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GEOCHEMICAL CHARACTERISTICS OF ORGANIC COMPOUNDS IN A PERMAFROST SEDIMENT CORE SAMPLE FROM NORTHEAST SIBERIA, RUSSIA

Genki I. MATSUMOTO[1], E. Imre FRIEDMANN[2] and David A. GILICHINSKY[3]

[1]*Department of Environmental Information Science, School of Social Information Studies, Otsuma Women's University, Karakida, Tama-shi, Tokyo 206*
[2]*Department of Biological Science, Florida State University, Tallahassee, FL32306, U.S.A.*
[3]*Institute of Soil Science and Photosynthesis, Russian Academy of Sciences, Pushchino, Moscow region, 142292, Russia*

Abstract: We studied total organic carbon (TOC), hydrocarbons and fatty acids in a permafrost sediment core sample (well 6–90, length 32.0 m, 1.5–2.5 Ma BP) from northeast Siberia (approximately 70°N, 158°E), Russia, to elucidate their geochemical features in relation to source organisms and paleoenvironmental conditions. Long-chain *n*-alkanes and *n*-alkanoic acids ($>C_{19}$) were most predominant hydrocarbons and fatty acids, respectively, so organic matter in the sediment core was derived mainly from vascular plants and, to a much smaller extent, from bacteria.

Low concentrations of unsaturated fatty acids revealed that organic matter in the sediment core was considerably degraded during and/or after sedimentation. The predominance of vascular plant components, the major ionic components of nonmarine sources, and geological data strongly implied that the sediment layers were formed in shallow lacustrine environments, such as swamp with large influences of tundra or forest-tundra vegetation. Also, no drastic changes in paleoenvironmental conditions for biological activity or geological events, such as sea transgressions or ice-sheet influences, occurred at the sampling site approximately 100 km from the coast of the East Siberian Sea during the late Pliocene and early Pleistocene periods.

1. Introduction

Organic compounds in sedimentary environments generally arise from *in situ* biological sources and reflect biological activity and biological composition. MATSUMOTO and coworkers have studied biogeochemical features of organic compounds in water, sediments, soil and rock samples from biologically extremely harsh environments, such as extremely cold regions of the McMurdo Dry Valleys in Antarctica (*e.g.*, MATSUMOTO *et al.*, 1979; MATSUMOTO, 1993) and acid hydrothermal environments in Japan (MATSUMOTO and WATANUKI, 1990, 1992). Cryptoendolithic microbial communities have been discovered in the same area of Antarctica (see, *e.g.*, FRIEDMANN and OCAMPO, 1976; FRIEDMANN, 1982).

Viable microorganisms have also been found in arctic permafrost sediment layers even at ambient temperatures lower than the freezing point of water (ZVYAGINTSEV *et al.*, 1985, 1990; GILICHINSKY *et al.*, 1989, 1992; GILICHINSKY and WAGENER, 1994). Interestingly, long-chain *anteiso*-alkanes and *anteiso*-alkanoic acids (C_{20}-C_{30}) have been detected in antarctic communities (MATSUMOTO *et al.*, 1992a). As part of a series of studies of biologically extremely harsh environments, here we report biogeochemical features of organic compounds, such as hydrocarbons (including normal, cyclic and acyclic isoprenoids) and fatty acids, in an arctic permafrost sediment core sample from a very cold site in northeast Siberia (Russia) in the Northern Hemisphere. We also discuss paleoenvironmental conditions of sedimentation.

2. Materials and Methods

2.1. *Sampling site and sediment core sample*

The drilling site (well 6-90) is situated approximately 100 m from the right river bank of the Bolshaya Chukochya River in the Kolyma-Indigirka lowland (approximately 70°N, 158°E) of northeast Siberia, Russia, at about 3.5 m above sea level. This area has a cold subarctic climate and a mean annual air temperature of -13.4°C. At the site, the depth of the seasonal thawing is 0.5 m. The vegetation is tundra dominated by dwarf birch (*Betula* sp.), dwarf willow (*Salix polaris*), dwarf larch (*Pinus* sp.), berries, grasses, mosses and lichens.

