Plains Anthropologist

ISSN: (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/ypan20

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Margaret E. Beck, Brandi L. MacDonald, Jeffrey R. Ferguson & Mary J. Adair

PLAINS ANTHROPOLOGIST, 2022, 1–26

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Red pigment in the Central Plains: A Pawnee case at Kitkahahki Town

MARGARET E. BECK
University of Iowa

BRANDI L. MACDONALD
University of Missouri

JEFFREY R. FERGUSON
University of Missouri

MARY J. ADAIR
University of Kansas

James Murie, early twentieth century ethnographer and member of the Pawnee Nation, once wrote that the "things that are most acceptable to the Pawnee gods are smoke, fat, paint, and flesh" (Murie 1981:466). Here we describe red paint at Kitkahahki Town, a late eighteenth–early nineteenth-century Kitkahahki Pawnee village in north-central Kansas. Using laser ablation inductively coupled plasma mass spectroscopy and Raman spectroscopy, we compare archaeological paint and pigment samples to three pigment materials – pipestone powder, vermilion, and ochre – all documented in the Great Plains after European colonization. We ultimately find no evidence of pipestone powder or vermilion as pigment at Kitkahahki Town and conclude that ochre (some of which may be from the Lower Cretaceous Dakota formation) is the most likely pigment material at the site. Ochre may have been especially significant because of links between this earth pigment and Pawnee sacred geography.

KEYWORDS Pawnee, paint, ochre, pipestone, vermilion

The Pawnee Nation, based in Oklahoma since 1876, has a traditional homeland centered on the Platte and Loup river valleys in east-central Nebraska. Their territory included not only earthlodge villages in river valleys but also sacred sites and hunting lands, stretching to the Niobrara River to the north and the Arkansas River

 DOI 10.1080/00320447.2022.2108601

to the south (Blakeslee et al. 1986; Echo-Hawk 1992; Parks and Wedel 1985; Roper 2006; Wedel 1936, 1986; Weltfish 1971). Pawnee people regard certain places in this landscape as "holy ground" (Parks and Wedel 1985:144) and as constituent parts of a larger "map of the sacred on this earth" (Parks and Wedel 1985:143). Here we investigate pigment materials within the "sacred geography" described by Parks and Wedel (1985; Figure 1). These materials were presumably chosen not only for their availability and working properties but also for their links to meaningful places and relationships.

This study is set around the time of the expeditions of Lewis and Clark in 1804–1806 and Pike in 1806, when Pawnee people numbered "perhaps seven to ten thousand" in total and "lived in two to five or six principal towns, each estimated to consist of 40–200 earth lodges, containing from 800 to 3,500 inhabitants" (Parks and Wedel 1985:146). Pawnee people used red paint in a variety of domestic and sacred contexts, including on painted parfleches (Morrow 1975) and gaming pieces (Hadley 2023). Red paint is particularly common on Pawnee ceramic

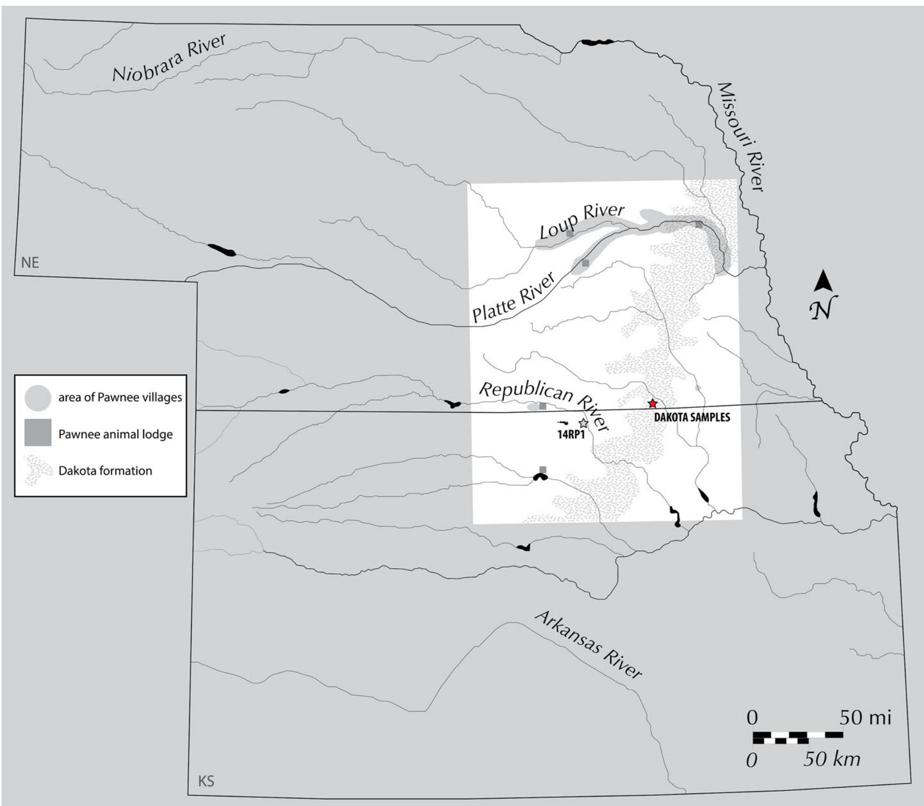

FIGURE 1. The study area (in white) in relation to modern Kansas and Nebraska, with boundaries from Parks and Wedel's (1985:figure 1) "sacred geography" and Dakota formation exposures from Ludvigson and others (2010:figure 1). Kitkahahki Town (14RP1) is shown in the study area in relation to sampled ochre in Jefferson County, Nebraska.

vessels after AD 1750 (Beck 2020; Dunlevy 1936; Grange 1968; Wedel 1936) including at Kitkahahki Town where nearly 20 percent of identifiable rim sherds had traces of red paint on the interior (Beck and Roper 2023). It has been described as "a thick, deep-red coloring matter. . . [that] in nearly all cases can be readily rubbed off" (Wedel 1936:70) and as "the residue of material kept in the vessels," particularly when found in bowl interiors (Figure 2; Grange 1968:67). The paint does not seem to have been fired on to vessel surfaces; it was deliberately applied in some cases (Beck 2020) and probably a remnant of vessel contents in others, such as "the bowl of reddened grease" used in the Skiri Pawnee New Fire Ceremony (Murie 1981:144).

