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Identifying and sourcing *pyrometamorphic* artifacts: Clinker in subarctic North America and the hunter-gatherer response to a Late Holocene volcanic eruption

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ABSTRACT

Pyrometamorphic rocks produced by natural coal combustion appear at archaeological sites across North America but have received little archaeological attention regarding provenance studies. Tertiary Hills Clinker is a distinct pyrometamorphic rock from Subarctic Canada utilized by hunter-gatherers from 10,000 years ago to European contact. We employ X-ray diffraction, thin section analyses, and electron probe microanalyses to characterise Tertiary Hills Clinker and inform archaeometric studies of rock produced by combustion metamorphism. We geochemically compare pyrometamorphic rocks used by pre-contact people across North America to demonstrate that Tertiary Hills Clinker can be sourced using portable X-ray fluorescence. Results indicate that Late Pleistocene/Early Holocene exchange networks in North America were larger than previously thought. A later change in the distribution of Tertiary Hills Clinker may relate to a Late Holocene volcanic eruption (White River Ash east) that fragmented modes of lithic exchange and associated social networks with potential stimulus for a subsequent large-scale migration of northern hunter-gatherers across the continent. Provenance studies of pyrometamorphic artifacts offer untapped opportunities to study social networks in coal-bearing regions across the world.

1. Introduction

Pyrometamorphism (also called combustion metamorphism) generally occurs when coal, oil, or gas burn with sufficient energy to bake or fuse neighbouring rock (Allen, 1874; Bentor et al., 1981; Cosca et al., 1989; Grapes, 2011:21; Stracher et al., 2010). Beds of fused rock were targeted for stone tool production because of the raw materials' internal uniformity (Cinq-Mars, 1973; Clark, 1986; Curran et al., 2001; Fredlund, 1976). Pyrometamorphic rock has received geological attention in North and South America (Hefern and Coates, 2004; Henao et al., 2010), Europe (Žáček et al., 2015), Russia (Sokol et al., 1998), China (Song and Kuenzer, 2017), and Africa (Pone et al., 2017). Despite the global distribution of rock produced by coal combustion and the use of it by people, comparatively few efforts have been made by archaeologists to formally identify pyrometamorphic rock in archaeological assemblages (Estes et al., 2010; Hughes and Peterson, 2009; Le Blanc, 1997; Vapnik et al., 2015).

We use X-ray diffraction (XRD), thin sections, and electron probe microanalyses (EPMA) to characterise and identify a distinct pyrometamorphic rock called Tertiary Hills Clinker (THC) that was distributed over 1.25 million km^2 and utilized for over ten millennia in North America (Andrews, et al., 2012; Cinq-Mars, 1973). We geochemically compare pyrometamorphic rocks used by pre-contact people across the continent with portable X-ray fluorescence (pXRF) to verify

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provenance of THC and infer hunter-gatherer exchange networks from the Late Pleistocene to European contact. A volcanic eruption dated at 846–848 CE called the White River Ash east event (Jensen et al., 2014; Lerbekmo, 2008) may have severed social relationships between people in two major river basins in North America: the Mackenzie River that flows north to the Arctic Ocean and the Yukon River that flows north and west to the Pacific Ocean (Gordon, 2012; Workman, 1979). This may have provided the initial stimulus of one of the largest pre-contact migrations of people (Athapaskan) in the New World (Haskell, 1987; Ives, 2003, 2010, 2014; Magne and Matson, 2010; Moodie et al., 1992; Seymour, 2012).

2. Pyrometamorphism and North American clinkers

Pyrometamorphism by coal combustion typically occurs near geological fracturing that exposes flammable strata to oxygen (Cosca et al., 1989). Natural fires spread horizontally along exposed coal seams and can burn underground for over 100 m. Pyrometamorphism products range from thermally altered but unmelted rocks (dubbed burnt or baked rocks), partially fused rocks (termed clinker), or totally melted rocks (called paralava or slag) (Grapes, 2011). Porcellanite is a specific type of clinker formed from shale or siltstone that is heated near the point of melting; the rock recrystallizes (sinters) and takes on a ceramic or porcelain texture (Hefern and Coates, 2004). The term porcellanite has been used in other geological contexts, e.g., formation as a siliceous duricrust (McNally et al., 2000), but in North America it is generally limited to pyrometamorphic origins with major outcrops on the Northern Plains of the US (Fig. 1). Flaked porcellanite artifacts dominate some Holocene assemblages in Montana and Wyoming (Clark, 1985; Fredlund, 1976). A related pyrometamorphic rock called non-volcanic natural glass (NVNG) was also used in pre-contact times on the US Northern Plains (Frison, 1974; Hughes, 2007a). In addition to coal sources, clinkers can also form from combustion of carbonaceous sediment like Cape Bathurst Clinker (CBC) (Mathews and Bustin, 1984) that outcrops in the Canadian Arctic (Fig. 1). CBC was used for several thousand years by coastal hunter-gatherers (Le Blanc, 1991). Hefern and Coates (2004) note that clinkers vary due to: 1) grain size and mineralogy of parent rock; 2) degree of heat alteration; and 3) degree of oxidation or reduction during and after heating. Different thermal regimes can create a diverse array of clinkers within a single outcrop.

Outcrops of THC are 30 km west of the second largest fluvial system in North America – the Mackenzie River of Canada's Northwest Territories (Fig. 1). Subarctic hunter-gatherers quarried THC from approximately 10,000 years before present to European contact (Andrews et al., 2012; Hanks and Pokotylo, 2000). Specifically, THC has been reported in association with a Late Pleistocene fluted point in Alberta (Bereziuk, 2016), middle Holocene microblades in Yukon and Northwest Territories (Andrews, 1999; Clark, 1986; Le Blanc, 1997), and Late Holocene copper in Yukon (Thomas, 2003) (Fig. 2). Like other clinkers used by hunter-gatherers in North America, little archaeological research has been published about identification, provenance, and overall significance of THC. In this study, we present summaries of XRD, thin section investigations, and EMPA to support the pyrometamorphic origin of THC prior to discussion in the remainder of the paper of our pXRF provenance results and their implications for detecting human responses to a Late Holocene volcanic eruption.

3. Identification of Tertiary Hills clinker

Vitreous rocks produced by coal combustion are particularly easy to

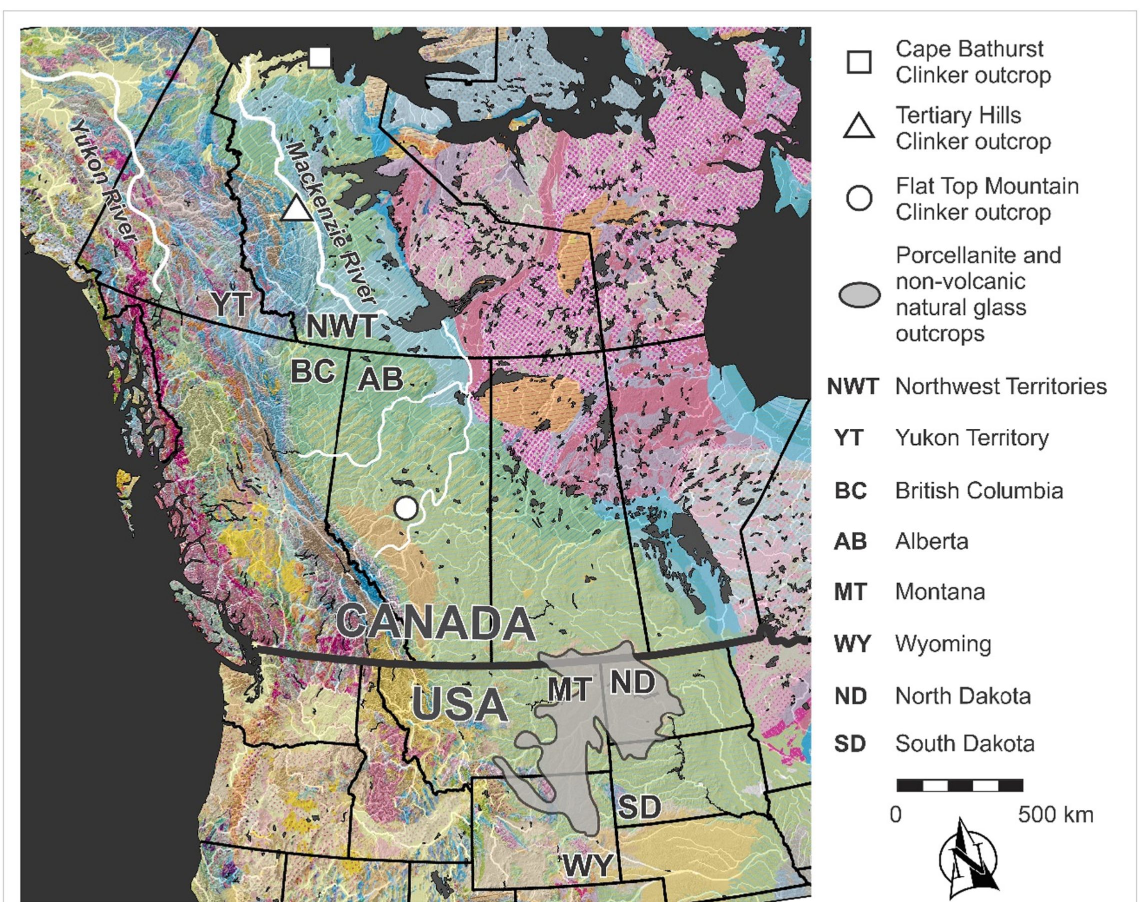

Fig. 1. Outcrops of recorded vitreous clinkers in Canada and northern United States discussed in text (bedrock geology data from USGS, 2014).