We obtained a permafrost sediment core sample by drilling to 32.0 m depth with a portable gasoline-powered drill, which operates without a drilling fluid. The sediment core was composed mainly of sandy-loam texture. The temperature of the sediment core sample was lower than -7°C. The water (ice) contents and pH values of the sediment core ranged from 20 to 30% including 5% of unfrozen water, and from 6.4 to 7.5, respectively (GILICHINSKY, 1993; GILICHINSKY *et al.*, 1993a). The redox potential results of the sediment core varied from -250 to $+280$ mV (SAMARKIN *et al.*, 1994; RIVKINA and GILICHINSKY, 1995). The core was split into ca. 5 cm-long segments, placed in metal boxes and transported frozen to Moscow, Florida State University, U.S.A. and then to Tokyo, Japan.

2.2. *Analytical methods*

Total organic carbon (TOC) of the permafrost sediment core sample was determined by a wet oxidation method after treatment with hydrochloric acid to remove inorganic carbon (ARINUSHKINA, 1970a). Major ionic components (Ca, Mg, HCO_3 and SO_4) in water extracts of the sediment core were determined by the methods of ARINUSHKINA (1970b).

Analytical methods for organic compounds in the sediment core were essentially similar to those in previous studies (*e.g.*, MATSUMOTO *et al.*, 1979; MATSUMOTO, 1994). Briefly, wet sediment samples (4.6–4.7, 8.5, 14.2, 19.0, 22.7, 28.5 and 32.0 m) were saponified with 0.5 M potassium hydroxide in methanol (70°C, 4 h). Hydrocarbons and fatty acids were extracted with ethyl acetate after acidification. The ethyl acetate extracts were chromatographed on a silica-gel column (160×5 mm i. d., 100 mesh, 5% water). Fatty-acid fractions were methylated with diazomethane. Hydrocarbons and fatty acid methyl esters were analyzed by the use of a JEOL JMS Automass 150 gas chromatograph-mass spectrometer (GC-MS) equipped with a fused-silica capillary column (J & W Scientific, DB5, 30 m×0.25 mm i. d., film thickness 0.25 μm). The column oven temperature was programmed from 60 to 120°C at 20°C/min, 120 to 300°C at 5°C/min and maintained for 10 min. The temperatures of injection block, GC-MS interface and ion source were maintained at 330, 300 and 250°C, respectively. The ionization energy, filament current and detector gain were 70 eV, 0.30 mV and −0.65 kV, respectively.

Hydrocarbons and fatty acids were identified by the comparison of retention times and mass spectra with those of authentic standards and published literature (*e.g.*, PHILP, 1985; MATSUMOTO *et al.*, 1989). Quantitation was by the measurement of peak height of the gas chromatogram or mass chromatogram.

3. Results and Discussion

3.1. Primary productivity

The concentrations of TOC, hydrocarbons and fatty acids in the sediment core sample are shown in Table 1. TOC contents of the sediment core ranged from 0.57 to 1.6%. TOC is a measure of primary productivity. Relatively low TOC contents of the sediment core suggested that conditions at the sediment site were those of an oligotrophic or mesotrophic shallow lacustrine environment, as discussed below. The carbon in bacterial cells, calculated from total bacterial counts, was less than 0.01% of the TOC (RIVKINA and FRIEDMANN, unpublished). Total concentrations of hydrocarbons and fatty acids varied from 1.3 to 5.0 and 8.2 to 85 μg/g dry sediment, respectively. Concentrations of fatty acids were much higher than those of hydrocarbons, as is the case in other environments on the planet.

3.2. Features of organic compounds

Normal alkanes with a predominance of odd carbon numbers (C_{15}-C_{35}) were detected in the 4.6-4.7-m section of the permafrost sediment core, together with *n*-alkenes (*n*-$C_{21:1}$-*n*-$C_{26:1}$) and acyclic isoprenoid alkanes (pristane, phytane and squalane, Fig. 1). The

Table 1. Concentrations of total organic carbon (TOC), hydrocarbons and fatty acids, and major ionic components in a permafrost sediment core sample from northeast Siberia, Russia.*

