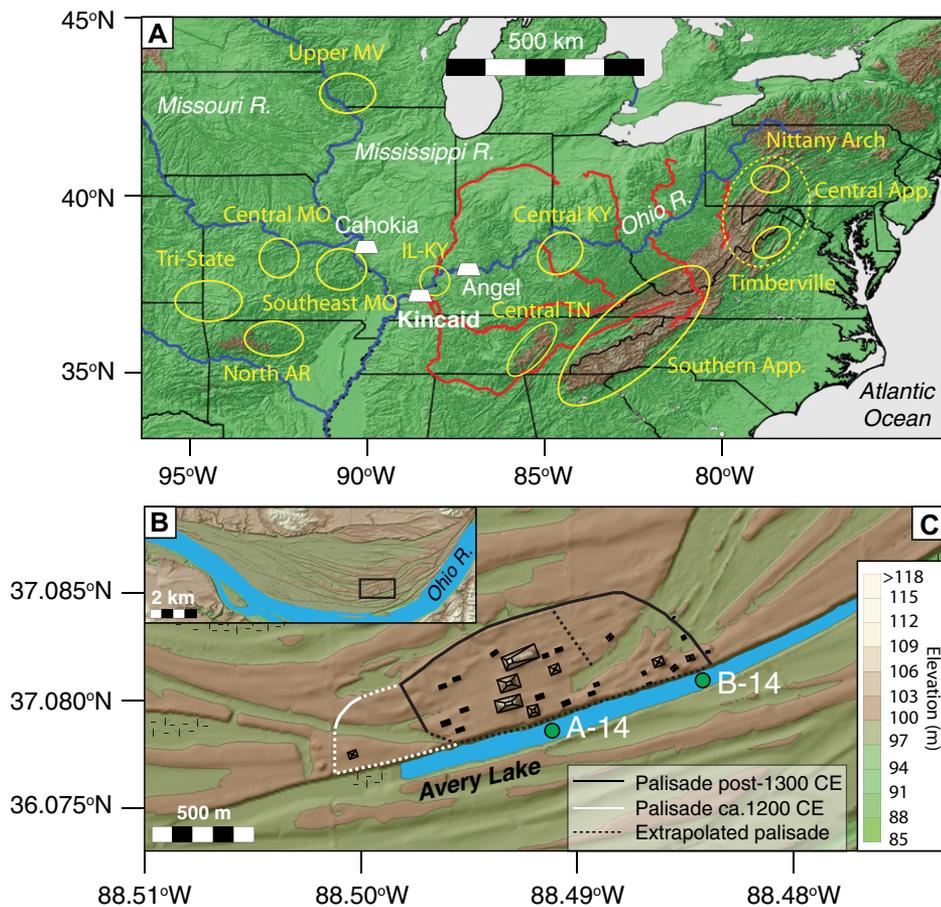
Figure 1. (A) Map of midwestern and eastern United States showing the distribution of galena-bearing ore bodies for which Pb isotopes have been determined (yellow circles). Dotted yellow line indicates central Appalachian Mountains region. Ore bodies from southeastern and central Missouri and upper Mississippi River valley have previously been identified as the primary sources from which Mississippian galena was derived. Major Mississippian settlements, including Cahokia, Angel Mounds, and Kincaid Mounds (focus of this study) are shown with white trapezoids. Major tributaries of the Ohio River are shown in red. MV—Mississippi Valley; MO—Missouri; AR—Arkansas; IL—Illinois; KY—Kentucky; TN—Tennessee; App.—Appalachians. **(B)** Topographic map of the Black Bottom floodplain on the lower Ohio River, showing the location of Kincaid Mounds (black box). **(C)** Expanded view of Kincaid Mounds archaeological site, including Avery Lake. Location of sediment cores used in this study are shown with labeled green circles. Structures (black rectangles) and fortification walls (black and white lines) are shown after Butler et al. (2011).

(Bird et al., 2019). For example, the most intensive pre-Columbian occupations identified in the archaeological record (the Baumer and Mississippian) were each characterized by extensive land clearance as evidenced by simultaneous lows in arboreal (tree) pollen and peaks in *Ambrosia* (ragweed) pollen, the latter being an indicator of landscape disturbance (Wright, 1967). Low population densities during the Baumer occupation and significantly increased population densities during the Mississippian occupation (Butler and Wagner, 2012) were additionally reflected in bulk sediment $\delta^{15}N$ variations (Bird et al., 2019). Whereas $\delta^{15}N$ was relatively invariant during the Baumer occupation, indicating low population densities, it abruptly increased by ~4‰ to over 6.5‰ between 1130 and 1185 CE, suggesting that population densities reached ~60 people/km² (Cabana and Rasmus-

sen, 1996). $\delta^{15}N$ values subsequently decreased, but remained elevated until after ca. 1460 CE, which agrees with the site being abandoned between ca. 1400 and 1450 CE.

