RECLAMATION OF SULPHIDE TAILINGS USING MUNICIPAL SOLID WASTE COMPOST: LABORATORY STUDIES

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SUMMARY

Three compost cover layer models were examined that might be beneficial in the suppression of tailings oxidation and acidic drainage. Laboratory studies were done to assess the potential compost cover layer benefits, and to facilitate the design of field tests. Two grades of compost were compared: immature (4 week old) and mature (14 week old) municipal solid waste (MSW) compost. Analyses of the leachate quality from compost cover layers and tailings were done since MSW compost may release trace metals, organic chemicals and pathogens into the watershed outside the tailings area. A laboratory simulation experiment to compare the three cover layer models and two grades of compost was conducted in plexiglass columns over 290 days.

The compost and the sand cover layer models appeared to be substantially better than the ploughed mode1 as cover layer designs for suppressing tailings acidic drainage. Oxygen concentrations were greatly depressed below atmospheric concentration in the compost and tailings layers in all three cover models. Substantial compaction pressure on the compost cover layer appeared to be of little benefit. Medium to strong reducing conditions developed, especially under an immature compost cover layer, and this enhanced the dissolution of oxides and increased the mobilization of iron, sulphate, and several trace metals. The formation of black iron sulphide precipitate and methane at the compost-tailings interface showed that acid mine drainage (AMD) processes and acidity were being reversed under the immature compost cover. However, reductive dissolution of tailings oxides was still proceedmg in the tailings layer at the end of the nine month simulation. This may be a transient condition until iron and sulphur become immobilized as less soluble precipitates such as sulphides.

Compost quality tests showed that leachates fi-om mature and fi-esh MSW compost present a low environmental risk for use on mine lands. Furthermore, very low hydraulic conductivity of the fresh compost cover layer will inhibit vertical water flow through the compost layer, and thus reduce the leaching of acids, metals and nitrogen compounds from the compost-tailings system.

From this preliminary study it is concluded that the compost model, using immature MSW compost, appears to be the simplest and most cost-effective tailings cover layer solution. Long

term field tests or large lysimeter pilot experiments are needed to determine whether the beneficial characteristics of the compost cover layer can be maintained. Long term compaction and decomposition of fresh compost must be examined to see ifits favourable hydraulic characteristics and anaerobic conditions will be maintained under natural conditions. In particular, the relative importance of the physical oxygen barrier and biological oxygen consumption benefits of different grades of MSW compost needs to be determined.

SOMMAIRE

Trois modèles de couverture de compost susceptibles de supprimer l'oxydation des résidus et le drainage acide ont été examinés. On a réalisé des études en laboratoire pour évaluer le potentiel qu'offrent les couvertures de compost ainsi que pour faciliter la conception d'essais sur le terrain. Deux types de compost ont été comparés : le compost frais (4 semaines de maturité) et le compost mature (14 semaines de vieillissement) de déchets solides municipaux (DSM) Des analyses de la qualité du lixiviat provenant de couvertures de compost et de résidus ont été menées pour la raison que le compost de DSM peut libérer des métaux en traces, des substances chimiques organiques et des pathogènes dans le bassin hydrographique à l'extérieur du parc à résidus. Une expérience de simulation en laboratoire pour comparer les trois modèles de couverture de compost et deux catégories de compost a été réalisée dans des colonnes en plexiglas pendant 290 jours.

Les modèles de couverture de compost et de sable se sont avérés beaucoup plus efficaces que le modèle de sol travaillé comme concepts de couverture du fait qu'ils permettent de supprimer le drainage acide des résidus. Les concentrations d'oxygène ont diminué bien en-deça de la concentration atmosphérique dans les couches de compost et de résidus dans les trois modèles de couverture. Compaction par une pression substantielle sur la couverture de compost n'a pas semblé procurer beaucoup d'avantages. Des conditions de réduction moyenne à forte se sont dévelopés, en particulier sous une couverture de compost fiais; ces conditions ont favorisé la dissolution des oxydes et ont augmenté la mobilisation du fer, des sulfates et de plusieurs métaux en traces. La formation d'un précipité de sulfure de fer noir et de méthane à l'interface compost-

résidus a mis en évidence l'inversion des processus du drainage minier acide lorsque la couverture de compost est fraîche. Cependant, la dissolution par réduction des oxydes contenus dans les résidus se poursuivait toujours dans les résidus à la fin du neuvième mois de simulation. Cette condition pourrait n'être que temporaire en attendant que le fer et le soufre soient immobilisés sous forme de précipités moins solubles comme les sulfures.

Les essais de qualité du compost ont indiqué que les liiviats provenant du compost de DSM mature et frais présentent un faible danger de pollution de l'environnement lorsqu'on l'utilise sur les sites miniers. De plus, une très faible conductivité hydraulique de la couverture de compost frais empêchera l'écoulement vertical de l'eau à travers la couche de compost de sorte qu'elle réduira la lixiviation des acides, des métaux et des composés azotés provenant du système compost-résidus.

De cette étude préliminaire, il ressort que le modèle utilisant du compost de déchets solides municipaux frais semble être la solution la plus simple et la plus rentable de couvrir les résidus. Pour déterminer si les caractéristiques avantageuses de la couverture de compost peuvent se maintenir, il faudra mener des essais à long terme sur le terrain ou de vastes expériences lysimétriques pilotes. La compaction à long terme et la décomposition du compost frais doivent être analysées pour établir si ses caractéristiques hydrauliques favorables et ses conditions anaérobies peuvent être maintenues dans un milieu naturel. En particulier, l'importance relative de la barrière physique contre l'oxygène et de la consommation biologique en oxygène qu'ofrent les différents types de compost de DSM reste à être déterminer.

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1. INTRODUCTION

A literature review of what is known about the physical and chemical characteristics of municipal solid waste (MSW) compost and other organic materials (Pierce 1992), revealed that a compost layer on sulphide tailing might be beneficial in the suppression of tailing oxidation and acidic mine drainage, in five ways:

(1) *Physical* oxygen *barrier* - the compost would be saturated with water over at least part of its depth so that the limiting factor in oxygen diffusion would be the diffusivity of oxygen in water;

(2) Oxygen-consuming *barrier-* the continued decomposition of compost would create a large biological oxygen demand that acts as a sink for atmospheric oxygen or dissolved oxygen

(3) *Chemical* inhibition - compounds and decomposition products in the MSW compost that leach into the tailing inhibit the growth and metabolism of sulphate-producing bacteria;

(4) Chemical *amelioration* - organic constituents in the MSW compost can cause the reductive dissolution of ferric oxides, leading to precipitation of metal sulphides and the consumption of acidity;

(5) *Reduced water infiltration* - reduced hydraulic conductivity of compacted, decomposing compost may prevent infiltration of precipitation, thus decreasing tailing groundwater flow.

Three compost cover layer models were proposed to produce and maintain these functions (Figure 1). The least expensive cover solution, the Compost model, consists of a deep layer'of compost placed directly on the tailing and topped with a minimal, protective soil layer (Figure 1 a). Revegetation requirements have not been considered in these models. The other two cover models consist of a layer of compost that is highly compacted by a thick revegetation layer, such as soil or sand and gravel (Figure 1 b & 1 c). The main purpose of the soil layer is to provide pressure to keep the compost layer permanently compacted so that gas-filled pore space is minimized and incoming precipitation cari produce a high degree of saturation. The top layer will also be a protective layer against erosion, evaporation and runoff that could destroy the compost layer as a uniform oxygen barrier. In the Ploughed model (Figure 1 b), compost is ploughed into the upper layer of tailing before the compost layer is placed, so that there is close chemical contact between the compost and the upper portion of the oxidized tailing. In the Sand model (Figure lc), the bottom of the compost layer is separated from the tailing by a coarse sand layer to isolate it hydraulically from the tailing.

(a)	(b)	(c)
Revegetation Layer	Revegetation Layer	Revegetation Layer
Compost	Compost	Compost
	Ploughed-in Compost	Coarse Sand
Tailing	Tailing	Tailing
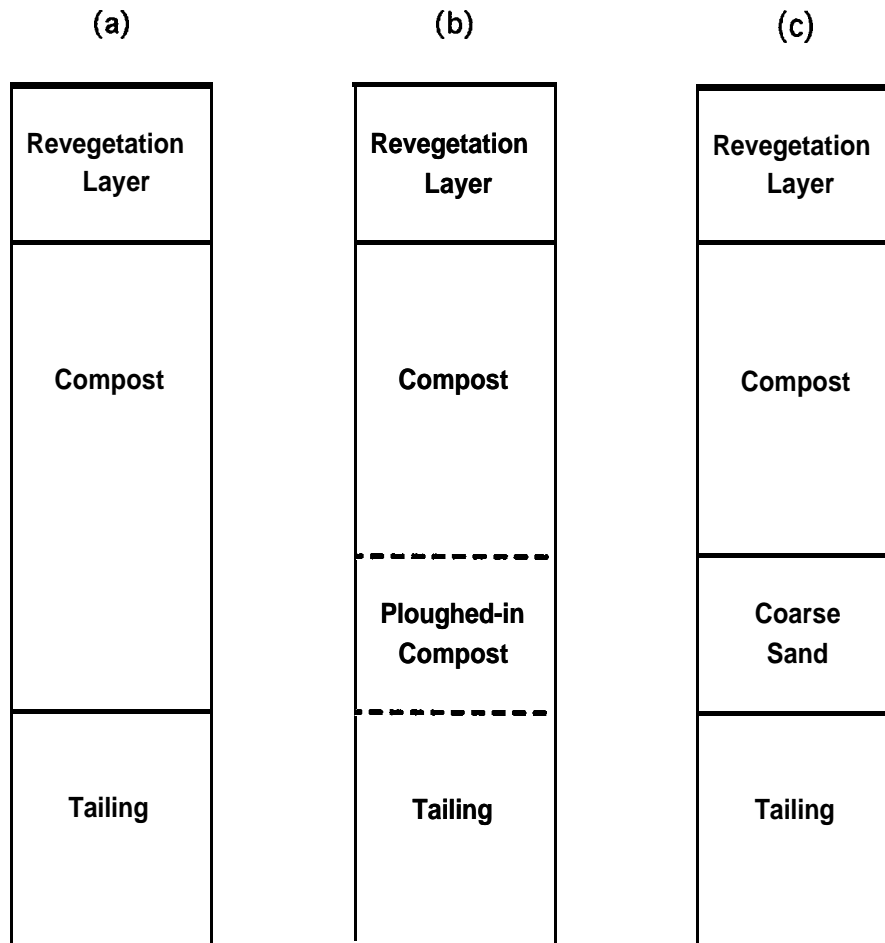
Figure 1. Diagrammatic representation of the three compost cover layer models studied: (a) Compost cover model; (b) Ploughed cover model; (c) Sand cover model.

Experimental investigations in the laboratory were done to assess whether the compost cover layer models will function as expected in field tests. Analyses of the leachate quality from compost cover layers and tailing were necessary since MSW compost may release trace metals, organic chemicals and pathogens into the watershed outside the tailing area. The risks of unacceptable levels of contaminants are largely a function of both the quality of the compost feedstocks and the optimization of the composting process. For MSW compost, separation of organic waste from non-biodegradable garbage and hazardous chemicals at the source is the best method for producing uncontaminated, high-quality compost (Pierce 1992). Sewage sludge compost may be another source of high-quality material if pathogens are killed and if the wastewater stream is uncontaminated by industrial effluents. Clean, immature (uncured) compost offers advantages over mature, agricultural-grade compost in tailing reclamation because of its high oxygen-consuming demand and the presence of a wide variety of organic compounds that could help in chemical amelioration of acidic mine drainage (AMD) processes. Immature compost could also be diverted much earlier in the composting process, and thus would be much cheaper for a municipality to produce. Both qualities of compost were studied.

2. PROJECT OVERVIEW

2.1 Project Goal

The goal of this project is to obtain answers to technical questions about the optimal cover layer design and safety of using a cover of municipal solid waste compost as a permanent solution to acid drainage from sulphide tailing. The results of the laboratory work will be useful in the design of field studies of compost cover layers on tailing at the Nickel Rim tailing site. Further laboratory work may be recommended to investigate the use of other organic wastes and to complement the field experiments.

2.2 Project Objectives

A detailed discussion of the project is found in the attached copy of the Proposal for Laboratory Studies (Appendix A). The following outline is provided to show the major lines of inquiry in this study.

A. Physical Aspects and Optimization of Compost Cover Layers

    a) Study of the effects of various depths of surface layer and compost maturity on compost compaction and other physical characteristics, including bulk density, porosity, degree of saturation, and saturated hydraulic conductivity.

    b) Measurement of oxygen concentrations within the compost-tailing column to determine the extent of anoxic conditions.

    c) Determination of the water balance and pattern of saturation of the compost cover system for a multi-week simulation of wetting/drying cycles.

B. Compost Leachate Composition from Compost of Varying Degrees of Maturity

    a) Regulation 347 leaching test (Environmental Protection Act).

    b) Trace metal and organic chemical spiking of compost to determine the degree of retention.

    c) Simulated acid rain effects on leaching of toxic compounds.

C. Chemical Processes and Changes Within the Compost and Tailing Layers

    a) Characterization of the crystallinity of oxidized tailing and the distribution of adsorbed trace elements within the tailing.

b) Effects of redox potential and acidity changes on solid phase chemical speciation and the composition of tailing pore water and leachate.

c) Effects of the optimal compost cover layer designs on solid phase chemical speciation and the composition of tailing pore water and leachate.

2.3 Experimental Design Overview

Four concurrent lines of investigation addressed the basic questions concerning the optimal compost cover layer characteristics, compost quality, the redox chemistry of the tailing and the leaching column methodology to be used in a final compost-tailing simulation experiment:

A. Physical Aspects and Optimization of Compost Cover Layers

B. Leachate Composition from Compost of Varying Degrees of Maturity

C. Chemical Processes and Changes Within the Compost and Tailing

D. Leaching Column Design and Sampling Methodology

The simulation experiment, described as part E, followed the completion of some of the preliminary   experiments.

E. Compost Cover Layer-Tailing Simulation

The compost-tailing models were treated to three wetting/drying cycles over approximately 42 weeks. Periodic chemical analyses on pore water and gases were done to follow the progress of chemical change in the compost and tailing. In addition, physical analyses were performed to understand the changes that may be taking place in the properties of the compost cover layer as a barrier to oxygen and water infiltration.

