

MINERALOGICAL REACTIONS IN LYSIMETERS CONTAINING TAILINGS WITH DIFFERENT SULFIDE CONTENTS

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Abstract

In June of 1993 INCO Ltd installed three outdoor lysimeters at Sudbury, Ontario, each holding 180 tonnes of tailings, and in which the only significant variable was the sulfide content (0.35, 0.98, and 2.3 wt% S), present almost wholly as pyrrhotite. The primary objective was to monitor oxidation reactions in each lysimeter, principally to determine whether the low-sulfide tailings could be used as a relatively unreactive cover on the existing 450 Mt of impounded tailings, and on future impoundments. Mineralogical examination of cores from the three lysimeters after 2½ years of weathering showed oxidation profiles of variable intensity correlative with S contents. Pyrrhotite altered to goethite, lepidocrocite, native sulfur, and an iron sulfate; the formation of secondary jarosite indicated partial solubilization of K-bearing silicates, confirmed by vermiculitic alteration of phlogopite. To determine the alteration progression, particularly among the silicate and aluminosilicate minerals that might be contributing to acid-neutralizing reactions, the lysimeters have been re-cored, approximately 4½ years after installation. The new mineralogical results indicate that downward oxidation has progressed significantly; moreover, a less intense but readily detectable upward oxidation is also occurring.

Introduction

The disposal of wastes at a mine site affects the planning, capital costs, and operation costs. As well, there is a need to ensure that a mine will not release acid drainage and metals that will be environmentally harmful, either during the life of the mine or after its decommissioning. Various options are available to render mine wastes to an environmentally benign status. Among the options considered by INCO Ltd at its Copper Cliff tailings facility near Sudbury, Ontario, was the flotation production of a low-sulfide tailings product that could be used to build tailings dams, as well as to provide a relatively unreactive cover for impoundments containing tailings that have a high sulfide content. To assess the environmental viability of employing the low-sulfide product, in June of 1993 three outdoor lysimeters, each holding 180 tonnes of tailings, were installed at the Copper Cliff tailings containment site. The lysimeters were cored for mineralogical examination after 2½ years of natural weathering, and were re-cored in 1997, after approximately 4½ years of natural weathering. In this paper we describe the progression of mineralogical alteration that has occurred.

Lysimeters and Starting Materials

The design and site layout of the lysimeters are shown in Figures 1 and 2. The lysimeters are within INCO's Copper Cliff tailings area, a facility that is Canada's largest and that currently contains about 450 Mt of tailings in which pyrrhotite is the principal sulfide mineral. The lysimeters were constructed to hold three products (main tailings, total tailings, and low-sulfide tailings) that are subjected to natural weathering, but which are isolated from each other and the local regime by impermeable geomembranes. The bottom of each lysimeter has a drain that facilitates downward movement of the pore waters and their subsequent sampling (Fig. 2). The pore waters are sampled semi-annually, in the spring and early fall, so that changes over the summer, during which the moisture content is low and sulfide oxidation is at a maximum, can be determined (6). The "main tailings" contain ~1 wt% S and represent tailings that have been discharged by INCO since 1994. The "total tailings" contain ~2.3 wt% S and represent about 85% of the tailings that have been accumulated since the 1930s; the higher sulfide contents of the total tailings are present because pyrrhotite had not been separated in the concentration process, as has been done since 1994. The pyrrhotite-rich concentrate produced since 1994 contains 10-12 wt% S ($\geq 40\%$ pyrrhotite), and these wastes are apparently deposited beneath a water cover in a separate part of the tailings-storage area (10). The "low-sulfide" tailings represent the residuals after production of the pyrrhotite concentrate; the particular sample of low-sulfide tailings used to fill the lysimeter was produced in 1993 during plant trials at INCO's Clarabelle mill (10).

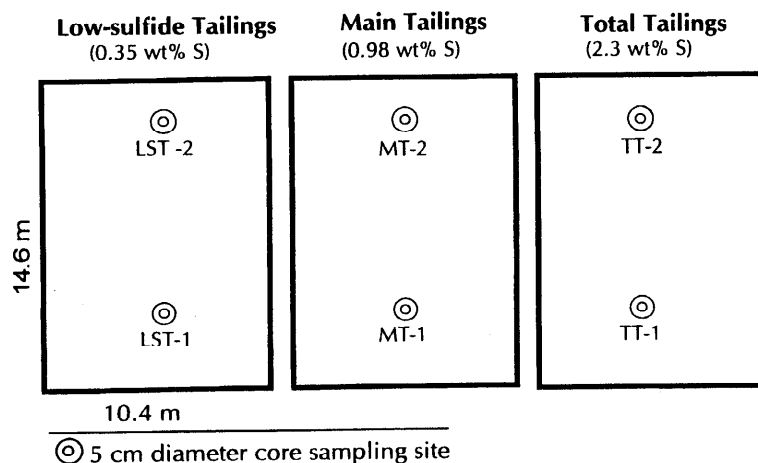


Figure 1: Plan view of the lysimeters, showing the approximate locations cored for mineralogical studies. Tailings were discharged into the lysimeters from near the sites labelled 1, and sites labelled 2 are distal.