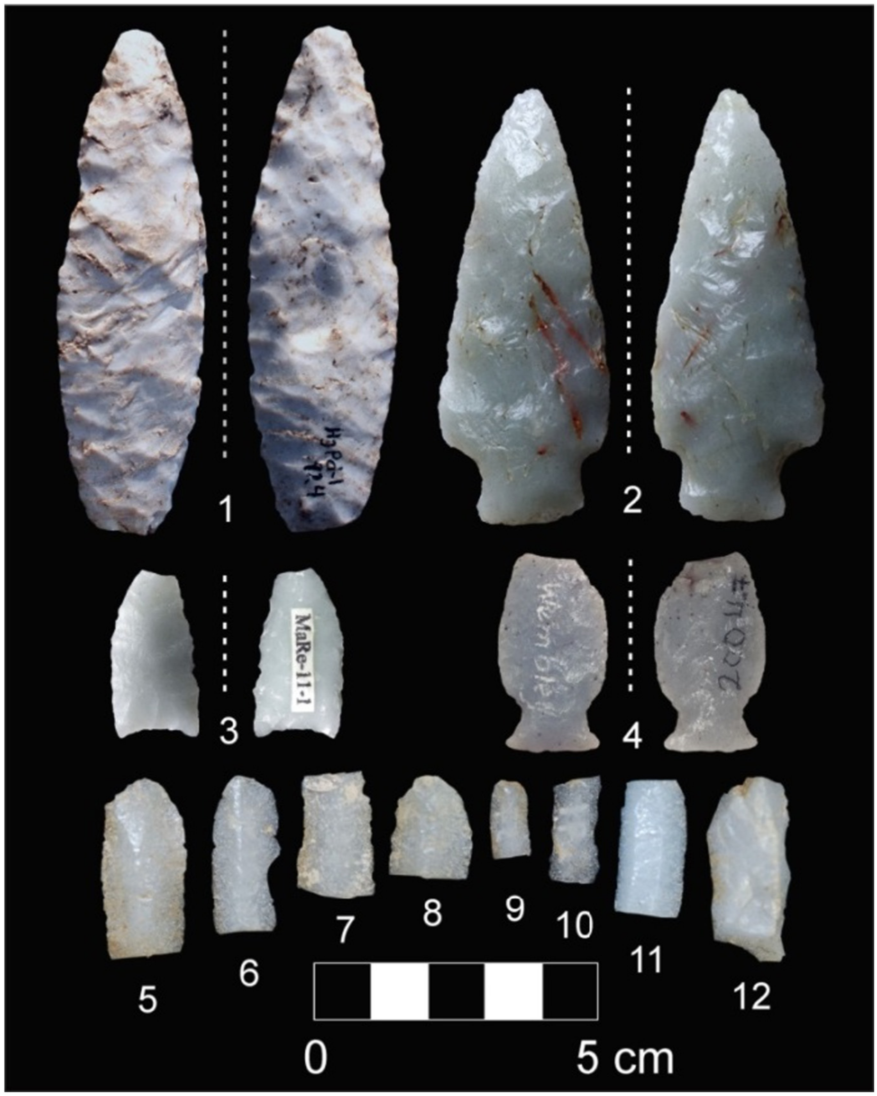

Fig. 2. Tertiary Hills Clinker artifacts. Specimen 1: HjPd-1:924, from northern Alberta (Royal Alberta Museum); Specimen 2: No catalogue number, from north central Alberta (Athabasca Archives); Specimen 3: MaRe-11:1, from western Northwest Territories (Prince of Wales Northern Heritage Centre); Specimen 4: 2004.7, from northwest Alberta (Grande Prairie Pioneer Museum); Specimens 5–12: KaVa-3:25–28, 56–57, 91, 102 (Yukon Heritage Branch).

overlook or misidentify in knapped stone tool assemblages because of superficial similarities to non-pyrometamorphic materials such as chert, quartzite, quartz, obsidian, or chalcedony (Hughes, 2007b; Hughes and Peterson, 2009; Kristensen et al., 2016). Tertiary Hills Clinker (THC) was variously called Keele River Obsidian (MacNeish, 1954:248), ignimbrite (a welded pyroclastic flow) (Millar, 1968), Tertiary Hills Welded Tuff (presumed by Cinq-Mars (1973) to be a volcanic ash welded by subsurface contact with magma domes), and Tertiary Hills Tuffaceous Clinker (ash fused by naturally ignited coal seams) (Pokotylo and Hanks, 1989). Systematic analyses to support the identification of THC have not been published and uncertainty persists concerning its parent materials and formation processes. Additionally, a lack of archaeological knowledge of THC, and clinkers in general, has limited reconstructions of their significance in North America.

THC is white, grey, brown, or purple (Kristensen et al., 2016) and varies from translucent to opaque with vitreous to glimmering lustre (Ives and Hardie, 1983). Cobbles display variable degrees of fusion, often with cracks and cortices reddened by oxidation, which is comparable to other North American vitrified clinkers (Fig. 3). Artifacts made from THC are typically a uniform white variety that has been informally described as glassy with a powdered sugar texture. A general diagnostic trait of clinkers is a high density of polydisperse circular vesicles (from $< 10\,\mu m$ up to 2 mm in diameter) produced by gas trapped during combustion (Table 1 and Fig. 4). THC fractures in a similar fashion to obsidian and was presumably highly sought after because of its unique appearance, hardness, and excellent workability (Hanks, 1993).

3.1. XRD materials, methods, and results

X-ray diffraction (XRD) is used to identify crystalline material based on the pattern produced by the elastic scattering of monochromatic radiation by the crystal structure of the material (Calvo Del Castillo and Strivay, 2012). Identifications are made by comparison of the experimental pattern to a database of diffraction patterns of known materials. Three samples of THC (two artifacts from Alberta and one natural outcrop piece) were ground to fine powders with an agate mortar and pestle, and XRD patterns acquired using Bragg-Brentano parafocussing reflection geometry with a Rigaku Ultima IV θ-θ diffractometer. This instrument has a Co X-ray source ($K\alpha$ 1.78899 Å) and Fe filter and was operated at 38 kV and 38 mA. The detector was a 1D silicon strip (D/tex Ultra). Each diffraction scan was run from 5 to 90° 2θ in continuous mode with a step size of 0.02° 2θ, and a count time of 0.6 s per step.

XRD results indicate that THC is heavily dominated by non-

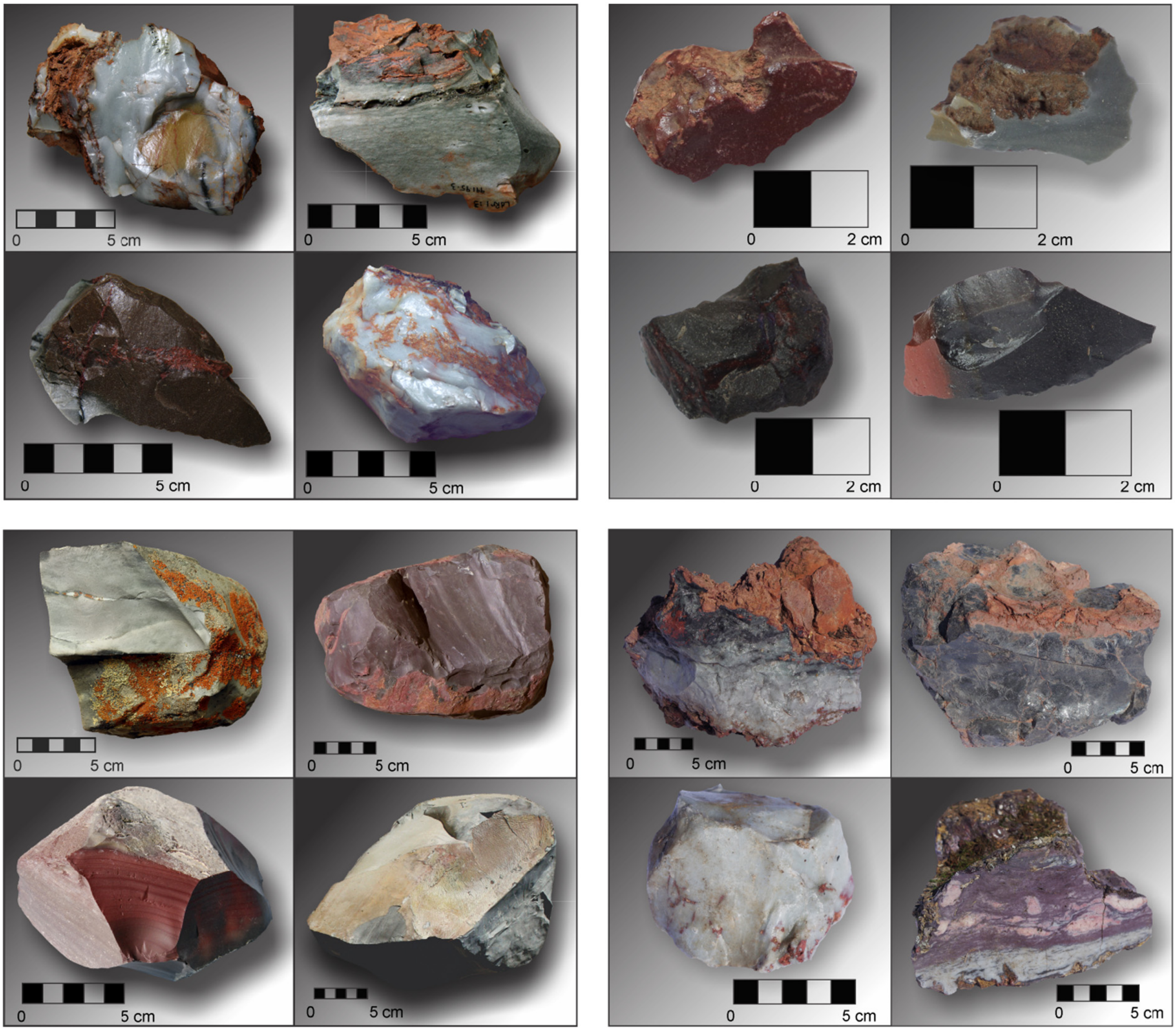

Fig. 3. Unmodified clinker cobbles. Top left quarter: Tertiary Hills Clinker, from top left clockwise: LcRq-7:45, LdRr-1:3, LcRq-7:38; and uncatalogued specimen likely from LdRq-3, Prince of Wales Northern Heritage Centre, Yellowknife, Northwest Territories. Top right quarter: Non-volcanic natural glass from southeast Montana, specimens uncatalogued, courtesy of Craig Lee. Bottom right quarter: Flat Top Mountain Clinker, specimens uncatalogued, Archaeological Survey of Alberta, Edmonton, Alberta. Bottom left quarter: porcellanite from southeast Montana, specimens uncatalogued, images courtesy of Jim Miller, Patrick Rennie, and James Keffer.

Table 1
Proportions (%) of vesicles and inclusions in Tertiary Hills Clinker samples from back-scattered electron images determined with ImageJ.

Sample	KfTd-3	GbPt-11	HhoU-113
Vesicles (void space)	1.9	2.6	2.7
Dark (in BSE) inclusions	2.3	12.8	3.8
Bright (in BSE) inclusions	0.1	0.2	0.2
Matrix glass	95.7	84.4	93.3

crystalline amorphous material with minute amounts of cristobalite, quartz, and mullite (a needle-shaped aluminosilicate mineral characteristic of high-temperature non-volcanic conditions) (see Cosca et al., 1989, and Clark and Peacor, 1992 for comparative mullite detection in pyrometamorphic rock). The presence of amorphous material (glass) explains why THC was used as a toolstone: it fractures conchoidally with sharp edges. XRD results support tentative identifications of several minerals that may have existed in the parent sedimentary rock (e.g., muscovite, dolomite) or were produced as a result of pyrometamorphism (e.g., olivine) or weathering (e.g., rozenite). The XRD results support an origin of THC via fusion of sedimentary rock from the combustion of coal seams.