3. METHODS

3.1 Experimental Material

3.1.1 Tailing

Oxidized and non-oxidized tailings were collected from the Nickel Rim tailing site of Falconbridge Ltd. in October and December 1992. The samples are being kept in sealed plastic containers indoors. A representative subsample was mixed and oven dried at $70^{\circ}C$ overnight before being crushed in a mortar. The solid was used in the chemical characterization experiments (part C).

3.1.2 MSW Compost

About 200 litres of municipal solid waste compost were obtained through the municipal compost research program of Dr. Lambert Otten at the University of Guelph. The compost was produced in a southern Ontario city which ran a two-year pilot study using source-separated, organic, household and yard waste from about 900 homes. Two grades of compost were studied: 4 week old, immature (uncured) compost, and 14 week old, mature, agricultural grade compost.

A comprehensive set of analyses have been done at the University of Guelph during the maturation of this compost. These data are being made available to our project in the Guelph project final report. Dr. Otten said that even mature compost varies within a batch, but shows general trends in composition on a seasonal basis because of changes in the source material. He has observed that all their compost batches have met Ministry of the Environment specifications for trace metal concentrations.

3.2 Experimental Procedures

3.2.1 Compost Compaction Experiments

b) Physical characterization of compost

Investigations of the physical properties of mature and immature compost were to have been done on 30 cm deep layers of compost. Measurements of bulk density, porosity and saturated hydraulic conductivity were planned to assess the effects that different degrees of compaction and initial moisture content have on water saturation. The degree of water saturation relates

directly to the function of the compost layer as an oxygen barrier. Apart from some preliminary, non-replicated trials, these experiments were not done as proposed. The short time frame of the project and limitations on equipment and space prevented us from conducting long-term compaction experiments in addition to the simulation run. Instead, physical, characterization of the compost physical properties was done on the compost-tailing system in the simulation experiment at about three months into the experiment. It was decided, based on the preliminary experiments, that these results would provide more useful information about compost compaction and hydraulic properties than would a separate set of short-term experiments.

Physical characterization was done on core samples extracted from the leaching columns of the simulation experiment using a one centimetre inside-diameter cork borer. It was necessary to sample the compost across the 9.4 cm inside diameter of the tube because the in *situ* volume of the sample is required. The sample was weighed and oven dried at $75^{\circ}C$ overnight to obtain the dry weight. Bulk density (i.e., the dry weight per unit volume of the *in situ* sample) and degree of water saturation were determined. Particle density was determined by measuring the volume of water displaced by a finely-ground sample of the oven dry compost material. Total porosity, P_T, was then determined using the formula (Vomocil 1965),

$$P_T = 100 [(D_p - D_b) / D_p]$$

where D_p is the particle density and D_b is the dry bulk density. Gas-filled porosity was calculated by subtracting the volume fraction of water in the sample from the total porosity.

The saturated hydraulic conductivity of the compost-tailing system was measured in the week alter day 42 when excess water was added to all the columns. Saturated hydraulic conductivity was determined by the falling head method, which involved flooding the saturated compost with a layer of water (i.e., a head of water) and measuring the time it took for the water level to drop a certain distance (Klute 1965). For time periods of up to several days, evaporation of water from the head of water on the compost was considered to be negligible. Saturated hydraulic conductivity was determined using the following formula,

$$K_s = (aL/At) \, l \, n \, (H_1/H_2)$$

where in the leaching column the cross section of the standpipe(variable a) is equal to the cross sectional area of the sample (variable A), L is the length of the sample, and t is the time for the hydraulic head to decrease from height H_1 to Hz.

3.2.2 Compost Leachate Composition

It was important to determine whether it is safe to use MSW compost on a tailing site. As a first step, we analyzed the composition of compost leachate water that could carry organic chemicals, excess nutrients, trace metals and pathogens into the watershed. The Lakefield Research labs of Falconbridge Ltd. did two types of standard leaching tests on samples of the same mature and fresh compost obtained by the Laurentian University group. The Methods section and the Results section of the Lakefield Research report is presented in Appendix B. The following is brief description of the analyses the Lakefield Lab performed:

a) Background content characterization of received composts

Background levels of metals (Ni, Cu, Pb, Zn, Cd, As, Cr, Co, Mo, Se, Hg) and pesticides, herbicides and PCB (Ontario Regulation 347, section 4) were measured in duplicate in the mature and fresh composts.

b) Background content characterization of spiked composts

Both compost grades were spiked with 10 times the Ministry of the Environment allowable levels of metals, pesticides, herbicides and PCB. The metals and spiked organics were measured without replication in the mature and fresh composts.

c) Ontario Regulation 347 leaching tests

Leaching tests, specified by Regulation 347, were done in duplicate on both grades of compost. Analytical results from a study at the Region of Waterloo have indicated that pesticides, herbicides and PCB's are not present in MSW compost above the limits of detection. Since the background analyses confirmed this to be true for the compost samples to be used in this study, it was not necessary to conduct analyses for these components in the leachates from non-spiked samples. Full analyses of leachates for the spiked samples was carried out.

d) Toxicity Characteristic Leaching Procedure tests

Leaching tests on both grades of compost were also be done using another standard procedure, the Toxicity Characteristic Leaching Procedure (TCLP) for toxic materials. Duplicate analyses were be done as described above for Regulation 347.

3.2.3 Chemical Processes In Tailing

Although it is possible for acids and trace metals to be introduced to the compost-tailing system from the compost cover layer, the bulk of the trace metal release and acid generation will take place in the existing tailing. Thus, it was important to characterize the composition and reactivities of trace metals in the oxidized and non-oxidized tailing layers from the Nickel Rim site.

a) X-Ray diffraction analysis

A portion of the tailing solid has been analyzed by x-ray diffraction to detect any crystalline structure in the oxidized tailing. The results of a detailed analysis of the tailing structure were useful in understanding the adsorptive properties of the tailing components and their chemical reactivities.

b) Sequential leaching analysis

The distribution of each trace element within the oxidized tailing provided important information on the reactivity of the tailing and on the potential metal mobility (solubility) under different redox conditions, as may be present in the compost-tailing system.

Triplicate samples of the dried solid tailing were submitted to a sequential leaching procedure to determine the distribution of trace metals within the oxidized and non-oxidized tailings. The procedure included five steps to obtain five extract fractions:

Fraction 1:   The solid is extracted with a solution of 1 M magnesium chloride adjusted to pH 7 and heated in a microwave oven for 4 minutes. The solution is filtered on a 0.2 μm membrane and acidified before further analysis. The solid is washed and returned to a teflon container for step 2. Elements extracted in this fraction are called "Exchangeable".

Fraction 2:   A solution of 1 M sodium acetate adjusted to pH 5 with acetic acid was added to the residue from step one, then microwaved for 5 minutes. The solution is filtered on a 0.2 μm membrane and stored for further analysis. The residue is washed and transferred to a teflon container for the next step. Elements recovered in this fraction are called "Carbonate Bound".

Fraction 3:   A reducing solution of $0.04M$ hydroxylamine hydrochloride in 25% (v/v) acetic acid is added to the residue and microwaved for 4 minutes. A 5 ml portion of the

extractant is added and the mixture is re-microwaved for 4 minutes. This **procedure** is repeated twice more. The solution is filtered and the residue is submitted to the next extraction after the usual washing. Elements extracted in this fraction are called "Bound to Iron-Manganese Oxides".

Fraction 4:   The residue is then extracted with a solution of 30% (v/v) hydrogen peroxide in 0.02M nitric acid for 4 minutes in the microwave. Since this solution is strongly oxidizing, a solution of 3.2M ammonium acetate is added after the filtration to prevent any readsorption of elements on oxidized tailing. The residue is washed and transferred to a teflon container for the last step. Elements extracted in this fraction are called "Bound to Organic Material and Sulphides".

Fraction 5:   The residue is digested with a fresh solution of 4:1 (v/v) nitric:hydrochloric acid and microwaved for 5 minutes. The solution is filtered and stored for further analysis. This fraction is called the "Residual Fraction".

The five different extracts we obtained were analyzed for Fe, Mn, Cu, Ni, Cr, Co, Zn, Pb, and Cd by Inductively Coupled Plasma Atomic Absorption Spectrometry (ICP). ICP analyses were performed by the Chemical Laboratory at Falconbridge Ltd., Sudbury Operations.

c) Chemical simulation of redox and acidity effects

Various redox conditions in the tailing system were simulated by using chemical reducers. The results obtained from these simulation experiments provided information on the effects on metal mobility in the oxidized tailing under reducing conditions. Slurry solutions were prepared by mixing known quantities of oxidized tailing solid with solutions of chemical reducers (e.g., sodium dithionite). The redox potential (E_h) was monitored with a combined platinum electrode system. Subsamples of the solution were collected and filtered through a 0.2 μm membrane. Metals released in the solution were measured by ICP analyses at the Falconbridge Laboratory and the concentrations correlated to the E_h value measured when the sample was taken.

Similar experiments were also performed on a slurry of oxidized tailing material using mature MSW compost as a reducing agent. Fresh compost was not used as a reducing agent in this type of experiment because of the coarse, heterogeneous nature of the material and the expectation that fresh compost would not stay in its initial state for very long because of rapid decomposition, and thus would be an unrepresentative material. The effects of fresh compost was examined only in the compost-tailing system during the simulation experiment (Section 3.2.4).

3.2.4   Compost Cover Layer-Tailing Simulation

a) Leaching column design

The simulation of a compost cover layer on oxidized tailing was based on a one-dimensional model of transport of atmospheric oxygen and water through the cover layer into the tailing. To provide the experimental environment on a scale suitable for a laboratory, cylindrical columns were employed (Figure 2 and Photo 1). The leaching columns were constructed from clear acrylic (plexiglass) tubes 183 cm high and 10 cm outside diameter with 3 mm thick walls. The clear tube walls allowed examination of the quality of packing of tailing and compost in the tube, and monitoring of visible changes in the material during the simulation experiment. Changes observerd were porosity and colour changes in the compost, and colour changes in the oxidized tailing as a result of reductive dissolution of oxides and the formation of precipitates. There is a marked colour change from blue-gray to red-brown as the Nickel Rim tailing go from a non-oxidized to oxidized state, and a reverse colour change would be expected if the oxidized tailing is being chemically reduced.

In order to obtain core samples of the compost and tailing material inside the leaching columns, two vertical rows of sample holes were drilled through the tube walls (Figure 2 and Photos 3 & 4). The holes were 13 mm diameter and were sealed with number zero rubber stoppers. The vertical positions of the potential sample holes are given in Figure 3. The vertical rows are separated by 90 degrees around the circumference of the tube. Another vertical row of six sampling holes was made on the backside of each tube at the level of the compost layer, with the holes mid-way between the other sampling holes. These holes were used at the end of the tenth week of the experiment to extract cores of compost in order to determine the degree of water saturation, bulk density, and gas-filled porosity. Each tube was seated into a polyethylene funnel which was sitting in a 9.5 cm holes cut in a heavy plywood shelf. The hole was bevelled so that the stress from the combined weight of the filled tube and compaction weight would be transferred directly through the funnel wall to the plywood shelf. Each tube was held vertically near its upper end in 10.5 cm holes made in a thin plywood shelf. The hole around each tube was caulked with a strip of foam rubber to seal off the nitrogen compartment but still allow the tube and funnel to be rotated to facilitate sampling.
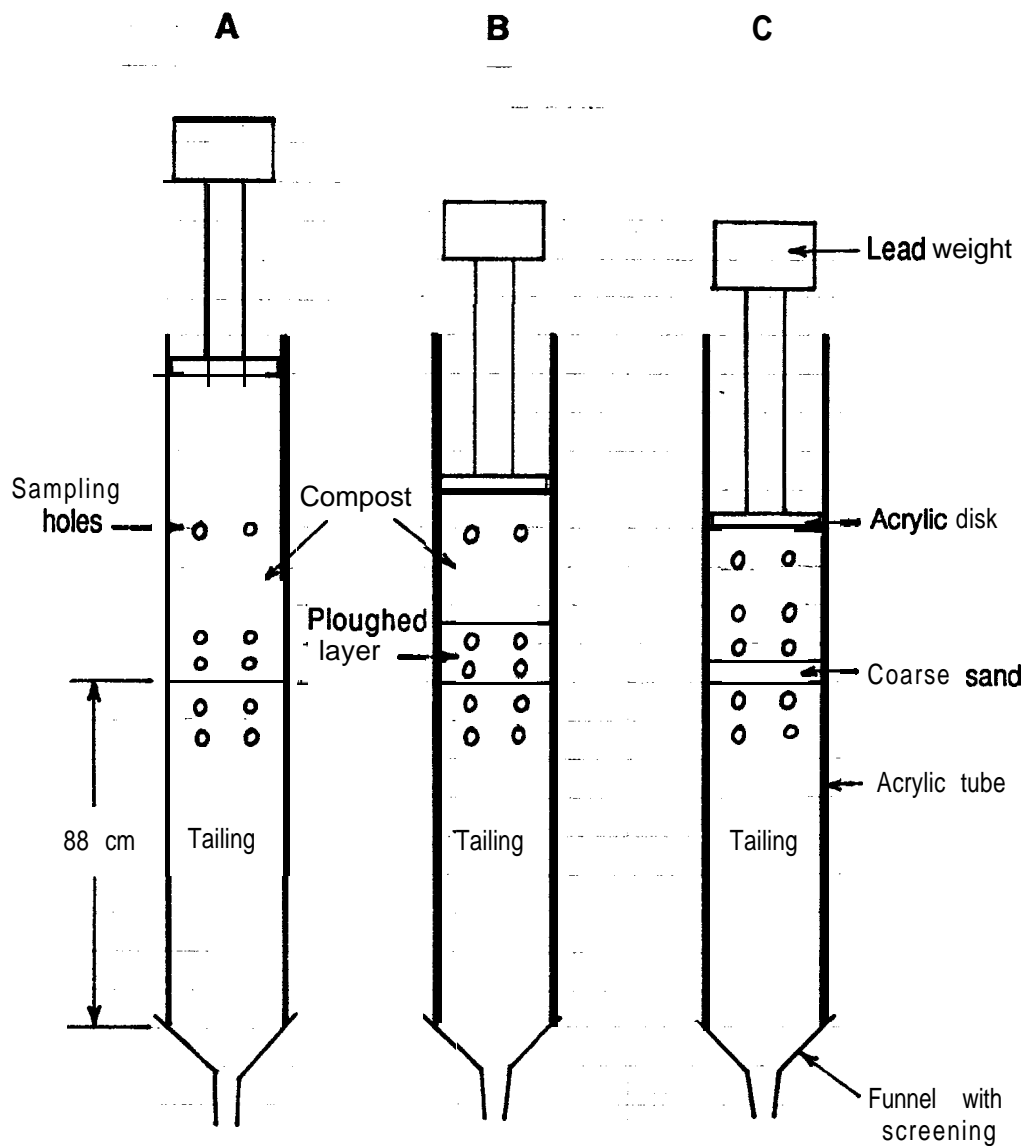
Figure   2. Diagrammatic representation of the leaching column design used in the cover layer simulation experiment, showing: (A) Compost model, (B) Ploughed model, and (C) Sand model.