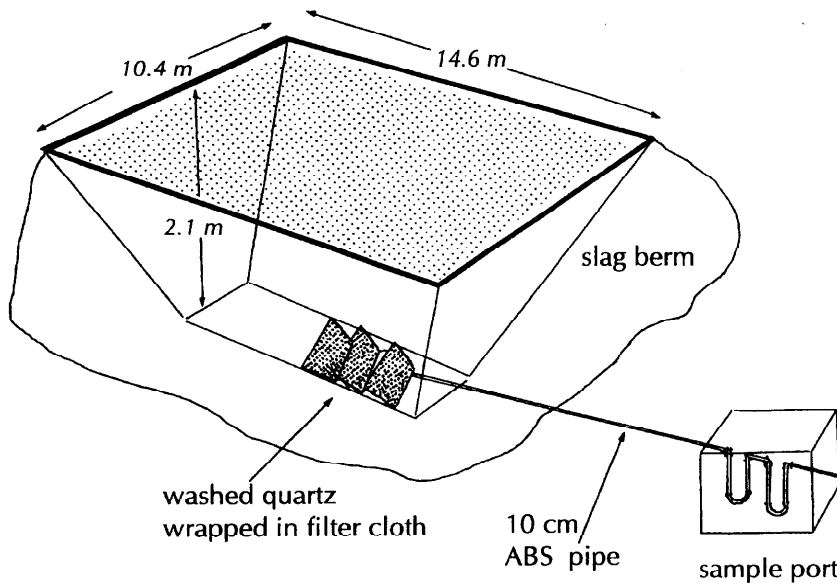


Figure 2: General design of the lysimeters; re-drawn from (10).

Various whole-rock ICP analyses of the lysimeter tailings are given in Table I. The cored tailings were obtained using thin-walled aluminum tubes, 5 cm in diameter. After coring, the tubes and their contents were cut into easily managed lengths, generally 30 cm or less. These were sealed with plastic caps, and were frozen. For the mineralogical study, the frozen cores were cut along their length, were extruded onto plastic sheeting, and were dried at room temperature.

Columns 1, 2, and 3 in Table I are representative of the unaltered tailings in each lysimeter, and column 4 represents the oxidized zones cored in 1995. The low-sulfide lysimeter was filled by side-discharge, but deposition in the others was from the T1 end of the lysimeters (Fig. 1);

TABLE I. Whole-rock Chemical Analyses of Lysimeter Tailings

wt%	Low-sulfide Tailings				Main Tailings				Total Tailings			
	1	2	3	4	1	2	3	4	1	2	3	4
SiO ₂	56.05	58.7	58.4	56.2	54.12	55.6	55.8	52.6	54.33	55.6	55.1	52.5
Al ₂ O ₃	13.17	14.5	14.4	11.9	12.90	13.6	13.8	11.9	12.85	13.4	13.2	11.6
TiO ₂	n.a.	0.76	0.75	0.89	n.a.	0.77	0.79	0.84	n.a.	0.78	0.77	0.80
Cr ₂ O ₃	0.06	0.04	0.04	0.04	0.06	0.05	0.05	0.06	0.05	0.04	0.04	0.05
FeO	11.11	10.2	10.0	10.7	12.23	12.2	12.5	12.0	13.25	12.9	13.3	12.5
MnO	0.17	0.16	0.16	0.16	0.16	0.16	0.16	0.15	0.16	0.16	0.16	0.14
CoO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
NiO	0.11	0.09	0.09	0.09	0.14	0.13	0.13	0.12	0.20	0.18	0.18	0.15
CuO	0.09	0.12	0.12	0.05	0.12	0.10	0.10	0.08	0.14	0.14	0.14	0.12
MgO	5.04	5.02	4.98	4.14	5.67	5.77	5.8	4.93	5.04	5.38	5.41	4.18
CaO	6.24	6.4	6.4	4.28	6.67	6.71	6.83	4.92	6.27	6.23	6.24	4.32
K ₂ O	1.13	1.28	1.28	1.24	0.99	1.25	1.25	1.21	1.05	1.18	1.15	1.24
P ₂ O ₅	n.a.	0.15	0.14	0.17	n.a.	0.15	0.15	0.16	n.a.	0.15	0.15	0.16
CO ₂	0.37	n.a.	n.a.	n.a.	0.40	n.a.	n.a.	n.a.	0.37	n.a.	n.a.	n.a.
S	0.35*	0.4	0.4	0.17	0.98*	0.98	0.95	0.80	2.3*	2	2	1.28