3.2. Thin section materials, methods, and results

Samples of THC, porcellanite (found mostly in the Powder River Basin of Montana and Wyoming), and NVNG (also found across southeast Montana and northeast Wyoming) were cut, ground, and polished for thin section viewing. The behaviour under cross-polarized light indicates that THC, like NVNG and porcellanites, is composed primarily of isotropic material, interpreted to be amorphous (glass) (Fig. 5). THC lacks any remnants of internal bedding, which are often visible in NVNGs, and has generally undergone more thorough fusion and vitrification than porcellanites. NVNG vesicles tend to be more elongate than THC, which suggests that NVNG experienced minor flow during formation whereas THC did not appear to undergo lateral movement. Porcellanite is more uniformly opaque indicative of less intense thermal alteration during formation (Fredlund, 1976). The thin section results support the formation of THC in close proximity to an underlying coal bed as opposed to the more mobile chimney structure (i.e., gas vent) associated with the formation of NVNG (dubbed a glassy paralava by Cosca et al., 1989). Microscope images (Fig. 6) help

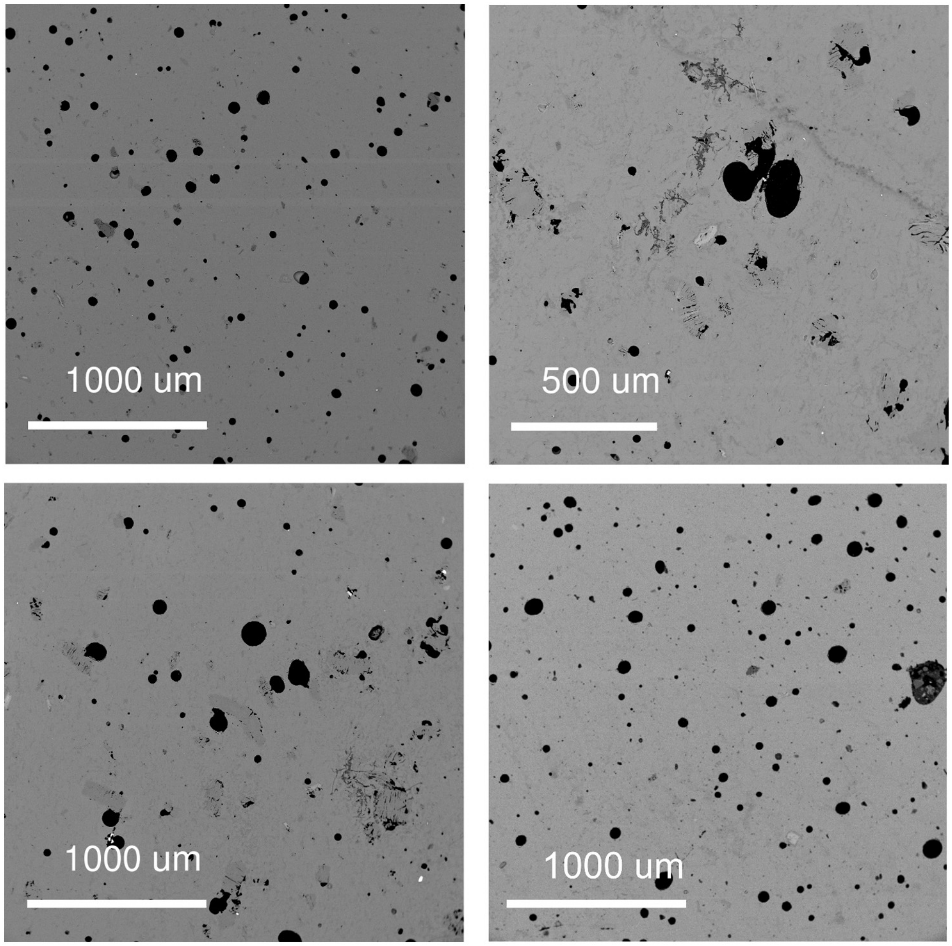

Fig. 4. Back-scattered electron images of THC. Vesicles are shown as black. Inclusions are either darker or lighter in grayscale than the medium grey glass matrix. Top left: KfTd-3; top right: GbPt-11 area 1; bottom left: GbPt-11 area 2; bottom right HhOu-113.

illustrate different thermal regimes and the influence of different parent materials in the formation of North American clinkers. Porcellanite, THC, and NVNG derive from coal combustion of fine-grained sedimentary rock while CBC is a more heterogeneous material formed from combustion of carbonaceous sediment.

3.3. EPMA materials, methods, and results

An electron probe microanalyzer (EPMA, or electron microprobe) uses a high-voltage focussed electron beam to generate characteristic X-rays in a polished sample. The intensity of these X-rays is measured with wavelength dispersive spectrometers and converted to elemental abundances with respect to standard materials after correction for matrix effects (Potts, 1987). The electron beam can be focussed to < 1 μm, which permits separate examination of matrix material vs. inclusions. Fig. 4 depicts back-scattered electron images (BSE) acquired with a JEOL 8900R electron microprobe in beam-scan mode with a focussed electron beam operated at 20 kV and 10 nA beam current. The back-scattered-electron signal is proportional to the mean atomic number of the material analysed: materials with more heavy elements will therefore appear brighter in such images (Lloyd, 1987). Analysis of the BSE images using the program ImageJ (Schneider et al., 2012) yields the area (volume) percentages of vesicles, dark- and bright-inclusions, with respect to the glass matrices (Table 1).

A total of 88 points in the glassy matrices and 22 inclusions were selected for more detailed examination from three round samples of THC fragments. A JEOL 8900R electron microprobe operated at 20 kV and 10 nA with a beam diameter of 10 μm was used for analysis of the glass matrices (Table 2). Count times for wavelength-dispersive spectrometry were 30 s on peaks and 15 s on backgrounds for the $K\alpha$ lines of: Si, Ti, Al, Cr, Fe, Mn, Mg and K, whereas conditions of 40 s on peak and 20 s on backgrounds were used for Na $K\alpha$.

Following Grapes (2011), liquidus temperatures (the temperature at which the material would have been completely molten) were calculated for the average anhydrous glass compositions at 1 bar (1 atm) pressure using the Excel spreadsheet rhyolite-MELTS v1.0 (Gualda and Ghiorso, 2015). The glass compositions are reminiscent of high-K rhyolites (Table 2). The solid inclusions in the glasses were identified as sekaninaite (the iron analog of cordierite, Grapes et al., 2011), metakaolin (Sperinck et al., 2011), feldspar, and silica.

Clinker melting temperatures range in general from 400 °C to 1600 °C (Grapes, 2011); the presence of metakaolin and sekaninaite in THC, along with the calculated liquidus temperatures of glasses, suggest melting between 800 and 1130 °C. Studies of coal reflectance in the Tertiary Hills (Sweet et al., 1989) produced Ro random values of 0.39 to 0.59 indicating ranks ranging from lignite to high volatile-C bituminous coal, which would be capable of high temperature combustion.

This assemblage of phases, in conjunction with the high calculated liquidus temperatures of the glasses, is consistent with a pyrometamorphic origin for THC (Grapes, 2011), as opposed to an igneous origin as a tuffaceous rock. Therefore, THC most likely formed through the combustion of coal and resultant pyrometamorphism of surrounding shale or mudstone parent materials.

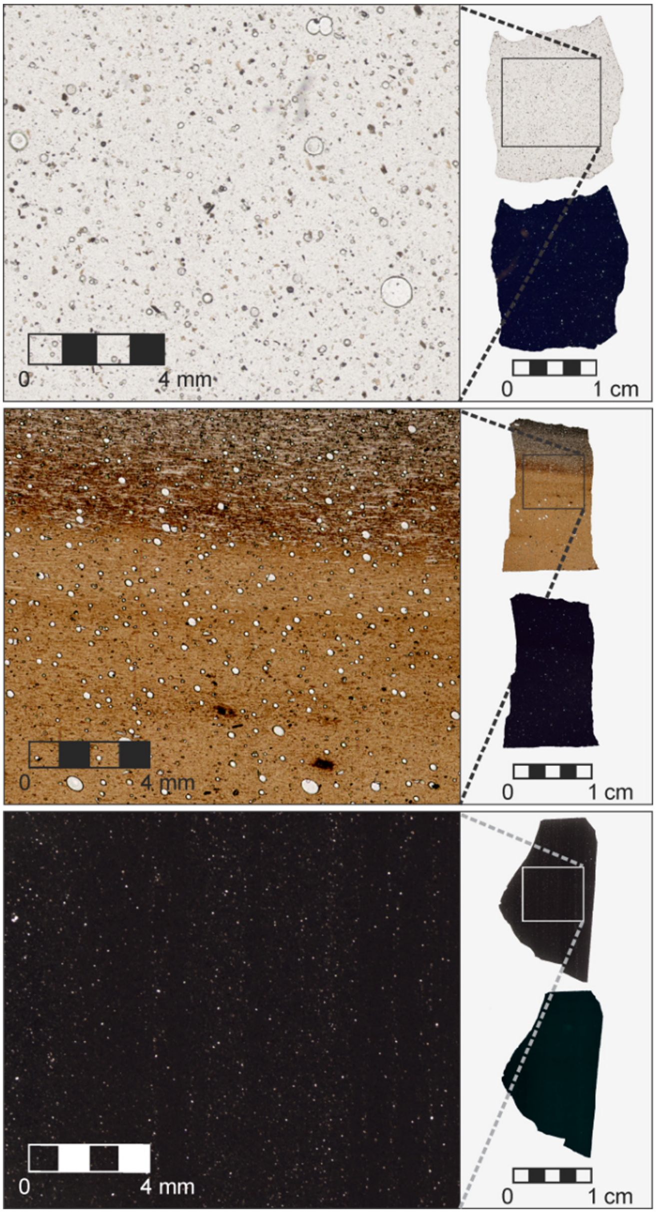

Fig. 5. Thin sections of THC (top), NVNG (middle), and porcellanite (bottom). At right are images of each thin section flake under normal light (top) and cross-polarized light (bottom).

4. Geological origins and formation processes

We here combine XRD, thin section, and EPMA results with field studies to infer geological origins and formation processes of THC, which have archaeological implications both in terms of pre-contact hunter-gatherer exploitation of localized outcrops and the ability to perform provenance studies on clinkers. THC outcrops in the Summit Creek Formation (Fig. 7): a roughly 3000 km^2 Late Maastrichtian (Late Cretaceous) to Paleocene (roughly 66 to 53 mya) succession of conglomerate, sandstone, ash beds, carbonaceous shale, and low grade coals (Fallas et al., 2013; Sweet et al., 1989; Yorath and Cook, 1981). The Summit Creek Formation formed as an alluvial fan that was subsequently uplifted, folded, and faulted during several phases of the Laramide Orogeny (Sweet et al., 1989; Yorath and Cook, 1981). Sections of the Summit Creek Formation contain small lens-like bodies (< 20 cm thick) of THC surrounded by baked, red siliceous mudstone (Fig. 8) (Hanks, 1993; Yorath and Cook, 1981). XRD, EPMA, and thin section results (e.g., spherical vesicles) are consistent with field evidence that the parent material of THC is clay or mudstone shale. Neither stratigraphy nor laboratory analyses suggest any evidence of formation of THC from contact with volcanic ash or magma (as suggested by Cinq-Mars, 1973; Millar, 1968; and Pokotylo and Hanks, 1989).

Coal and bituminous sediments associated with clinkers are typically exposed by stream cutting or glacial activity (Grapes, 2011). The Tertiary Hills experienced numerous cycles of glaciation and scour (Duk-Rodkin et al., 1996) that may have also exposed coal beds to some form of ignition (e.g., forest fires). While clinkers in Montana and Wyoming formed 4 mya (Hefern and Coates, 2004), the metastable nature of the THC minerals detected by EPMA suggests formation in the last 12,000 years. During in situ coal burning, active smoke vents would be visible and a likely source of curiosity to Holocene tool makers. Smoking vents (bocannes) are thought to have similarly attracted pre-contact people to Cape Bathurst Clinker outcrops (Le Blanc, 1991). The Shuhtagot'ine Dene First Nations, whose traditional territory encompasses the Tertiary Hills, are well aware of modern active coal burns and interpret them to be remnants of burning fat that dripped down from a giant beaver killed by culture hero Yamǫ́ria (Blondin, 1990).

Archaeometric and field studies suggest that THC is a more localized and uniform material compared to widely distributed and variable outcrops of porcellanite and NVNG (Fig. 1). These latter materials form in complex chimney and bed structures across the US Northern Plains (Cosca et al., 1989; Hefern and Coates, 2004) with a predictably broader variety of parent materials and combustion dynamics. Spatial confinement of THC outcrops to a comparatively small area, and the likelihood of greater geochemical consistency compared to US clinkers, make THC a candidate for provenance studies.

