

Leonardite-Type Material at Red Lake Diatomite Deposit, Kamloops Area, British Columbia

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INTRODUCTION

“Leonardite” and “humate” are loosely used terms covering a variety of naturally occurring lithologies with high humic acid content, including weathered (oxidized) lignite, sub-bituminous coal and a variety of carbonaceous rocks such as mudstones, shales and claystones (Kohanowski, 1957 and 1970; Hoffman *et al.*, 1993). These raw materials are used mainly as soil conditioners, however they also have use in wood stains, drilling fluid additives and as binder in iron pelletizing (Broughton, 1972a and Hoffman *et al.*, 1993). To our knowledge, this is the first publication describing leonardite in British Columbia, therefore in addition to describing the deposit, this paper also provides background information about leonardite, humic acids and the methodology used in this study.

BACKGROUND

Humic substances are formed from the chemical and biological degradation of plant and animal residues, and from microbial activity. Based on their solubility in alkali and acids, humic substances are partitioned into three main fractions: humic acid, fulvic acid and humin. Humic acid is soluble in dilute alkali but is coagulated by acidification of the alkaline extract. The fraction which remains in solution when the alkaline extract is acidified (i.e. soluble in both dilute alkali and dilute acid), is termed fulvic acid. Humin is the fraction which cannot be extracted by either dilute base nor by dilute acid (Martin, 1997).

Carbon is the major element in humic and fulvic acids. Typical elemental analysis of humic acid is presented in Table 1.

Given the diversity of synthesis and degradation, the humic acid structure is loosely defined as a mixture of complex macromolecules having polymeric phenolic

TABLE 1
COMPOSITION OF SOIL HUMIC ACID (BUFFLE, 1988)

Elemental composition	(%)
Carbon	53.8-58.7
Hydrogen	3.2-6.2
Nitrogen	0.8-5.5
Sulfur	0.1-1.5
Oxygen	32.7-38.3
Functional group content	(meq/g)
Total acidity	5.6-8.9
#NAME?	1.5-5.7
Phenolic OH	2.1-5.7
Alcoholic OH	0.2-4.9
> C=O Quinones	.4-2.6
> C=O Ketones	0.3-1.7
-O-CH ₃	0.0-0.8
Structural Composition	(%)
aromaticity	69
%H: Aromatic	16-43
%C: Aromatic	21-35
	10-30
fraction of total OH (%)	
aliphatic COOH	9
aromatic COOH	20
phenolic OH	14

structures consisting of aromatic rings joined by alkyl chains of various lengths (Merck Index, 1996). The degree of aromaticity of humic acid has been estimated at 70% on the basis of oxidative degradation products. Several purely hypothetical structural models have been proposed in response to observed chemical and physical behaviour. The humic acid structure is presumed to contain voids of various dimensions that could trap and bind other organics.

The determination of relative molecular mass, size and shape depends on polydispersity, charge effects, aggregation properties and hydration properties. Consequently, the reported values of humic acid molecular mass range from 200 to 10 000. The values are a strong function of pH (Filella, Parthasarathy, and Buffle, 1995).

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Humic acids promote the decomposition of rocks and minerals in soil, increasing the acidity of alkaline soils which liberates nutrients that then become available to plants. For this reason the majority of leonardite products are used as soil conditioners in agriculture and in tailings rehabilitation (Cooley *et al.*, 1970; Hoffman and Austin, 1993).

Materials with high humic acid content may be further processed for use as water-soluble wood stains, drilling fluid additives (Odenbaugh and Ellman, 1967, Roybal *et al.*, 1986), binders in iron ore pellets and lignite briquettes.

The biggest growth potential for humic acid-based products is in soil conditioning and agricultural applications. Although humic acid or similar materials can be derived by controlled oxidation of coal, such products are considered synthetic and therefore, are not used in organic growing operations. Leonardite and humates on the other hand, are considered to be natural products. Because of its industrial and soil conditioning applications, leonardite is typically considered an industrial mineral (Gulliov, 1991; Hamilton, 1991 and Hoffman and Austin, 1993). In most cases the energy value of leonardite and humate is too low to be considered as a fuel.