We draw upon the archaeological record at Kitkahahki Town (14RP1), a late eighteenth-early nineteenth-century Kitkahahki Pawnee village in north-central Kansas (Figure 1), and use laser ablation inductively coupled plasma mass spectroscopy (LA-ICP-MS) and Raman spectroscopy to compare red paint on ceramic surfaces to powdered pigment and to possible pigment materials. The only explicit ethnographic reference to a Pawnee pigment material is from Weltfish (1971:478), who describes working red pipestone and states, "All red powder resulting from the sawing process was saved to be used as red paint." Contemporary Indigenous carvers also save pipestone powder to make red paint (Hadley 2015, 2016). In addition to pipestone powder, we consider two other mineral pigments: vermilion and ochre. Vermilion, or mercuric sulfide (HgS), was exported by the French and English to North America beginning in the seventeenth century and became a "a staple of the transatlantic fur trade" (Lozier 2017:45). Use of ochre, or earth pigment, is suggested during Pawnee ceremonies in which people used "red earthen paint" (Murie 1981:400), and "a ball of buffalo fat mixed with red earthen clay" (Murie 1981:427).

Red paint in the Pawnee ethnographic record

> In one of his dreams he dreamed of a man who was painted red all over and who told him that he was the sun. (Murie 1981:364)

George Catlin (1842:102; Letter No. 14) describes red paint as an integral part of "classic dress" and as one of several attributes with "some definite importance or meaning which an Indian could explain to us at once. . . every streak of red paint covered a wound which he had got in honorable combat." It could also be used for sun protection according to Maxídiwiac, a Hidatsa elder also known as Buffalo-bird-woman. As she recalled older customs of face painting, she noted that "[i]n olden times the husband carried a paint bag and every morning painted his face, as did his wife and children also. . . I painted every morning because the wind and air made our faces dark, tanned them as you say, so we painted that our complexions would not darken" (Wilson 1924:257).

Red paint was incorporated into daily hairstyles for both men and women. Catlin (1842:95; Letter No. 13) described women's hairstyles as broadly similar across the Great Plains, with women "parting the hair on the forehead, and always keeping

FIGURE 2. Bowl from Horse Creek with red pigment (25NC2-6).

the crease or separation filled with vermilion or other red paint." Along the Lower Missouri River, Pawnee and other men often had.

> the hair being cut as close to the head as possible, except a tuft the size of the palm of the hand, on the crown of the head, which is left of two inches in length: and in the centre of which is fastened a beautiful crest made of the hair of the deer's tail (dyed red) and horsehair, and oftentimes surmounted with the war-eagle's quill. In the centre of the patch of hair. . . is preserved a small lock, which is never cut, but cultivated to the greatest length possible, and uniformly kept in braid, and passed through a piece of curiously carved bone; which lies in the centre of the crest, and spreads it out to its uniform shape. . . Through this little braid, and outside of the bone, passes a small wooden or bone key, which holds the crest to the head. . . Amongst those tribes who thus shave and ornament their heads, the crest is uniformly blood-red; and the upper part of the head, and generally a considerable part of the face, as red as they can possibly make it with vermilion. (Catlin 1842:23-24; Letter No. 34)

The "beautiful crest made of the hair of the deer's tail (dyed red) and horsehair" described above is known as a roach (Figure 3), and the "curiously carved bone; which lies in the centre of the crest" as a roach spreader. A roach spreader from the Hill site (25WT1), a Kitkahahki Pawnee site in the Republican River valley occupied 1775–1809 (O'Shea 1989:table 2), "bears several rows of tiny pits filled with red pigment" (Wedel 1936:85) probably representing pigment on the head of the wearer.

Red paint is also among "the things most acceptable to the Pawnee gods" (Murie 1981:466). Throughout Native North America, red paint is "one of the most powerful animating substances in the universe, with divine origins and properties ranging from protective to transformative and from interactive to integrative" (Zedeño 2009:412). It figures prominently in many of the Pawnee ceremonies and sacred bundles documented by James Murie, early twentieth century ethnographer and member of the Pawnee Nation (see also Dorsey 1904).

One example of sacred red paint use comes from the White Beaver Ceremony of the Chawi, which has the goal of "renewing. . . powers" and specifically "reviving … hibernating animals, particularly those living in the waters" (Murie 1981:201). The two men leading the ceremony created the altar, including the White Beaver (represented by a beaver skin with the skull and tail), and then painted their faces and bodies. This paint included "mysterious *red clay* mixed with medicine," which they put on the lower half of their faces (Murie 1981:204; emphasis added). Afterward the beaver was washed with water and then had red paint applied around its nose and mouth (Murie 1981:208). Early on the third day of the ceremony, one of the leaders made the following remarks:

> Doctors and errand men, we do this as our forefathers were told by the animals. Since the animal was put away for the winter as if dead, it must now receive its life and power from the sun. Some of you will paint with the

FIGURE 3. George Catlin, *La-wáh-he-coots-la-sháw-no, Brave Chief, a Skidi (Wolf) Pawnee*, 1832. Smithsonian American Art Museum, gift of Mrs. Joseph Harrison, Jr., 1985.66.100.

> red paint, for *red paint is from the sun*. Others will paint with white clay, for this animal is white. (Murie 1981:225; emphasis added)

Another example comes from the Buffalo Dance of the Pitahawirata as learned from the "Buffalo people," spirits of deceased bison who required gifts in exchange for knowledge of the ceremony. The man who first met the Buffalo people received instructions in his dreams, including the directive "You must always have a buffalo skull in your lodge" (Murie 1981:396). The Buffalo Doctors conducting the ceremony every year purified the skull on the first day by washing and then painting it with both white clay and red paint. Similar practices may be thousands of years old in the Great Plains, as suggested by red paint on a bison skull at the Folsom Cooper site (34HP45) in Oklahoma (Bement 1999).