5. Sourcing

5.1. PXRF materials and methods

PXRF analyses were conducted to determine if different clinkers have distinct geochemical signatures that could lend reliable quantitative support to connections drawn between clinker artifacts and outcrops. XRF can provide a geochemical summary of the elements present within individual specimens and their respective concentrations. When employed on silica-rich rocks like clinkers, pXRF is particularly effective at detecting and quantifying concentrations of the elements Mn, Fe, Zn, Ga, Rb, Sr, Y, Zr, Nb, and Th (Glascock et al., 1998; Speakman et al., 2011).

THC artifacts from Northwest Territories ($n = 45$), Alberta ($n = 10$), and Yukon (n = 10), and 35 raw outcrop samples from the Tertiary Hills were analysed along with samples of Montana porcellanites (n = 10), CBC ($n = 7$), and Montana NVNG (n = 10). Twelve samples of clinker from Flat Top Mountain in north central Alberta (FTMC) were also included for comparative geochemistry although there is currently no evidence that it was quarried in pre-contact times. In addition, samples of quartzite, quartz, chalcedony, and other materials that may superficially resemble clinkers ($n = 20$ in total) were analysed to assess whether or not they can be differentiated by pXRF.

XRF analyses were completed using a Bruker AXS Tracer III-SD handheld spectrometer attached to a laptop computer running Bruker software S1PXRF. The Bruker AXS Tracer III-SD instrument is equipped with a Rh X-ray tube and a 10 mm^2 Silicon Drift Detector (SDD) with a resolution of 145 eV FWHM for 5.9 keV X-rays. To optimize determination of elements of interest, a Bruker AXS excitation filter (comprised of 0.1523 mm Cu, 0.0254 mm Ti, and 0.3047 mm Al) was used. Data were collected for 300 s live-time count periods with the device set at 40 kV and 30 µA. Manganese, Fe, Zn, Ga, Rb, Sr, Y, Zr, and Nb were quantified via their $K\alpha$ X-ray emissions, while Th was determined using its $L\alpha$ X-rays. The proprietary obsidian calibration supplied by Bruker AXS was employed for THC elemental analyses. Speakman et al. (2011)

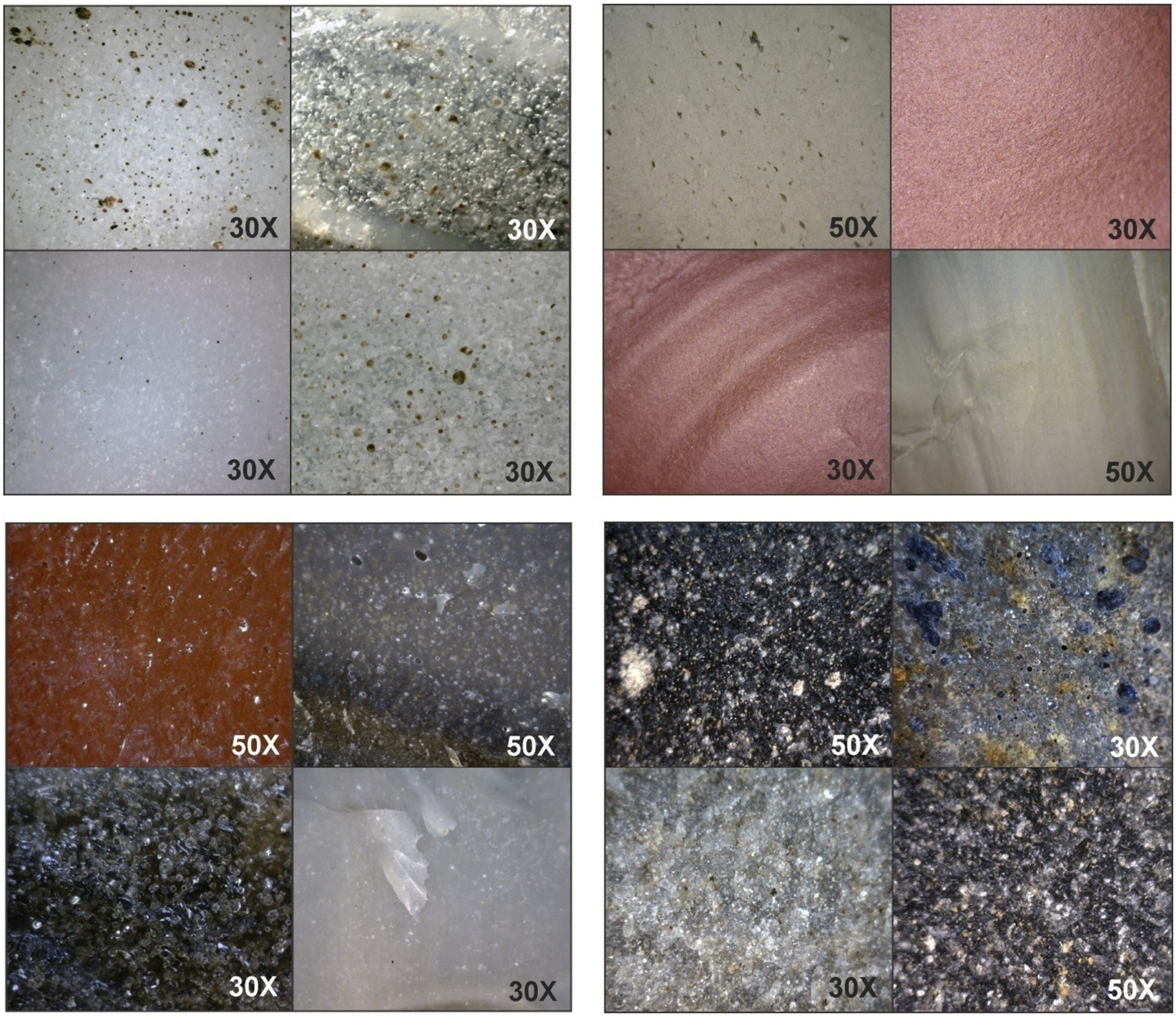

Fig. 6. Microscope images and magnification; THC (top left), Montana porcellanites (top right), CBC (bottom right), and Montana NVNG (bottom left). Porcellanite provided by Jason Roe, NVNG provided by Craig Lee.

Table 2
EPMA average analyses of the glass matrices of three THC samples. Cr was sought but not found above the limit of detection. Mean weight-percent compositions are listed with standard deviations in brackets.

Sample	KfTd-3	GbPt-11	HhOu-113
# of points	28	31	29
SiO_2	78.22 (0.19)	76.98 (0.85)	77.85 (0.32)
TiO_2	0.06 (0.02)	0.07 (0.01)	0.06 (0.01)
Al_2O_3	12.77 (0.10)	13.16 (0.45)	12.62 (0.09)
FeO_{total}	1.03 (0.04)	1.25 (0.10)	0.94 (0.08)
MnO	0.05 (0.01)	0.04 (0.01)	0.05 (0.01)
MgO	0.04 (0.01)	0.09 (0.02)	0.03 (0.02)
CaO	0.53 (0.02)	0.89 (0.10)	0.48 (0.02)
Na_2O	0.46 (0.03)	1.62 (0.08)	0.45 (0.04)
K_2O	6.56 (0.12)	5.09 (0.33)	6.63 (0.14)
Total	99.72 (0.19)	99.19 (0.34)	99.11 (0.32)
Liquidus °C (1 bar)	1126	1079	1128

found the obsidian calibration gave relatively accurate results for the analysis of ceramic data, which they considered reasonable because obsidian and pottery are silica-rich materials. Given the expected elemental similarity between THC and obsidian, we deemed it reasonable to use the obsidian calibration in this study. Furthermore, pXRF analysis of a powdered sample of NIST 278 (obsidian), NIST 2710a (soil), and the USGS rock reference materials RGM-2 (rhyolite), QLO-1 (quartz latite), and GSP-2 (granodiorite), for quality assurance purposes, gave results for the elements listed above in good agreement with their certified or recommended values.

5.2. PXRF results

The analysed clinkers were determined to have distinct but variable elemental signatures (Table 3 and Appendix A), which is predictable given that different thermal regimes create a wide variety of pyrometamorphic rocks within a single outcrop (Grapes, 2011:30). High levels of iron and sulphur (not quantified in this study) are likely due to the presence of pyrite (FeS_2) from coal combustion; Sweet et al. (1989) found that the average sulphur content of coal in the Summit Creek Formation was 0.6%. We found that clinkers in general contain levels of Zr, Rb, Sr, and Y, and other trace elements commonly associated with coal-baked origins (although comparable concentrations can occur in volcanic materials). The relatively high Rb concentration also points to an original shale or mudstone (argillaceous) precursor of THC. As in obsidian-sourcing studies, trace element comparisons including Rb, Sr, Zr, Ga, Y, Th, and Mn are useful for differentiating THC from similar-looking materials, as well as other clinkers (Figs. 9–10 and Table 3).

The majority of THC artifacts from Northwest Territories and Yukon fall within 95% confidence ellipses of several element bivariate plots of THC outcrop material (Figs. 9-10). NVNG and porcellanite from

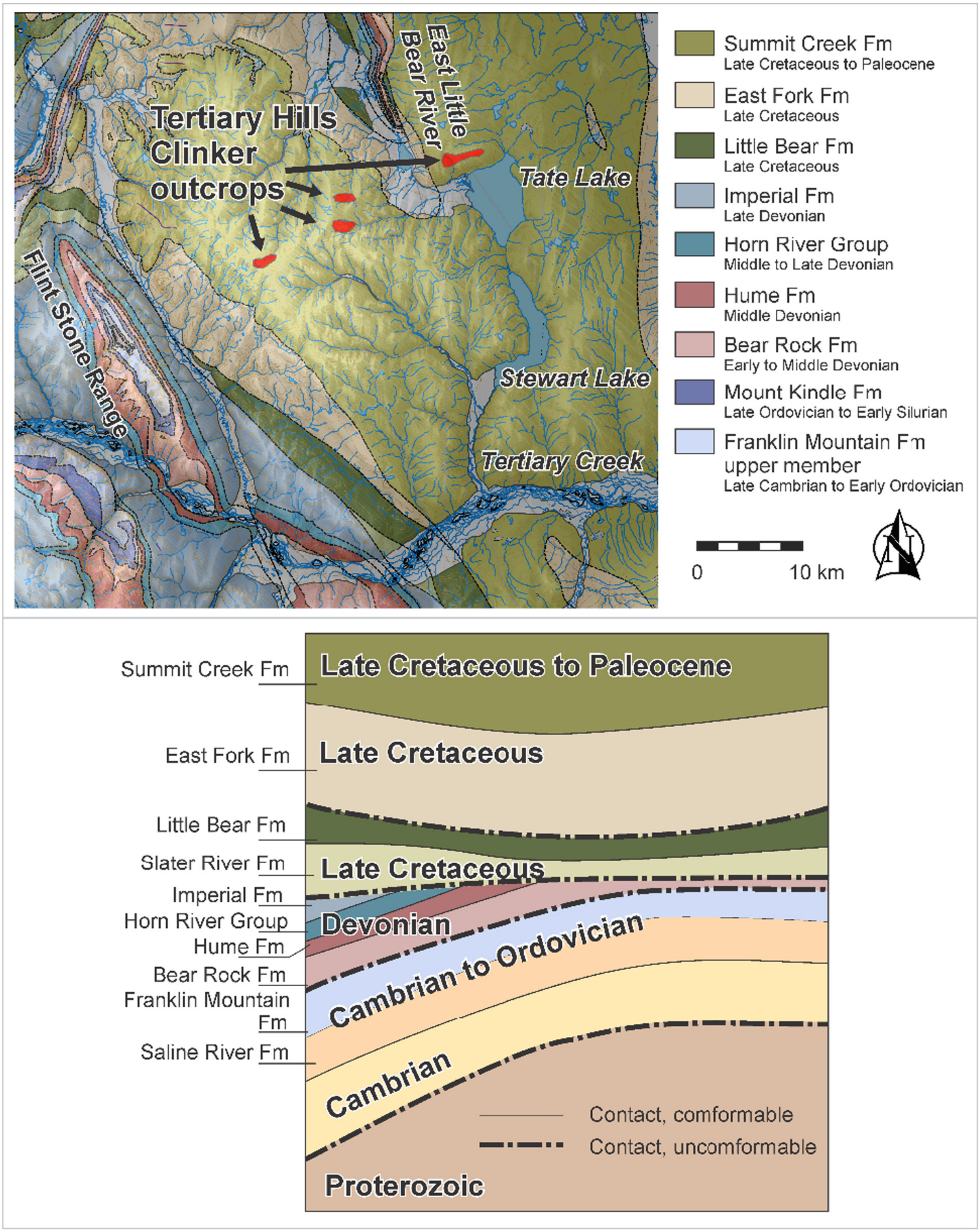

Fig. 7. THC outcrops (top) in the Summit Creek Formation. An adapted schematic stratigraphy of bedrock including the Summit Creek Formation (from Fallas et al., 2013).