Sample depth (m)	TOC (%)	Hydrocarbons (μg/g)	Fatty acids (μg/g)	Major ionic components (me/100 g sediment)
4.6	1.6	4.21	84.8	Ca (0.2), HCO_3 (0.27)
8.5	0.85	1.33	13.6	Ca (0.2), Mg (0.22), HCO_3 (0.4)
14.2	1.2	1.33	19.7	Ca (0.2), Mg (0.38)
19.0	ND	4.98	16.3	ND
22.7	0.85	1.37	27.1	Ca (0.1), Mg (0.1), SO_4 (0.18)
28.5	0.57	2.16	8.15	Ca (0.07), Cl (0.1), HCO_3 (0.1), SO_4 (0.1)
32.0	1.1	1.73	23.2	Ca (0.12), Cl (0.27)

* Dry base.
ND: No data.

most predominant hydrocarbon was n-C_{27} alkane. No bimodal distribution of n-alkanes was detected. Hydrocarbon distributions in other sections of the sediment core were generally similar to that of the 4.6–4.7-m section, except for the 19.4-m section, in which the most predominant hydrocarbon was n-C_{23} alkane. Triterpanes and triterpenes (C_{27}-C_{31}) were detected, but no steranes were found anywhere in the other sections of the sediment core sample (Fig. 2). The major compounds were all unmatured triterpanes and hopenes, including hop-22(29)-ene.

Normal alkanoic acids (C_{10}-C_{32}) with a predominance of even carbon numbers were

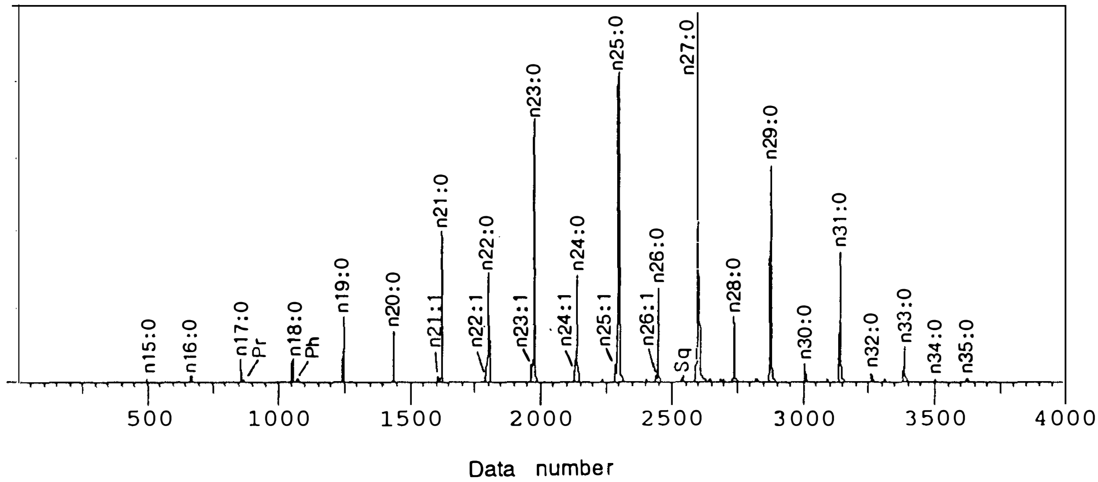

Fig. 1. Mass chromatogram (m/z 57) of the hydrocarbon fraction of the 4.6–4.7 m depth sample in a permafrost sediment core from northeast Siberia, Russia. Arabic figures on the peaks denote carbon chain length of n-alkanes. Pr, Ph and Sq are pristane, phytane and squalane, respectively. m : n = Carbon chain length : number of unsaturation.