In North America, known, potentially economical deposits are reported in Arkansas, Florida, Louisiana, New York, North Dakota (Dove, 1926), Michigan, Minnesota, Texas and Wyoming (Burdick, 1965). Deposits in Wyoming, New Mexico (Shomaker and Hiss, 1974; Siemers and Wadell, 1975a, b and 1977, Roybal and Barker, 1986), North Dakota, Alberta and Saskatchewan (Figure 1) have received the most attention (Broughton, 1972a, Carter, 1982; Hoffman *et al.*, 1993; Hamilton, 1991 and Gulliov, 1991).

Raw humate or leonardite should contain a sufficient concentration of humic acid to be of economic interest. New Mexico's agricultural humate typically contains 12 to 18% humic acid. The material should also have low Hg, As, Se, Cd, Ba, Pb, Zn and radionuclide content (Hoffman *et al.*, 1993). The materials used for drilling fluid applications typically contain over 65% humic acid (Hoffman *et al.*, 1993).

The Red Lake deposit is currently mined for diatomite-bearing rocks and the ore is shipped for processing to the Western Industrial Clay Products plant in Kamloops. At the plant, the ore is processed and blended with other materials to produce a variety of industrial absorbents and pet litter products.

Depending on the average humic acid content of the Upper Carbon-rich unit (*Muc*) currently exposed in the floor of the operation (Photo 1), the Red Lake deposit could become a significant producer of humic acid-bearing material marketable as leonardite or humate.

LOCATION AND REGIONAL SETTING

The Red Lake diatomite mine (092INE081), originally operated by D.E.M. Resource Processors Ltd., be-



- 1- Red Lake, BC; 2- Battle River, Alta;
- 3- Sheerness, Alta; 4- Estevan, Sask;
- 5- North Dakota; 6- Glenrock, Wyoming;
- 7- San Juan area, New Mexico

Figure 1. Leonardite-type deposits in North America.

longs to Western Industrial Clay Products. It is located approximately 40 km northwest of Kamloops (Figure 2).

The deposit is accessible by a combination of paved and gravel roads. Mining is seasonal due to the relatively high elevation of the deposit (1300 m above sea level), which results in unpredictable road conditions in the winter.

Tertiary Basins are indicated by shaded areas. The deposit is interpreted to lie near the base of the Miocene Deadman River Formation. The andesite or basalt flow basement that underlies the deposit is believed to belong to the Eocene Kamloops Group (Read, 1995).

DEPOSIT GEOLOGY

The presence of diatomite in the Red Lake area was known since 1928 (Eardley-Wilmot, 1928 and Cockfield, 1948). The geology of the deposit was described recently by Read (1995). The general form and geology of the deposits can be conveniently summarised by north-south and east-west sections (Figure 3a and 3b).

These sections indicate that the lithologies at the mine site include Grey Andesite, Basal Carbon-rich unit (*Mbc*), Basal Diatomaceous Earth (*Mbd*), Upper Carbon-rich unit (*Muc*), Upper Diatomaceous Earth (*Mde*)



Photo 1. View of the Red Lake mining operation. Upper carbon-rich unit (*Muc*) containing leonardite is exposed within the bench in the central portion of the photo.