Montana and CBC from the Mackenzie Delta consistently plot outside the geochemical variability of THC and can be distinguished by pXRF. The relatively tight spatial clusters of CBC, porcellanites, NVNG, and FTMC are supportive of future sourcing work with these clinkers. On a broader level, pXRF offers rapid and non-destructive means to quickly distinguish clinkers from similar-appearing materials such as quartzites and chalcedonies.

A plot of Ga vs. Sr/Nb indicates that some Yukon, Alberta, and Northwest Territories artifacts are beyond the 95% confidence ellipse of THC outcrop material suggesting that the outcrop samples analysed in this study may not capture the full variability of these elements and that these elements may be of future utility to distinguish particular THC outcrops. Purported THC artifacts in central Alberta (GbPt-11 and GfPt-3) exhibit levels of Mn, Sr, and Ga outside the variability displayed by

Fig. 8. THC outcrops are found in the Summit Creek Formation visible here as the upper reddened strata in this exposure, people at centre for scale (image courtesy of David Pokotylo).

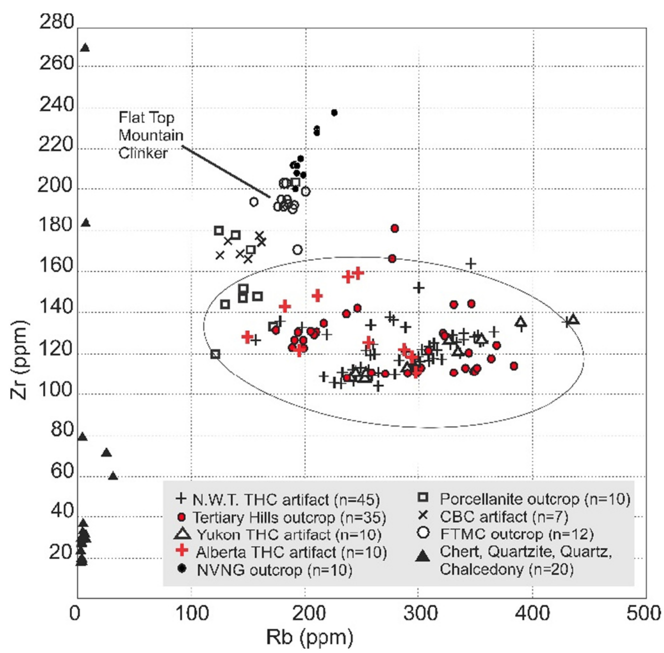

Fig. 9. Bivariate plot of element concentrations with 95% confidence ellipse around THC outcrop material.

THC outcrop material and could indicate that this clinker was quarried from a particular outcrop in the Tertiary Hills that was not captured in this study. Variability in the detected elements is to be expected considering the variability in source rock over the distribution of THC. Lastly, Fe should be interpreted with caution: it is suspected that two THC outcrop samples yielded abnormally high Fe levels due to the presence of cortex within areas of the samples analysed by pXRF. The cortex covering THC tends to consist largely of iron oxides. Results of the pXRF analysis indicate that clinker artifacts across Subarctic North America can be reliably differentiated from one another, and can be sourced to THC outcrops in Northwest Territories.

6. Archaeological significance

6.1. Distribution

THC artifacts were transported 600 km both east and west of Tertiary Hills, 530 km to the north, and 1200 km south of their outcrops with a total area that encompasses roughly 1.25 million km^2 (Figs. 11 and 12). In comparison, we estimate that Edziza obsidian artifacts from northern British Columbia extend 1.5 million km^2 into Yukon and northern Alberta (Potter et al., 2017; Woywitka, 2017) while Knife River Flint from North Dakota extends 3.7 million km^2 across the Northern Plains into Alberta (Ahler, 1986:105; Kristensen et al., 2018). A particularly wide distribution across irregular and forested terrain (not known for ease of human movement) likely relates to THC's proximity to the second largest river network in North America.

6.2. Frequency

THC is comparatively rare but when it does occur, it is often a significant portion of site assemblages (Table 4). Roughly one quarter of all THC-bearing sites in Northwest Territories contain a THC tool (THC tools comprise roughly 6.5% of site assemblages with THC) but it most commonly occurs as small bifacial reduction or tertiary flakes indicating that THC generally moved in the form of curated tools. Tool to debitage ratios do not differ significantly with distance from the source. A similar pattern prevails in Alberta and Yukon where nine of the 28 sites with THC have assemblages in which the only THC artifact recovered is a curated tool. All THC debitage recovered beyond Northwest Territories is late stage debris. THC is relatively brittle and would not be a suitable raw material for repetitive blunt force tasks like chopping or early stage scraping of hides and this presumably limited its utility to piercing and slicing; typical THC tools are projectile points, knives, and microblades.

6.3. Chronology

Fourteen sites with THC artifacts have yielded radiocarbon dates that span the Holocene (Table 5 and Fig. 13). Sites in Alberta (GdQn-1) and Yukon (NcVi-3) produced THC flakes (see Appendix 1 for pXRF results) and fluted points (Bereziuk, 2016; Clark and Clark, 1983; Esdale, 2008; Le Blanc, 1997) while JcRw-3 in Northwest Territories (Millar, 1968) produced a date from 10,124 to 9534 cal years BP (Stuiver et al., 2018) although stratigraphic control is poor. HhOu-113

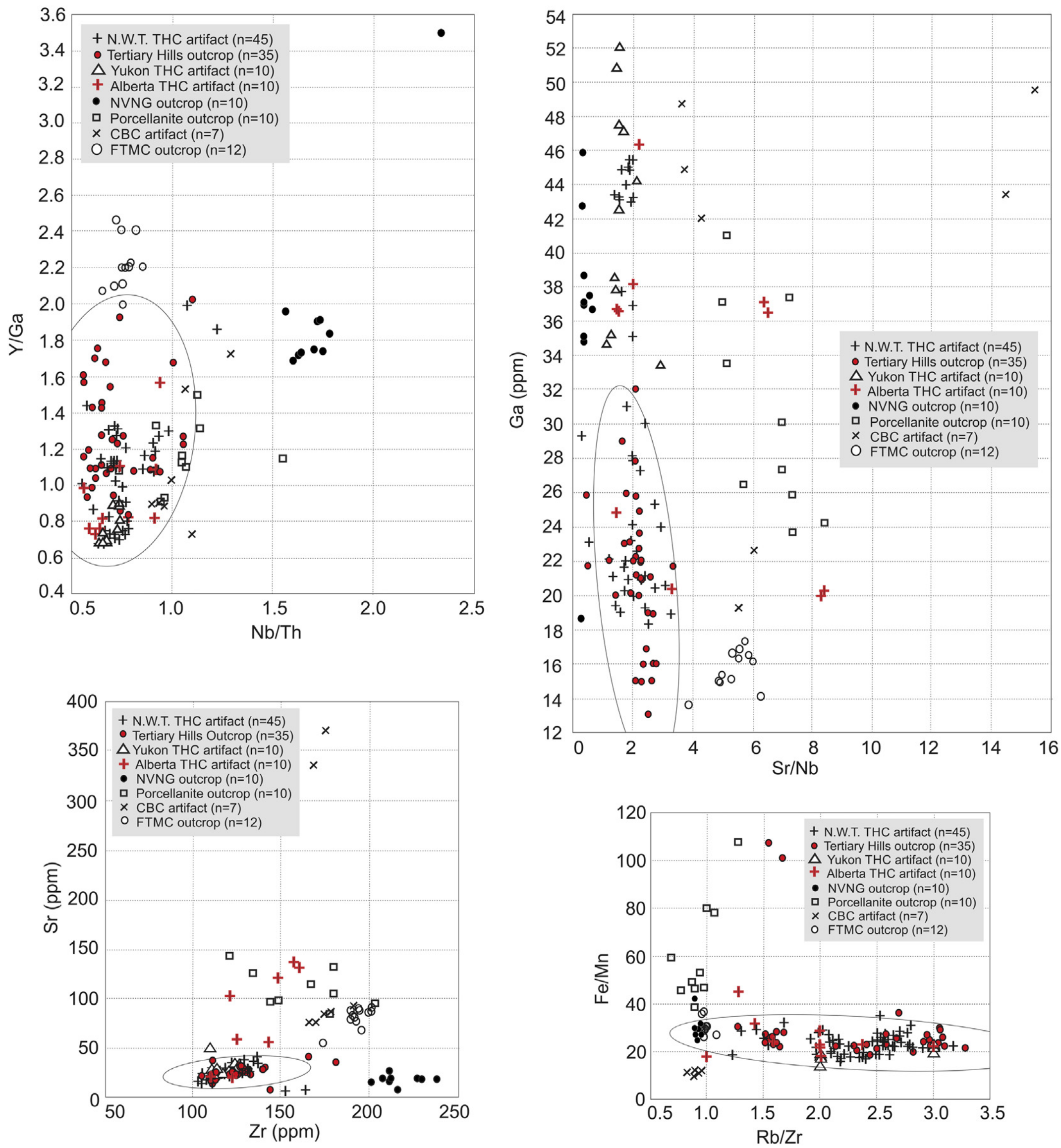

Fig. 10. Bivariate plots of element concentrations with 95% confidence ellipses around THC outcrop material (cherts, quartzites, quartzes, and chalcedonies were excluded).

is a single occupation site in Alberta (Roskowski, 2012) that yielded THC flakes (see Appendix 1) and a date of 8150 to 7970 cal years BP (Stuiver et al., 2018). THC occurrences at Late Pleistocene/Early Holocene sites suggest a wide Subarctic social network during deglaciation when continental ice sheets had not fully melted to the west and east. If the Tertiary Hills deglaciated earlier than surroundings (Hanks, 1993), they may have been connected to Alberta and Yukon through a navigable corridor as early as 12,000 cal years BP (Dawe and Kornfeld, 2017). Fluted KRF points have been found in Alberta (Kristensen et al., 2018) at a similar latitude to the fluted point and THC site at GdQn-1: fluted point makers in Alberta appear to have maintained contact from North Dakota to Northwest Territories (a linear distance of 2500 km

Table 3
Average element concentrations in parts per million (i.e., μg/g) (standard deviation in brackets) as measured by pXRF (n = number of samples of each group analysed).