1. Data from (10); n.a. – not analyzed or not reported; total Fe as FeO; Na₂O not determined

2, 3. Stuparyk (1996, unpublished) for unoxidized tailings in 1993; S is total S

4. Oxidized zone in core from 1995, after ~2½ years of weathering; S is total S; sample is a composite of T1 and T2 cores from 0 to 15 cm

*Sulfur analyses from (6)

TABLE II. Image-analysis Results (Wt%) for Lysimeter Tailings

Mineral	Low-sulfide Tailings	Main Tailings	Total Tailings
quartz	17	13	18
plagioclase	28	27	26
orthopyroxene	5	6	4
clinopyroxene	6	6	4
amphibole	28	28	24
phlogopite	6	5	8
K-feldspar	2	2	1
muscovite	<0.5	<0.5	<0.5
chlorite	3	4	3
epidote	1	1	1
titanite	<0.5	nd	nd
ilmenite	1	1	2
magnetite	<0.5	<0.5	1
total sulfides	1	4	6
- pyrrhotite	1	3	6
- pyrite	<0.5	<0.5	<0.5
- chalcopyrite	<0.5	<0.5	<0.5
- pentlandite	<0.5	<0.5	<0.5

nd: not detected

the result of end-discharge has been a grain-size variation, with the silt-size fines partly concentrated at the T2 (distal) ends of the lysimeters. Although this variation affects the amount of moisture that is retained by the tailings (6), the size distribution emulates that obtained by spigot discharge of tailings.

In addition to whole-rock analyses, splits of the unoxidized parts of the cored samples, both proximal and distal, were combined, and the composites were used to obtain the mineralogy of the unoxidized tailings in each lysimeter. Quantification of the mineralogy was done at CANMET, Natural Resources Canada, Ottawa, using a MP-SEM-IPS image-analysis system in which the minerals were first discriminated on the basis of their grey level in a backscattered-electron image (12). Minerals with overlapping grey levels were subsequently distinguished on the basis of the count rates for various elements as detected by energy-dispersion spectra. The results of the image-analysis study (Table II) confirm that the various tailings are mineralogically similar,

but with an appreciable difference in their pyrrhotite contents. Mineralogical studies have shown that the small amounts of P_2O_5 reported in Table I, but not accounted for in Table II, are attributable to the presence of apatite (8), and that CO_2 is largely attributable to dolomite.

Acid-Base Accounting

Figure 3 summarizes the results of five static tests on each of the three tailings types (10). According to acid-base accounting guidelines for British Columbia (5), none of the tailings falls within the category of "not potentially acid-generating"; however, all of the results for the low-sulfide tailings are well within the field of uncertainty between acid-generating and not acid-generating (Fig. 3). To achieve a benign (not acid-generating) status, the low-sulfide tailings would have to contain about 1 wt% calcite, or alternatively, would have to be completely free of sulfide. Thus, little neutralization potential seems to be contributed by the aluminosilicate assemblage even though 32 to 40% of it consists of pyroxene and amphibole, both of which weather moderately rapidly in field conditions (2) and in experimental dissolution studies at pH 5 (11). This result raises questions as to which, if any, of the common rock-forming aluminosilicate minerals furnish significant neutralization potential, and whether any of them react sufficiently rapidly to prevent the generation of ARD (acid rock drainage).