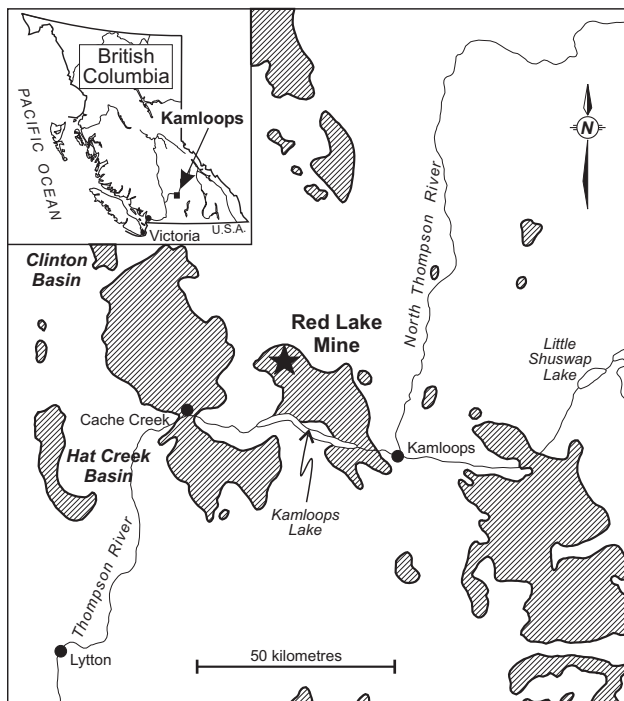


Figure 2. Geological setting of the Red Lake deposit, British Columbia, Canada. Tertiary basins are indicated by shaded areas.

and overburden (*Qs*). These units are described in stratigraphic order (from oldest to youngest) below.

Grey Andesite (possibly basalt) is the oldest unit that outcrops in the mine area. It is a grey, locally purplish, in places vesicular or amygdaloidal. This andesite forms the basin that holds the diatomite deposit and it probably belongs to the Eocene Kamloops Group (Read, 1995). It consists essentially of plagioclase laths (50-70%), measuring less than 0.5 mm, set in a glassy matrix. Altered ferromagnesian minerals form less than 7% of the rock. The materials observed to fill vesicles are calcium carbonate, zeolite (probably analcime), celadonite, hematite and amorphous silica.

Medium grey, vesicular breccia consisting of angular clasts ranging from a few millimetres to a few centimetres (rarely 20 centimetres) was described by (Read, 1995), however it is not shown on the cross sections of the deposit. Read (1995) interprets it as sedimentary in origin.

Basal Carbon-rich unit (*Mbc*) forms lenses up to 2 m in thickness (Figure 3a and 3b). This layer was not exposed at the time of our visit and therefore not studied or sampled.

Basal Diatomaceous Earth (*Mbd*) is a brown diatomite layer overlaying the lower organic-rich horizon. It may locally reach over 5 m in thickness. This layer is relatively porous, soft and relatively ductile. In places, one can drive a geological pick into it without breaking it. Because of its water content, this rock appears denser than the diatomite-bearing rock from the *Upper Diatomaceous Earth unit*. It has a massive appearance and consists mainly of clay (probably montmorillonite) and diatoms.

Upper Carbon-rich unit (*Muc*) is described as carbonaceous shale, coal-like material and black wood fragments (Read, 1995). The unit is over 2 metres thick locally and it separates the previously described brown Basal Diatomaceous Earth from the Upper Diatomaceous Earth unit. This organic-rich layer was exposed in the floor of the mine at the time of our visit and nine samples were collected from it. The upper 15 centimetres is particularly rich in light-weight woody fragments. The unit also contains crumbly, sand-like diatomite-bearing lenses or layers.

Upper Diatomaceous Earth (*Mde*) unit is mostly beige to pale grey in colour, but it may also be brownish. It locally reaches up to 7 m in thickness (Figure 3). It is commonly laminated or massive, but in many places blocky. Centimetre-scale parting is a dominant texture. This highly absorbent, light-weight unit (density 0.61 g/cm³) consists of montmorillonitic clay, and contains 20 to 35% of diatomite skeletons. This rock has provided, up to now, the bulk of the raw material for the Western Industrial Clay Products plant.

Overburden (*Qs*) consists mainly of unconsolidated glacial and alluvial deposits, however, it may consist locally of material that was relocated during mining.

GEOCHEMISTRY OF THE UPPER CARBON-RICH UNIT

Nine representative samples were selected and described in the field. All samples came from the Upper carbon-rich unit (*Muc*). These samples weighed 3 to 5 kilograms each.