Three distinct Pb concentration peaks are also apparent in the Avery Lake data during each of the major occupations in the Black Bottom, i.e., Baumer, Mississippian, and the Euro-American occupation since ca. 1800 CE (Bird et al., 2019). These Pb anomalies remained even after normalization to the conservative elements zirconium (Zr) and titanium (Ti), indicating that they represent excess Pb in the environment (Boës et al., 2011). Given their temporal association with human occupations, and that they did not occur under any other conditions, the Pb anomalies are attributed to anthropogenic pollution. Anthropogenic sources of Pb during the past ~200 yr have been extensively

investigated, with wood and coal combustion (1800s and 1900s), ore smelting (1800s to present), and the use of leaded petroleum products (1940s to the 1970s) generally implicated as the sources of modern Pb pollution (Graney et al., 1995). Here, we focus on the pre-Columbian Pb signals, as little is known about the occurrence and/or sources of early Pb pollution in North America.
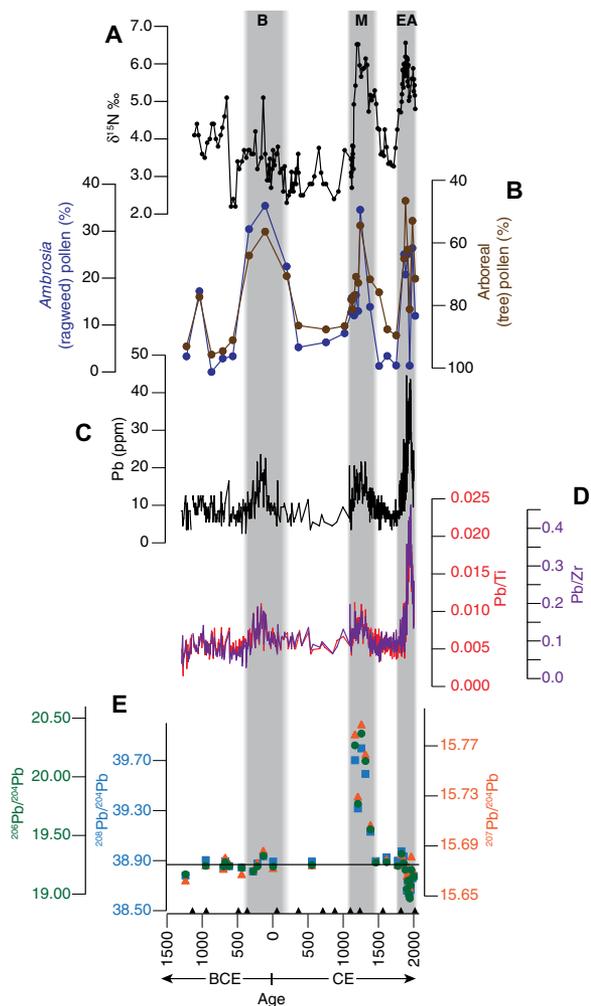
METHODS

We selected 30 samples from Avery Lake sediment core A-14 (Bird et al., 2019) for Pb isotope analyses at the Department of Geological Sciences, University of Florida (Gainesville, Florida, USA). Samples were processed and measured in an ISO 6 clean lab equipped with ISO 4 laminar-flow hoods following Kamenov et al. (2009). About 0.05 g of sediment was weighed in acid-cleaned Teflon vials and digested with Optima-grade HF and HNO_3. The sample solution was then evaporated, and the residue was dissolved in 1N Optima-grade HBr. This solution was loaded on columns packed with Dowex 1X-8 resin to separate Pb for isotope analysis. Samples were washed 3× with 1 ml of 1N HBr, and the Pb fraction was collected in 1 ml of 3N HNO_3. Pb isotope compositions were determined on a Nu-Plasma multicollector–inductively coupled plasma–mass spectrometer (MC-ICP-MS), with Tl normalization. The reported Pb isotope data are relative to the following long-term values of NIST (U.S. National Institute of Standards and Technology) standard NIST 981: $^{206}Pb/^{204}Pb = 16.937$ (± 0.004, 2σ), $^{207}Pb/^{204}Pb = 15.490$ (± 0.004, 2σ), and $^{208}Pb/^{204}Pb = 36.695$ (± 0.009, 2σ). The Avery Lake age model and proxy results discussed below (X-ray fluorescence [XRF], pollen, $\delta^{15}N$) were previously developed by Bird et al. (2019) (see the GSA Data Repository[1]).