Photo 1. Nitrogen atmosphere cabinet containing six leaching columns - one replicate of the three cover layer models for the two grades of compost.

Compost Model   **Ploughed Model**   **Sand Model**

170
150   **150**
130   **130**   **130**
120   **120**   **120**
115 compost   **1   1   5**   compost**115** compost
110   **110**   **110**

                    ■ ■ ■ ■ ■ ■ ■ ■ ■ ■ ■ ■

105   **105**   **105**
100   **100** compost-   **100**
95   **9 5** tailing   **9 5**
                                    mixture   ■ ■ ■ ■ ■ ■ ■ ■ ■ ■ ■

9 0   **90**   coarse sand

top of tailing ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪ - ▪

85   **85**   **85**
80   **80**   **80**
70   **70**   **70**
60   **60**   **60**
40   **40**   **40**
20   **20**   **20**

Figure 3. Positions of sampling holes (centimetres from the bottom of the tube) in the leaching columns for the three cover layer models. The vertical row of five holes actually sampled during this simulation experiment are indicated in bold. (Compare with Figure 1)

Compaction of the compost in the leaching columns was achieved using a piston made of a 9.4 cm diameter disk of 13 mm thick plexiglass attached to a pine post which reached up above the top of each tube (Figure 2). Each disk had eighteen 3 mm holes drilled through it to allow the free passage of gases and water (Photo 2). Lead fishing net weights were attached to the top of the pine posts (Photo 1). In the Compost model, initially containing one metre depth of compost, a single 9 kg lead weight applied a pressure of approximately 12.5 KPa (0.125 atmosphere) to the top of the compost in order to simulate the weight of a sandy soil layer of 50 cm which could be revegetated to prevent erosion (Figure 1 a, Figure 2A). In the Ploughed and Sand cover layer models, initially containing 30 cm of compost (Figure 1 b & 1 c), four 9 kg lead weights were attached to each post by chains to give a pressure on the compost of 50 KPa (0.5 atmosphere). This amount of weight was applied to simulate the effect of a surface revegetation layer of about two metres of gravel and sandy soil.

b) Cabinet design

The cabinets to house the leaching columns were designed to support a large amount of weight (Photo 1). The cabinets wold hold a nitrogen atmosphere in the bottom two compartments below the shelves for the duration of the 10 week experiment, except for short periods when the cabinets are opened for sampling. This was done to ensure that oxygen enters the compost-tailing system only from the air above the compost layer (Le., a one-dimensional vertical flux). The cabinet width and height dimensions are based on a standard sheet of plywood (244 cm by 122 cm) and the depth was 30 cm. The frame was constructed of pine boards (widths 3.8 cm by 3.8 cm) and this was screwed to a heavy plywood base (40 cm by 122 cm). Six heavy duty rubber casters were attached to the bottom of the base to give support and mobility to the cabinet. The front of each cabinet had two full-height doors (244 cm by 61 cm) which were closed shut with barrel bolts at the top and bottom. All external joints inside the cabinet were caulked with silicone and a gasket of plastic foam-filled weather stripping was stapled inside the frame to seal each door against loss of the nitrogen atmosphere inside. Nitrogen was injected into the cabinet at the top because it is slightly less dense than air and was expected that the cabinet air would be displaced downwards as the nitrogen was injected. The cabinet was divided vertically in the middle by a polyethylene plastic sheet to create two compat-tments. This was done to avoid the wastage of nitrogen gas from both sides when only one door is opened for sampling.

Photo 2. Close view of a plexiglass disk attached to a pine post inside a leaching column. With the addition of weights at the top of the post this became a piston for compacting the compost layer.

c) Loading of material into the columns

The base of each plexiglass tube was placed into a polyethylene funnei and secured with duct tape to form a unit that could be easily handled (Photo 3). A piece of fibreglass screen lining the funnel prevented tailing material from draining out of the funnel. About 500 ml of wet, oxidized tailing material was added to each funnel at a time and was then tamped firmly with a pine post to remove any large air spaces. An 88 cm depth of tailing was put into each of the 18 columns and the tailing layer was then flooded periodically with distilled water until solution drained freely from the funnel at the bottom. This was done to ensure that the tailing would have a uniform, high level of saturation at the start of the simulation exoeriment.
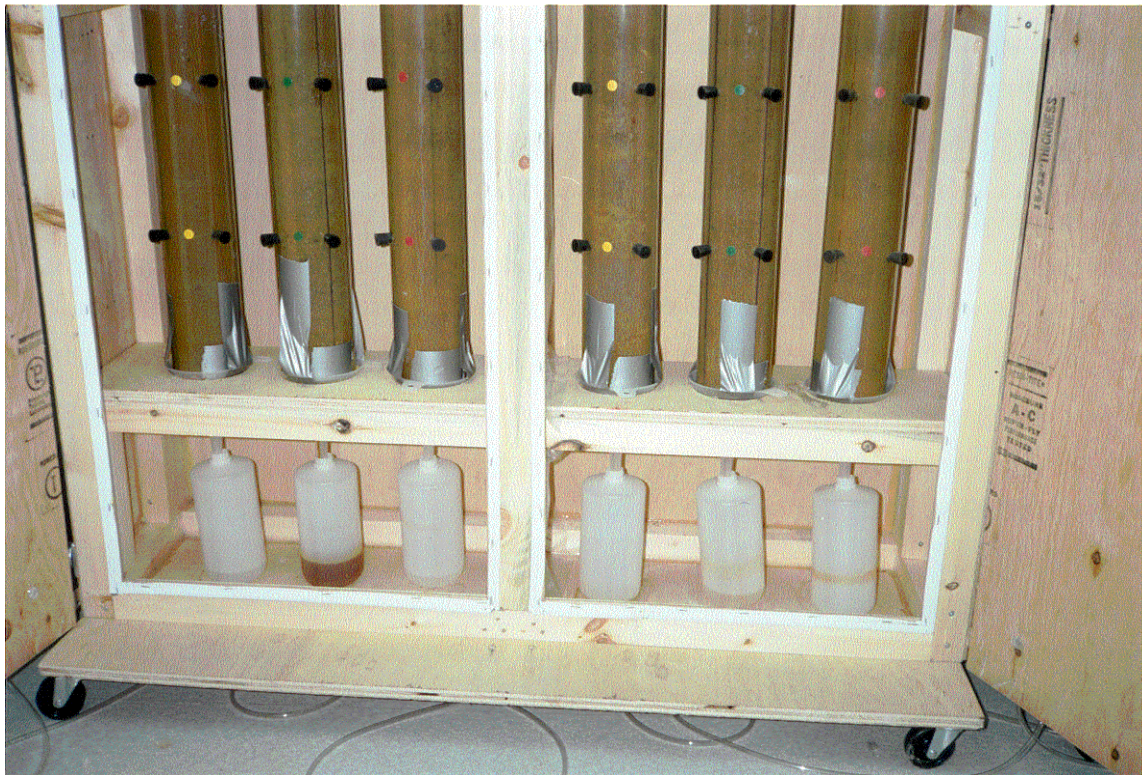
Photo 3. Placement of leaching columns into polyethylene funnels which are supported on a strong plywood shelf. Each funnel drained into a one litre bottle so that effluent could be collected for analysis.

Photo 4. Three leaching columns containing fresh compost. The Ploughed model is on the left, Compost model is in the middle, and the Sand model is on the right. Note the formation of black iron sulphide precipitate near the compost-tailing interface.

Photo 5. Three leaching columns containing mature compost. The Ploughed model is on the left, Compost model is in the middle, and the Sand model is on the right. Notice the finer texture of mature compost compared to fresh compost (compare Photo 4).

In the Compost cover layer model, a 100 cm layer of moist compost (40-50% water content) was packed into a section of tube which had been attached with duct tape to a funnel. A pine post was used to firmly tamp down about 500 ml of compost 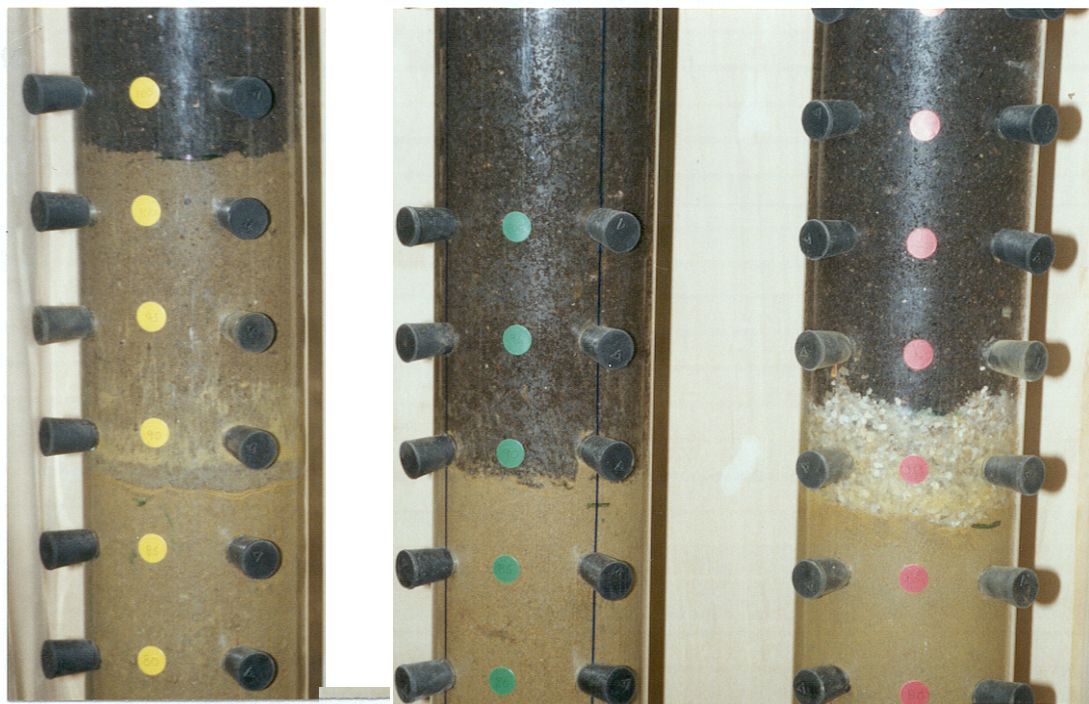at a time to remove large air spaces. In the Ploughed model, containing a layer of compost mixed with tailing (Figure 1 a, Photos 4 & 5), compost was mixed with oxidized tailing on a 1: 1 ratio by volume before adding it to the tube. Each tube of the ploughed model was packed, 500 ml at a time, with a 20 cm layer of the compost-tailing mixture. The Sand model (Figure 1 b) was created by putting a 5 cm layer of coarse, non-toxic, aquarium sand on top of the tailing. All remaining columns were then packed with a final 30 cm layer of moist compost (40-50% water content). After the compaction pistons were inserted, all columns were flooded at the top with a 3 cm layer of distilled water.

d) Sampling methodology

During the simulation experiment, small samples of tailing and compost were taken for gas analysis and redox potential (Eh) measurement. Since atmospheric oxygen was introduced into the pure nitrogen atmosphere when the cabinet door was opened, it was necessary to reestablish an anoxic atmosphere around a column before removing the rubber stopper. A plexiglass glove box was constructed which would fit half way around the column and provide access to the two sampling holes at each level (Photo 6). The glove box was supported on the column by clips that attached on each side of the box to two chains hanging from the upper shelf, and by an elastic tord wrapped around the back of the column which pressed a weather stripping seal to the column (Photo 7). The experimenter then moved the nitrogen line feeding into the cabinet to the port on the top of the glove box (Photo 7).

Inside the glove box were placed a 4 ml pre-weighed glass vial, a 15 ml pre-weighed plastic tube (containing 5 ml of distilled, deoxygenated water), a redox potential probe and a cork borer (with wooden plunger). The experimenter waited a few moments for the nitrogen gas flow to establish anoxia in the box. He then flushed the glass vial with nitrogen gas before removing the stopper from a sampling hole. Using the cork borer, a l-2 cm long core of material was extracted from the column. Before replacing the stopper, the hole in the tailing or ploughed layer was filled with a piece of glass rod. Sampling holes in the compost layer were not refilled because of the difficulty and risks of handling compost material inside the glove box. Inside the anoxic glove box, half of the material was put in the glass vial and sealed with a rubber septum and screw cap. The rest of the material was inserted into the plastic tube containing water. Sometimes a second core had to be taken to provide this second sample. The tube was agitated for about half a minute before a redox potential probe (Fisher Scientific 13-620-82 with Accumet pH meter) was inserted into the liquid. The E_h was recorded after three minutes, and then the

tube was capped. Both containers were removed from the glove box and reweighed. After gas analyses were compieted, the glass vial was oven dried at $75 \, ^oC$ overnight to determine the dry weight of material sampled.