All samples were split; one half was kept for further studies and the other was sent to Loring Laboratories in Calgary for sample preparation and chemical analyses.

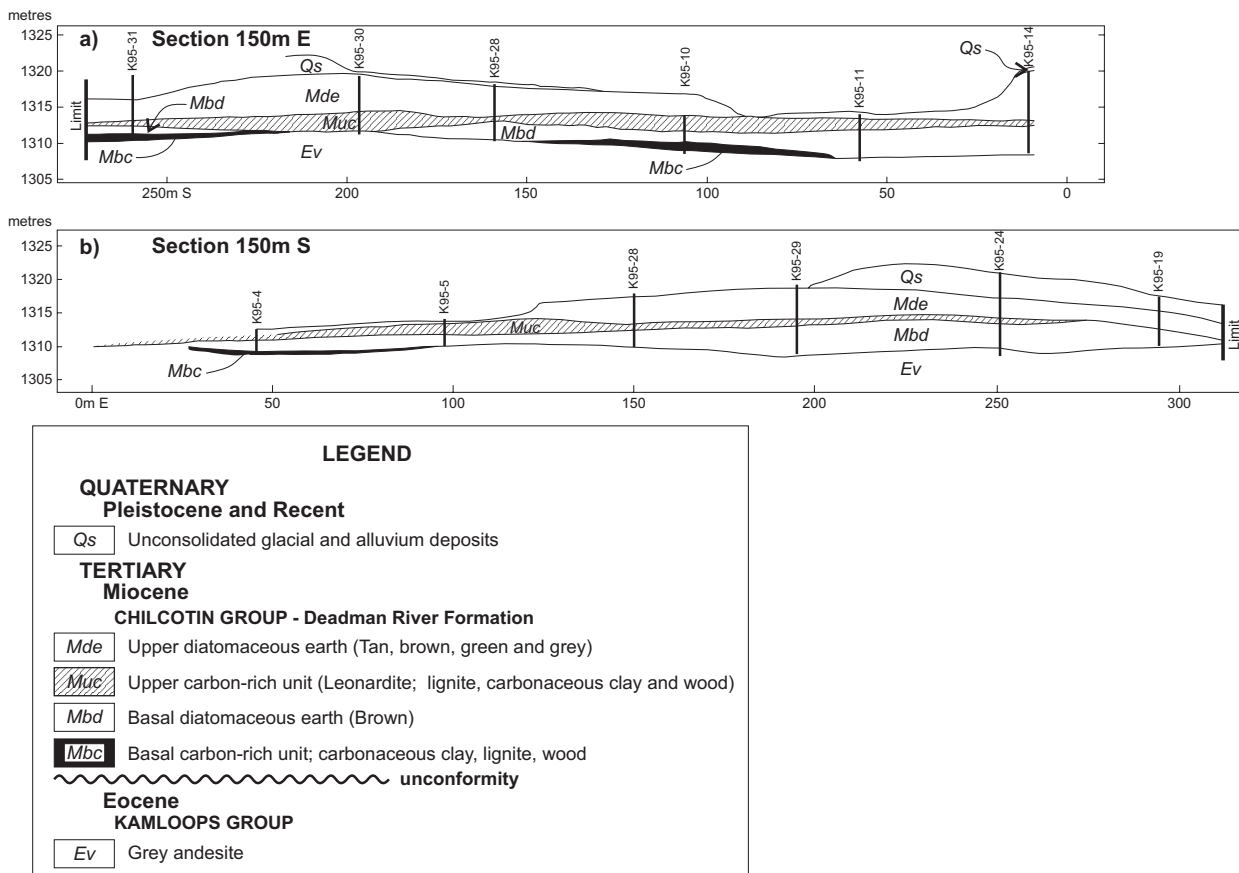


Figure 3. North-south (a) and east-west (b) sections of the Red Lake deposit (modified from Read (1995)).

Analytical Procedure

The sample preparation and analytical methods are described below.