Mineral Compositions

Within the Sudbury igneous complex that hosts the Cu-Ni ores, compositions of plagioclase range from An_{61} to $An_{<10}$, the $Fe/(Fe+Mg)$ mol ratio of orthopyroxene ranges from 0.31 to 0.38, and that of augite varies from 0.27 to 0.60 (4). For the lysimeter tailings, electron-microprobe analyses (8) gave compositions of An_6 to An_{70} for plagioclase ($n = 5$), and $Fe/(Fe+Mg)$ values of 0.26 to 0.39 for orthopyroxene (enstatite; $n = 5$). Amphiboles ($n = 12$)

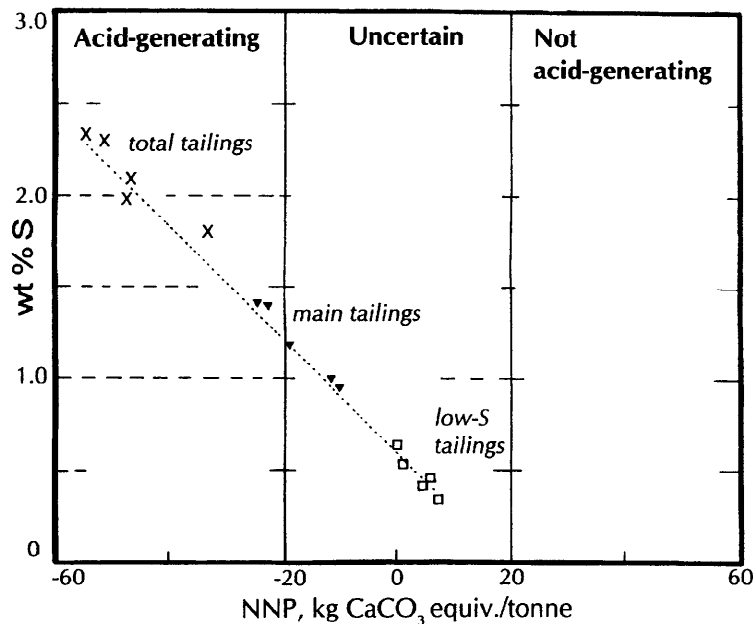


Figure 3: Wt% S versus NNP (net neutralization potential) of the three lysimeter tailings. Data from (10).

are Ca-rich and are predominantly actinolite, but include magnesiohornblende, ferrohornblende, ferrotschermakite, and ferro-edenite (nomenclature of Leake *et al.*, 3). Phlogopite, ideally $\text{KMg}_3\text{AlSi}_3\text{O}_{10}(\text{OH})_2$ (7), has formula Mg variable from 1.66 to 2.86 ($n = 15$). Chlorite ($n = 16$) is almost equally divided between ferroan clinocllore and chamosite, with the latter commonly near the end-member, *i.e.*, $\text{Fe}/(\text{Fe}+\text{Mg}) = \text{up to } 0.94$.

Among the non-silicate minerals, analyses (8) of magnetite ($n = 11$) showed up to 0.49 wt% Cr_2O_3 , 0.85 wt% V_2O_3 , 0.27 wt% MnO , and 0.19 wt% NiO ; averages are 0.24, 0.44, 0.10, and 0.08, respectively. Ilmenite ($n = 19$) averages 0.08 wt% Cr_2O_3 , 0.13 wt% V_2O_3 , and 2.1 wt% MnO . Both the hexagonal and the monoclinic varieties of pyrrhotite are present in the tailings. Compositions ($n = 14$) range from $(\text{Fe}_{6.79}\text{Ni}_{0.06})_{\Sigma 6.85}\text{S}_8$ (monoclinic) to $(\text{Fe}_{7.34}\text{Ni}_{0.07})_{\Sigma 7.41}\text{S}_8$ (hexagonal). All grains were found to be nickeliferous, to a maximum of 0.99 wt% Ni. Cobalt contents are low, and most Co resides in pentlandite (up to 1.76 wt% Co, $n = 11$).

Progress of Alteration

The three lysimeters have been exposed to identical field conditions for identical periods of time. The primary mineralogy is similar for each lysimeter, except that the amount of pyrrhotite increases progressively from the low-sulfide tailings, to the main tailings, to the total tailings. The principal variable in the solids is therefore the pyrrhotite content.

Hydrogeochemical Effects

After 18 months of exposure that involved two peak periods (two summers) of oxidation, the lysimeters had developed a water table for which the unsaturated zone was about 50 cm deep (10). At this time, the total tailings (highest sulfide) and main tailings had already developed a low pH in the near-surface zone, and elevated levels of Fe and SO_4 were evident in the unsaturated zone of the total tailings. In contrast, the same zone in the low-sulfide tailings remained neutral. After 25 months, pore-water pH in near-surface samples of both the total and main tailings had decreased to ≤ 4 , and in the total tailings the maximum pore-water concentrations of Fe and Ni each exceeded 1500 mg/L (6). Although pore-water pH of the low-sulfide tailings remained near neutral even after 40 months of tailings weathering, in subsequent months these tailings also became acid-generating.