Paint and pigment materials

Paint is made from pigment, a liquid such as water, and often a binder to help the paint adhere to the underlying surface (Munson 2020; Rapp 2009). The resulting mixture has the insoluble pigment particles suspended throughout. In pigments with a high clay content, the clay may contribute to its binding properties (Eiselt et al. 2011). Other binders in North America included animal fat and blood, salmon eggs, and human saliva (Ancheta 2013; Eiselt et al. 2011; Grinnell 1962; Scott et al. 1996; Stephen 1898). Animal fat was a binder in some Pawnee paint, as described during the Skiri Pawnee New Fire Ceremony:

> Meanwhile the leading warrior mixed some *consecrated fat and red earthen clay.* After rubbing this mixture over his own body, the leading warrior passed it. . . until all five priests had anointed themselves with the *bowl of reddened grease*. (Murie 1981:144; emphasis added)

Lipid analysis of two Kitkahahki Town body sherds with interior red paint revealed the presence of animal fat most consistent with a medium-size mammal, rather than a large herbivore, and low levels of the fatty acid C18:0 suggest beaver in particular (Malainey and Figol 2020).

We consider three pigment materials in this study: pipestone, vermilion, and ochre. Each of these materials is described in more detail below.

Pipestone

Pipestone is carvable clay-rich stone, such as lightly metamorphosed claystone or shale, that does not disperse in water (Hughes et al. 1998; Wisseman et al. 2012), and red pipestones gain their color from dispersed hematite throughout the rock. Over the last five millennia, Indigenous people in the midcontinent have collected pipestone from sources in Kansas, Missouri, Minnesota, Wisconsin, Illinois, and Ohio (Wisseman et al. 2012). Different pipestone sources in North America are distinguished by their mineral composition (Wisseman et al. 2012) as characterized with X-ray diffraction (XRD) or a Portable Infrared Mineral Analyzer (PIMA) spectrometer (Emerson and Hughes 2000, 2001; Emerson et al. 2002, 2003; Gunderson 1991, 1993; Hughes et al. 1998; Wisseman et al. 2002, 2012). The mineralogical differences are associated with chemical differences, which can also be assessed (Hoard et al. 2014).

Two pipestone types dominate Pawnee pipestone use: Minnesota pipestone and Kansas pipestone. "Minnesota pipestone" or "catlinite," a very fine-grained red pipestone that lacks quartz and is particularly easy to carve, is found only in and around Pipestone National Monument in southwestern Minnesota (Gunderson and Tiffany 1986; Hughes 1995; Scott et al. 2006). The southwestern Minnesota quarries and the pipestone itself are sacred for Indigenous groups in the northern Midwest and Plains, as described in the nineteenth century by Lewis and Clark (Thwaites 1904:115, 1905:44–45) and Catlin (1842; Letter No. 55). The Indigenous contemporary carvers interviewed by Hadley (2015) hold permits to quarry at Pipestone National Monument. “Kansas pipestone” is quartzose red pipestone transported in glacial till from the Jasper, Minnesota area and deposited in northeastern Kansas, southeastern Nebraska, and northwestern Missouri (Gunderson and Tiffany 1986; Hadley 2015, 2016).

Minnesota pipestone was extensively quarried, crafted, and traded by Oneota groups after AD 1400 (Fishel et al. 2010; Gunderson and Tiffany 1986), and its use markedly increased in the Middle and Upper Mississippian regions after AD 1450–1550 (Emerson and Hughes 2001; Penman and Gunderson 1999). It is not well represented in the Central Plains for several more centuries, however. Prior to AD 1700, ancestral Wichita (Great Bend) and ancestral Pawnee (Lower Loup) groups mostly had Kansas pipestone. After AD 1700, although Kansas pipestone use continued, most of the characterized pipestone in Pawnee and Wichita sites is Minnesota pipestone (Hadley 2018; Gunderson and Tiffany 1986). This increase in Minnesota pipestone after AD 1700 may reflect increased participation in shared exchange and religious practices across the Midwest and eastern Plains, including belief in the “aura of spiritual power” (Fishel et al. 2010:191) of the Minnesota quarries.

Vermilion

The pigment vermilion is a commodity, moving through international markets before arriving in North America as a unit of exchange (Hart 1982; Lozier 2017). Originating from the mineral cinnabar (Gettens et al. 1972), it consists of mercuric sulfide (HgS) and can be identified using XRF to recognize the presence of mercury (Hg) as well as through Raman spectroscopy (Pearlstein et al. 2009). Vermilion may also be extended or supplemented with red lead (Gettens et al. 1972; Lozier 2017).

Vermilion is mentioned in both French and English accounts in North America as early as the 1680s (Lozier 2017), and in 1724 Sieur de Bourgmont included vermilion among merchandise for trade with the “Padouca” or Plains Apache in Kansas (Johnson 2016:482). Throughout the eighteenth century, it “was abundantly traded and offered as a diplomatic present in every North American frontier zone” (Lozier 2017:47). There was enough vermilion at Grand Portage National Monument in northeastern Minnesota, where the Northwest Fur Company operated (White 2005), that soil and water in the area is still contaminated with mercury (Rolfhus et al. 2015). A prominent example of vermilion use in the Great Plains is the Rosebud winter count, a late nineteenth-century pictograph calendar of the Brulé band of the Lakota Sioux (Pearlstein et al. 2009; Thornton 2007).

The Pawnee participated substantially in the Missouri River fur trade by 1803–1804, based on Lewis and Clark’s estimates of the annual dollar value of trade (Blakeslee 1975; Thwaites 1904, 1905). Vermilion was well established as a commodity in this trade, and the Lewis and Clark expedition carried it among the trading items recommended by Auguste and Pierre Chouteau (Buckley 2019).