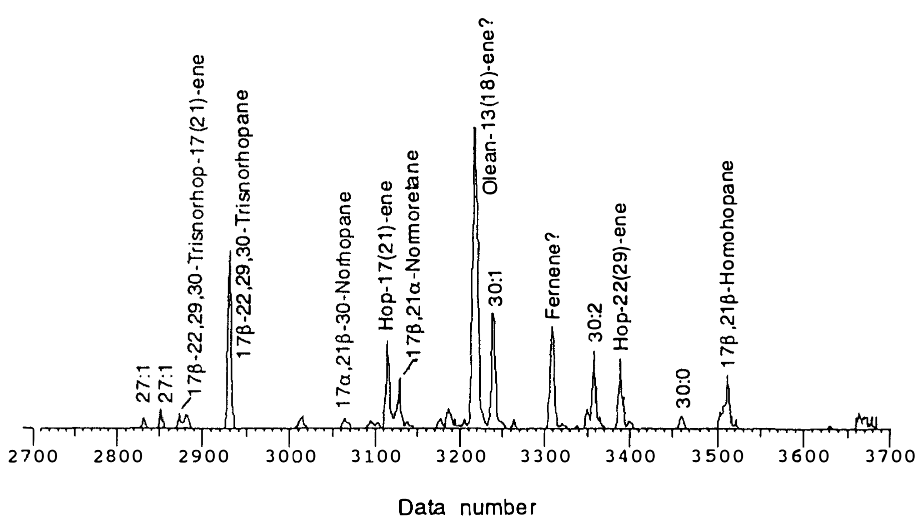

Fig. 2. Mass chromatogram (m/z 191) of triterpanes and triterpenes in the hydrocarbon fraction of the 4.6–4.7 m depth sample in a permafrost sediment core from northeast Siberia, Russia.

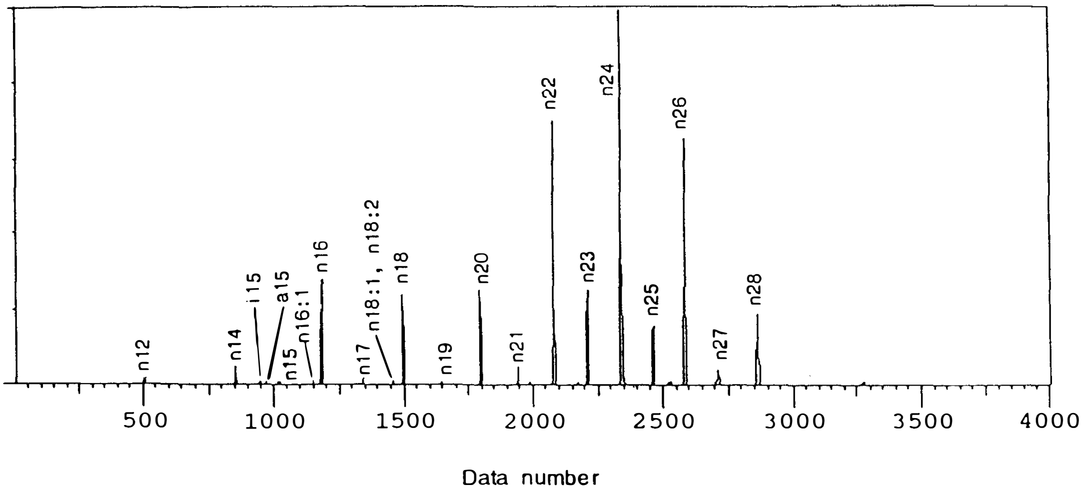

Fig. 3. Mass chromatogram (m/z 74) of the fatty acid fraction of the 4.6–4.7 m depth sample in a permafrost sediment core from northeast Siberia, Russia. Arabic figures on the peaks denote carbon chain length of n-alkanoic acids. i and a are iso- and anteiso-alkanoic acids, respectively. m : n = Carbon chain length : number of unsaturation.

found, together with small amounts of *n*-alkenoic acids (*n*-$C_{16:1}$, *n*-$C_{18:1}$ and *n*-$C_{18:2}$) and *iso*- and *anteiso*-alkanoic acids (C_{13}-C_{17}, Fig. 3). These *n*-alkanoic acids showed bimodal distributions with peaks at *n*-C_{16} or *n*-C_{18} and at *n*-C_{24}. The distributions of fatty acids in other sections of the sediment core were generally similar to that of the 4.6–4.7-m section. The most predominant fatty acid was all *n*-C_{24} alkanoic acid throughout the sediment core sample.