Sample	(n)	Mn	Fe	Zn	Ga	Th	Rb	Sr	Y	Zr	Nb
Tertiary Hills Clinker	35	380 (40)	11,000 (6100)	106 (70)	21 (4)	17 (1)	280 (63)	25 (6)	27 (5)	125 (16)	12 (2)
Flat Top Mountain Clinker	12	340 (20)	10,600 (700)	79 (17)	16 (1)	19 (1)	188 (14)	77 (10)	35 (1)	193 (8)	14 (1)
Cape Bathurst Clinker	7	780 (290)	32,100 (35200)	355 (424)	39 (12)	20 (6)	149 (18)	161 (123)	39 (6)	174 (8)	20 (5)
Natural Non-Volcanic Glass	10	450 (80)	12,900 (1300)	96 (18)	36 (7)	33 (5)	198 (11)	19 (4)	69 (4)	216 (11)	57 (3)
Porcellanite	10	660 (240)	36,200 (9100)	118 (38)	31 (6)	16 (5)	147 (21)	112 (19)	32 (3)	160 (24)	18 (4)

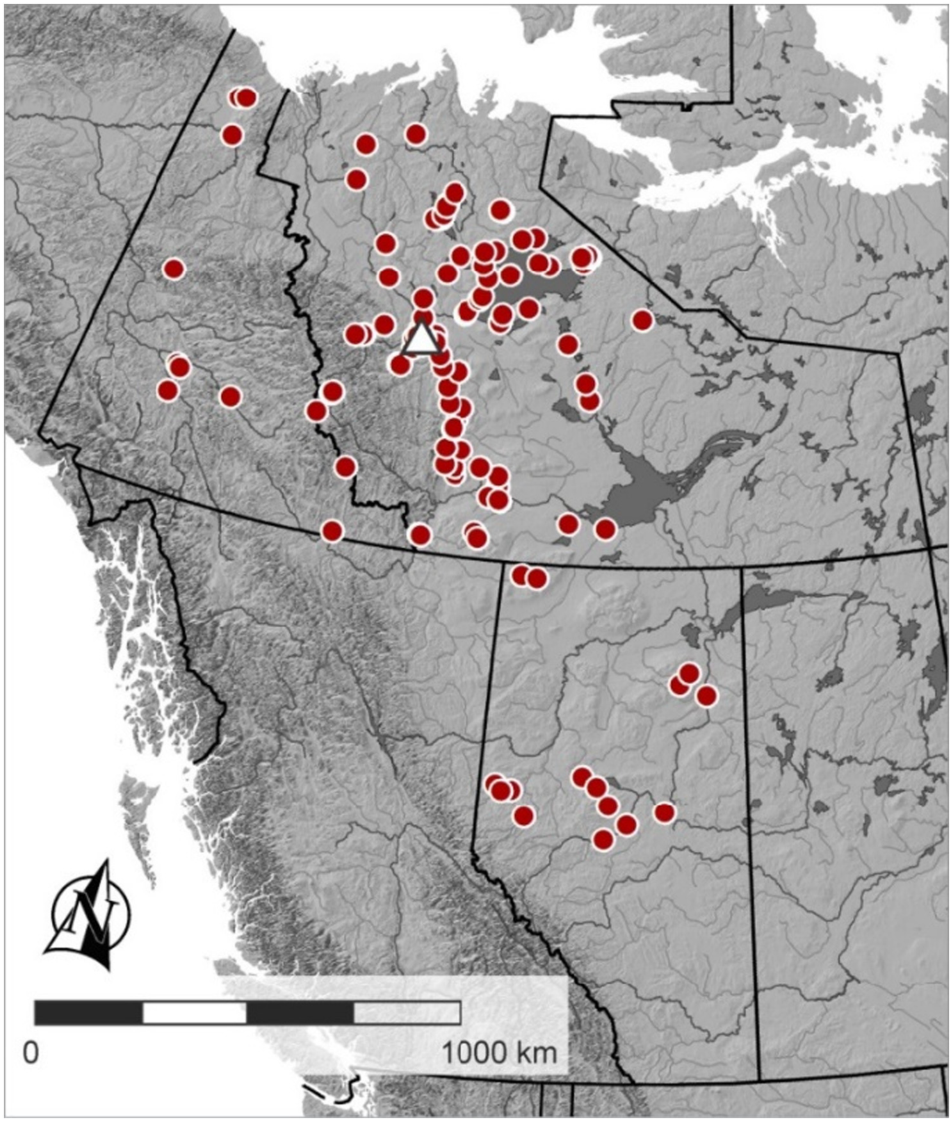

Fig. 11. All confirmed occurrences of pre-contact sites (n = 160) with THC artifacts in Yukon, Northwest Territories, and Alberta. More sites likely exist but THC was not mentioned in site records or collections were inaccessible.

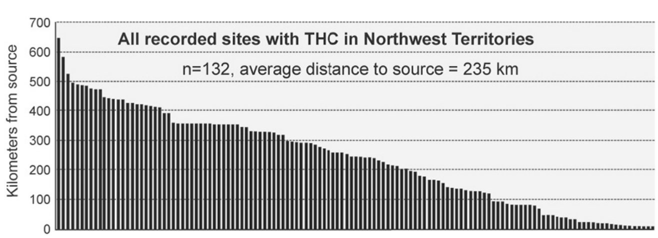

Fig. 12. Distance drop-off chart. The x-axis consists of archaeological sites, each represented by one bar.

equivalent to that between southern Greece and northern Denmark). The geographic connection highlighted by THC movement through the Middle to Late Holocene demonstrates conduits of exchange that, with future identification and provenance analyses of THC, can inform models of how communal hunting, the bow and arrow, linguistic groups, and DNA families spread across interior North America.

6.3.1. *White River Ash east and volcanic eruptions*

The majority of archaeological sites containing THC lack datable material due to taphonomic processes but many contain a well-defined stratigraphic marker that can be used for relative dating of occupations. The White River Ash east (WRAe) eruption dated to 846–848 CE (Jensen et al., 2014) created an ash lobe that extended across Yukon into Northwest Territories over 600 km east from its origin at or near Alaska's Mount Churchill (Fig. 14). An estimated 47 km^3 of ash descended across roughly 1 million km^2 making this one of the largest Holocene eruptions in North America (Lerbekmo, 2008; VanderHoek and Nelson, 2007). Permit reports, site forms, and publications were analysed for all available THC-bearing sites to assign pre- vs. post-WRAe ages (Table 6) based on the presence of ash and typological information (e.g., projectile points and microblades).

A comparison of pre- vs. post-WRAe assemblages indicates that the eruption altered hunter-gatherer mobility and exchange patterns. When pre- vs. post-WRAe chronologies could be assigned to Yukon and Alberta assemblages with THC, all sites are pre-WRAe despite the fact that a relatively high percentage of pre-contact sites in Subarctic Canada are from the last 1000 years (e.g., due to visibility, connections to oral history, and erosion factors). It appears that long-distance relationships with people of Northwest Territories broke down after the eruption based on the absence of post-WRAe THC in Yukon and Alberta.

Changes in THC movement before and after WRAe suggest that the eruption weakened contact and material exchange across the

Table 4
Comparison of assemblages with THC from Northwest Territories, Alberta, and Yukon. Sites with less than ten artifacts are excluded in the fourth row to remove a skew caused by small lithic scatters.

Jurisdiction	Northwest Territories	Yukon	Alberta	Total
Number of sites with THC	132	12	16	160
Frequency of THC in assemblages with THC	33.3%	12.3%	12.8%	32.1%
Frequency of THC in assemblages with THC excluding sites with < 10 artifacts	16.6%	5.3%	5.4%	16.0%
Rough total of pre-contact sites in territory/province	~4000	~4000	~36,000	–
Percentage of pre-contact sites that contain THC in territory/province	~3%	< 1%	< 0.1%	–
Average distance from source	235 km	585 km	1067 km	341 km

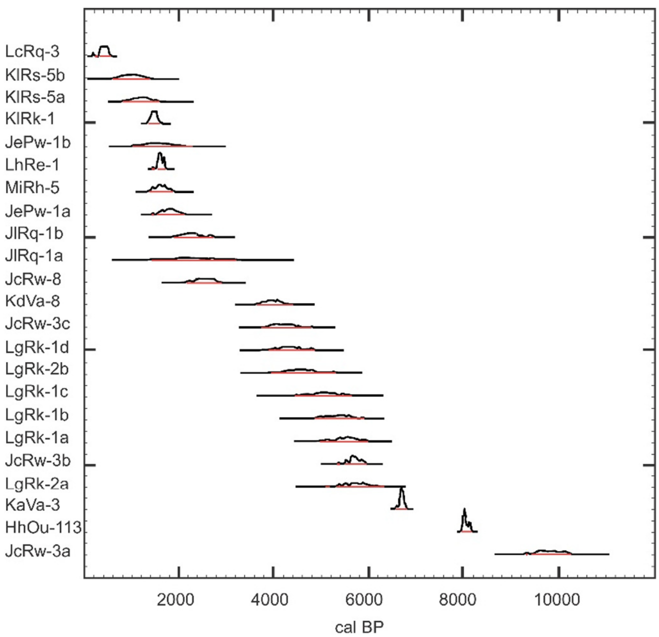

Fig. 13. Probability distribution of radiocarbon dated archaeological sites with THC. Probability distributions based on calibrated 2σ range generated using Calib 7.1 (Stuiver et al., 2018) and CORELdraw 6.0.

Mackenzie Mountains that separate Yukon and Northwest Territories. While landscapes and biota may have been minimally affected by ash in the Mackenzie Basin (MacDonald, 1987; Slater, 1985; Szeicz et al., 1995), human adaptations in the Yukon River Basin, particularly regions with over 5 cm of ash deposition, were likely heavily stressed (e.g., Anderson et al., 2005; Bunbury and Gajewski, 2013; Gajewski et al., 2014; Kuhn et al., 2010). In general, volcanic eruptions in the arctic/subarctic with significant ash deposition (5 cm or greater) experienced more dramatic impacts on ecosystems and people than in temperate environments because of relatively simpler trophic pyramids and more fragile landscapes (Dumond, 2004; Fitzhugh, 2012; Grishin et al., 1996; Jacoby et al., 1999; Pendea et al., 2016; Sheets, 2012; VanderHoek and Nelson, 2007). Oral history indicates that in the last 500 years, people maintained regular contact and kin networks across the Mackenzie Mountains (Gillespie, 1981; Hanks, 1993; Michea, 1963); with a lack of access to Yukon River resources and social networks caused by ash and ecological stress, people of the Mackenzie Basin may have shifted to a more insular economy and endogamous kin network (Ives, 1990). This may explain a drop in the movement of curated THC tools away from the source and an increase in domestic production because the Tertiary Hills were more frequently visited during seasonal rounds of Mackenzie Basin hunter-gatherers. A lack of access to social networks to the west (towards Yukon), may have weakened Northwest Territories hunter-gatherer economic systems that, in turn, had once helped support long-distance exchange south to Alberta. We argue that the WRAe eruption destabilized a social landscape that had previously fostered long-distance exchange.

Within Northwest Territories, sites with THC increase from roughly one per 200 years (pre-WRAe) to one site per 50 years (post-WRAe).

Table 5
Sites with THC recovered from radiocarbon dated components. CARD is the Canadian Archaeological Radiocarbon Database (Martindale et al., 2016).