The E_h that we measured must be treated as a relative redox potential value since the redox transformations in the compost-tailing system would be complex and thus could not be calibrated against a redox potential standard. The redox probe was checked periodically against a standard solution to guard against malfunction and drift in its response. The redox probe was too fragile to insert directly into compost or tailing material in the columns, so the method to measure an equilibrium E_h between a solid sample and deoxygenated water was devised. Therefore, the redox potential measurements made in the compost and tailing material are a relative E_h that are assumed to be consistent between experimental treatments. Similarly for acidity measurements, since pore water was not being extracted, an accurate measurement of in situ sample pH could not be obtained. However, a relative pH was measured in the deoxygenated water used to determine relative E_h for the solid core samples. We are assuming that a similar equilibrium situation existed between the solid phase and the deoxygenated water in all the core samples
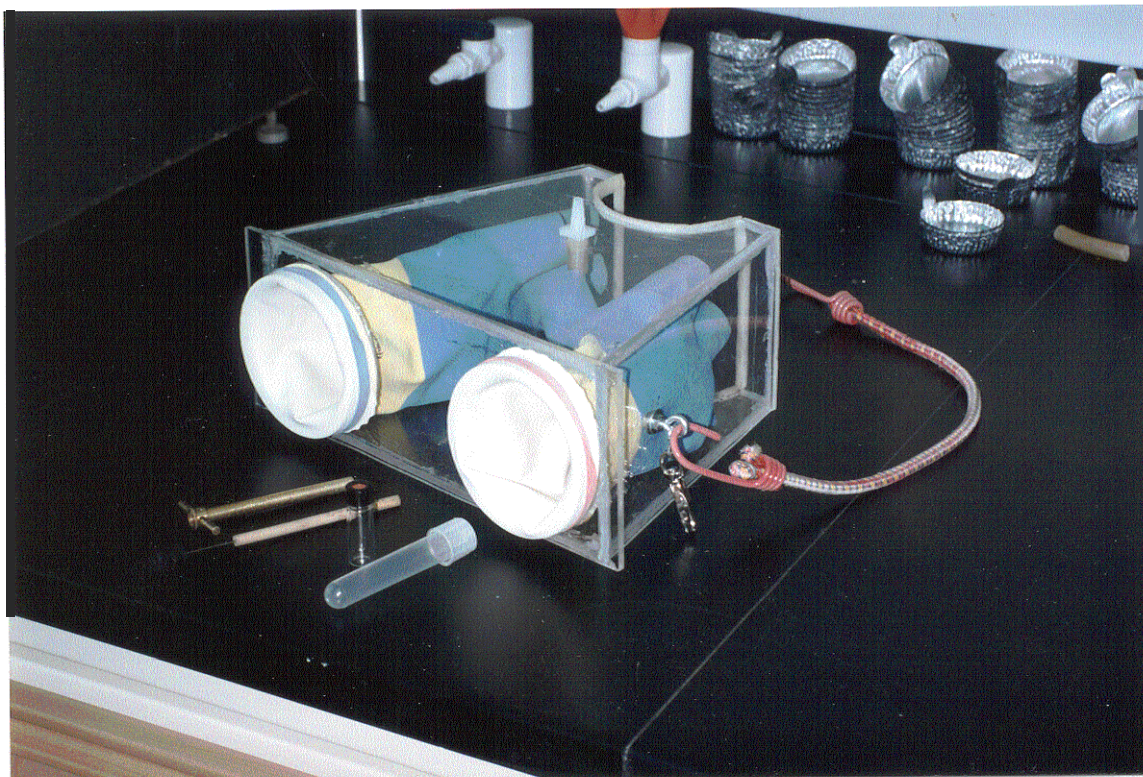
Photo 6. Plexiglass glove box showing the semicircular channel, elastic cord and weather stripping designed for sealing against the leaching column, the nitrogen inlet port on top, and the sampling containers and cork borer sampler.

Photo 7. Sampling glove box in use on a leaching column. Notice the two chains and elastic tord supporting the box while the experimenter takes the samples. The nitrogen line is visible feeding into the top of the box.

The laboratory study involved a simulation run over 290 days. The triple replication of leaching columns, each with two rows of holes, allowed for six separate profiles to be sampled from each of the six treatments during the simulation run. Abbreviations for the treatment matrix in the simulation are given in the following table:

Compost Type	Compost Cover Layer Model		
	Compost	Ploughed	Sand
Mature	CM	PM	SM
Fresh	CF	PF	SF

e) Gas analysis technique

The glass vials containing compost or tailing samples were transported to another lab for gas chromatography and ion chromatography analyses. It was found that a sealed anoxic vial would absorb some atmospheric oxygen if left overnight before gas measurement. To avoid the possibility of this contamination error, only as many samples were collected from the columns as could be analyzed the same day. Vials were stored in the refrigerator until shortly before starting gas analysis to minimize further chemical changes.

A precision syringe was used to sample 100 microlitres of gas in the vial. This sample was injected into a gas chromatograph (Varian Aerograph 90-P3) equipped with a thermal conductivity detector and a column designed for low molecular weight gases. Gases of interest to this study that could be measured with this instrument include: oxygen, carbon dioxide, carbon monoxide and methane. It was not possible to detect hydrogen sulphide with the thermal conductivity detector. Nitrogen produced by the denitrification of nitrate and nitrite could not be distinguished from the background nitrogen atmosphere. The temperature of the GC oven was changed manually from $35°C$ to $180°C$ at a rate of $20°C$ per minute starting at the four minute point following sample injection in order to separate the peak for oxygen from the peaks for the other gases. The gas chromatograph system included an integrator-plotter (Hewlett-Packard Integrator 3396A) which expressed each detected component as a percentage of the total sample.

f) Anion analysis

The levels of nitrates and sulphates were also analyzed in each sample of deoxygenated water using a high pressure ion chromatography system controlled by the Millenium 2010 software.

g) Experimental schedule

Period 1- Initial Draining

Following the loading of the columns and their saturation with water, the columns were allowed to drain for seven days. No attempt was made to exclude the oxygen in the atmosphere since the cabinets were being opened frequently to observe the effects of compaction and initial leaching on the layers of material. After the first set of samples was taken on day 7, the cabinets were closed on day 14 and nitrogen gas was injected into each cabinet using a system of tygon tubes hooked to a pressure-regulated nitrogen tank. Once the cabinets were filled with nitrogen, a low flow rate of nitrogen was maintained to replace gas leaking out. The cabinet atmosphere was sampled occasionally and analyzed for oxygen by gas chromatography to check that anoxic conditions were being maintained.

Period 2 - Futther Draining for 5 Weeks then Rewetted

A second set of 30 samples was taken statting on day 39. A five point profile in one replicate of each cover model treatment was sampled for gas analysis, water content, redox potential, nitrate and sulphate. All columns were then rewetted by adding 5 cm of distilled water (about 400 ml) to the top of each column, in order to simulate a heavy rainfall.

Period 3 - Draining for 2 Weeks

A third set of samples was taken starting on day 54.

Period 4 - Further Draining for 2 Weeks

A third set of samples was taken starting on day 67.

Period 5 - Further Draining for 13 Weeks

A fourth set of samples was taken on day 160. When the decision was made to extend the simulation experiment, all columns were rewetted on day 194.

Period 6 - Further Draining for 18 Weeks

A final set of samples was taken on day 290. A full set of measurements was made. Dried solid tailing samples were also analyzed using sequential extraction analysis.

4. RESULTS AND DISCUSSION

4.1 Physical Aspects of Compost Cover Layers

The experiments on compost compaction (Experimental Design, Section A) were set aside after some preliminary compaction trials, in order to begin the compost cover layer simulation experiment. In working with the compost-tailing system in the leaching columns it became evident that the compactions experiments, as described in the project proposal, were too limited in scope, time frame, and realism. It was decided that physical analyses of the compost layers at the end of three months into simulation experiment would give more useful information than would separate compaction experiments.

4.1.1 Degree of Compost Compaction

Compaction of the compost was not a rapid process, even in treatments under the considerable pressure of 50 KPa (0.5 atmosphere), the equivalent of about two metres of mineral revegetation layer. After an initial rapid settling of compost over a few minutes, further subsidence was evident over several days, and it continued at a much slower rate for months. Table 1 shows the degree of compaction that had occurred in the columns after three months of simulated surface layer pressure.

Table 1. Degree of compaction of fresh and mature compost layers by a constant surface pressure for 94 days (average of three replicates). The same bold letter symbol denotes no significant difference between means (p=.05).

Cover Layer Treatment	Surface Layer Pressure (KPa) ,	Initial Compost Layer Dept h (cm)	Degree of Compaction (%)	
Ploughed Fresh	50	30	24.7	**a**
Ploughed Mature	50	30	9.8	**b**
Sand Fresh	50	30	14.1	**a**
Sand Mature	50	30	8.8	**b**
Compost Fresh	12	100	4.9	c
Compost Mature	12	100	4.2	c

The trends in these data show that the fresh compost was compacted to greater degree than the mature compost in the Ploughed and Sand cover layer models. The fresh compost was rather slimy in texture and this may produce lower internal friction and thus less resistance to deformation in the material. A decrease in the amount of large pore spaces and a higher degree of water saturation in the more strongly compacted Ploughed and Sand models were seen initially. However, later in the experiment all fresh compost layers showed the development of large voids, presumably caused by the accumulation of gases from decomposition (see Section 4.4.3).

The processes of compost compaction as a function of initial water content and swelling were not examined. Unless the compost was either very wet or very dry, the initial water content appeared to have little effect on the degree of compactability of the compost layer. Swelling of the compost layer was not seen, even under conditions of long term saturation.

4.1.2 Effects on Compost Water Content, Bulk Density and Porosity

On day 94 of the compost cover layer simulation run, the bulk density, total porosity, water content, particle density, and gas-filled porosity in the compost layers were determined in all 18 columns. An inverse relationship was found between bulk density and both percentage water content (Figure 4) and total porosity (Figure 5). It should be noted that the four Ploughed model data points (Figures 4 & 5, square symbols) with highest bulk densities are samples containing 50% tailing material of much higher density and thus cannot be directly compared to compost samples. Fresh compost maintained a much higher water content and had a lower bulk density than mature compost under the same compaction treatment (Figure 4). The trend towards higher water contents with decreasing bulk density are believed to be the result of more pore space being present to hold water in those samples.

A corresponding inverse relationship between gas-filled porosity and bulk density is shown in Figure 6. Gas-filled porosity is the amount of pore space that is filled by gas, originating either from the atmosphere or produced by chemical processes in the compost-tailing system. Since gas-filled porosity was strongly correlated to total porosity $(r=0.94)$, the variability and importance of pore water were relatively small. Similarly, Figure 6 shows that the profiles of gas-filled porosity, determined on day of the experiment, were also a strong function of the bulk density. Thus, the compaction pressure of 50 KPa, equivalent to about 2 metres of mineral surface layer, was not effective in squeezing much of the pore space out of the compost matrix during the relatively short simulation experiment. Work by Chow et al. (1992) on sphagnum peat showed that a compaction pressure of 100 KPa was not sufficient to produce complete saturation in that organic material, although it should be noted that their compaction test was also of short duration.

Figure 4. Relationship of compost layer total porosity and bulk density on day 94. Each point is the mean of three replicates. (Fresh compost - solid symbols, Mature compost - open symbols, Ploughed model - squares, Compost model - diamonds, Sand model - triangles).

Figure 5. Relationship between the dry bulk density and percentage water content for compost layer samples on day 94. Each point is the mean of three replicates. (Fresh compost - solid symbols, Mature compost - open symbols, Ploughed model - squares, Compost model - diamonds, Sand model - triangles).
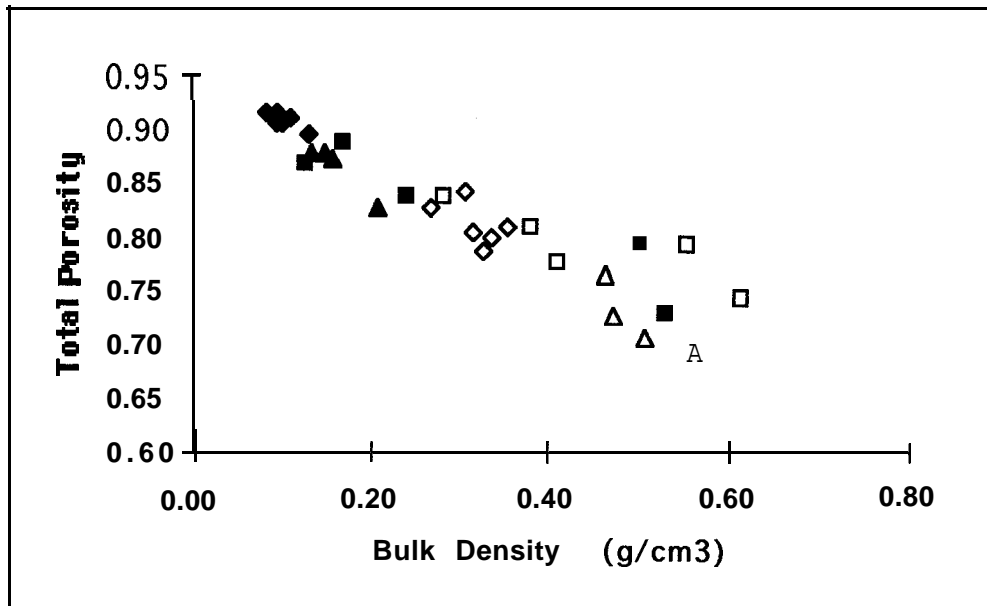
Figure 6. Relationship of compost layer gas-filled porosity and bulk density on day 94. Each point is the mean of three replicates. (Fresh compost - solid symbols, Mature compost - open symbols, Ploughed model - squares, Compost model - diamonds, Sand model - triangles).
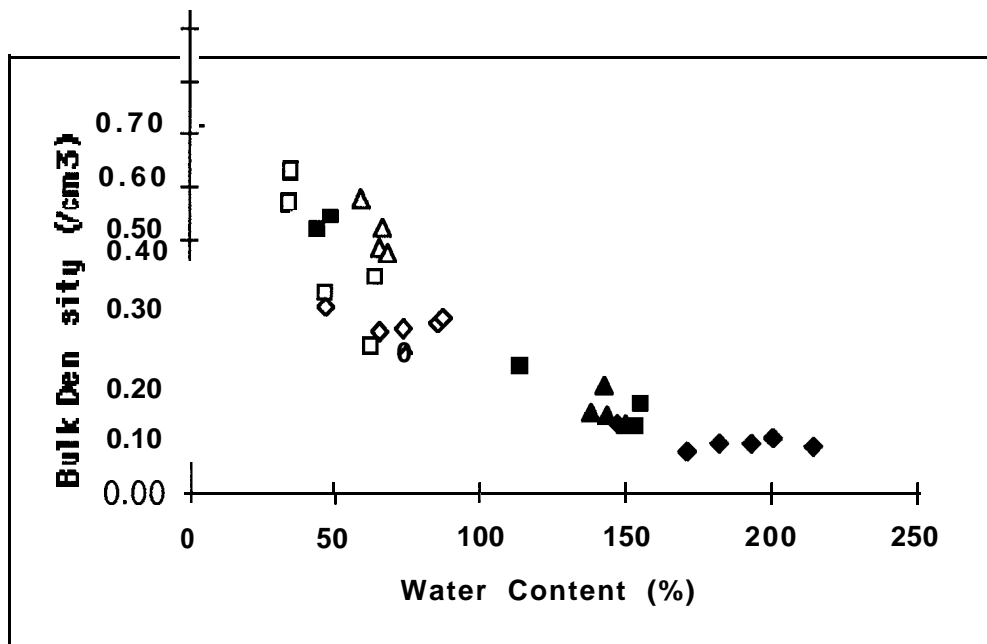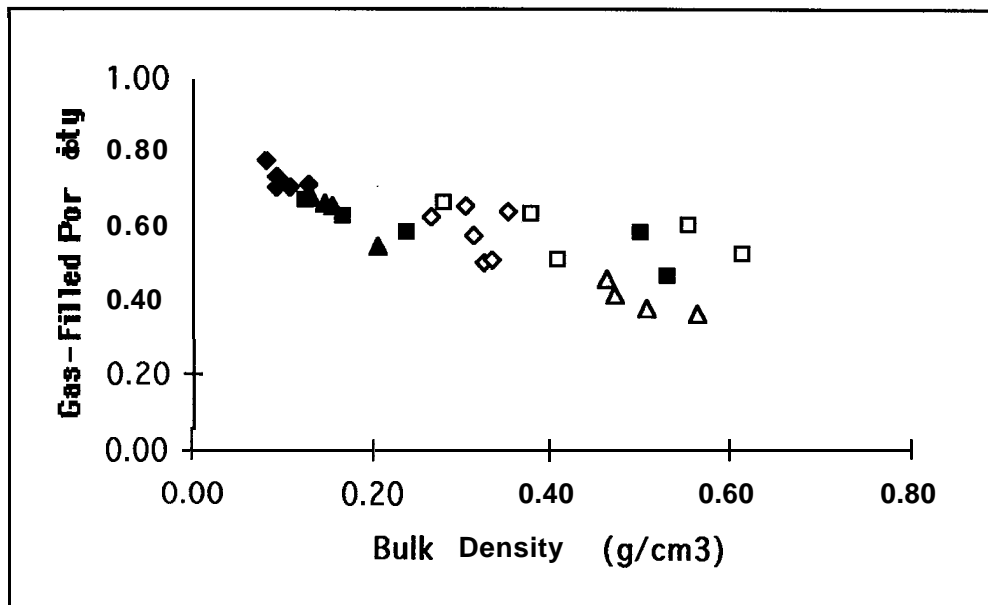
A faster rate and greater degree of compaction would be expected in the field under the more frequent wetting-drying cycles and freeze-thaw cycles of spring and fall.