Ochre

Ochre is a naturally occurring pigment with a very deep history of human use (Siddall 2018). It is also very broad in geological terms, encompassing

> a diversity of rocks, minerals, and sediments that contain Fe-oxide/oxyhydroxide mineral phases that can produce a red or yellow streak or stain, including hematite, limonite, goethite, ferricrete, siderite, scoria, laterite, and iron-enriched clay and silt . . . The mineralogical composition of ochre typically includes one or more phases of a Fe-oxide/oxyhydroxide matrix with major impurities such as quartz, mica, clay minerals, sulfides, or carbonates. (MacDonald et al. 2018:477)

Its color can be manipulated by heating, changing color from yellow (goethite) to red (hematite) and creating a range of red and brown shades—a process documented as early as the European Upper Paleolithic (Jezequel et al. 2011; Pomies et al. 1999; Salomon et al. 2015) and observed throughout North America (Ancheta 2013; Densmore 1918; Ewers 1979; Morrow 1975).

Ochre appears throughout Plains and Midwest ethnography, such as the "earth pigments" or "ferruginous clays" that provided red, yellow, and brown colors for painting hides across the Plains and elsewhere in North America (Ewers 1979:3; see also Morrow 1975). Maxídiwiac described "red paint, such as we [Hidatsa] obtained in the hills" (Wilson 1924:219), and the Omaha word "waçezhide" for red paint translates to red clay, which Omaha speakers also "applied to the trader's article" (Fletcher and La Flesche 1911:615).

When earth pigments are encountered in the archaeological record, it is not always clear how they were prepared and applied. Were they added to a binder and used as paint, or used instead as dry pigment? Dry powdered red ochre may have been sprinkled on the floor of Lodge 1 of Kitkahahki Town (Table 1) and on living floors in the Plains region over 10,000 years ago (Roper 1991). The earliest people in the Plains (and the continent) also applied red ochre to human remains, mortuary objects, and cached items (Roper 1991; Trabert and Hollenback 2021:45–48) – uses that may include dry pigment, prepared paint, or both. A man at the Late Paleoindian site of Horn Shelter (41BQ46) in Texas was buried with tools "used in paint production, body painting, and scarification" and possibly tattooing (Jodry and Owsley 2014:590). These tools include a worked ochre nodule and associated items with traces of ochre, such as two sandstone abraders and two turtle carapace bowls.

Red ochre sources in the Great Plains include deposits in both sedimentary and metamorphic rock. The Powars II Paleoindian quarry (48PL330) in Wyoming is a well-known deposit in metamorphic rock, appearing in the Precambrian Good Fortune Schist formation (Frison et al. 2018; Southerland and Cola 2015; Stafford et al. 2003; Tankersley et al. 1995; Zarzycka et al. 2019). Sedimentary examples include mudstones from the Lower Triassic Chugwater Formation and the Lower

TABLE 1.

ARCHAEOLOGICAL SAMPLES FROM 14RP1.

LA-ICP-MS ID	Cat No	Type	Institution	Recovery context	Notes
MEB21	5153	ceramic	KSHS	Lodge 5, southwest barrow pit	
MEB22	5169	ceramic	KSHS	Lodge 5, southwest barrow pit	
MEB23	3635	ceramic	KSHS	Lodge 6	
MEB24	4392	ceramic	KSHS	Unit 586, F629 (artifact concentration)	
MEB25	4402	ceramic	KSHS	Unit 586, F629 (artifact concentration)	
MEB26	3897	ceramic	KSHS	Unit 586, no feature	See Malainey and Figol 2020
MEB27	5018	ceramic	KSHS	Unit 593	
none	5022	ceramic	KSHS	Unit 593	See Malainey and Figol 2020
MEB28	5024	ceramic	KSHS	Unit 593	
MEB101	14023	powder	KU	Lodge 1	
MEB102	14304	powder	KU	Lodge 1, south side	
MEB103	14319	powder	KU	South field? (Nystrom donation)	

Cretaceous Kootenai Formation in Montana (Kingery-Schwartz et al. 2013). In Kansas and elsewhere in the Central Plains, ochre (hematite and limonite) occurs in sedimentary deposits. Sources include "clays and shales" and "concretions (particularly in the Dakota formation)" (Tolsted and Swineford 1984:72, 75).

In this study, we focus on red ochre available in the sedimentary Lower Cretaceous Dakota formation (O'Connor 2005), which is the only potential source of ochre in Kansas noted by Stein (2006). One particularly large deposit, described by Kinney (1942:104), is now apparently under Wilson Lake (Stein 2006):

> Possibly the best known prospect [of "iron from hematite and limonite"] is in the SE1/4 sec. 13, T. 13 S., R. 11 W., Russell county. Here a bed of hematite and limonite eight feet thick crops out in the top of the Dakota formation of Cretaceous age. . . It is somewhat contaminated by thin interbedded layers of sandstone parallel to the stratification, although in places individual layers of iron oxide as thick as ten inches free of sandstone can be observed. (Kinney 1942:104)

Red mudstone, clay and ironstone are forms of red ochre in the Dakota formation. These materials appear in multiple defined facies of the Dakota formation in Kansas: Facies 1, with "[v]ariegated mudstones with thin siltstone and sandstone lenses;" and Facies 5, with "[s]ideritic, clay-ironstone-bearing deposits" including "burrowed, thin sideritic clay ironstone seams and concretionary layers" (Hattin and Siemers 1987). The concretions in Facies 5 are noted as paint material in the geological field guide to Jefferson County, Nebraska: "The American Indian utilized the iron oxides in these concretions as paint pigments, and occasionally even today's artists gather these concretions for the same purpose" (Pabian 1977:15). Dakota mudstones and clays, which "contain a large admixture of

TABLE 2.

COMPARATIVE SAMPLES.

