



Iron oxide geochemistry in the Great Lakes Region (North America): Implications for ochre provenance studies



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ABSTRACT

Evidence for ochre use has been identified at archaeological sites in the eastern Great Lakes Region (North America) dating from the Paleoindian through to the Late Woodland periods. Yet, little is known regarding its procurement practices or if it was a component of established exchange networks. Addressing archaeological evidence for ochre provenance requires an assessment of the geochemical variability of Fe-oxide bearing deposits to determine if they can be differentiated on the basis of their chemical compositions, and if their chemical signatures satisfy the provenance postulate. We present the results of a geochemical characterization and multivariate statistical analysis of Fe-oxide deposits and ochre artifacts. Using Neutron Activation Analysis (NAA) and X-ray Diffraction (XRD), we determined the elemental and mineralogical characteristics of ten Fe-oxide sources in the Great Lakes Region. In addition, we analyzed and compared twenty-three ochre nodule artifacts from the Haney-Cook villages (BcHb-27) and the Ball village (BdGv-3) to determine if they could be geochemically linked to any of the source deposits. The results presented here demonstrate that the Fe-oxide bearing sources each possess unique geochemical signatures, and, that ochre artifacts recovered from the archaeological sites can be linked to source deposits or source *zones*. Inhabitants of Haney-Cook were most likely engaged in direct procurement of local Fe-oxides, while those at the Ball site used ochres that were acquired either by mid-distance (< 100 km) direct procurement, or intra-group exchange. Our findings illustrate the potential for ochre-based provenance studies in the Great Lakes Region.

1. Introduction

Ochre is commonly recovered from archaeological sites in North America, and evidence for its use dates as early as the Early Paleoindian period (12,000 years BP). One of the earliest documentations of ochre use for painted grave goods includes Clovis Anzick burials in Montana (Lahren and Bonnicksen, 1974: 148). The Lindenmeier Folsom fluted point habitation site in New Mexico produced 61 pieces of ochre exhibiting grinding facets, pigment grinding stones, and a carved turtle plastron disc with residual ochre paint (Wilmsen and Roberts, 1978: 132–134). Specific to the North American Great Lakes region, its applications include decoration for pottery and other portable objects, including Paleoindian quartz crystal fluted points (Roberts 1984: 251), as pigment for pictograph painting (Dewdney and Kidd, 1967), and in association with mortuary and ceremonial practices. Ochre nodules, sometimes referred to in the regional literature as “paintstones” or “crayons” (Clermont and Chapdelaine 1998: 92), are a commonly

identified artifact class in archaeology of the eastern Great Lakes area, and have been recovered from sites including Constance Bay (Savage, 1972; Watson, 1972), Martin-Bird (Dawson, 1987), Haney-Cook (Fox, 1979; Garrad, 2010), Ball (Fox, 2011), Rumrill-Naylor (Rumrill, 1985), and Witch Point (Gordon, 2013: 116). There is abundant evidence of iron oxide use in mortuary contexts spanning the Archaic Period (10,000 BP–2800 BP), including the use of ochre “crayons”. Caches or deposits of ochre have been identified in association with human or canine internments at the Mantle Site (Birch and Williamson, 2013: 153), Morrison Island-6 (Clermont and Chapdelaine, 1998: 92), Liahn II and Bruce Boyd (Spence et al., 1978; Spence and Fox, 1986; Williamson, 1980), Donaldson-1 (Spence et al., 1990), Cameron's Point, Picton (Walker, 2015: 43), Killarney Bay I (Greenman, 1953), and Michipicoten (Bishop and Smith, 1975: 60). At the Jacob Island site, chunks of ochre were concentrated in the cranial and pelvic regions of human burials, and ochre-stained deer bone elements were found (Conolly et al., 2014: 116, 125). At Lake Temagami and Frank Bay,

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Gordon (2013) describes ochre-covered canine burials, and Pearce (2005) describes painted turtle shell rattles.

This summary, which is by no means exhaustive, underscores the point that ochre use was widespread and sustained for over 12,000 years of human history in the eastern Great Lakes region. Despite its ubiquitous presence at archaeological sites, quarrying practices and provenance of ochre are poorly if at all understood. No systematic geochemical provenance study of Fe-oxide deposits or artifacts in the Great Lakes Region currently exists. However, a growing body of literature demonstrates the potential for ochre provenance research via elemental, mineralogical, and isotopic techniques (Dayet et al., 2016; Huntley et al., 2015; Kingery-Schwartz et al., 2013; MacDonald et al., 2011; MacDonald et al., 2013; Pradeau et al., 2016; Zipkin et al., 2017, and others). The purpose of this study was to determine if Fe-oxide bearing deposits in the Great Lakes Region possessed unique geochemical signatures that would satisfy the provenance postulate (Glascock and Neff, 2003; Weigand et al., 1977). Furthermore, we assessed if ochre artifacts from two archaeological contexts, Haney-Cook and Ball sites, could be chemically linked to source deposits. This study demonstrates the value of mineralogical characterization as a preliminary tool to sort categories of Fe-oxide types, and how phase analysis can inform and enhance the interpretation of elemental trends in Fe-oxides.