Site	Conventional RC date BP	Lab number	Material	$\delta^{13}C$ (per mil)	Location	Reference
JcRw-3	8720 ± 190 4920 ± 110	GAK 1275	Charcoal	−25.0	Northwest Territories	Millar, 1968
	3780 ± 160	I-3190	Carbonaceous soil	Not known		
		GAK 1274	Charcoal	−25.0		
HhOu-113	7220 ± 40	Beta-33,309	Calcined bone	−23.1	Alberta	Roskowski, 2012
KaVa-3	5870 ± 40	Beta-86,359	Charcoal	−26.1	Yukon	CARD
LgRk-2	4965 ± 220	S-5	Peat	−27.0	Northwest Territories	Clark, 1986
	4065 ± 220	S-8	Plant remains	−27.0		
LgRk-1	4430 ± 240	RIDDL-322	Caribou bone collagen	−20.0	Northwest Territories	CARD
	3890 ± 180	RIDDL-323	Caribou bone collagen	−20.0		
	4800 ± 200	S-10	Charcoal	−25.0		
	4650 ± 200	S-9	plant remains	−25.0		
KdVa-8	3630 ± 140	AECV-1560C	Charcoal	Not known	Yukon	Thomas, 2003
JcRw-8	2460 ± 160	GSC 844	Charcoal	−25.0	Northwest Territories	CARD
JlRq-1	2225 ± 170	S-691	Charcoal	−25.0	Northwest Territories	CARD
	2265 ± 385	S-703	Charcoal	−25.0		
JePw-1	1860 ± 135 1635 ± 280	S-2873	Charcoal	−25.0	Northwest Territories	Hanks and Irving, 1986
		S-2875	Charcoal	−25.0		
MiRh-5	1690 ± 110	S-922	Calcined bone	−20.0	Northwest Territories	Clark, 1975, CARD
LhRe-1	1690 ± 50	Beta-099129	Charred plant material	−27.0	Northwest Territories	Toews and Pickard, 1997
KlRk-1	1570 ± 60	S-704	Charcoal	−25.0	Northwest Territories	CARD
KlRs-5	1285 ± 205 1070 ± 215	S-2873	Charcoal	Not known	Northwest Territories	Hanks, 1993
		S-2877	Charcoal	Not known		
LcRq-3	335 ± 80	I-7788	Charcoal	−25.0	Northwest Territories	Cinq-Mars, 1975

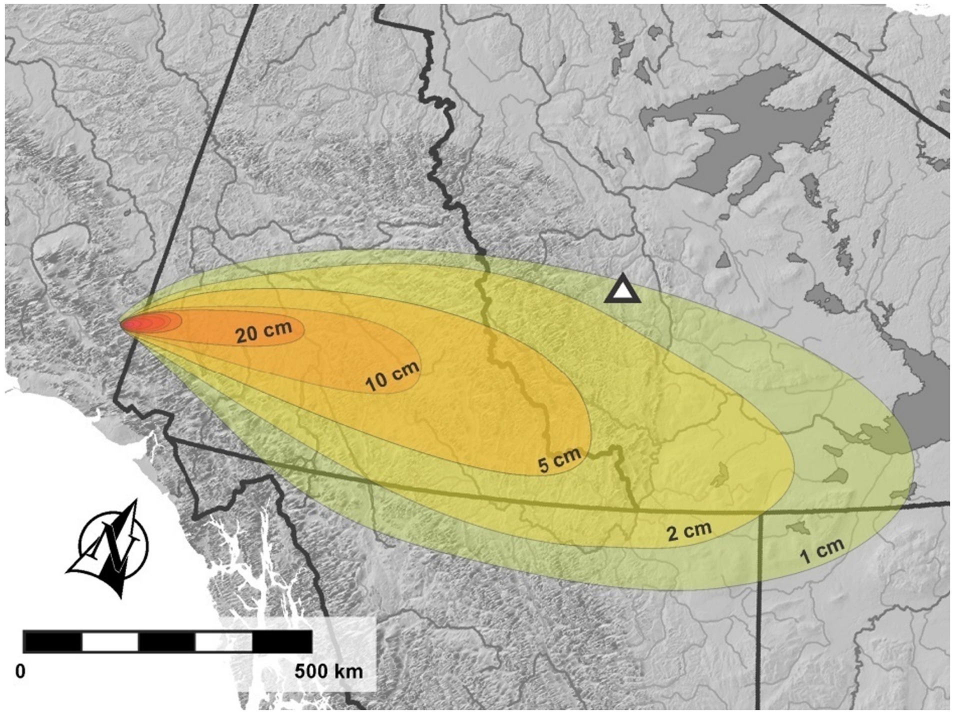

Fig. 14. WRAe isopach map of tephra depth (adapted from Lerbekmo, 2008). The white triangle is the THC outcrop.

The frequency of THC tools drops significantly from pre- to post-WRAe while the relative percentage of THC in Northwest Territories assemblages significantly increases from pre- to post-WRAe (Table 6). Overall, the movement of curated THC artifacts drops while domestic production (increased percentages of debitage) increases after the eruption. Spatial data also indicate that post-WRAe long distance movement of THC drops (Fig. 15). However, several archaeological sites in Northwest Territories with THC in both pre- and post-WRAe components indicate a local continuity of exploitation (e.g., LgRk-1, LcRq-3, and KlRs-5 on Fig. 15).

7. Discussion

Long-term exploitation of pyrometamorphic raw material outcrops (Tertiary Hills Clinker) implies some degree of stability and continuity in Subarctic hunter-gatherer adaptations in Northwest Territories. Knowledge of THC outcrops was presumably passed down from generation to generation with high fidelity despite cultural and technological changes over 10,000 years. Conversely, the visibility of burning bocannes and vents may have been a continuous beacon for raw material exploitation. The identification of THC and provenance results support the assertion that for much of the Holocene, contact between cultures has at least sporadically existed from Alberta to Yukon.

The Mackenzie River and its tributaries appear to have been conduits of contact and exchange, which supports historic records of the river acting as a meeting ground of Dene people (Gillespie, 1981; Michea, 1963). Fig. 16 is an overlay of Dene First Nation territories at the time of European contact (Ives, 1990) superimposed on THC site distribution and illustrates the significance of the Mackenzie River as a definer of social boundaries and possible mode of exchange. If extended into pre-contact times, the majority of sites with THC artifacts (Fig. 12) could have been produced by hunter-gatherers who personally visited outcrops: First Nations of the Mackenzie Mountains regularly moved distances of 300 km in traditional seasonal rounds (Andrews et al., 2012; Hanks, 1993). The majority of sites beyond a few hundred kilometers from the source can be explained by a single exchange between two neighbouring bands. The location of Tertiary Hills near the junction of four traditional territories may explain its movement within them. In times of need, Shuhtagot'ine Dene travelled beyond their traditional territory to the northeast and if this practice is of antiquity, it would explain the pre-contact presence of THC in this area. The Shuhtagot'ine Dene often travelled over the Mackenzie Mountains into Yukon to exchange goods and fish for salmon (Gillespie, 1981; Hanks, 1993; Michea, 1963) so the occurrence of THC (prior to White River Ash east deposition) at archaeological sites along rivers that drain west from the Mackenzie Mountains is not surprising.

With an average distance of 1070 km from the source, sites in Alberta with THC are more difficult to explain and likely involved the exchange of goods multiple times. All sites in Alberta with THC are located on or between major rivers that flow north to join the Mackenzie River (Fig. 16). It appears that rivers were routes of exchange in Subarctic pre-contact times with a corollary implication that boat technology was a major means of maintaining social connections, perhaps as early as the Late Pleistocene (Engelbrecht and Seyfert,

Table 6
Comparison of pre- vs. post-WRAe assemblages with THC in Northwest Territories.

Chronology	Pre-WRAe	Post-WRAe	Unknown age	Total or average
Sites with THC	44	26	62	132 (total)
Frequency of THC in all sites with THC	10%	48.3%	48.1%	33.3% (average)
Frequency of THC in assemblages with THC (but excluding sites with < 10 artifacts)	5.2%	42.5%	16.9%	16.6% (average)
Number of sites with THC tools	17	1	12	30 (total)
% of total assemblage that is a tool	5.5%	0.2%	10.1%	6.5% (average)

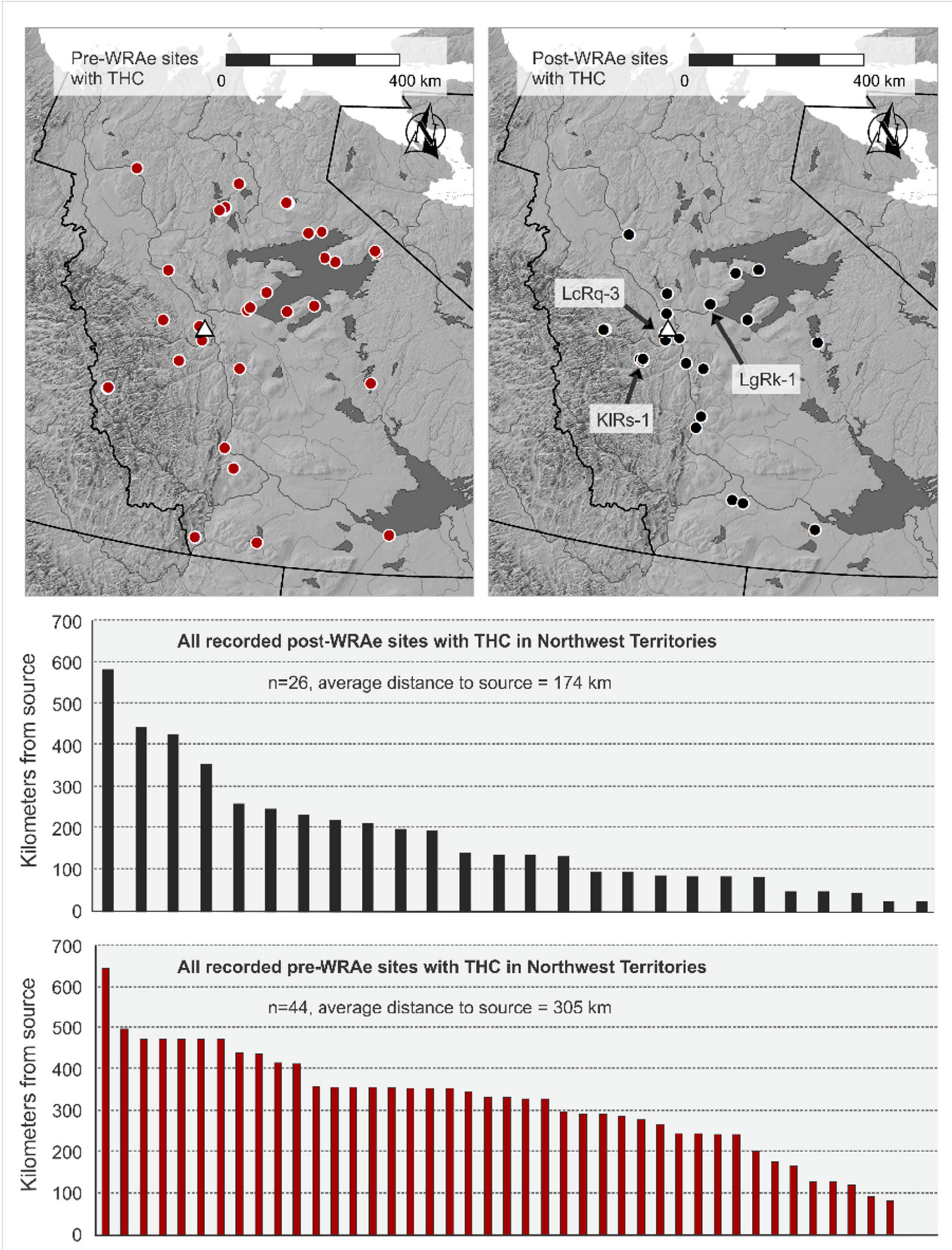

Fig. 15. Distance drop-off chart comparison and maps of pre- vs. post-WRAe archaeological sites with THC. The x-axis consists of archaeological sites, each represented by one bar. The white triangle in the maps above is the outcrop of THC.