LA-ICP-MS ID	Material	Location	Notes
MEB32	MN pipestone	Pipestone National Monument, MN	unprocessed and unfired
MEB33	MN pipestone	Pipestone National Monument, MN	unprocessed and unfired
MEB39	Dakota red clay	Hwy 15; Jefferson County, NE	unprocessed and unfired
MEB40	Dakota yellow shale	Rose Creek pit; Jefferson County, NE	unprocessed and unfired
MEB104	Dakota red concretion	Hwy 15; Jefferson County, NE	unprocessed and unfired
MEB105	Dakota red concretion	Hwy 15; Jefferson County, NE	unprocessed and fired to 800 C
MEB106	Dakota red concretion	Hwy 15; Jefferson County, NE	levigated and unfired
MEB107	Dakota red concretion	Hwy 15; Jefferson County, NE	levigated and fired to 800 C
MEB108	Dakota yellow shale	Rose Creek pit; Jefferson County, NE	unprocessed and fired to 800 C
MEB109	Dakota yellow shale	Rose Creek pit; Jefferson County, NE	levigated and unfired
MEB110	Dakota yellow shale	Rose Creek pit; Jefferson County, NE	levigated and fired to 800 C
MEB111	Dakota red clay	Hwy 15; Jefferson County, NE	unprocessed and fired to 800 C
MEB112	Dakota red clay	Hwy 15; Jefferson County, NE	levigated and unfired
MEB113	Dakota red clay	Hwy 15; Jefferson County, NE	levigated and fired to 800 C
MEB115	KS pipestone	Doniphan County, KS	unprocessed and unfired; teaching collection (cat 2169)
MEB116	KS pipestone	Doniphan County, KS	unprocessed and unfired; teaching collection (cat 1210)

reddish and yellowish ocher," also appear in a review of ochre sources suitable for commercial paint (Condra 1907:30–31; see also Burchett 1991). These include clays historically mined for brickmaking (Gould 1899–1900:149–151; Kinney 1942:132–133), such as those near Endicott, Nebraska (Brenner et al. 2000; Joeckel et al. 2008) and Concordia, Kansas (Plummer 1959).

Sample and methods

This project investigates red paint at Kitkahahki Town, a fortified Kitkahahki Pawnee village along the Republican River (Adair and Hofman 2022; Beck et al. 2022; Kessler et al. 2021; Roper 2006). During the period AD 1777–1831, "groups of *Kitkahahki* splintered from the main Pawnee settlements in the Loup to establish and reoccupy villages in the Republican valley" (Kessler et al.

2021:484–485). Kitkahahki Town's primary occupation dates to approximately AD 1777–1802, with probable brief reoccupation in the following decades (Kessler et al. 2021). Residents were actively engaged in the fur trade, as suggested by abundant beaver remains in the faunal collection and other material culture at the site (Bozell and Latham 2023).

Our research goal was to evaluate four hypotheses about paint materials, using LA-ICP-MS and Raman spectroscopy at the Archaeometry Lab at the University of Missouri Research Reactor.

- Hypothesis 1: Sampled red paint at Kitkahahki Town (in one or more cases) is similar to Minnesota pipestone.
- Hypothesis 2: Sampled red paint at Kitkahahki Town (in one or more cases) is similar to Kansas pipestone.
- Hypothesis 3: Sampled red paint at Kitkahahki Town (in one or more cases) is similar to vermilion.
- Hypothesis 4: Sampled red paint at Kitkahahki Town (in one or more cases) is similar to ochre from the Dakota formation in the Central Plains.

In our analysis, we draw upon archaeological material from twentieth century site investigations at Kitkahahki Town, including two lodges (Lodges 1 and 2) excavated by the University of Kansas (KU) in 1949 and nine lodges (Lodges 3, 4, 5, 6, 7, 22, 23, 24, and 25) and external units excavated by the Kansas Historical Society (KSHS) in 1965–1968 (Adair and Hofman 2022; Roberts 1978; Smith 1950: Witty 1968). The archaeological samples include body sherds with red paint on the interior (Figure 4) and pigments collected in powder form. We submitted eight sherds from the KSHS excavations for compositional analysis (Table 1) along with three powdered pigments from the KU excavations.

We also submitted possible pigment materials – Minnesota pipestone, Kansas pipestone, and ochre from the Dakota formation – for comparison. Both Minnesota and Kansas pipestone appear at Kitkahahki Town as finished pipes (three pipes of Kansas pipestone and one of Minnesota pipestone) but all identified manufacturing debris was Kansas pipestone (Hadley 2023). Kansas pipestone therefore seems more likely to be a pigment source at this site than Minnesota pipestone. The two analyzed samples of Kansas pipestone in this study come from a teaching collection at the University of Kansas with donated artifacts from northeastern Kansas. The analyzed samples of Minnesota pipestone were purchased at Pipestone National Monument from people with a quarry permit (n = 2) or previously collected and archived at MURR (n = 4; see Mead 1999).

Dakota formation ochre was collected from previously identified exposures in Jefferson County, Nebraska (Figure 1): red clay and iron oxide concretions from Hwy 15 south of Fairbury, Nebraska ("Exposure 3;" Pabian 1977:6) and yellow shale from the Rose Creek Pit ("Stop 3;" Joeckel et al. 2008:14). These collection sites are approximately 40 miles (64 km) to the northwest of Kitkahahki Town. Future research will explore similar Dakota formation exposures in Washington and Cloud counties in Kansas that are closer to the site.

FIGURE 4. Sampled sherds with red paint on interior.

The red clay, iron oxide concretions, and yellow shale all qualify as "ochre" as defined here and could all be used as pigment for red paint. We were uncertain about what form of ochre might have been preferred, if any, and if variation in ochre form and processing would have a noticeable effect on the chemical data. We therefore submitted all three forms in four states (unprocessed and unfired, unprocessed and fired to 800 °C, levigated to concentrate clay-sized particles and unfired, and levigated and fired to 800 °C) for a total of 12 LA-ICP-MS samples of ochre. The yellow shale oxidizes to a color very similar to the red clay at temperatures of 500 °C and higher.