3.3. Sources of organic compounds

Long-chain *n*-alkanes and *n*-alkanoic acids ($>C_{19}$) with a predominance of odd and even carbon numbers, respectively, are commonly distributed in the waxes of vascular plants (*e.g.*, KOLATTUKUDY, 1970; MATSUMOTO and WATANUKI, 1992). In the sediment core, long-chain *n*-alkanes (73.1–91.6%) and long-chain *n*-alkanoic acids (62.9–82.5%) were the most predominant hydrocarbons and fatty acids, respectively (Table 2). Thus, organic matter in the sediment core sample was mainly derived from vascular plants. Usually, *n*-C_{29} alkane is the most predominant long-chain *n*-alkane in sedimentary environments of the middle latitudes, whereas the most predominant *n*-alkane in the sediment core was mainly *n*-C_{27} alkane. The analysis of leaves of *Betula* sp. from east Siberia showed that *n*-alkane composition with the maximum of *n*-C_{27} alkane was similar to those of the sediment core (MATSUMOTO, 1994; MATSUMOTO *et al.*, unpublished). Also, palynological studies showed that generally *Betula* sp. pollen was the most predominant pollen throughout the sediment core and other sediment cores in this region (SHER and KAPLINA, 1979; KARTASHOVA, 1985; GILICHINSKY and KARTASHOVA, unpublished). Thus, it is most likely that hydrocarbons in the sediment core were mainly derived from *Betula* sp. Although living organisms having *n*-alkanes with the peak at *n*-C_{23} are not common, a water plant (*Zannichellia* sp.) from Lake Baikal was found to contain *n*-C_{23} alkane as its most prominent hydrocarbon (MATSUMOTO *et al.*, unpublished). Thus, certain water-plant-like

Table 2. Compositions of hydrocarbons and fatty acids found in a permafrost sediment core sample from northeast Siberia, Russia.

Sample depth (m)	Hydrocarbon (%)				Fatty acids (%)			
	n-Short n-C_{15}-n-C_{19}	*n*-Long n-C_{20}-n-C_{36}	*n*-Alkenes C_{19}-C_{29}	Isoprenoid*1 Pr, Ph, Sq	*n*-Short n-C_{12}-n-C_{19}	*n*-Long n-C_{20}-n-C_{32}	*n*-Alkenoic n-C_{16}-n-C_{18}	Branched*2 C_{13}-C_{17}
4.6	5.3	84.4	9.8	0.42	14.7	82.5	1.8	1.0
8.5	3.8	91.6	3.6	0.57	24.1	67.3	6.5	1.6
14.2	7.5	88.4	2.9	0.61	22.7	71.6	3.3	2.1
19.0	5.4	84.4	9.4	0.54	26.5	66.0	2.5	3.8
22.7	16.5	73.1	9.6	0.48	21.2	73.2	4.6	0.69
28.5	12.3	80.1	7.0	0.42	28.5	62.9	6.7	1.6
32.0	7.6	85.4	4.7	1.7	31.0	64.3	1.6	2.7

*1 Pr: Pristane, Ph: Phytane, Sq: Squalane.
*2 *Iso* and *anteiso*-alkanoic acids.

organisms may be a major source of the hydrocarbons in the 19.4-m section of the sediment core.

Long-chain *n*-alkenes with a predominance of odd carbon numbers are often found in lake sediments in the temperate zone and are believed to originate from certain vascular plants, such as reeds and ferns and/or peat deposits (GIGER and SCHAFFNER, 1977; CARDOSO *et al*., 1983; CRANWELL *et al*., 1987). Also, long-chain *n*-alkenes found in lake sediments of Antarctica, where vascular plants are absent, are attributed to microalgae (MATSUMOTO *et al*., unpublished). Hence, long-chain *n*-alkenes found in the sediment core can be explained by the contribution of certain vascular plants, microalgae and/or peat deposits.

Pristane and phytane may be derived from phytyl side chain of chlorophylls. Squalane is often distributed in archaebacteria, such as methanogenic bacteria as discussed elsewhere (LANGWORTH, 1985; MATSUMOTO and WATANUKI, 1990, 1992; MATSUMOTO *et al*., 1992b). Methanogenic bacteria and methane are commonly distributed in permafrost sediment layers in Siberia. Methanogenic bacteria (<107 cells/g of dry sediment) and methane (2–40 ml/kg of dry sediment) were detected in the sediment core and other sediment cores in this region (RIVKINA *et al*., 1992; GILICHINSKY *et al*., 1993b; SAMARKIN *et al*., 1994; RIVKINA and GILICHINSKY, 1995). Thus, it is very likely that squalane originated mainly from *in situ* methanogenic bacteria.

Branched (*iso*- and *anteiso*-) alkanoic acids are only found in bacterial lipids (see, *e.g*., O'LEARY, 1982), so the alkanoic acids in the sediment core were produced by bacteria, although their contribution appears to be small, because of low percentages (Table 2).