Compaction of the compost layer would have been inhibited somewhat by adhesion of compost to the plexiglass tube wall which provided rigid support. In the field, the compost layer would lack this artificial support and would be free to compact more quickly. We became concerned that the slow rate of compaction, especially in the fresh compost treatments which were visibly porous, might be caused by a sealing effect of the compaction piston (Photo 2) so that gases could not escape from the compost, thereby allowing water to infiltrate. This would also cause a 'hydraulic' resistance to compaction as positive pressure developed in the compost layer. To investigate this hypothesis, the weights were removed from the Ploughed Fresh replicate B and the piston was moved a few centimetres up the tube above the layer of water sitting on the compost layer. During the next three days it was observed that, (1) no gases were seen escaping from the compost, (2) the head of water above the compost did not decrease, and (3) the compost layer thickness did not change (i.e., no rebound or swelling occurred). When the 50 KPa piston pressure was reapplied after three days, there was no additional, short term compaction. It was concluded that the Fresh compost layer was stable, very resistant to further change, and had some property that effectively sealed off the layer from both the escape of gases and the infiltration of water.

Water that infiltrated the compost layers was held in a relatively small proportion of the pore spaces. It is possible that gases produced by decomposition were being trapped in the pore spaces thereby excluding water. This hypothesis was suggested by the work of Buttler et *al.* (199 1) where they concluded that the low hydraulic conductivity of peat in the field is determined by the blockage of pore space by trapped methane gas bubbles produced by decomposition. The gas analyses in this experiment showed only trace amounts of methane but much larger amounts of carbon dioxide were being produced, especially in the fresh compost (see Section 4.4.3). Some large voids developed and were visible in the fresh compost layers causing a great deal of variability in the physical characteristics. If the voids contained nitrogen gas, presumably caused by the denitrification of compost nitrate (see Table 2), our method of gas chromatographic analysis using nitrogen carrier gas was unable to measure it.

There were no apparent trends of dry bulk density or gas-filled porosity with depth in the compost layer. As was indicated by the inverse relationships shown in Figure 7, the profiles of gas-filled porosity and bulk density are mirror images of each other. The fresh compost is consistently lower in bulk density and higher in gas-filled porosity than the mature compost in each cover layer model. Only the Sand Mature treatment showed a substantially lower gas-filled porosity and higher bulk density than the other treatments, but the significance of this is unknown and the degree of variability was high among the three replicate Sand model samples.

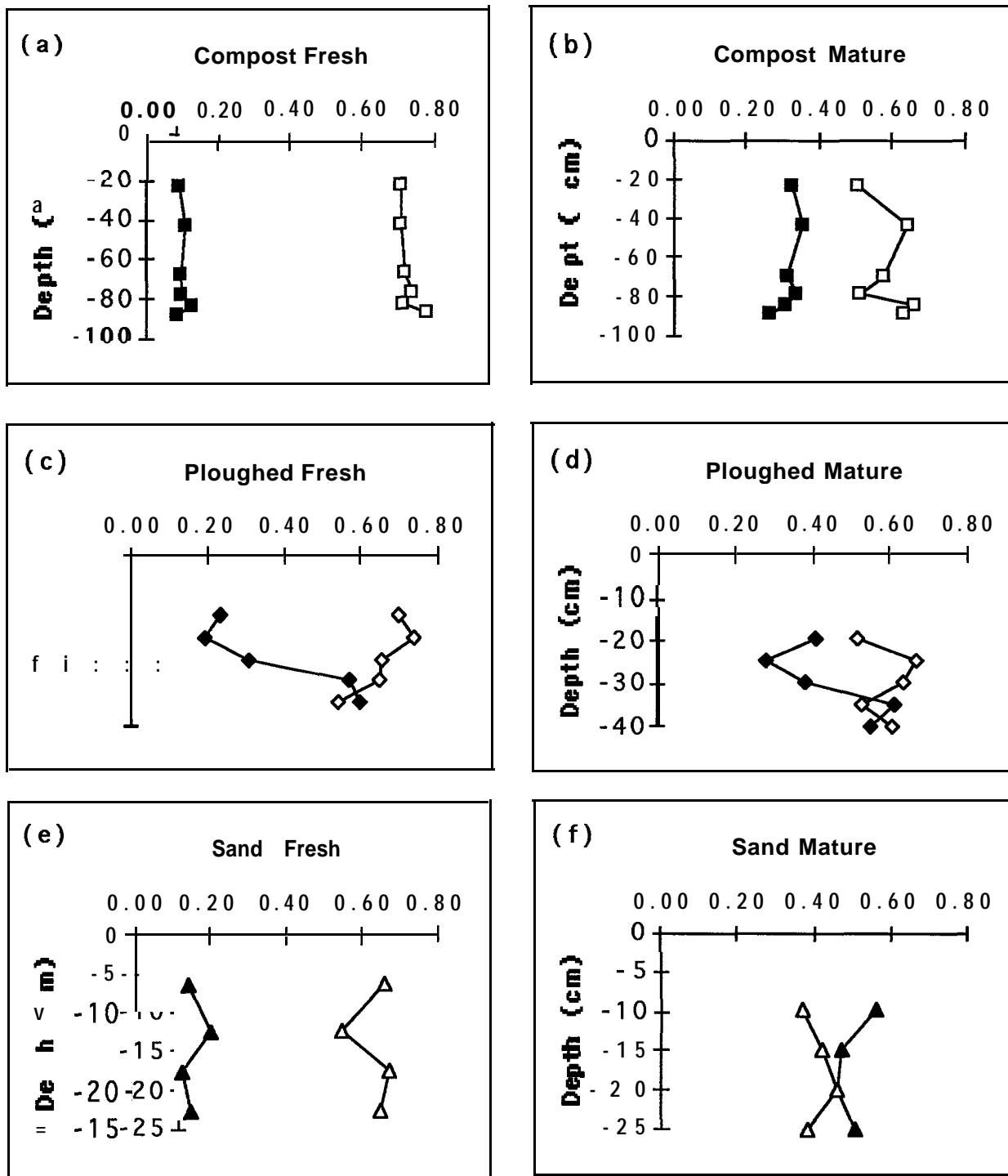**Figure 7. Profiles** of dry bulk density (g cm^{-3}, solid symbols) and gas-filled porosity (cm3 cm^{-3}, open symbols) in the compost layers of the six treatments, measured on day 94 of the experiment. Each point is the average of three replicates.

4.1.3 Effects on Hydraulic Conductivity

There was a striking difference in hydraulic conductivity between the mature and fresh compost treatments. Figure 8 shows a decrease over several orders of magnitude in saturated hydraulic conductivity, from a maximum of 4.4×10^{-4} cm/s in mature compost to a minimum of 2.2×10^{-6} cm/s for fresh compost columns. By contrast, tailing and ploughed (mixed) layers had much higher saturated hydraulic conductivities of 1.3×10^{-3} cm/s and 0.6×10^{-3} cm/s, respectively. Conductivity decreased from tailing, to the ploughed layer (50:50 mature compost-tailing mixture), to mature compost, to fresh compost. There is also evidence in the data of a reduction of conductivity with greater compost layer compaction, as shown by the lower conductivities of the more compacted Ploughed and Sand models in contrast to the less compactedCompost model.

A large degree of variability was seen in the conductivities for each treatment, even for the tailing alone. The source of the variability is not clear but it may be the result of the packing technique, the small cross-sectional area of the column, or heterogeneity of material selected for each column. Boelter (1969) found great variability in saturated hydraulic conductivity between peat types and samples at the same stage of decomposition. The hydraulic properties of the fresh compost continued to change throughout the experiment. When the fresh compost treatments were wetted after loading (day 1), the water infiltrated at a slow but visible rate. Later in the experiment, about day 90, three of the fresh compost columns still had a shallow head of water on the compost from the rewetting on day 42, indicating a further decrease in hydraulic conductivity to a value near zero in the Fresh treatments.

The values of saturated hydraulic conductivity for fresh compost (e.g. 0.01 cm h-1 or 0.3×10^{-5} cm s-1) were in the lower range of the most decomposed and least conductive peat samples measured by Boelter (1969), and 100 times less conductive than Minnesota peats (Chason & Siegel 1986). In a general sense, however, peat and compost cannot be compared because peat becomes less conductive as it decomposes while this compost (made by aerobic processes) appears to be two orders of magnitude more conductive when it is mature. If fresh compost, like peat, were kept in a state of anaerobic decomposition as a result of compaction and high water content, it is possible that its low hydraulic conductivity could be maintained. The applicability of this hypothesis to the compost-tailing system can be tested realistically only under field conditions and over a much longer time period.

As we discussed in the previous section, despite its higher water contents and greater porosity, the fresh compost layer effectively seals off the system from infiltration of water or the escape

of significant amounts of gas. Thus, the compost layer forms the largest and most limiting resistance of the water drainage pathway through the compost-tailing system. If the sealing property of a fresh compost layer could be maintained permanently, and possibly enhanced by long term compaction, then the very low hydraulic conductivities it produces would form an effective barrier to the infiltration of precipitation and groundwater flow into the tailing from the surrounding terrain, and thus decrease the flow of dissolved oxygen they contain.
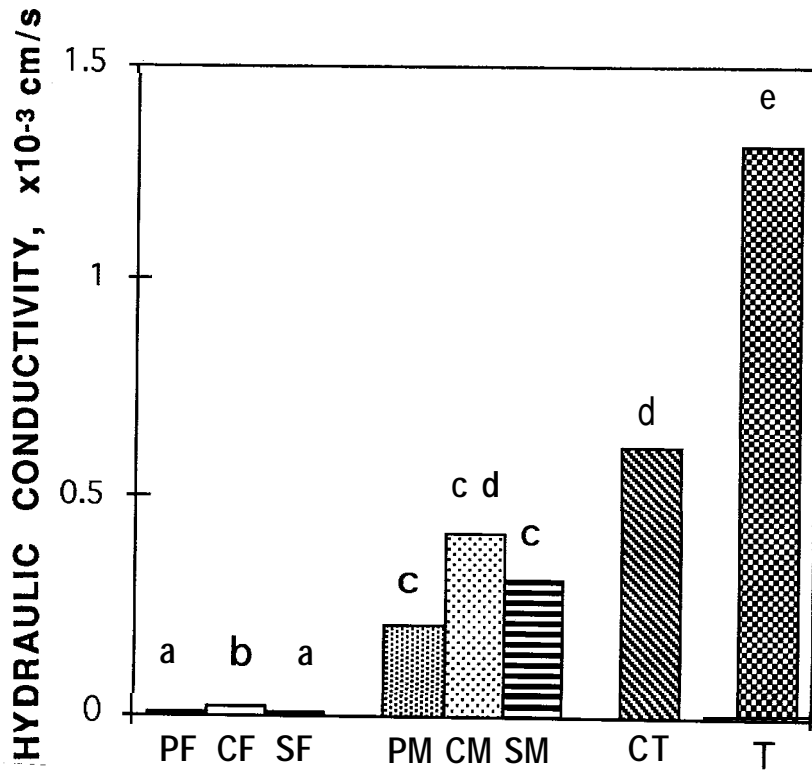

Figure 8. Saturated hydraulic conductivity of the compost cover layer-tailing system. Each bar is the average of three replicates and the same bold letter indicates no significant difference at the p=0.2 level. (PF - Ploughed Fresh; CF - Compost Fresh; SF - Sand Fresh; PM - Ploughed Mature; CM - Compost Mature; SM - Sand Mature; CT - Compost and Tailing mixture; T - Tailing alone).

4.2 Leachate Composition from Compost of Contrasting Degrees of Maturity

The report of the compost leachate quality analyses by Lakefield Research Ltd. is presented in Appendix C. Results selected from their analyses are reproduced here and discussed in the context of the compost-tailing simulation experiment.

4.2.1 Background Content Characterization

The concentrations of selected trace elements and nitrogen compounds in the mature and fresh (immature) compost is presented in Table 2. It may be seen that most trace elements were present at concentrations of one-third to one-half the suggested allowable concentrations for finished compost (Ontario Ministry of the Environment 199 1). Molybdenum is the only element which exceeded the guideline limit. The source of the molybdenum is unknown and this is an unusual contaminant for source-separated household compost. There are no published standards for the background concentrations of Fe, Mn or nitrogen compounds, but Lakefield Research Ltd. notes that these are present at relatively high concentrations in the mature and fresh compost samples. More important than the background concentration is the leachability of these elements and compounds.