Initial Mineralogical Effects

The initial coring of the lysimeters for mineralogical studies was done in October, 1995, approximately 2½ years after weathering of the tailings had begun. By that time, sulfide oxidation had resulted in the local development of yellow to yellowish brown, ochreous coatings on the main and total tailings, but the low-sulfide tailings remained dark grey.

Mineralogical examination of the 1995 cored tailings showed that partial oxidation of pyrrhotite had occurred in the uppermost 30-50 cm of all three lysimeters, and that alteration of the sulfides was the most intense in the total tailings (highest pyrrhotite content) at the proximal (coarsest grain size) end of the lysimeter (9). In all cores, including those from the proximal and distal ends of the low-sulfide lysimeter, the pyrrhotite had locally undergone substantial alteration, as manifested mainly by various degrees of pseudomorphic replacement by Fe oxyhydroxides (chiefly goethite), native sulfur, and Fe sulfates (mainly $\text{FeSO}_4 \cdot n\text{H}_2\text{O}$). Other secondary precipitates detected in the tailings include small amounts of covellite, derived from the alteration of chalcopyrite, and small amounts of gypsum. The latter may have been partly or largely derived from the mill process-water. Except for biotite, alteration of aluminosilicates was not clearly evident. Alteration of biotite, detected only in the oxidized portion of the total-tailings lysimeter, is marked optically by lessened color intensity and pleochroism, is evident compositionally by decreases in the contents of K, followed by losses of Mg and Fe, and is XRD-detectable by the appearance of vermiculite-type alteration.

Subsequent Effects

Re-coring of the lysimeters in the fall of 1997, approximately 4½ years after lysimeter installation, showed that substantial changes had taken place in the 2-year interval since the initial coring. In the low-sulfide tailings, weak ochreous coloration of some layers is evident to a core depth of 38 cm in the distal sample, thus indicating that much more oxidation of sulfides and concomitant precipitation of secondary minerals has occurred. The main tailings (intermediate S) are intensely ochreous over most of the core length, with local weakly cohesive layers indicating that slight cementation by secondary minerals has occurred. The total tailings (highest sulfide) are similarly strongly oxidized to a depth of ~30 cm, but are succeeded downward by a grey, megascopically unaltered interval that continues to near the bottom of the hole; the bottom 3-4 cm of the core, however, has been strongly oxidized and is yellowish.

The megascopic appearance of the cores indicates that oxidation effects are weakest in the low-sulfide tailings, as is expected. Oxidation of the tailings with the highest sulfide content is much more intense, and an anomaly is present in that a thin, but strong and clearly demarcated oxidation zone occurs at the bottom of the hole, beneath unoxidized tailings (Fig. 4). The equivalent anomaly is even more pronounced in the main tailings; all of this core has been altered, with the middle depth of the core less affected, thus suggesting a merging of upward and downward oxidation (Fig. 4). The net effect is that the main tailings are the most strongly altered. A possible explanation for this peculiarity is offered in a later section.

Mineralogical Changes

The mineralogical changes after 2½ and 4½ years of weathering were compared by X-ray diffractometry, and by optical microscopy of polished thin sections in transmitted and reflected light. The sulfide alteration index (S.A.I.), which is a relative indication of the extent to which the primary sulfide minerals have been replaced (1), and which has been modified for application to the lysimeter cores (Table III), is illustrated in Figure 5.

As has been noted, the proximal main tailings and total tailings are coarser grained than their distal counterparts. In the cored low-sulfide tailings, however, this variation is less conspicuous, and the moisture contents at the two ends of this lysimeter have been found to be similar (6). In the other two lysimeters, however, the proximal (coarser grained) portions have