LA-ICP-MS

Pieces of $< 1\ cm^2$ were removed from each sample using clippers or a tile saw, gently rinsed in deionized water, and mounted on standard thin section slides alongside standard reference materials from NIST (SRM-610, SRM-612, SRM-690, SRM-679), and Corning glasses Brill-B and Brill-D. For NIST SRM and Corning glasses, we used reference values published by Brill and Rising (1999) and Pearce et al. (1997). NIST certified values were used for selected elements for SRM-690 and SRM-679.

Data were collected using a PerkinElmer SCIEX NexION 300 Quadrupole ICP-MS coupled with a Teledyne Instruments Inc. Analyte Excite HelEx 193 nm laser ablation system. We selected five 40 µm wide and 80 µm long ablation pass lines on each standard reference material, quality control, and unknown sample. After each ablation, the laser was paused for 25 s while the ICP-MS continued to collect signal intensity data. The laser moved at a rate of 5 µm/s firing laser bursts at a rate of 10/s. Laser power was set to 42.6% of the maximum output. The procedure bracketed ten unknown samples with a set of standards and quality control samples at the beginning and end of each to monitor instrument stability and drift throughout analytical runs (approximately every 30 mins). The ablated sample vapor traveled through tubing in a helium transport agent. It was mixed with argon gas at the ICP-MS torch, where the sample was ionized and passed through two detectors that measured the signal intensity in counts per second for 60 isotopes: ^{7}Li, ^{9}Be, ^{11}B, ^{23}Na, ^{24}Mg, ^{27}Al, ^{29}Si, ^{31}P, ^{34}S, ^{35}Cl, ^{39}K, ^{44}Ca, ^{45}Sc, ^{47}Ti, ^{51}V, ^{52}Cr, ^{55}Mn, ^{57}Fe, ^{59}Co, ^{60}N, ^{63}Cu, ^{66}Zn, ^{71}Ga, ^{75}As, ^{77}Se, ^{85}Rb, ^{88}Sr, ^{89}Y, ^{90}Zr, ^{93}Nb, ^{98}Mo, ^{107}Ag, ^{115}In, ^{118}Sn, ^{121}Sb, ^{133}Cs, ^{138}Ba, ^{139}La, ^{140}Ce, ^{141}Pr, ^{146}Nd, ^{147}Sm, ^{153}Eu, ^{157}Gd, ^{159}Tb, ^{163}Dy, ^{165}Ho, ^{166}Er, ^{169}Tm, ^{172}Yb, ^{175}Lu, ^{178}Hf, ^{181}Ta, ^{182}W, ^{197}Au, ^{205}Tl, ^{208}Pb, ^{209}Bi, ^{232}Th, and ^{238}U.

Switching the laser on and off for a pause of 25 s between scans formed a series of curves for each isotope. Each peak represents a single ablated spot, whereas the baseline between peaks serves as the sample blank. An Excel-based macro script subtracted the sample blank (the average of 5 baseline replicates before and after the ablation signal) from each replicate. The blank-subtracted replicates within each peak were summed and averaged. Anomalous peaks were identified and eliminated. The average data for element isotopes in counts per second were converted to a total elemental signal by comparing the signal intensity measured for each element in the sample for the same element measured in the standard reference materials. We used isotope Si^{29} as our internal standard. The standard signals are then referenced to published values for standard reference materials to calculate the *Ky* where

K is the conversion factor for element y.

$$Ky = \frac{Standardized\ signal\ for\ Y}{[Y]in\ the\ reference\ material} * [internal\ standard]in\ the\ reference\ material$$

The standard signal was then divided by the Ky and the sum of all elements is normalized to 100 percent oxide, as proposed by Gratuze and others (2001). Results for the sample unknowns are reported in Supplemental Information Table 1.

Raman spectroscopy

Samples were analyzed using a Bruker Bravo handheld Raman spectrometer. The instrument is equipped with two laser diodes (excitation wavelengths 785 and 852 nm), operates at a maximum (combined) laser output of 100 mW and a spot size of ~1 mm, and records signal intensity data at a spectral range of 300 - 3200 cm^{-1} at 10–12 $cm-^{1}$ resolution. One unique capability of the dual-laser system is sequentially shifted excitation (SSE), a technique which minimizes the effects of sample fluorescence on the resulting spectrum. It does this by operating the two lasers at sequentially shifted wavelengths (< 1 nm), during which the fluorescence bands and unwanted spectral artifacts remain at fixed positions in the spectrum while the positions of the Raman bands will shift slightly in response to the minor variations in excitation wavelength. During a single measurement, the instrument collects six independent interferograms that are subsequently compiled into a single calculated spectrum. This capacity enables the instrument software to then identify and subtract the unwanted fluorescence bands, resulting in significantly reduced spectral interferences. This capability is especially desirable for the analysis of materials with fluorescence-inducing components, which are commonly encountered in archaeomaterials analysis. See Jehlička and Culka (2021) and Culka and Jehlička (2019) for detailed evaluations of Bravo's SSE functionality.

The laser spot was targeted on the red pigmented areas with highest visible color opacity. Between 1–3 measurements were taken on each sample, and the samples were rotated after each assay. The resulting interferograms were imported into Bruker OPUS software, converted to a single composite spectrum, and underwent the following standard manipulations: Rubbberband baseline subtraction (64 baseline points) and smoothing (5 smoothing points) using the Savitzky–Golay algorithm (Savitzky and Golay 1964). Each of the six interferograms and the calculated single spectrum were manually screened to identify unwanted features that may have resulted in over- or under- subtraction of the baseline and the presence of signal saturation features. Once satisfied with the calculated spectra for each sample, the peaks were identified and compared to multiple reference spectra and databases to confirm mineral assignments. These reference databases include Bruker Art and Archaeology reference library, RRUFF project database (Lafuente et al. 2015), and Pigments Checker v.5 by Cultural Heritage Science Open Source (Caggiani et al. 2016). A summary table of peak identifications for all samples is provided in Supplemental Material Table 2.