Sample preparation: All samples were weighed, partially dried to bring the moisture near equilibrium with the atmosphere, crushed and split using a riffle. A representative sample was obtained and crushed to -60 mesh and mixed again. The detailed methodology is described under ASTM Designation D 2013 - 86.

Humic acid determination (colorimetric method): A 0.5 g sample was extracted with 50 ml of 0.5 N NaOH solution for 10 minutes. Two ml of the filtered solution was transferred to a 100 ml flask and brought to bulk volume. The absorbency was read on the Bausch and Lomb Spectronic 20 spectrometer and compared to the standard carried with the analysis.

Humic acid determination (chemical precipitate method): A 2 g sample was extracted with 50 ml of 0.5 N NaOH solution for 1.5 hours and centrifuged. The supernatant liquid was transferred to another weighed bottle and acidified to pH < 1 and centrifuged again. The solid was dried and weighed as humic acid solid.

Major elements analysis: 0.2 g of coal ash was fused with lithium metaborate, dissolved in 5% HNO₃ and analysed using the Thermo Jarrell Ash Model IRIS high reso-

lution ICP unit. Consequently the results are reported on an "in ash" basis.

Trace elements analysis: 1 g of coal was ashed and then transferred to a Teflon beaker and digested using HF, HNO₃ and HCl until near dryness. The sample was then boiled with 20% HCl and transferred to a 100 ml flask and made up to a final volume of 100 ml. The sample was analyzed by the Thermo Jarrell Ash Model IRIS high resolution ICP unit.

Ultimate analysis: This analysis involves the determination of the water (H₂O), carbon (C), hydrogen (H), nitrogen (N), sulphur (S) and ash content. The value of oxygen is not measured, but determined by difference. The standard method for ultimate analysis of coal and coke as described under ASTM Standard Designation D 3176 - 84 was used.

Broughton (1972b) describes a low-angle X-ray scattering method for identification of leonardite. There was no time to test this method and to compare the results with above described methods used to determine humic acid content, but this analytical procedure may be a worthwhile as an exploration tool.

TABLE 2
HUMIC ACID CONTENT OF THE UPPER
CARBON-RICH UNIT (MUC) DETERMINED USING
COLORIMETRIC AND CHEMICAL
PRECIPITATION METHODS

SAMPLE ID	BASIS	% Humic	
		% Humic Acid Colorimetric	Acid Chemical ppt
RED-00-7	A.D.	5	-
RED-00-15	A.D.	12	-
RED-00-16	A.D.	52	75.0
RED-00-17	A.D.	43	45.2
RED-00-19	A.D.	9	5.8
RED-00-20	A.D.	47	48.8
RED-00-22	A.D.	22	7.0
RED-00-23	A.D.	13	3.7
RED-00-24	A.D.	20	13.8

Results of Analyses

The results of the humic acid determination using both the colorimetric and chemical precipitate methods are shown in Table 2.

Nine samples were analysed using the former and only seven using the latter method, as a cost saving measure. Both types of determinations were done on air dried samples. The same seven samples as used above were also analysed for major (Table 3) and trace elements (Table 4). Table 5 shows the results of the ultimate analysis. For every sample H₂O, C, H, N, ash, S and oxygen content are reported as A.R. (as received), A.D. (air dried) and Dry (oven dried).

DISCUSSION OF THE DATA

The Red Lake diatomite deposit contains the Upper carbon-rich unit (*Muc*) which is the first documented oc-

currence of humic acid-rich material, that can be described as leonardite or humate, in British Columbia. The correlation coefficient (*r*) method described by Harrell (1987) was used to compare the results of the colorimetric and chemical precipitate methods. In this correlation *r* varies from -1 to 1. These values correspond to either perfect negative or positive correlation, respectively. If *r* = 0, it means that there is no correlation at all. In our case, *r* = 0.96, indicating excellent correlation.

Figure 4 indicates that the low-cost colorimetric method is probably sufficient for exploration purposes, and suggests that the chemical precipitate method should be used where precision is required. The line with 45° slope on Figure 4., originating at the intersection of X and Y axis, is shown for reference purpose only. If the two, humic acid determination methods that we used gave identical results, all the data would plot directly on this line.