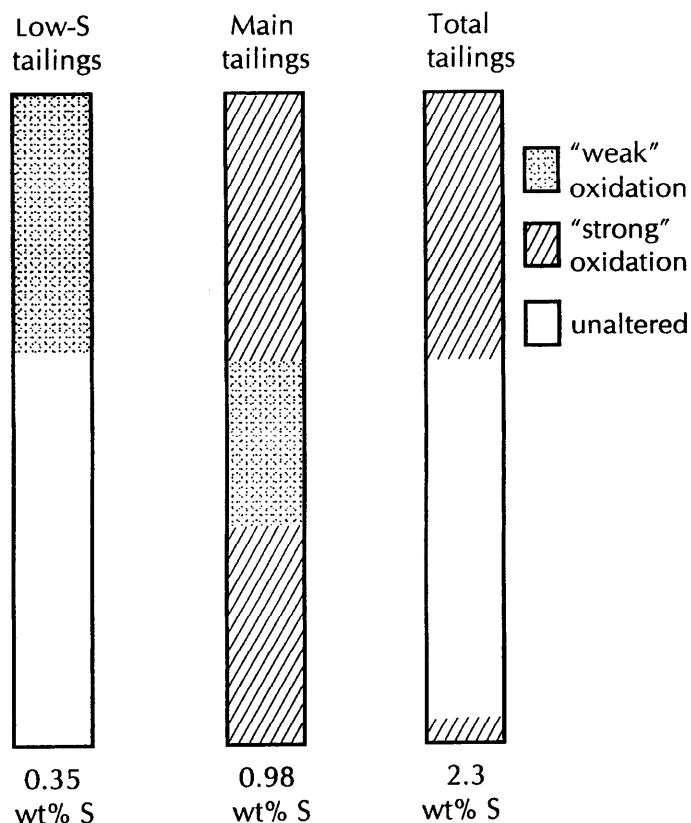


Figure 4: Schematic representation of alteration in the lysimeter cores, chiefly to illustrate the unusual alteration pattern of the proximal portion of the main tailings.

the (variable) depths at which the sampling had been done.

Figure 5 shows that the S.A.I. of the low-sulfide tailings is highest nearest the surface and decreases downward. Although spatial differences in the moisture content of the low-sulfide tailings were not observed (6), the S.A.I. suggests that oxidation of the proximal tailings has been more extensive than that in the distal tailings.

Main Tailings. Pronounced differences in the S.A.I. of the proximal and distal portions of the main tailings are evident. In the proximal (coarser) tailings, the inflection at about 50 cm (Fig. 5) marks the position at which downward and upward oxidation merge, and this position is the

moisture contents only half those in the distal portions (6). The progress of sulfide alteration was therefore examined in cores from a proximal as well as a distal site in each lysimeter.

Low-sulfide Tailings. The S.A.I. of the low-sulfide tailings is difficult to determine with accuracy because not only is the sulfide content relatively low, but a high proportion consists of locked grains of chalcopyrite and pentlandite. The mineralogy and the Cu and Ni contents (Table I) indicate that about half of the total S is attributable to chalcopyrite and pentlandite; thus, no more than 0.2 wt% S is present as pyrrhotite and other minerals, including sulfates. To offset potential bias in the microscopic determinations of the S.A.I., it would have been preferable to code the polished thin sections, using random numbers. This was not done, but bias was at least minimized by doing the microscopy of the samples in sequence, without prior knowledge of

Table III. Sulfide alteration index for the lysimeter tailings

Scale	Description of degree of sulfide alteration
10	Pyrrhotite and pentlandite completely obliterated; traces of pyrite and chalcopyrite may still be visible
9	Pyrrhotite absent, but pentlandite with thick alteration rims may be present
8-7	Pyrrhotite in traces as surviving cores or rods in pseudomorphs; pentlandite still rimmed
6-4	Pyrrhotite has broad alteration rims (thinning from scale 6 to scale 4) that are commonly multiphase; pentlandite grains may have thin rims (at scale 6) or show slightly altered edges (at scale 4)
4-2	Alteration rims on pyrrhotite thin as scale grades down to 2, at which many grains appear unaltered; pentlandite appears fresh
1-0	Very few grains of pyrrhotite are altered at an index of 1, usually along fractures or discontinuously at grain margins; at a scale of 0, all grains are pristine

Adapted from (8) and (9)