Analyses of mature and fresh compost for common toxic organic chemicals (see list in Appendix C, page 8) revealed no detectable concentrations of these contaminants in the compost. As a result, the leaching tests did not analyse for these compounds. The lack of any pesticide or herbicide residues in this compost, which is composed partly of yard waste, may be a function of the late autumn season when it was collected. Contamination by organic chemicals must be considered a potential problem with the use of MSW compost until the seasonal variability of the feedstock is better understood.

4.2.2 Compost Leachate Analyses

The results of two different leaching tests, Regulation 347 and TCLP, reveal similar patterns in the mobility of trace metals and nitrogen compounds (Table 3). Concentrations of nitrate and nitrite in the mature compost, and organic nitrogen concentrations in both the mature and fresh compost, were quite variable but often significantly higher than the Ontario Drinking Water Objectives (ODWO), the human consumption standard usually used as a more strict comparison to Regulation 347. Iron and manganese also showed an elevated leachate level, especially in the fresh compost.

Table 2. Concentrations of elements and nitrogen compounds in fresh and mature compost in comparison to the Ontario Ministry of the Environment (199 1) Guidelines for compost production. (From Lakefield results, Appendix C, page 1). TKN ▪ Total Kjedhal Nitrogen and organic N = TKN ▪ (NH4 + NH_3). All concentrations in μg/g dry weight.

Analytes	Fresh Compost Average	Mature Compost Average	Ontario Compost Guidelines
As	2.5	3.5	10
Cd	1.9	1.7	3
Co	10.3	10.3	25
Cr	28.0	21.4	50
CU	31.1	33.5	60
Fe	10445	11100	
Hg	0.02	0.05	0.15
M n	507	435	
M o	37.8	52.6	2
Ni	16.5	12.7	60
Pb	32	53	150
se	<1	<1	2
Zn	155	179	500
NO_3	520	2968	
NO_2	<10	<10	
TKN	16507	11461	
NH_4+NH_3	672	92	
organic N	15835	11369	

Table 3. Concentrations of elements and nitrogen compounds leached from fresh and mature compost in accordance with Ontario Regulation 347 hazardous waste test (R347) and the Toxicity Characteristic Leaching Procedure (TCLP). (From Lakefield Research results, Appendix C, pages 2 & 3). TKN - Total Kjedhal Nitrogen and organic N = TKN - $(NH_4 + NH_3)$. All concentrations in mg/L (Note: dash = element or ion not detected). Leachate concentrations are compared to the Mine Effluent Limits of the Municipal and Industrial Strategy for Abatement in Ontario (MISA).

Analytes	Fresh Compost		Mature Compost		MISA
	R347	TCLP	R347	TCLP	Limits
As		-	-		0.5
Cd		-	-		
Co		-	-		
Cr		-	-		
Cu					0.3
Fe	0.60	0.69	0.71	0.29	
Hg		.002		.001	
Mn	5.24	0.12	1.29		
Mo					
Ni		-	-		0.5
Pb		-	-		0.2
Se					
Zn	0.13	0.04	0.09	0.03	0.5
NO_3	0.42	-	49.4	104	
NO_2	0.55		77.3		
TKN	46.2	15.6	11.1	5.29	
NH_4+NH_3	14.0	5.49	0.24	0.45	
organic N	38.2	10.1	10.8	4.8	

The leaching results indicate that inorganic and organic nitrogen compounds are mobilized in the compost especially once it has matured. However, this was a 'worst case' leaching test that did not simulate the 'real world' situation of the compost cover layer on tailing. Leaching will be important only where the infiltration and subsurface flow of water are significant. Measurements of saturated hydraulic conductivity for the compost-tailing system (see Section 4.1.3) suggest that the water flow rate could be very low through a fresh compost cover layer. Changes in the hydraulic properties of the compost with compaction and further humification may further suppress the ground water flow through the compost layer, and either into the tailing or laterally out of the compost layer itself, so that leaching of nitrogen compounds and trace metals will not be a problem for the watershed outside the tailing area. Monitoring of nitrate level in samples in the simulation experiment (section 3.24) addressed what actually happens to nitrate in a simulation of the compost-tailing system. One of the benefits of the compost cover layer is the establishment of reducing conditions in the tailing which would lead to nitrate reduction and the denitrification of nitrates and nitrites to produce harmless nitrogen gas.

4.2.3 Leachate Composition from Spiked Compost

Mature and fresh compost were spiked with high levels of inorganic and organic compounds in order to investigate how a highly contaminated batch of compost would retain toxic constituents. The leaching results from the Lakefield spiking experiments are given in Appendix C, pages 4-7 and 9-l 2. The percentage of the spiked material which appeared in the leachate of the fresh and mature compost samples is shown in Table 4. The results for some analytes are quite different between the two standard leaching tests.

The pattern of spiked leaching results is difficult to interpret and reveals some problems with both the premise of the experiment and the methodology. The inorganic and organic spiking materials were water soluble and were added to the compost at ten times the maximum allowable concentrations for compost material (see Appendix C, pages 4 and 9). However, the short incubation time of 24 hours would not have been enough time for equilibria to develop between the analytes in solution and the solid phase. Thus, it is not surprising that a significant portion of some spiking compounds were leached out. Additionally, many of the compounds were already present in significant quantity in the background composition of the compost and may have already been occupying most of the adsorption sites on the solid phase.

These factors make the spiking experiment unrealistic and cast doubt on the applicability of the leaching test results to the safety issue of using MSW compost as a tailing cover layer. It would

have been more useful to have obtained a batch of compost which was 'naturally' contaminated by toxic source materials. This compost would have had time to establish an equilibrium between the solid and liquid phase. Hence, leaching analyses of such a compost would have given a better indication of the long term quality and safety of the material by itself.

Table 4. Percentage of material used to spike fresh (raw) and mature compost which subsequently appeared in the leachate using the Regulation 347 test and the TCLP test. Percentages have been rounded to the nearest integer. Note: dash ▪ not detected (Derived from Lakefield Research analyses, Appendix C.)

Analyte	Percentage of Spiked Material Leached			
	Fresh Compost Samples		Mature Compost Samples	
	R347	TCLP	R347	TCLP
As	19	20	20	22
Cd	7	6	8	7
Co	21	18	23	19
Cr	↑	↑	4	3
CU	↑	↑	↑	↑
Fe	3	81	3	86
Hg	3	3	↑	2
Mn	71	72	61	77
Mo	110	5	71	5
Ni	13	11	14	12
Pb	0	0	0	0
se	26	20	36	33
Zn	15	16	18	18
Aldrin	4	↑	24	3
Deldrin	0	0	0	0
Chlordane	14	14	14	14
DDT	0	0	↑	0
Endrin	3	2	5	4
Heptachlor		-		
Heptachlor epoxide	4	4	5	4
Lindane		-		
Methoxychlor	28	16	16	16
Toxaphene	14	14	14	14
2,4-D	31	18	39	37
Silvex	22	28	29	44
PCB	56	56	52	40

4.3 Chemical Processes and Changes Within the Compost and Tailing

4.3.1 X-Ray Diffraction Analyses of Oxidized and Non-oxidized Tailing

One oxidized tailing sample and one non-oxidized (reduced) tailing sample were analyzed by x-ray diffraction. The complete results of the analyses are presented in Appendix D (Table D. 1 and Table D.2), for the oxidized and non-oxidized samples, respectively. The limited sensitivity of the instrument permitted us to detect only obvious differences between the two samples.

In the non-oxidized tailing, there was a noticeable presence of:

anot-thite	$(Ca, Na) (Si, Al)_4 O_8$
albite	$(Na, Ca) (Si, Al)_4 O_8$
pyrrhotite	$Fe_{1-x} S$
other aluminosilicates.	

In the oxidized tailing:

aluminosilicates were replaced by quartz (SiO_2)
pyrrhotite was replaced by oxidized forms of Fe, such as Fe_3PO_7, K_2FeO_4, $Mg_2Fe_2O_4$, $FeAl_2O_4$.

4.3.2 Chemical Simulation of Redox and Acidity Effects

Simulation experiments of reducing conditions on oxidized and non-oxidized tailing are summarized in Table 5. Chemical reductants were effective in decreasing Eh, but it is not known what effects this had on the mobility of certain trace elements in the tailing.

The redox simulation results show that the solubility of iron and most metals reached a maximum at -350 mV, a moderate reducing environment. The similar peak in release of other trace elements at -350 mV indicates that most of the trace metals are adsorbed or coprecipitated with ferric oxides and are released at the same time these oxides are reductively dissolved. Highly reducing conditions (-400 to -500 mV) caused a decrease in the solubility of iron and some trace metals, with the exception of Co, Mn, and Ni, as less soluble metal sulphides were formed. It should be noted that these were simulation experiments that did not involve naturally-occurring reductants or organic matter that may be present in the compost-tailing system. Thus, the results can indicate only the general trend toward decreasing solubility of many trace elements with decreasing redox potential.

Table 5. Concentrations of trace metals released into solution at different absolute redox potentials by exposure of oxidized tailing to chemical reducers (average of triplicate results with a 5-10% relative error). Note: dash - not detected

Redox Potential (mV)	Concentrations of metals released from tailing ($\mu g/g$)									
	Fe	Mn	Cu	Ni	Cr	Co	Cd	Zn	Pb	Hg
-209	▪	▪	20	▪	▪	▪	▪	▪	▪	▪
-300	46000	20	25	113	10	4	6	24	-	-
-350	58000	38	75	225	35	5	8	75	▪	▪
-400	47000	27	21	158	13	5	8	39	▪	-
-450	35000	14	▪	100	9	3	6	13	▪	▪
-500	22000	97	20	720	▪	19	3	42	▪	▪

In a preliminary, non-replicated experiment, mature compost was used as a reducing agent in a slurry with oxidized Nickel Rim tailing. The addition of compost to tailing also brought about a modest decrease in E_h of about 100 mV over several weeks (Figure 9). The decrease in relative E_h was an encouraging sign that the compost material would provide a beneficial cover layer. However, we believe that the weak reducing condition that developed because of the compost chemical properties would not, by itself, cause significant reductive dissolution of oxides, and thus not result in the release of large amounts of iron or trace metals into solution.
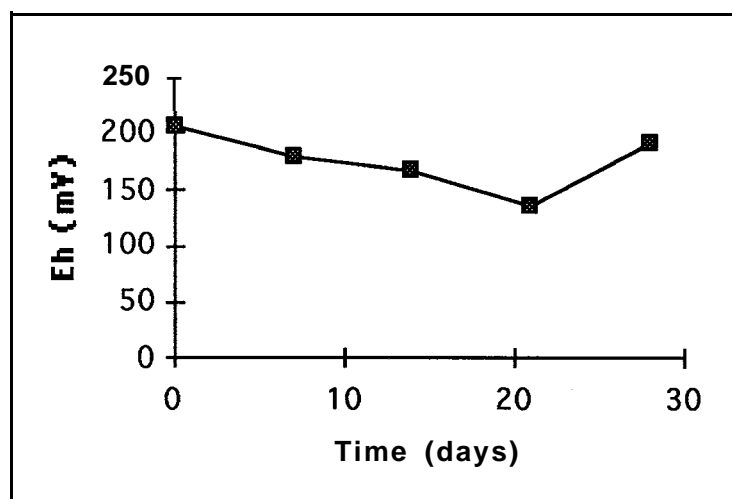

Figure 9. Simulation of mature compost as a reducing agent in oxidized tailing on a 1 :1 ratio by weight.

4.3.3 Sequential Extraction of Oxidized and Non-Oxidized Tailing

Sequential extraction analyses for trace metals were performed on original samples of unoxidized and oxidized tailing from the Nickel Rim site. The same oxidized tailing material was used in the compost cover layer simulation experiment. The sequential analyses were done to provide information on the mobility of trace metals under different redox conditions, as may be experienced under the different cover layer treatments in the simulation experiment. For clarity, the complete picture of the distribution of trace metals in the original unoxidized and oxidized tailing, and tailing at the end (day 290) of the simulation experiment are presented and discussed together in section 4.4.5.

4.4 Compost Cover Layer-Tailing Simulation

4.4.1 General Observations

a) Experimental schedule and treatments

From the outset of the 10 week simulation experiment, it was apparent that this was too short a time frame in which to see the full benefits of a compost cover layer on tailing. Additionally, the low hydraulic conductivity and poor infiltration characteristics of the fresh compost treatments made it impossible to keep to the schedule of wetting-drying period that was originally proposed. The simulation experiment was not terminated after 10 weeks as planned to allow sectioning of the columns, but rather, it was decided to maintain the columns as long as available sampling points and use of the lab space allowed. The simulation experiment was ended following a sampling period on day 290.

b) The maintenance of anoxia

We were unable to maintain an anoxic atmosphere inside the cabinets using reasonable flow rates of nitrogen gas. This caused us some concern since our design was based on a one-dimensional model of oxygen transport from the atmosphere through the cover layers to the tailing, and thus we wanted to avoid the lateral diffusion of atmospheric oxygen through the plexiglass walls of the leaching columns. Attempts were made to better seal the cabinets but these modifications were not successful enough.

However, in the third week of the simulation experiment, a darkening coloration appeared near the compost-tailing interface in the fresh compost treatments (see Photo 4). The discolouration continued to develop as a black solid in the fresh compost columns and moved upward into the compost and ploughed layers, and later downwards into the tailing layer. This was identified by chemical analysis as iron sulphide precipitate. The iron sulphide would not have been able to develop at the walls of the leaching column if significant lateral oxygen diffusion through the column walls was occurring. Late in the experiment, it was observed that iron sulphide was also being precipitated at the interface zone in some of the mature compost treatments. At about the same time it was noticed that, in one of the Ploughed model columns containing fresh compost, the extensive iron sulphide layer had disappeared. Investigation revealed that one of the rubber stoppers at this level had been improperly replaced after the last sampling period several weeks before, and atmospheric oxygen must have leaked into the column and caused oxidation of the iron sulphide precipitate. The incident demonstrated that the plexiglass tube, with stoppers

properly seated, was effective in excluding atmospheric oxygen from the experimental material.

c) Data collection changes

In its conception, this study was meant to be a relatively short-term project of moderate cost, which could be done over the winter of 1992 and prepare the way for comprehensive field work. In retrospect, the time frame of the study as it was proposed was too short to accommodate the variety of experimental work and the number of samples that could have been analyzed. As a result, some changes were made to the experimental design that was proposed (Appendix A) in order to obtain significant results in time for an autumn 1993 field experiment startup. (The simulation experiment was finally terminated in January 1994.) However, we believe that the simulation results, presented below, have revealed significant benefits in using compost cover layers as a solution in reducing acidic tailing AMD, and have satisfied the project goal of allowing the design of the Phase 3 field work to proceed.