RESULTS AND DISCUSSION

The Pb isotope results show consistent trends across all time series, with two primary features standing out. The first is a long-term baseline, which averages 19.250, 15.676, and 38.885 for $^{206}Pb/^{204}Pb$, $^{207}Pb/^{204}Pb$, and $^{208}Pb/^{204}Pb$, respectively (Fig. 2). The second is an abrupt, positive Pb-isotope-ratio excursion toward higher values during the Mississippian period between 1150

Figure 2. Avery Lake (Illinois, USA) proxy results from core A-14 (Bird et al., 2019). (A) $\delta^{15}N$ **as indicator of population density before 1830 CE and combination of population and agricultural activity after 1830 CE. (B) Pollen results for** *Ambrosia* **(ragweed; blue line), a disturbance indicator, and arboreal (tree) pollen (brown line). Note that y-axis is reversed for arboreal pollen abundances such that increased disturbance (high** *Ambrosia***) coincide with decreases in arboreal pollen abundances. (C) Pb concentrations from X-ray fluorescence (XRF). (D) XRF Pb abundances normalized to Ti (red) and Zr (purple) in order to remove Pb variations resulting from changes in terrestrial erosion. Resulting peaks are Pb pollution derived from anthropogenic sources. (E) Pb isotope results for** $^{206}Pb/^{204}Pb$ **(green),** $^{207}Pb/^{204}Pb$ **(orange), and** $^{208}Pb/^{204}Pb$ **(blue). Solid black horizontal line indicates the Pb isotopic background not including Mississippian or Euro-American excursions. Gray vertical boxes denote archaeologically determined pre-Columbian Native American occupations at Kincaid Mounds during Baumer (B) and Mississippian (M) phases as well as current Euro-American occupation (EA). Black triangles on the x-axis indicate positions of accelerator mass spectrometry (AMS)** ^{14}C **ages (Bird et al., 2019).**

and 1400 CE that was contemporaneous with an excess Pb peak. Notably, no Pb isotope excursions occurred during the Baumer phase, despite increased excess Pb. These Pb isotope results suggest distinct origins for the Mississippian and Baumer Pb peaks.

Spatial variations in Pb isotopes of regional ore deposits have been systematically investigated over the last several decades, providing a means with which to determine the provenance of Pb in soil and sediment samples based on their Pb isotope ratios (Figs. 1 and 3; Goldhaber et al., 1995; Heyl et al., 1966; Kesler et al., 1994a, 1994b; Kesler and van der Pluijm, 1990). The well-defined pre-anthropogenic Pb isotope background against which the Mississippian excursion occurred is consistent with Appalachian sediment sources (Fig. 3).

When plotted in Pb isotope space, the radiogenic Mississippian Pb isotope ratios plot along mixing lines that project toward values associated with radiogenic Pb ores from Missouri and the upper Mississippi River valley (Fig. 3). This suggests that the excess Mississippian Pb concentrations resulted from an influx of radiogenic Pb primarily from Missouri and to a lesser extent the upper Mississippi valley. This is consistent

with Walthall's (1981) seminal work that used trace elemental geochemistry to show that the majority (~83%) of Mississippian-period galena originated from the Old Lead Belt subdistrict in southeastern Missouri. Among the remaining, 12% was derived from upper Mississippi valley surface deposits and 5% from central Missouri.

The Avery Lake Pb isotope results demonstrate that Mississippian galena use was a source of environmental pollution that remains as legacy pollution today. Archaeological evidence indicates that galena was commonly crushed and that the metallic powder was ritually used as a cosmetic or to adorn sacred objects and/or structures (Homsey-Messer and Humkey, 2016; Walthall, 1981). It is therefore most likely that the source of Pb pollution in Avery Lake was galena powder that was washed or blown into the lake after its use and/or as part of its production by grinding.