SULFIDE ALTERATION INDEX

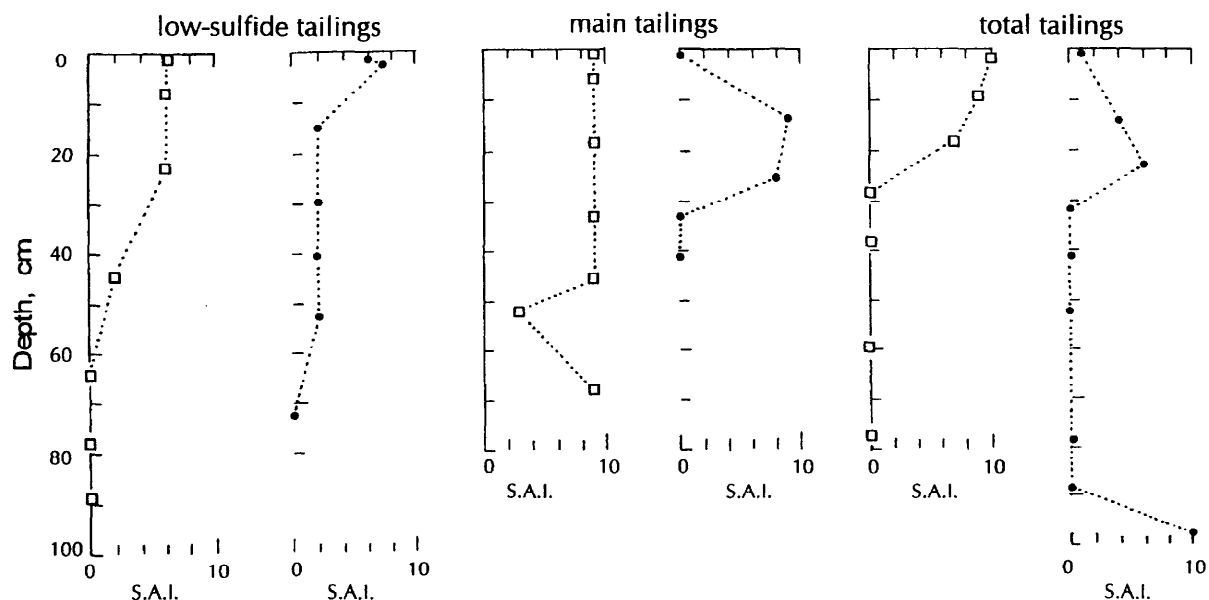


Figure 5: Sulfide alteration index of proximal (open squares) versus distal (solid circles) tailings.

only one at which pyrrhotite has not been obliterated. In the distal, much finer grained tailings, the near-surface tailings are grey rather than ochreous, and pyrrhotite within this interval is unaltered. An ochreous zone begins at ~13 cm depth, at which the S.A.I. is similar to that of the proximal tailings at the equivalent depth (Fig. 5). The total cored interval in the distal tailings may not have been deep enough to determine whether upward oxidation occurred at this site.

It is evident that grain size, and hence retained moisture and pore-gas oxygen concentration, has had a significant effect on the intensity of sulfide alteration in the main-tailings lysimeter. The effect of the finer grain size, and higher moisture content, of the distal tailings has been to inhibit alteration, both in overall intensity and in distribution (depth).

Total Tailings. The S.A.I. of the total tailings also shows the effects of the finer grain size of the distal tailings (Fig. 5). Whereas the near-surface interval in the proximal tailings is megascopically ochreous, reflecting strong oxidation, the equivalent interval in the distal tailings is megascopically grey, with only very minor wisps and streaks of yellowish, altered tailings. Upward oxidation is not evident in the core from the proximal area, but the distal core terminates in well-defined ochreous zone, 3-4 cm thick, wherein pseudomorphs after pyrrhotite are evident and a trace of chalcopyrite is the only surviving sulfide mineral.

Aluminosilicate Alteration

Although the whole-rock chemical analyses (Table I) show that the oxidized tailings have apparently decreased in Mg, Ca, Al, Si, and S relative to their fresh counterparts, it is difficult to assess the validity of these results because Na was not determined, and because the tailings do not show a significant or systematic increase in Fe. Optical microscopy, however, indicates that phlogopite has partly altered to a vermiculite-type mineral characterized by a 12 Å X-ray diffraction peak. Shaw (9) reported that, after 2½ years of oxidation, the vermiculitic mineral occurred only in near-surface samples of the total tailings. After 4½ years, however, the mineral is readily detected in all tailings types (Fig. 6). Alteration of phlogopite released K to form jarosite, which is also detectable now in all three tailings types.