The major changes to the experimental design and methodology involved simplifying the experiments and reducing the number of samples in order to better match the experimental material, the available instrumentation, the availability of technical assistance, and keep the project within its budget. For the sake of realism and time, much of the physical characterization and optimization of the compost cover layer design was simplified and incorporated into the cover layer simulation experiment. Sampling techniques to obtain *in situ* samples of pore water and gases proved to be unfeasible with the size of core samples that were taken. As a result, methods were developed to sample deoxygenated water and gases that were in equilibrium with core samples taken under anoxic conditions from the columns. Fewer samples were taken in column vertical profiles without sacrificing the quality of the data that were gathered. It must be mentioned that the gas chromatograph that we used was on loan from another laboratory and had to be returned following the analyses on day 69. Unfortunately, a replacement instrument could not be obtained at a cost that was justifiable within the project budget. Therefore, gas analyses were done for only the first four sampling periods.

4.4.2 Effects on Oxygen Concentration

It was hypothesized that the major potential benefit of a compost cover layer is the establishment a physical oxygen barrier produced by a permanently water-saturated layer between the atmosphere and the acid-generating tailing. A negligible rate of oxygen diffusion through the cover layer would suppress oxidation and allow a progression toward stronger reducing conditions in the tailing as different chemical species dominate the reduction processes

(Pierce 1992). The concentration of oxygen was measured in all samples of compost and tailing for the first four sampling periods (Figure 10). It became evident that oxygen concentration was below 2% in most samples but seldom was close to zero. Thus it was concluded that there was an oxygen contamination error in our samples, caused either by small air leaks into the column (in the absence of a 100% cabinet nitrogen atmosphere, as discussed above), or caused by oxygen introduced by the sampling and analytical methods. Some samples did show oxygen concentrations above 5%, but these samples were surrounded by other samples of low oxygen concentration, and thus are considered anomalous or the result of local oxygen leaks into the column. It is concluded from these data that both mature and fresh compost treatments led quickly to anoxic conditions in all the sample levels that were examined.
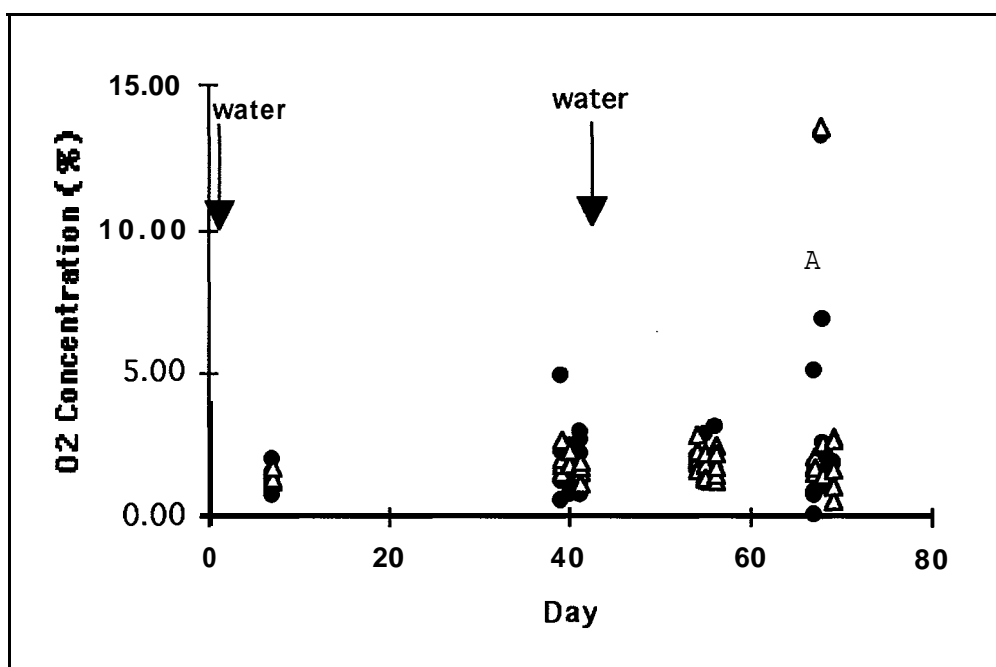
Figure 10. Oxygen concentrations measured in all compost and tailing samples during the first four sampling periods of the simulation experiment. Arrows indicate when water was added on days 0 and 42. (Fresh compost treatments - solid circles, Mature compost treatments - open triangles).

4.4.3 Effects on Water Content and Redox Potential

The relationship between relative redox potential (Eh) and water content for all the samples of the simulation run is shown in Figure 11. It is clear that the highest water contents and lowest E_h were found in the fresh compost layer (Fig. 11, solid squares). The lowest E_h in the tailing was produced under this cover (Fig. 11, solid circles). The water content of the tailing samples

was modified little by the wetting and drying treatments. The ploughed layer, with an equal mixture of compost and tailing (Fig. 1 1, triangles), shows an Eh-water relationship intermediate between the compost and tailing. Some of the Fresh treatment tailing and ploughed layer samples had E_h values as low as those for the fresh compost. This suggests that the properties of the Fresh compost cover layer produced the low E_h in the underlying tailing layer.
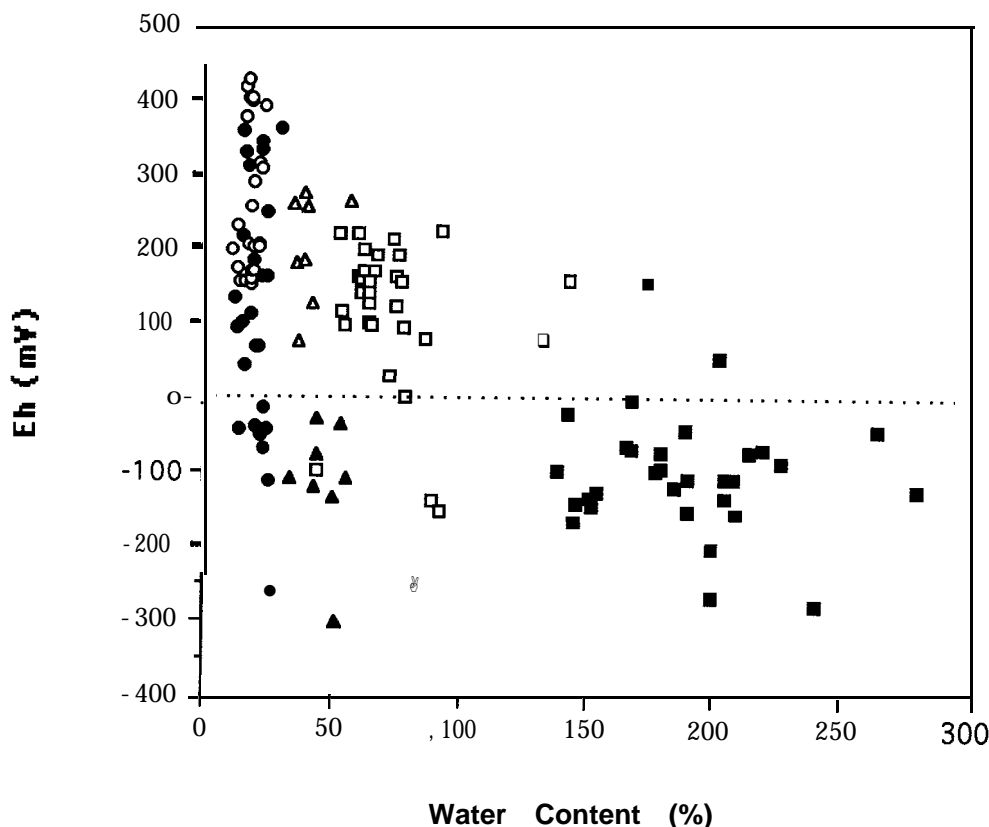
Figure 1 1. Relationship between relative redox potential (Eh) and water content for individual samples. (Fresh compost - solid symbols, Mature compost - open symbols, compost layer - squares, tailing - diamonds, ploughed layer - triangles).

A decreasing redox potential in the tailing with time would indicate the development of stronger reducing conditions which may lead to the reduction of sulphate and ferric ions. The progress of the average Eh for the tailing layer samples can be seen in Figure 12. Tailing under the Fresh compost treatments showed a continuous E_h decrease in the first five sampling periods, while the tailing under the Mature compost cover layer .demonstrated a significant decrease by day 160, but returned to high E_h values like those that were measured at the beginning of the experiment. It is clear that the Fresh and Mature compost covers produced quite different chemical environments in the tailing layer.
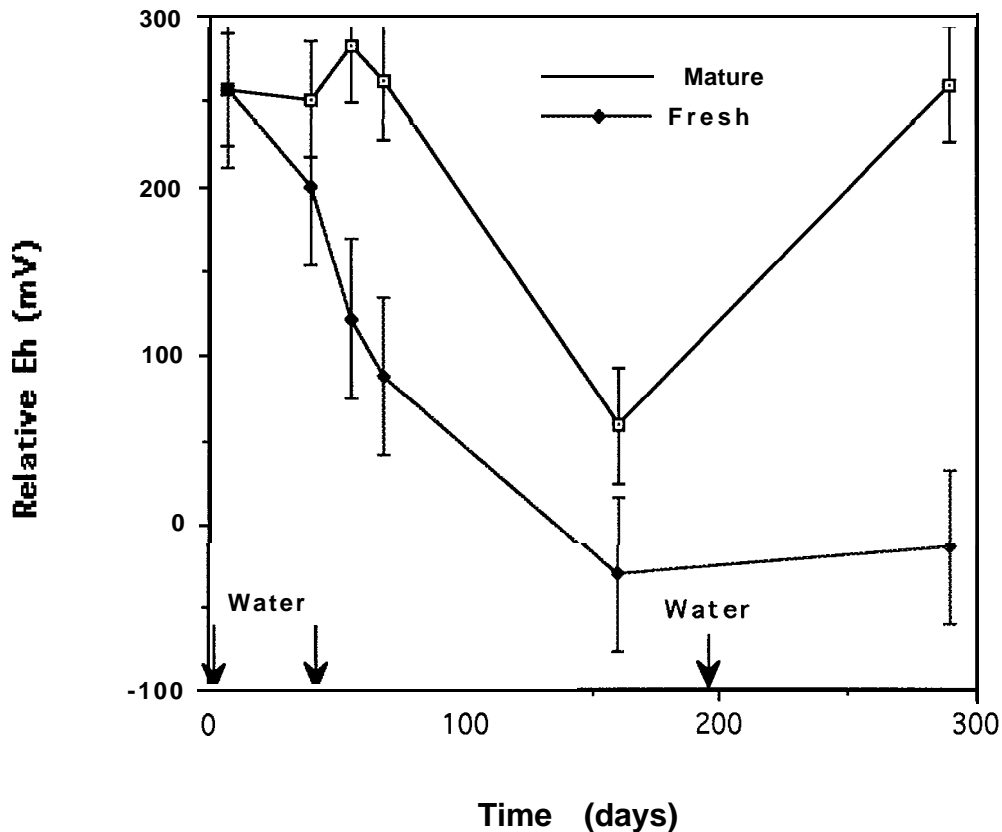
Figure 12. **Changes in tailing layer relative redox potential (Eh) during the simulation experiment.** Each **point is the average of** all **tailing samples for the indicated sampling periods (days 7, 39-41, 54-56, 67-69, 160, 290). (Fresh compost treatments ~ solid** circles, **Mature compost treatments ~ open squares. Standard error of the means is shown).**