To estimate the amount of galena-derived Pb contained in Avery Lake, we used a two-end-member mixing model that accounted for different end-member Pb concentrations and isotopic compositions (Faure and Mensing, 2005; see the Data Repository). Here, we use $^{206}Pb/^{204}Pb$ isotope ratios and Pb concentrations

of the background sediment and pure galena for end members A (E_A) and B (E_B), respectively. The isotopic composition and abundance of Pb for E_A (19.250 and 11.8 ppm, respectively) were estimated by averaging pre-Columbian background $^{206}Pb/^{204}Pb$ isotope values and Pb concentrations excluding the Mississippian $^{206}Pb/^{204}Pb$ isotope excursion. The $^{206}Pb/^{204}Pb$ isotopic value (21.1996) and concentration of Pb in E_B was estimated using an 83%-12%-5% mixture of southeastern Missouri, upper Mississippi valley, central Missouri galena (Walthall, 1981), which have respective average $^{206}Pb/^{204}Pb$ values of 20.958, 22.536, and 22.004), and the relative abundance of Pb in galena (86.6%). Lead isotope values for different mixtures of E_A and E_B were calculated as follows:

$$\left(\frac{^{206}Pb}{^{204}Pb}\right)_M = \left(\frac{^{206}Pb}{^{204}Pb}\right)_A f_A \left(\frac{[Pb_A]}{[Pb_M]}\right)$$
$$+ \left(\frac{^{206}Pb}{^{204}Pb}\right)_B (1 - f_A)\left(\frac{[Pb_B]}{[Pb_M]}\right),$$

(1)

where $\left(\frac{^{206}Pb}{^{204}Pb}\right)_M$ is the isotopic composition of the sediment resulting from the combination of sediments from the two end members, $\left(\frac{^{206}Pb}{^{204}Pb}\right)_A$ and $\left(\frac{^{206}Pb}{^{204}Pb}\right)_B$ are the respective isotopic compositions of E_A and E_B, $\left(\frac{[Pb_A]}{[Pb_M]}\right)$ and $\left(\frac{[Pb_B]}{[Pb_M]}\right)$ are the Pb concentrations in E_A and E_B relative to the mixture, and f_A is the abundance of E_A in the mixture.

The mixing hyperbola generated by Equation 1 indicates that galena deposition in Avery Lake contributed between 2.1 and 15.9 ppm Pb during the Mississippian occupation between ca. 1150 and 1400 CE (Fig. 3C). Accounting for dry sediment bulk density and sedimentation rate changes during the Mississippian period, we calculated the flux of galena-derived Pb to Avery Lake (area of 134,880 m^2). Our results indicate that a total of ~1.5 t (metric tons) of galena-derived Pb was deposited in Avery Lake as a result of Mississippian activities. Over the ~250 yr Mississippian occupation, this equates to ~6.2 kg Pb/yr. This estimate compares favorably with one derived from the XRF data (1.1 t of Pb; 4.5 kg Pb/yr), which was similarly calculated by integrating the flux of excess Pb during the Mississippian period across Avery Lake (see the Data Repository for calculations).