The samples from this study indicate extreme variations in humic acid concentrations perpendicular to the strike and possibly along strike. Such variation indicates that a much broader systematic sampling is required to obtain a representative humic acid content of the *Upper Carbon-rich unit* at the mine site. It also suggests that careful blending would be needed to obtain and maintain a consistent humic acid content if leonardite material from Red Lake were to be mined and marketed as a soil conditioner.

Trace element data, reported on "total rock" basis (not in ash), indicate that silver (Ag), arsenic (As), gold (Au), beryllium (Be), bismuth (Bi), cadmium (Cd) and selenium (Se) are below the detection limit. Other elements are present in trace, but detectable quantities including Ba (< 291 ppm), Co (< 96 ppm), Cr (< 19 ppm), Cu (< 32 ppm), La (< 119 ppm), Mn (< 187 ppm), Mo (< 44 ppm), Ni (< 107 ppm), P (< 0.087 %), Pb (< 47 ppm), Sb (< 16 ppm), Sr (< 86 ppm), Th (< 5 ppm), U (< 5 ppm), V (< 270 ppm), W (< 23 ppm), Zn (< 74 ppm). Boron levels (19 to 547 ppm) most likely reflect the original playa-type environment. The concentrations of ash from these samples (Table 4) indicate that there are no anomalous levels of trace elements including As, Se, base metals and

TABLE 3
MAJOR ELEMENTS ANALYSIS OF ASH DERIVED FROM THE UPPER CARBON-RICH UNIT (MUC), RED LAKE
DEPOSIT (WHOLE ROCK ICP ANALYSIS)

Sample No.	Al ₂ O ₃ (%)	Ba (ppm)	CaO (%)	Cr (ppm)	Fe ₂ O ₃ (%)	K ₂ O (%)	MgO (%)	MnO (%)	Na ₂ O (%)	Ni (ppm)	P ₂ O ₅ (%)	SO ₃ (%)	SiO ₂ (%)	Sr (ppm)	TiO ₂ (%)	V ₂ O ₅ (%)	Undet. (%)
RED-00-16	19.97	555	3.10	110	28.14	0.20	1.15	0.01	0.17	467	0.209	0.92	41.32	138	0.55	0.09	3.92
RED-00-17	15.31	621	1.67	38	26.43	0.45	0.83	0.02	0.56	92	0.535	0.33	48.87	137	0.99	0.13	3.27
RED-00-19	5.13	79	0.72	29	1.62	0.14	0.56	0.01	0.06	67	0.013	0.30	87.80	36	0.13	0.03	3.34
RED-00-20	14.75	283	13.64	79	10.88	0.23	6.34	0.16	0.13	401	0.093	3.03	45.09	393	0.32	0.07	5.16
RED-00-22	17.26	210	1.23	34	3.17	0.20	0.94	0.02	0.12	32	0.031	0.30	69.09	64	0.57	0.04	6.96
RED-00-23	19.25	163	1.26	61	4.56	0.18	1.08	0.02	0.15	353	0.046	0.48	67.40	65	0.64	0.04	4.76
RED-00-24	22.95	241	4.30	85	9.16	0.22	3.54	0.07	0.21	175	0.100	1.74	48.84	142	1.05	0.09	7.55

Whole rock analysis expressed on an "in ash" basis.

0.2 g Coal ash fused with lithium metaborate, and dissolved in 5% HNO₃.