Results

Hypotheses 1 and 2 – that Kitkahahki Town paint is similar to either Minnesota or Kansas pipestone – both appear to be false. Raman spectroscopy identified pyrophyllite (one of the distinguishing mineral phases in pipestones; Wisseman et al. 2012) in both the Minnesota and Kansas pipestone samples, but not in any of the archaeological samples. The absence of pyrophyllite in the archaeological samples conclusively rules out Minnesota and Kansas pipestone as the source of pigment. Both materials also differ chemically from the archaeological samples, as illustrated by a plot of the first two principal components of the element concentration data from LA-ICP-MS (Figure 5). All samples of Minnesota pipestone cluster together (Group 1) in the top left of Figure 5, away from the archaeological samples and the other possible pigment materials. Kansas pipestone also appears to separate from other materials, although we cannot describe its range of chemical variation with only two analyzed samples.

As noted above, there is no evidence that Minnesota pipestone was worked at the site, with all manufacturing debris identified instead as Kansas pipestone (Hadley 2023). It is possible that only relatively soft Minnesota pipestone was used as pigment, as described by Weltfish (1971), and that powder from coarser and more heterogenous Kansas pipestone was less desirable for this purpose.

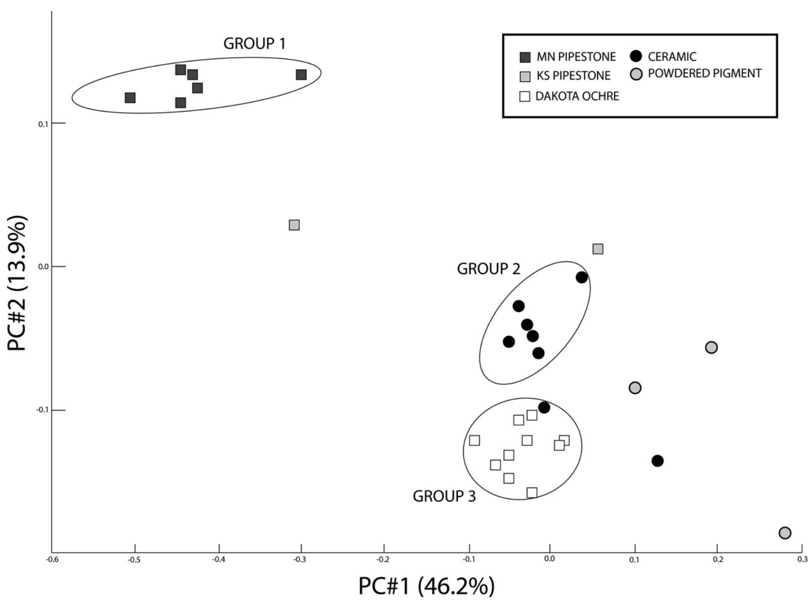

FIGURE 5. Scatterplot showing the results of a principal component analysis (PCA) of the LA-ICP-MS results. The samples compared here are Minnesota pipestone, Kansas pipestone, ochre collected from the Dakota formation, archaeological ceramics from Kitkahahki Town, and archaeological powdered pigment from Kitkahahki Town. PC1 (46.2%) is positively driven by the elements Zn, Cs, and Ni. Elements that drive the variation for PC2 (13.9%) include Nd, Sm, and Ba.

Hypothesis 3 – that Kitkahahki Town paint is similar to vermilion – is also rejected. There is no evidence in the LA-ICP-MS or Raman data of detectable amounts of vermilion (mercuric sulfide, HgS) or of two other commercial red pigments, red lead (minium, Pb_3O_4) or cadmium red (cadmium sulfoselenide, Cd [S]Se). The major Raman band for cinnabar (the mineral form of the pigment vermillion) at 1445 cm-1 was never observed in any of the archaeological samples.

Hypothesis 4 still appears viable – Kitkahahki Town paint may be similar to Dakota formation ochre. Both LA-ICP-MS and Raman data from archaeological samples support use of a clay-rich ochre, from the Dakota or another formation. LA-ICP-MS analysis reveals high aluminosilicate content (~20% Al_2O_3 and ~70% SiO_2), moderate Fe-oxide content (2.4-4.1%, enough to produce visible red pigment), and low amounts of minor trace elements found in earth minerals (K_2O, CaO, Na_2O, TiO_2) suggesting mixtures of Fe-oxides and clay. The Raman analysis identified minerals typical of an ochre mixture. For the Fe-oxide fraction, this includes hematite, some instances of goethite, magnetite, maghemite, and iron oxide with manganese impurities. For the non-Fe-oxide fraction, this is clear in the abundant presence of quartz, rutile/anatase, and Raman signals characteristic of aluminum-oxide and aluminum oxide stretching, bending, and deformation features, all of which are typical of clay minerals.

Limited evidence suggests some use of Dakota formation ochre in particular. All analyzed samples from the Dakota formation cluster in Group 3, regardless of ochre type (yellow shale, red clay, or iron oxide concretion) or processing type (unprocessed or levigated, fired or unfired). One archaeological paint sample from a sherd interior (MEB-27) also appears in Group 3, suggesting chemical similarity. Although the LA-ICP-MS data suggest that any of the Dakota ochre types could be the origin of this paint, the Raman data appear to rule out the yellow shale. The yellow shale has weak peaks for siderite ($FeCO_3$), common in low-grade, iron-rich sedimentary formations including the Dakota formation (Hattin and Siemers 1987) but entirely absent in the analyzed Dakota red clay and iron oxide concretions and from the archaeological samples.

All paint samples from Pawnee ceramics cluster in Group 2, with two exceptions (MEB-27, in Group 3 as noted above, and MEB-22). Surprisingly, none of the archaeological powdered pigments group with paint from vessel interiors. All three powdered pigment samples (and MEB-22) separate from each other and from the three identified groups (see Figure 5). This separation and the division between Groups 2 and 3 suggests that ochre collected at least from multiple locations, if not multiple geological formations, was used for pigment at Kitkahahki Town.