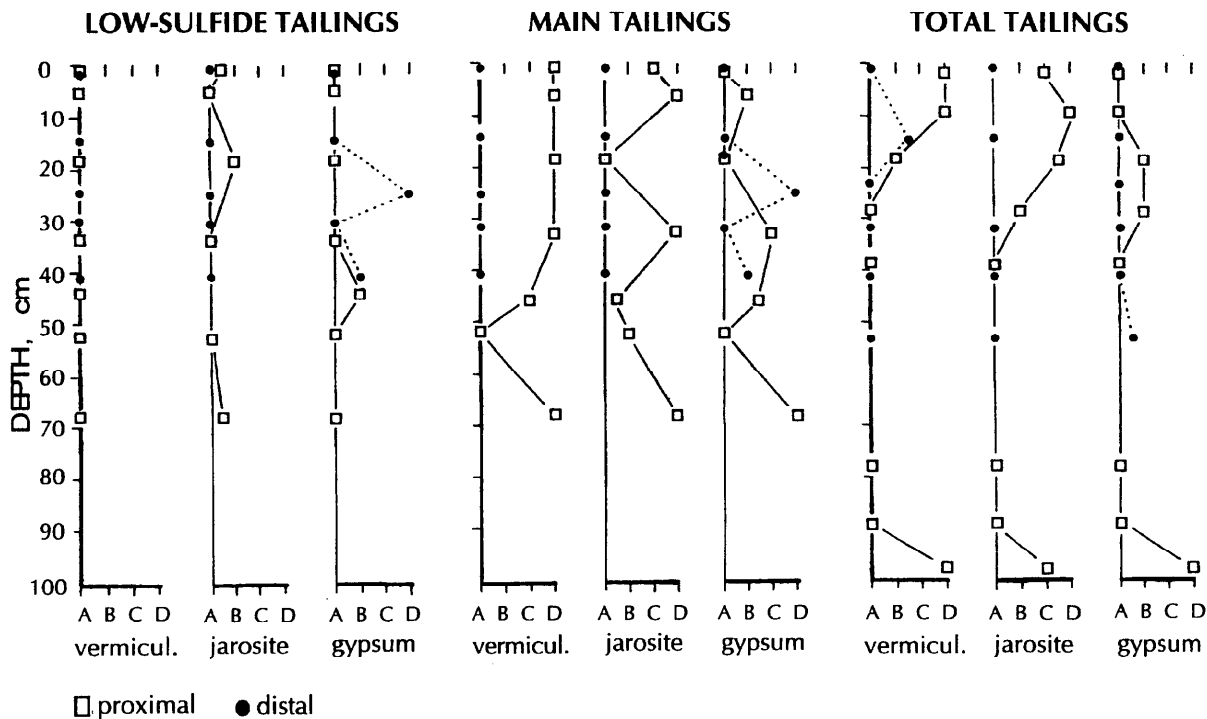


Figure 6. X-ray diffractometry results for the distribution of secondary vermiculite, jarosite, and gypsum in the tailings lysimeters. Horizontal scale: (A) not observed; (B) barely detectable; (C) readily detectable; (D) prominent.

Figure 6 illustrates the distribution of secondary jarosite, gypsum, and the vermiculitic mineral as determined by X-ray diffractometry. In addition to alteration of phlogopite, possible release of Ca from aluminosilicate or carbonate minerals is strongly suggested by the distribution of gypsum. The possible mineral source(s) of the Ca is under further study. The results in Figure 6 show not only that the least amount of alteration has occurred in the low-sulfide tailings, but also that the finer grain size of the distal tailings has significantly retarded alteration.

Upward Oxidation

Few explanations can account for the oxidized tailings that are present at the bottom of two of the lysimeters. Among the explanations considered was penetration of the geomembrane during coring, thus allowing upward oxygen migration. This seems unlikely because of presumably restricted oxygen inflow at the depth of the lysimeters at their mid-point. The most probable cause of upward oxidation is breakage in the ABS pipe to the sampler port (Fig. 2). Breakage occurred annually during freeze-thaw cycles, and although the breaks were repaired in the initial years of monitoring, changes in personnel duties led to a subsequent lack of maintenance; thus, especially in more recent years, oxygen may have been allowed to penetrate into the quartz sand and adjacent tailings nearest the centre-line of the lysimeters. Coring in late 1998 showed that the oxidation zone at the bottom of the tailings does not form an envelope beneath the tailings. This result supports the suggested explanation for upward oxidation, which is evidently strongest above the quartz sand drainage system.

Conclusions

Pyrrhotite in the lysimeters is undergoing rapid and extensive alteration. Pentlandite is more resistant to alteration, and chalcopyrite is the most resistant (Table III). The extent and intensity

of alteration are least in the low-sulfide tailings, but even these are acid-generating because the carbonate content of the tailings is low, and alteration of aluminosilicates is too slow to neutralize the acidity that rapidly develops from the oxidation of pyrrhotite. The effect of finer grain sizes in the lysimeters has been to inhibit alteration. In the coarser, sand-size tailings in one lysimeter, almost complete oxidation of pyrrhotite to a depth of ~45 cm has occurred in 4½ years. This core also has the greatest amount of secondary gypsum, jarosite, and vermiculitic alteration. Although the aluminosilicate assemblage contains several minerals that weather relatively rapidly, they have not prevented acid generation even at S contents as low as 0.35 wt%.

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