1.1. Terms and conditions: ochre, iron oxide, and provenance

It is important to emphasize here our use of certain terminology, including: ochre, pigment, iron oxide, and provenance. In literature on archaeology, art, and earth and materials sciences, the terms used to describe iron oxides are variable and inconsistent (Helwig, 2007:39). The term *ochre* is commonly understood in the archaeological literature as red or yellow pigment, yet more accurately encompasses 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 (Dayet et al., 2016; Henshilwood et al., 2009; Hodgskiss, 2010; Zipkin et al., 2017). 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. We recognize our present use of the term *ochre* to denote a cultural classification, used to broadly describe red, Fe-oxide bearing mineral or rock conglomerates found in archaeological contexts from which pigment could be produced. As such, our use of the term ochre also implies collection, modification, and intention on part of the individual(s) engaging with those minerals. Our study includes an assemblage of ochre artifact nodules, of which some show evidence of usewear (i.e. striations, facets, abrasion) which we associate with grinding on surfaces in the direct application of pigment, or grinding against an abrasive surface to collect the resulting powder.¹ Therefore, to be clear, in this paper we use the term Fe-oxide in reference to geologic source deposits, and the term *ochre* in reference to the Fe-oxide materials recovered from archaeological contexts. Finally, we use the term *provenance* in the spirit of the provenance postulate (Glascock and Neff, 2003; Weigand et al., 1977); that the elemental, isotopic, or mineralogical variability between sources or compositional groups must be greater than the variability within a source or compositional group.

2. Iron oxide sources: formation and mineralogy

Geologic samples from ten iron-oxide bearing deposits located in Ontario, Minnesota, New York, and Michigan were characterized by

NAA and XRD. The specimens were either field collected by one or more of the authors, or provided by researchers from existing reference collections. The sources were selected on the basis of multiple criteria, including ethnographic or historical reference to iron extraction (both pre- and post-dating European contact), their location in proximity to historically known trade routes in the eastern Great Lakes, and modern accessibility to source locations to gather reference material. Table 1 lists the sources by name and region, and Fig. 1 is a map showing their respective locations. Plate 1 is a series of macro- and micro-graphs of hand specimens from each source deposit, illustrating the diversity in colour, texture, luster, relative grain size, and mineral and fossiliferous inclusions. Table 2 summarizes the major and minor mineral phases identified by XRD (XRD raw data and spectra figures are available in SI Table 2 and SI Figs. 1–6). Based on the formation, diagenetic history, and mineralogy of the source deposits, we have loosely categorized them into three main types: marine-type, metamorphic/granitic-type, and iron-silicate type.

2.1. Ontario iron oxide sources

Mattawa River and Yorston River deposits are metamorphic-granitic type Fe-oxide, and are both located in the Southern Province of the lower Canadian Shield Geologic Complex (ON). Here, the surface geology is a patchwork of granite and granitic gneisses, and iron-rich ore deposits exposed most recently by the last glacial retreat (Douglas, 1969). The Mattawa River source is a red-brown-pink, angular and coarse-grained, consolidated conglomerate of iron-oxides and granitic accessory minerals. Its mineralogy is dominated by hematite (Fe₂O₃), and minor crystalline phases include K-feldspar (orthoclase [KAlSi₃O₈]), spessartine (Mn₃Al₂[SiO₄]₃), and zoisite (Ca₂Al₃O), all commonly associated with granite and granite-pegmatite rocks. Known locally as *Porte de l'Enfer* (Gates of Hell), the Mattawa deposit is located on a historically significant canoe route connecting the Ottawa River to Lake Nipissing (Tyyska and Burns, 1973: 32–48). Historic records indicate that it was a well-known sacred location and pigment source among local inhabitants prior to and at the time of European contact (Henry, 1809). Yorston River source is located approximately 40 km west of Lake Temagami on a portage route between Seagram and Long Lakes. In contrast to the nearby Mattawa source, it is finer-grained in texture and earthy in luster, with a muted orange-red-yellow hue. Its major mineral phases include hematite and quartz (SiO₂).

The Burnt Hills Fe-oxide outcrop, part of the historic Fourteen Island Lake Iron Mine, is located in the Frontenac Axis Region of the lower Canadian Shield. It is an iron-silicate type deposit, and is dominated by hematite and quartz with minor amounts of poorly crystalline iron oxide (Fe III) and iron-silicate phases. It is deep red-orange in colour, and has a coarse-grained, angular texture when homogenized with a mortar and pestle. The Lost Locale ochre outcrop is a thin, discontinuous bed of Fe-oxide, located north of Bobcaygeon (ON). Previously undocumented by official geologic surveys, it was incidentally discovered by Fox and P. LeBaron (Ontario Geological Survey) while searching for a nearby graphite source deposit. It is an angular, coarse-grained, red-pink conglomerate that is glossy, almost pearly, in luster. XRD confirms it is composed mainly of hematite, calcite (CaO₃), and dolomite (C₂CaMgO₆), with minor amounts clinocllore ([Mg₅Al][AlSi₃O₁₀[OH]₈]).

Osler Bluff outcrop is part of the discontinuous, Fe-oxide bearing shale component of the Cabot Head formation, which extends from southwestern Ontario into the Michigan Basin. This mid-Silurian formation was precipitated in a marine environment, and the outcrop of the Cabot Head shale at Osler Bluff is fossiliferous. It readily fractures in laminar sheets when moderate pressure is applied. It is purple-red in colour, and produces an orange-red streak. Specimens of Osler Bluff iron oxide were collected laterally across the deposit at approximately 500 m intervals where available at surface level. Hand samples that were collected from roughly the centre of the Osler Bluff formation

¹ Usewear analysis of the modified iron oxide nodules is presently in progress as a forthcoming thesis project led by co-author J.B.

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