1994).

Despite long-term stability of THC use, the WRAe eruption decreased the movement of curated tools and increased domestic production. We surmise that hunter-gatherers in the Yukon River Basin were negatively affected by heavy ash and this severed a connection with Mackenzie Basin hunter-gatherers that, in turn, reduced exchange networks and shifted THC exploitation to greater local consumption. High resolution temporal data from pre-contact weapons recovered from ice patches in Yukon indicate a clear technological shift after the WRAe from atlatl darts to bow and arrow technology (Hare et al., 2012) with a potentially associated cultural disruption. Caribou DNA records (Kuhn et al., 2010) indicate that the ash fall may have decimated ungulate populations on which the residents of southwest Yukon relied. The earliest bow and arrow record in Yukon ice patches is a bow made of coastal maple (Hare et al., 2012) suggesting that the WRAe event either stimulated exchange of a new technology in the Yukon River Basin or triggered the influx of new people from the west.

The spread of the bow and arrow around 1200 years ago may have influenced THC exchange in Northwest Territories and abroad to Yukon and Alberta. The bow and arrow are thought to have increased dietary breadth and reduced hunting band sizes (Angelbeck and Cameron, 2014; Bettinger, 2013; Churchill, 1993), and/or increased human conflict (Maschner and Mason, 2013), with a resultant decrease in hunting territories, although applications of these hypotheses have

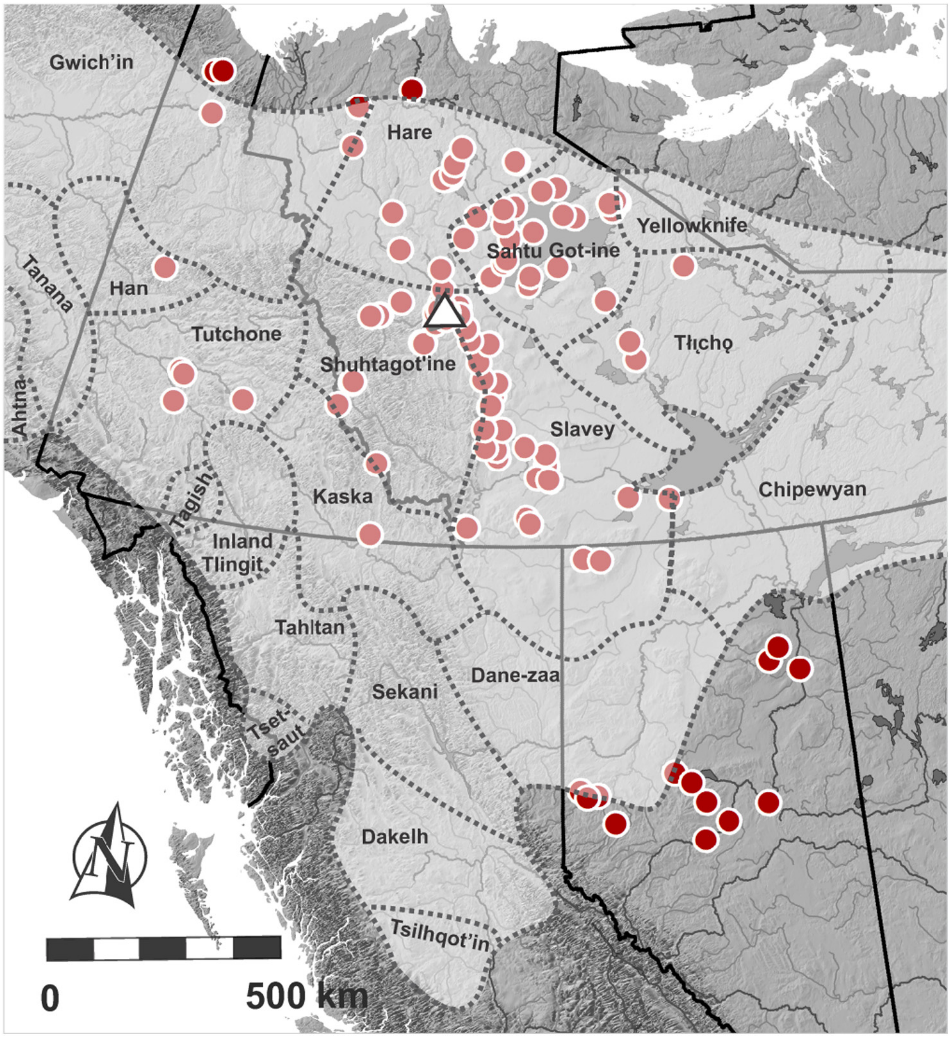

Fig. 16. An overlay of Dene First Nations territories at the time of European contact on the distribution of archaeological sites with THC (red circles). THC may have been limited to exchange between Dene ancestors if Dene territories at one time extended further south into Alberta in the Holocene than at the time of European contact.

been to cultural landscapes and ecosystems different from the Subarctic. However, large-scale hunting events, like communal caribou drives, persisted in the north to historic times, implying that hunting band sizes may not have been greatly influenced by the bow and arrow (e.g., Friesen, 2013; Gordon, 1990). Ice patch records indicate an antiquity of snare technologies for small game (Andrews et al., 2012), which suggests that dietary breadth may not have been significantly altered with bow and arrow use either.

The last 1200 years in Northwest Territories and Yukon (the Late Prehistoric Period) are generally marked by the introduction of small side-notched points (reduced in size compared to the earlier and presumed atlatl dart points), disappearance of microblades, and in Yukon, an increase in organic and copper tools (Clark and Gotthardt, 1999; Cooper, 2012; Gordon, 1996; Morrison, 1984). Land use patterns did not change significantly. It can be argued that local THC exploitation increased with adoption of the bow and arrow. However, other forms of archaeological evidence have yet to reveal a significant impact of the bow and arrow in Subarctic subsistence and social networks (Andrews et al., 2012; Morrison, 1984; Workman, 1979). It remains plausible that the WRAe event and bow and arrow spread across Subarctic Canada are related and therefore archaeologically challenging to differentiate in terms of human impact. Vegetation communities do not appear to have changed significantly in Northwest Territories in the last 3000 years (MacDonald, 1987; Slater, 1985; Szeicz et al., 1995), therefore shifting climates and changing ecosystems (long term) are unlikely explanations of the changes in pre- and post-WRAe networks of raw material exchange. Ice patch archaeology and palaeoenvironmental studies, when combined with changes in THC distribution, point to the WRAe volcanic event as a disruptive force in the Subarctic social landscape.

Changes in THC distribution offer two contributions to theories of a long debated origin and impetus of the migration of Athapaskan-speaking people (ancestors of modern Dene First Nations) from the Canadian Subarctic to the American Southwest and Great Basin (Derry, 1975; Gordon, 2012; Haskell, 1987; Ives, 2003, 2010, 2014; Matson and Magne, 2007; Moodie et al., 1992; Seymour, 2012; Workman, 1979). Firstly, the identification of THC in Alberta and Yukon indicates that the presumed ancestral Dene contact zone extended across thousands of kilometers from the Circumpolar North to the Northern Plains east of the Rocky Mountains. This connection persisted for perhaps several thousand years before Athapaskan migration began in the Late Holocene. Secondly, the WRAe eruption appears to have altered social dynamics as revealed by a geographic reduction of THC exchange above WRAe tephra. The volcanic event was a likely stimulus of culture change and may be implicated as one factor that ultimately dislocated a group of hunter-gatherers from their homeland, which initiated a much larger-scale movement of people across the continent.

Archaeologists have long sought links between WRAe and Athapaskan migration (Derry, 1975; Ives, 2003; Workman, 1979) or downplayed the significance of this ecological event (Gordon, 2012). Previous researchers have relied on oral history of volcanic events (Moodie et al., 1992), models of hunter-gatherer population density (Ives, 2003; Mullen, 2012; Workman, 1979), or have drawn on other hunter-gatherer responses to eruptions (Gordon, 2012) to infer impacts of WRAe on pre-contact people and then extrapolate the likelihood that this event stimulated out-migration. The identification of THC and provenance analyses offer some of the first reliable archaeological clues that ancestral Dene people maintained connections of deep antiquity from Yukon and Northwest Territories to the Northern Plains of Alberta, a valid path en route to the US Southwest and Great Basin regions. Our pXRF results and WRAe analyses also offer some of the first evidence beyond ice patch records that the volcanic event influenced northern hunter-gatherers and their social networks.

Our results are consistent with other analyses of northern hunter-gatherer responses to volcanic events (see Dumond, 2004; Fitzhugh, 2012; Jacoby et al., 1999; Pendea et al., 2016; VanderHoek and Nelson, 2007). Pre-contact people demonstrate a high resilience to ecologically rebound, either by temporarily relying on kin or altering subsistence strategies, but they existed in social landscapes where large-scale natural events tipped the balances or caused disturbances to cultural fabrics that some groups capitalized on to another's disadvantage (see Begét et al., 2008; Grattan and Torrence, 2007; Torrence, 2016; Williams, 2002; Ziedler, 2016). People of the Yukon and Mackenzie River Basins created kin and non-kin based alliances (Ives, 1990) to strengthen their socio-political hold on landscapes and buttress subsistence strategies. A large-scale event like the WRAe would have triggered alterations to social landscapes. Social processes are major means of adapting to large-scale ecological disturbances: provenance analyses when combined with the use of stratigraphic markers (pXRF analysis of THC and changes in distribution before and after the WRAe) provide a tool to evaluate social responses to large events.

8. Conclusion

We identify and characterise pyrometamorphic rocks using a suite of macroscopic, microscopic, and archaeometric techniques including XRD, thin section studies, and EPMA. We demonstrate that some clinkers produce geochemically distinct profiles of value for provenance studies. On the basis of results from pXRF, clinker artifacts from sites across Northwest Territories, Yukon, and Alberta can be confidently sourced to outcrops in the Tertiary Hills west of Mackenzie River. Results of these analyses (e.g., 1.25 million km^2 of THC extent that spans 10,000 years of human use) indicate that hunter-gatherer social networks beginning in the Late Pleistocene/Early Holocene were tethered to rivers and encompassed broader areas of Subarctic North America than previously thought. The White River Ash east volcanic eruption around 846–848 CE (Jensen et al., 2014) may have negatively influenced local hunter-gatherers in the Yukon River Basin that in turn severed modes of contact across the Mackenzie Mountains to the east with hunter-gatherers of the Mackenzie River Basin. This indicates that the volcanic event altered local hunter-gatherer social networks, and perhaps provided initial stimulus for the dislocation of a Dene hunter-gatherer group that culminated in a large-scale migration to the American Southwest and Great Basin. The application of provenance studies to pyrometamorphic rocks and the investigation of changing spatial and temporal distributions of raw materials offer means to reconstruct human movement and culture contact in global studies.

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jasrep.2018.11.039.

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Declaration of interest

The authors have no competing interests to declare and are not aware of any financial or personal relationships that could inappropriately influence this work.

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