TABLE 4
TRACE ELEMENTS ANALYSIS FROM THE UPPER CARBON-RICH UNIT (MUC),
RED LAKE DEPOSIT (32 ELEMENT ICP ANALYSIS)

Sample Name	Ag (ppm)	Al (%)	As (ppm)	Au (ppm)	B (ppm)	Ba (ppm)	Be (ppm)	Bi (ppm)	Ca (%)	Cd (ppm)	Co (ppm)	Cr (ppm)	Cu (ppm)	Fe (%)	K (%)	La (ppm)
RED-00-16	<0.5	2.57	<5	<5	64	148	<5	<5	0.44	<5	96	19	15	4.17	0.03	119
RED-00-17	<0.5	3.99	<5	<5	123	291	<5	<5	0.50	<5	76	12	22	7.41	0.19	50
RED-00-19	<0.5	2.42	<5	<5	80	53	<5	<5	0.42	<5	35	<5	20	0.85	0.08	16
RED-00-20	<0.5	1.52	<5	<5	19	67	<5	<5	1.54	<5	29	12	12	1.43	0.04	35
RED-00-22	<0.5	7.62	<5	<5	547	185	<5	<5	0.71	<5	18	19	77	1.83	0.19	42
RED-00-23	<0.5	8.28	<5	<5	470	120	<5	<5	0.70	<5	29	19	32	2.68	0.16	42
RED-00-24	<0.5	2.03	<5	<5	145	48	<5	<5	0.41	<5	25	<5	19	0.96	0.04	24

Sample Name	Mg (%)	Mn (ppm)	Mo (ppm)	Na (%)	Ni (ppm)	P (%)	Pb (ppm)	Sb (ppm)	Sr (ppm)	Th (ppm)	Ti (%)	U (ppm)	V (ppm)	W (ppm)	Zn (ppm)	Se (ppm)
RED-00-16	0.16	7	22	0.04	82	0.020	21	6	35	<5	0.08	<5	65	23	74	<5
RED-00-17	0.23	52	44	0.33	107	0.087	24	13	64	<5	0.25	<5	270	14	60	<5
RED-00-19	0.26	45	7	0.05	44	0.007	17	7	31	<5	0.06	<5	27	10	71	<5
RED-00-20	0.67	187	<5	0.03	101	0.009	14	<5	86	<5	0.04	<5	70	6	33	<5
RED-00-22	0.48	94	<5	0.23	32	0.016	47	16	54	<5	0.31	<5	89	8	52	<5
RED-00-23	0.53	95	<5	0.24	41	0.021	47	13	52	<5	0.34	<5	77	6	67	<5
RED-00-24	0.32	74	<5	0.05	38	0.008	18	<5	29	<5	0.11	<5	68	9	49	<5

Results expressed on an "in coal" basis.

1g of coal was ashed, and the ash was then totally dissolved using HF and aqua regia and taken to a final volume of 100 ml.

TABLE 5
ULTIMATE ANALYSIS OF UPPER CARBON-RICH UNIT (MUC)
RED LAKE DEPOSIT (A.R. - AS RECEIVED, A.D. - AIR DRIED)

SAMPLE ID	BASIS	H2O (%)	C (%)	H (%)	N (%)	ASH	S (%)	O (%)
RED-00-16	A.R.	67.56	14.08	0.97	0.44	8.60	0.17	8.17
	A.D.	10.98	38.65	2.67	1.20	23.60	0.47	22.43
	Dry	-	43.42	3.00	1.35	26.51	0.53	25.20
RED-00-17	A.R.	30.23	21.56	1.52	0.77	33.99	0.38	11.55
	A.D.	8.18	28.37	2.00	1.01	44.74	0.50	15.20
	Dry	-	30.90	2.18	1.10	48.73	0.54	16.55
RED-00-19	A.R.	53.31	4.80	0.85	0.38	37.81	0.22	2.64
	A.D.	4.2	9.85	1.74	0.77	77.57	0.45	5.42
	Dry	-	10.28	1.82	0.80	80.97	0.47	5.65
RED-00-20	A.R.	42.01	28.49	2.27	0.76	11.60	0.81	14.07
	A.D.	8.19	45.11	3.59	1.20	18.36	1.28	22.27
	Dry	-	49.13	3.91	1.31	20.00	1.39	24.26
RED-00-22	A.R.	39.78	6.09	0.65	0.48	47.70	0.21	5.09
	A.D.	3.97	9.71	1.04	0.77	76.06	0.33	8.12
	Dry	-	10.11	1.08	0.80	79.20	0.34	8.46
RED-00-23	A.R.	33.25	6.67	1.03	0.20	52.79	0.30	5.75
	A.D.	4.36	9.56	1.48	0.29	75.64	0.43	8.24
	Dry	-	10.00	1.54	0.30	79.09	0.45	8.62
RED-00-24	A.R.	33.25	34.24	3.07	0.01	12.58	0.71	16.15
	A.D.	5.73	48.36	4.33	0.01	17.76	1.00	22.81
	Dry	-	51.30	4.60	0.01	18.84	1.06	24.19