Discussion

Pipestone powder was not used as a pigment at Kitkahahki Town; it was found neither in vessel interiors nor as powdered pigment (see also Hadley 2023). Vermilion was not used either, despite residents' active engagement with the fur trade (Bozell and Latham 2023). Ochre is the only investigated material to remain plausible, including ochre from the Dakota formation within the boundaries of Pawnee sacred geography.

In his discussion of vermilion in Native North America, Lozier (2017:55) notes that "the imported pigment never entirely supplanted locally sourced ones." One of his possible explanations is price; even in the mid-nineteenth century, Schoolcraft (1851:234) describes vermilion as "generally too costly for habitual use" by Comanche people. Another is that "some groups. . . maintained relatively little contact with outside traders until the early twentieth century" (Lozier 2017:56). Ultimately Lozier (2017:56) argues that "some individuals and communities continued to prefer duller ochre. . . because they could control the ritual dimensions of its extraction and maintain a relationship with the supernatural forces that inhabited the landscape around them."

Even with the availability of vermilion, Pawnee people at Kitkahahki Town may have preferred ochre because of connections between earth pigments, Pawnee sacred geography, and relationships with other resources and other people. Many Indigenous communities in North America have complex, interdependent, and reciprocal relationships with landscapes and the biological and non-biological entities within (Basso 1996; Deloria 2003; Dylan and Smallboy 2016; Spillett 2021; Todd 2018). Connections to place – which anthropologists define as "framed space that is meaningful to a person or group over time" (Thornton 2008:10; see also Aucoin 2017) – may be expressed through earth materials such as pigment (Ancheta 2013; Munson and Hays-Gilpin 2020). In Pueblo communities in the U.S. Southwest, for example, "colorants were powerful materials, obtained from significant locations" (Munson 2020:25).

Relatively light and portable, pigments have been widely transported and traded in non-industrial settings throughout the world (Arnold 1985), including the Indigenous Great Plains (Zarzycka et al. 2019). To further explore the origins of pigment at Kitkahahki Town and other sites, we need a better understanding of ochre sources throughout the Plains and Midwest and the variation within and between them. The literature on chipped stone resources (e.g. Banks 1990, Ray 2007, Wyckoff 2005), which focuses on geological contexts rather than individual find locations, may provide a good model for documenting pigment materials in the midcontinent.

One formation worth further study is the Upper Cretaceous Niobrara Chalk to the west of Kitkahahki Town (Beck et al. 2022; O'Connor 2005). Around the turn of the twentieth century, the Indianola Ochre Mine in Red Willow County, Nebraska mined pigment and manufactured paint "from an 11-foot thick bed of ocher at a depth of 30 feet beneath the town. This bed is in weathered Niobrara" (Burchett 1991:2). This particular deposit may not have been an accessible ochre source without industrial digging equipment, and unfortunately the mine no longer exists (Sehnert 2009). It remains possible that other exposures of Niobrara Chalk contain accessible ochre.

Conclusion

> Paint was an article of trade between groups or individuals long before the white trader arrived. It was expensive, held in great respect, and cared for as carefully as gold dust. (Mable Morrow 1975:34)

We ultimately find no evidence of pipestone powder or vermilion as pigment at Kitkahahki Town and conclude that ochre is the most likely pigment material at the site. The ochre may originate from multiple locations, if not multiple geological formations, but at least some of it may come from nearby exposures of the Lower Cretaceous Dakota formation. As part of our ongoing research on Pawnee pigment use and regional ochre sources, we continue to sample the Dakota and other formations to better understand Plains pigment materials. Pigments are one important way that people embody their relationships to places.

Acknowledgements

This research was supported by grant NSF-1912776 awarded to the Archaeometry Lab at MURR and by funding from the Arts and Humanities Initiative at the University of Iowa to Margaret Beck. We thank Whitney Goodwin, April Oga, and Hannah Sears for their contributions, which include assistance with LA-ICP-MS sample preparation and data acquisition. We also appreciate feedback from *Plains Anthropologist* editor Bill Billeck and from two anonymous reviewers.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Funding

This work was supported by U.S. National Science Foundation: [Grant Number 1912776]; University of Iowa Arts and Humanities Initiative.

Notes on Contributors

Margaret E. Beck is Professor of Anthropology at the University of Iowa. Her research interests include households, cooking technologies, and raw materials for craft production. She has conducted ethnoarchaeological research in the Philippines, United States, and India. Her archaeological work includes studies of ceramic manufacture and use in the U.S. Great Plains and Southwest.

Brandi L. MacDonald is an archaeological scientist at the Archaeometry Laboratory at the University of Missouri Research Reactor. She specializes in provenance and materials characterization of pigments, glazes, pottery, and stone tools using various techniques in chemistry and physics.

Jeffrey R. Ferguson (University of Missouri, Department of Anthropology and the Archaeometry Laboratory at the University of Missouri Research Reactor) is an archaeologist focused on compositional analysis of archaeological materials to investigate many forms of social interaction and movement. Much of his current work focuses on large-scale exchange of ceramics and lithics in the American Southwest, particularly western New Mexico.

Mary J. Adair, PhD is Curator Emerita of Archaeology at the University of Kansas in Lawrence, Kansas. Her research foci include reconstructing diet from analyses of archaeological plant remains, documenting the rise and importance of agriculture, chronology and variations within Middle Woodland complexes of the central Plains, and early historic Pawnee occupations and response to European contact. She is active in the Plains Anthropological Society.

ORCID

Margaret E. Beck http://orcid.org/0000-0002-4591-4557
Brandi L. MacDonald http://orcid.org/0000-0003-2887-4351

Supplemental material

Supplemental material for this article can be accessed here: https://doi.org/10.1080/00320447.2022.2108601.

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Article received 8/2/2022; accepted 28/7/2022