In contrast to the Mississippian period, no Pb isotope excursion was associated with the Baumer Pb peak between 310 and 15 BCE (Fig. 2). We suggest that biomass burning plausibly accounts for the excess Pb deposited in Avery Lake during this time, given that Pb is an important component of biomass combustion products (Larson and Koenig, 1994) and pre-Columbian societies are known to have extensively used fire
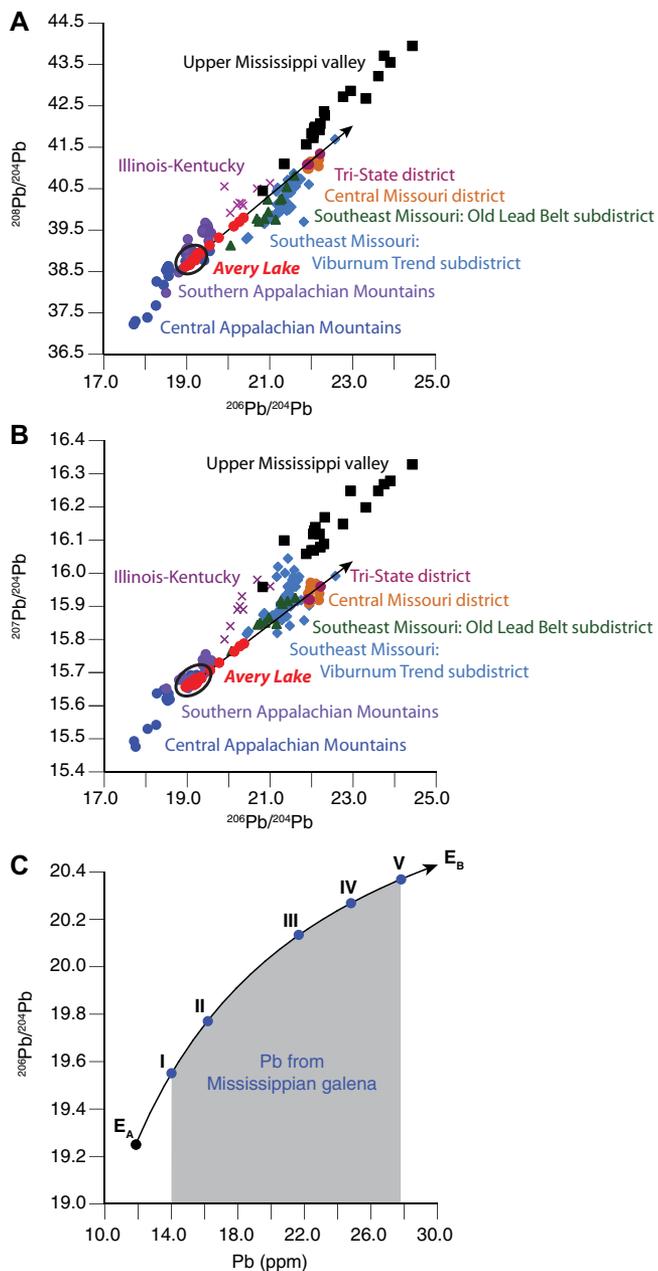
Figure 3. (A,B) Lead isotope plots of $^{208}Pb/^{204}Pb$ versus $^{206}Pb/^{204}Pb$ (A) and $^{207}Pb/^{204}Pb$ versus $^{206}Pb/^{204}Pb$ (B) showing that background Pb isotope values at Avery Lake (Illinois, USA; red circles) are consistent with Appalachian Mountains sediment source (black circled area). More-radiogenic Avery Lake Pb isotope values plot along mixing lines that trend most strongly toward Missouri (USA) ore sources, consistent with previous work indicating that most Mississippian galena originated from these sources (Walthall, 1981). (C) Hyperbola formed by mixing Avery Lake background sediments (end-member A, E_A) with galena (end-member B, E_B) derived from southeastern (83%) and central (12%) Missouri and upper Mississippi River valley (5%). Point E_A represents the Pb isotopic composition (19.250) and Pb concentration (11.8 ppm) background in Avery Lake sediments derived from the Appalachian Mountains. Points I through V represent the range of Pb concentrations resulting from Mississippian galena use. E_B (pure galena with an 83%-12%-5% mix of ore sources detailed above) plots off mixing hyperbola.

where galena trade, processing, and utilization occurred also contain legacy pre-Columbian Pb pollution, as suggested by increased Pb in Horseshoe Lake near Cahokia (near present-day East St. Louis, Illinois) during the Mississippian occupation (Pompeani et al., 2019). Detailing spatiotemporal and source variability in Pb pollution provides important information about how pre-Columbian Native Americans interacted with and utilized their landscape and its resources.

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to modify the landscape (Delcourt and Delcourt, 2004; Delcourt et al., 1998). We suggest that initial land clearance via fire was followed by frequent fire use to maintain the landscape and for domestic activities. Because the combusted biomass had originally grown in sediments from the Black Bottom floodplain, which share the same Appalachian source as those deposited in Avery Lake, it would have the same "local" isotopic signature, thereby not altering Pb isotopic ratios in Avery Lake. Nonetheless, the XRF data suggest that biomass burning resulted in the deposition of ~0.4 t of Pb at Avery Lake during the Baumer occupation.

CONCLUSIONS

Significant amounts of Pb were deposited in Avery Lake as a result of pre-Columbian Native American activities that involved both land use and long-distance trade or acquisition of galena followed by localized processing and utilization. During the Mississippian occupation between ca. 1150 and ca. 1400 CE, Pb isotopes indicate that galena processing and/or use resulted in ~1.5 t of anthropogenic Pb pollution. An earlier Pb pollution peak between 310 and 15 BCE during the Baumer occupation, which lacked an accompanying Pb isotope excursion, was likely the result of extensive local biomass burning. It is virtually certain that pre-Columbian Pb pollution was not restricted to Kincaid Mounds. However, the spatial distribution of pre-Columbian Pb pollution within the Black Bottom, and indeed at other archaeological sites across the midcontinental United States, is not known at this time. It is probable that other sites

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