Note: Hydrogen and Oxygen do not include H and O from sample moisture. Value of oxygen by difference.

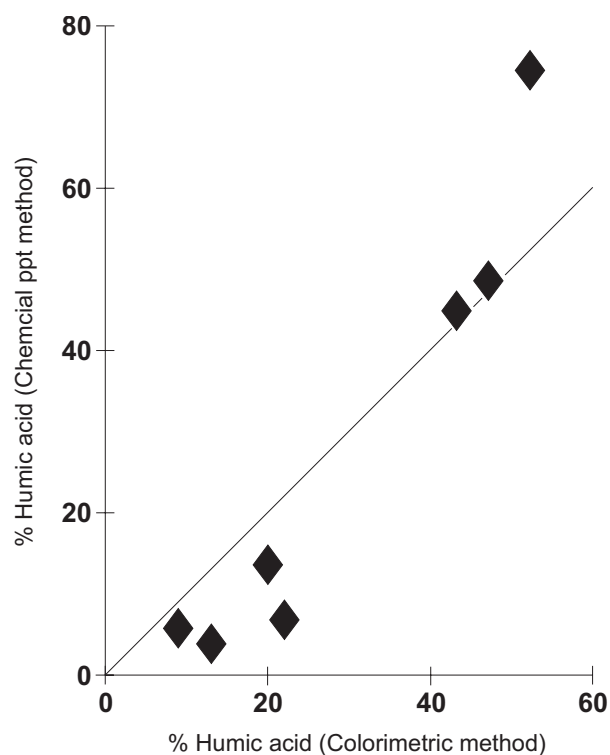


Figure 4. Relationship between humic acid determination results using the chemical precipitate and colorimetric methods.

radionuclides that would limit the use of this material in agricultural and horticultural applications.

The ultimate analysis also provided useful information. It indicates that within the pit, water may represent 30 to over 67% and ash 8.6 to over 50% of the unprocessed raw material from the Upper Carbon-rich unit (*Muc*). Sulphur content varies from 0.17 to 0.71%, nitrogen from 0.01 to 0.77% and oxygen from 2.64 to 16.15% on an as received basis. Upon drying the proportions of all of these constituents, except water, will increase substantially.

Major element analyses (Table 3) shows that SiO₂ (41 to over 80%), Fe₂O₃ (1.62 to 28.14%) and Al₂O₃ are the major constituents in the ash. High SiO₂ content was expected because the host is diatomite-bearing. High Al₂O₃ is due to clay content. There appears to be no relationship between Fe₂O₃ and SO₃ confirming the absence of sulphides previously established by visual observation and fully expected in the highly oxidized environment

SUMMARY

Organic material from the Red Lake diatomite mine can be described as leonardite or humate. This preliminary study is not based on systematic sampling, and indicates extreme fluctuations in humic acid content within the organic-rich layer. We can not speculate on the average humic acid content of the *Upper Carbon-rich unit* (*Muc*) exposed at the Red Lake mine. No high levels of trace elements that would preclude the use of the material

as a soil conditioner were detected. In summary, our data suggests that the *Upper Carbon-rich unit* currently exposed in the floor of the open pit has potential as a soil conditioner or at the very least, as a material for tailing re-habilitation. The *Lower Carbon-rich unit* was not sampled but it is possible that it may also have high humic acid content.

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