Triterpanes and triterpenes found in the sediment core (except for olean-13(18)-ene which is suggested to be present) are mainly due to prokaryotic organisms, such as cyanobacteria and bacteria as discussed elsewhere (*e.g*., VENKATESAN, 1988; MATSUMOTO and WATANUKI, 1990, 1992). Olean-13(18)-ene may come from certain vascular plants (*e.g*., PHILP, 1985; MATSUMOTO and WATANUKI, 1990, 1992).

The hydrocarbon and fatty-acid compositions of plants and other organisms in northeast Siberia are not yet sufficiently known; further studies on the sources of organic matter are required for detailed discussion.

3.4. Paleosedimentary environments

The sediment deposits in the Kolyma-Indigirka lowland, formed during the late Pliocene and Pleistocene periods and reaching a thickness more than 500 m, are the richest and least disturbed permafrost areas in the Northern Hemisphere and probably on the planet. The age of the sediment core was determined by paleomagnetic boundary (1.8 Ma), supported by paleontological evidence. The ages of the sediment core and those in similar sediment cores in this region ranged from 1.8 Ma to about 3.0 Ma (KAPLINA, 1981; VIRINA *et al*., 1984; MINYUK, 1986; GILICHINSKY *et al*., 1990; GILICHINSKY, 1995).

Major ionic components in the sediment core were Ca, Mg, HCO_3, Cl and/or SO_4 at low concentrations, which are ordinary levels in all Cenozoic sediment layers in this area (Table 1). They are typical of nonmarine components. Vascular-plant components were abundant throughout the sediment core (Table 2). Also, it is well known that all sediment layers in this region have lake-alluvial genesis (POPOV, 1953, 1982; SHER, 1971). These results strongly imply that the sediment layers were formed in shallow lacustrine environments, such as swamp, with large influences from vascular plants.

The presence of small amounts of *n*-alkenoic acids suggested that organic matter in the permafrost sediment core was considerably degraded during and/or after sedimentation, but the paucity of matured triterpenes and steranes showed that the sediment site was not affected by any thermal stress. The degradation of organic matter may have occurred largely in the sediment layers, before the formation of permafrost. Also, permafrost sediment layers contain, as a rule, considerable numbers of viable bacteria. Viable bacteria at 10^3–10^4 cells/g of dry sample were counted throughout the sediment core, and evidence is mounting that these bacteria are metabolically active, although at a low level (GILICHINSKY *et al*., 1993b; FRIEDMANN, 1994; GILICHINSKY and WAGENER, 1994). Thus, it is very likely that organic matter in the permafrost sediment layers was degraded by bacterial activity for a long period of time (1.8–3.0 Ma), even at ambient temperatures below 0°C. Further studies on the degradation rate of organic matter in permafrost sediment layers are needed.

No large changes in vertical distributions of TOC concentrations or in compositions of hydrocarbons and fatty acids were observed in the sediment core sample. Palynological data indicated that the fluctuations in the surroundings of the sediment core sampling site were between tundra and forest-tundra landscapes (SHER and KAPLINA, 1979; KARTASHOVA, 1985; GILICHINSKY and KARTASHOVA, unpublished). These results strongly suggest that drastic changes in environmental conditions, in either biological activity or geological events, such as sea transgressions and ice sheet influences, did not occur at the sampling site approximately 100 km from the coast of the East Siberian Sea during the late Pliocene and Pleistocene periods.

4. Conclusions

Geochemical studies of organic compounds combined with geological data in an arctic permafrost sediment core (32.0 m) from northeast Siberia ca. 100 km from the coast of the East Siberian Sea, Russia revealed:

1) Long-chain *n*-alkanes and *n*-alkanoic acids were the predominant hydrocarbons and fatty acids, respectively, throughout the sediment core (1.5–2.5 Ma BP).

2) Organic matter in the sediment core was mainly derived from vascular plants, such as *Betula* sp., plus a small contribution from bacteria.

3) Organic matter in the sediment core was considerably degraded during and/or after sedimentation.

4) Sediment layers throughout the sediment core were probably formed in shallow lacustrine environments, such as swamp, with large influences of tundra or tundra-forest vegetation.

5) No drastic changes in paleoenvironmental conditions for biological activity, or in geological events (*e.g.* sea transgressions and ice sheet influences) occurred at this site during the late Pliocene and early Pleistocene periods.

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