A more detailed comparison of the chemical environment produced by the different cover layer treatments is presented through relative E_h profiles (Figure 13), water content profiles (Figure 14), and relative pH profiles (Figure 15). On day 7, complete profiles were not sampled and not all treatments were sampled since it was assumed that the material would have uniform characteristics in all treatments at the start of the experiment.
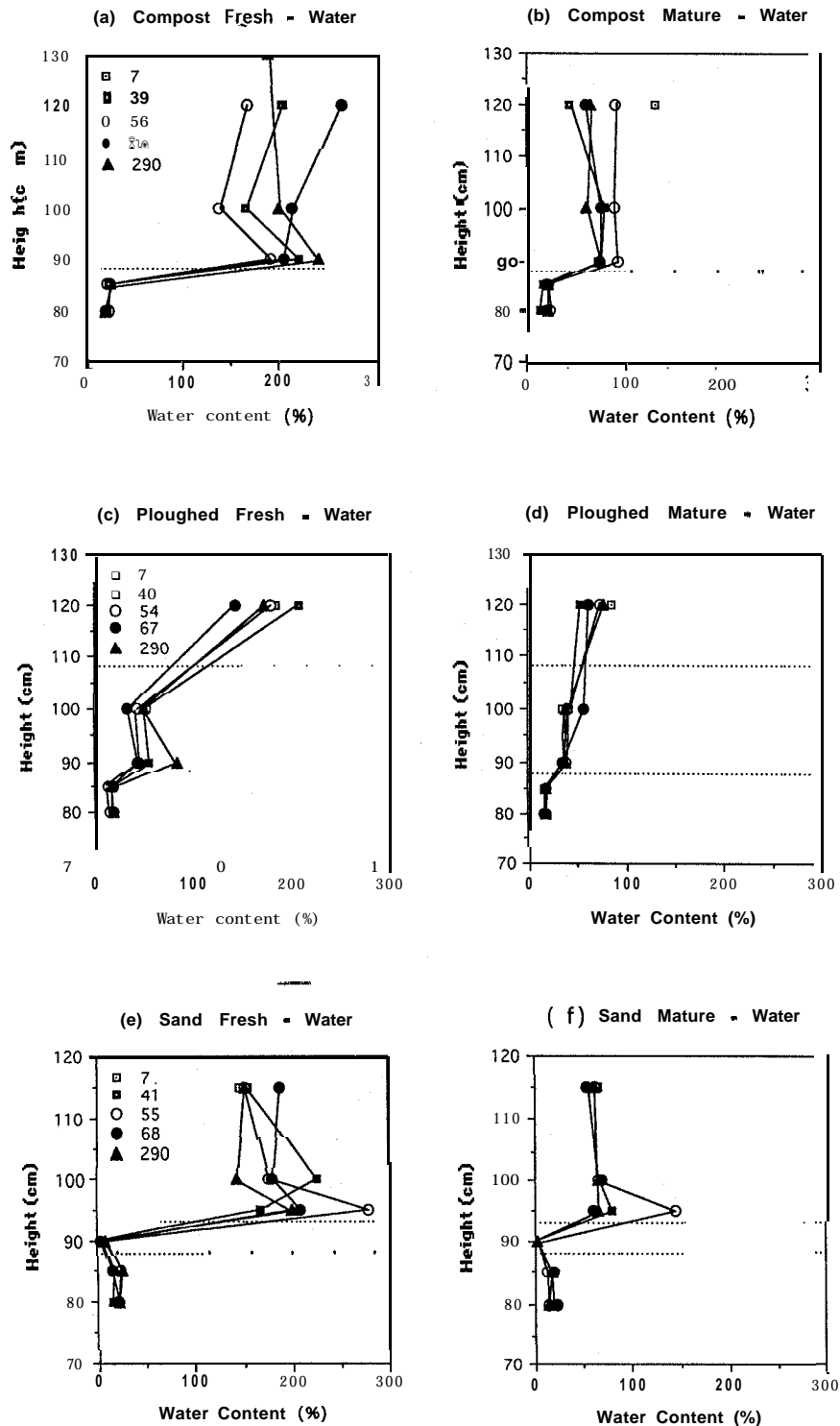
Figure 13. Water content profiles for the Compost, Ploughed and Sand cover models separated into Fresh and Mature compost treatments. (Day 7 - open squares, day 39 - solid squares, day 56 - open circles, day 69 - solid circles, day 290 - solid triangles. Tailing O-88 cm, ploughed layer 88-108 cm, sand layer 88-93 cm).
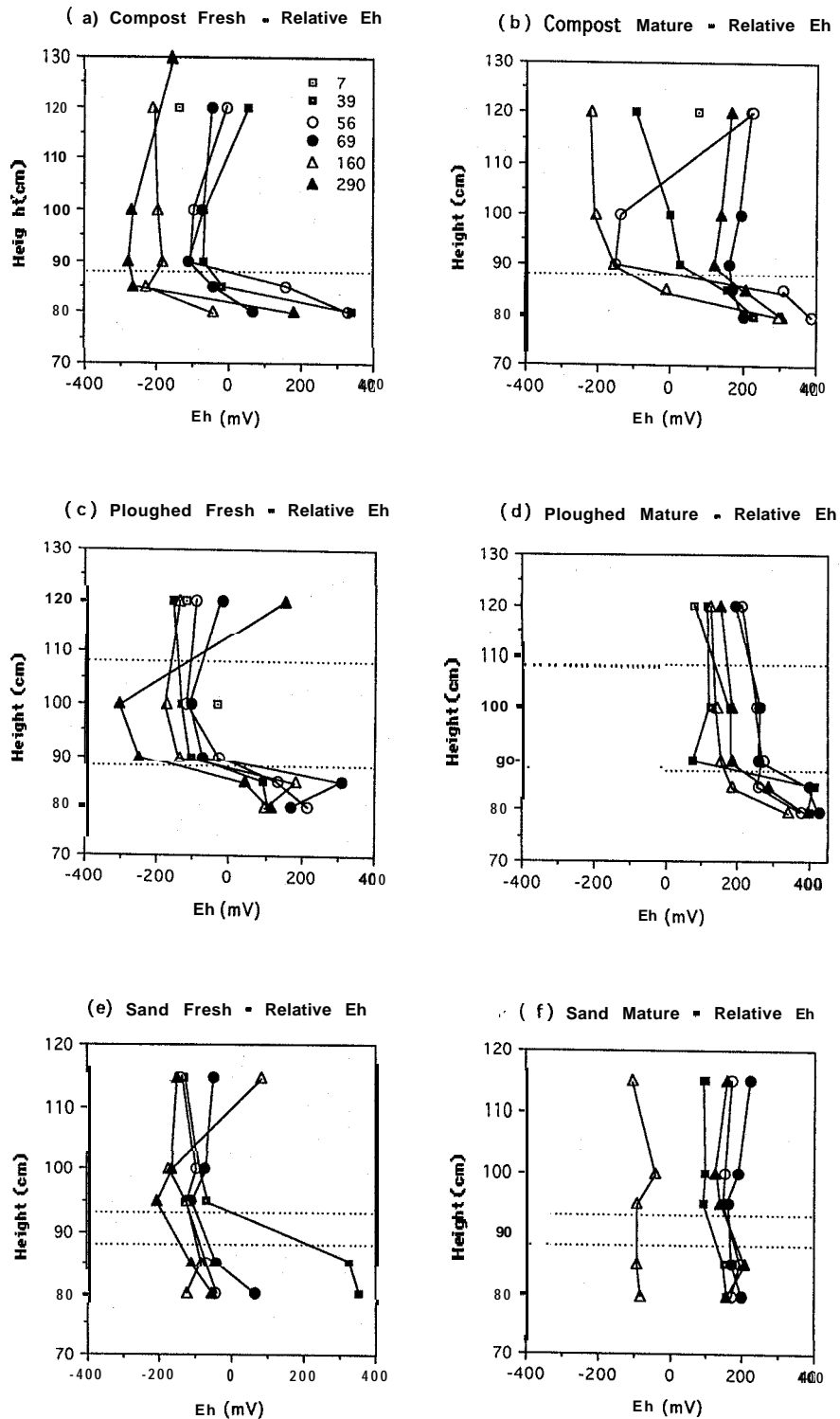
Figure   14. Relative redox potential profiles for the Compost, Ploughed and Sand cover models separated into Fresh and Mature compost treatments. (Day 7 - open squares, day 39 - solid squares, day 56 - open circles, day 69 - solid circles, day 160 - open triangles, day 290 - solid triangles. Tailing O-88 cm, ploughed layer 88-108 cm, sand layer 88-93 cm).

(a) Interface Water-Eh ▪ Day 40

(b) interface Water-Eh ▪ Day 55

(c) Interface Water-Eh ▪ Day 68

(d) Interface Water-Eh ▪ Day 290

Figure   15. Progression of the Eh-water content relationship across the compost-tailing interface during the simulation experiment for each   cover layer treatment. (Compost model ▪ squares, Ploughed model ▪ circles, Sand model ▪ triangles, Mature compost ▪ open symbols, Fresh compost ▪ closed   symbols.)

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The fresh compost layer in all three cover models maintained a higher water content than the corresponding mature compost layer (Figure 13). Tailing layers were uniformly low in water content in all treatments, while the ploughed layers, with a 50:50 mixture of tailing and compost material, had more variable, intermediate water contents.

There is some evidence in the Compost Fresh treatment profiles (Figure 13a) and both Sand Fresh and Sand Mature treatment profiles (Figure 13ef) of a pooling of water in the compost above the interface with the tailing and sand layers. This pattern 'is consistent with the capillary barr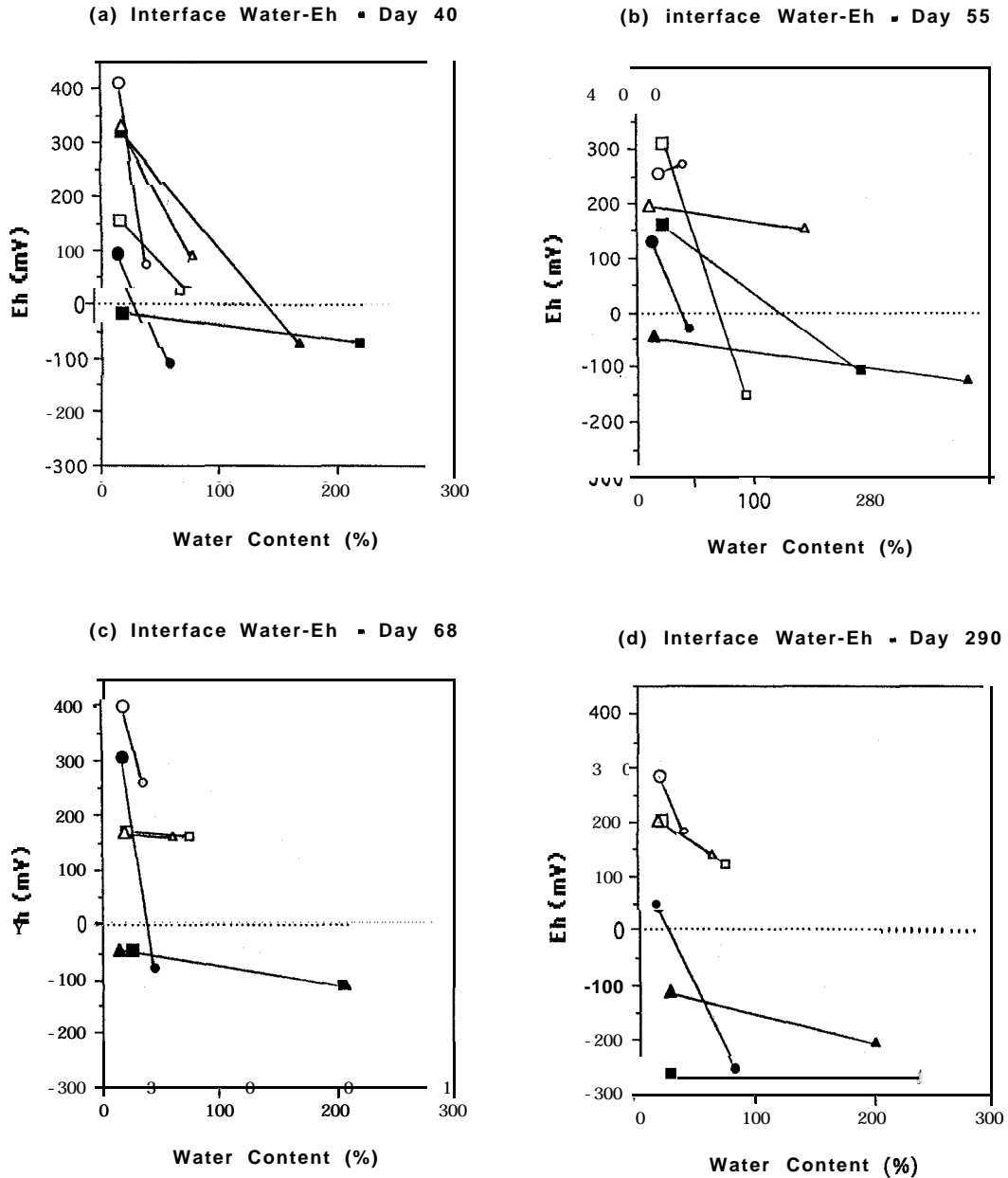ier role of a coarse layer in the Sand model in which water tension (suction) in the unsaturated lower layer could not exert a pull on the water in the much finer overlying layer. Thus, water would not drain and would be retained in the finer layer. However, downward drainage of water could continue in the saturated compost layer under the influence of gravity and result in an accumulation of water in the lower compost layer (Hillel 1971). The potential pooling effect was the main reason for evaluating the Sand model, but it may also be present in the Compost Fresh treatments on days 39, 56 and 290 (Fig. 13a). Since only one level in the compost was sampled in the Ploughed model (Figure 13ab), our data will not show if the water pooling effect also occurred in this treatment, however, the incorporation of compost into the ploughed layer might have created a transition zone between the hyraulic properites of the compost layer and the tailing that discouraged water pooling. In conclusion, these data suggest that following rewatering, the difference in hydraulic properties between the compost and the coarser tailing layer seemed to inhibit drainage of compost water and thus enhanced the benefit of the compost cover as a physical oxygen barrier, at least for a few weeks (Figure 13ef - compare days 55 and 68).

The development of low E_h did not closely follow the patterns of high water content, but. rather progressed with time to more strongly reducing conditions in all three models; through day 160 in the Mature treatments and day 290 in the Fresh treatments (Figure 14). It was noticed that the Ploughed model profiles taken on days 40 and 54 were sampled from the same column (Figure 14cd, closed squares and open circles). Curiously, the profiles in the Ploughed Mature and Ploughed Fresh treatments both showed an increase in Eh, despite the fact that the columns were rewetted on day 42. The Sand model profiles (Figure 14 ef) showed a strong vertical uniformity and tailing E_h reached low values more quickly than in the Compost or Ploughed models. The low Sand model E_h values may have been the result of the maintenance of a better physical oxygen barrier in the compost cover layer, as discussed above.

The pattern of E_h profiles cannot be totally explained from the available data, but it should be noted that each point in a profile was based on a single sample from different replicate leaching

columns and may reflect the inherent variability of the material used. In general, the E_h becomes lower toward the interface in both the tailing and cover layers, especially in the Fresh treatments. Exceptions to this pattern are the minimum E_h values shown in the ploughed layer in many of the Ploughed treatment profiles (Figure 14cd). The depression in E_h may reflect the BOD activity of compost microorganisms as well as the physical oxgyen barrier caused by high water content.

The relationship between E_h and water content in the upper (85 cm) tailing samples and the lowest cover layer sample may be examined in more detail in Figure 15. (Lines on the graphs connect the tailing Eh-water value in the tailing with the matching value in the cover layer.) These data reveal a much closer correspondence between tailing E_h and cover E_h in the Compost and Sand models than in the Ploughed model. Fresh treatments also appear to maintain a stronger linkage of tailing E_h to the cover E_h than the Mature treatments. This suggests that the Compost and Sand cover models are more effective at creating reducing conditions in the tailing under a Fresh compost cover layer. In contrast, there is little difference in the Eh-water relationship among the three cover models when Mature compost is used. The Sand Fresh treatment water content - E_h relationship showed the most clear results, with the water-Eh line becoming more horizontal and declining to lower E_h values from day 40 to 290.

4.4.4 Gas Analyses

Analyses of gas samples in equilibrium with the solid phase samples revealed patterns that were consistent with the relative redox potential and oxygen concentration results. Methane and carbon dioxide were the only gases besides oxygen that were detected. However, since the iron sulphide precipitate observed is formed from the reaction of hydrogen sulphide with Fe (II), it is certain that H_2S gas was present in the tailing-compost interface zone where iron sulphide precipitate was found (see section 4.4.1 and Photo 4). Hydrogen sulphide is produced by anaerobic sulphate-reducing bacteria which utilize small organic molecules as substrates. Methane and carbon dioxide are both decomposition products of anaerobic bacterial breakdown of organic substrates, that were provided here in abundance by the compost and its leachates. Therefore, the presence of methane in the columns was an indication of a strong reducing environment in a least part of the surrounding compost and tailing layer.

Profiles of methane (Figure 16) and carbon dioxide (Figure 17) demonstrated several patterns in the six cover model-compost treatments. No carbon dioxide or methane was detected by the gas analyses of samples from day 7.

Figure 16. Profiles of methane (CH4) in the Compost, Ploughed and Sand model columns during the first four sampling periods of the simulation experiment. (Fresh compost - solid symbols, mature compost - open symbols. Tailing layer O-88 cm, ploughed layer 88-108 cm, sand layer 88-93 cm).
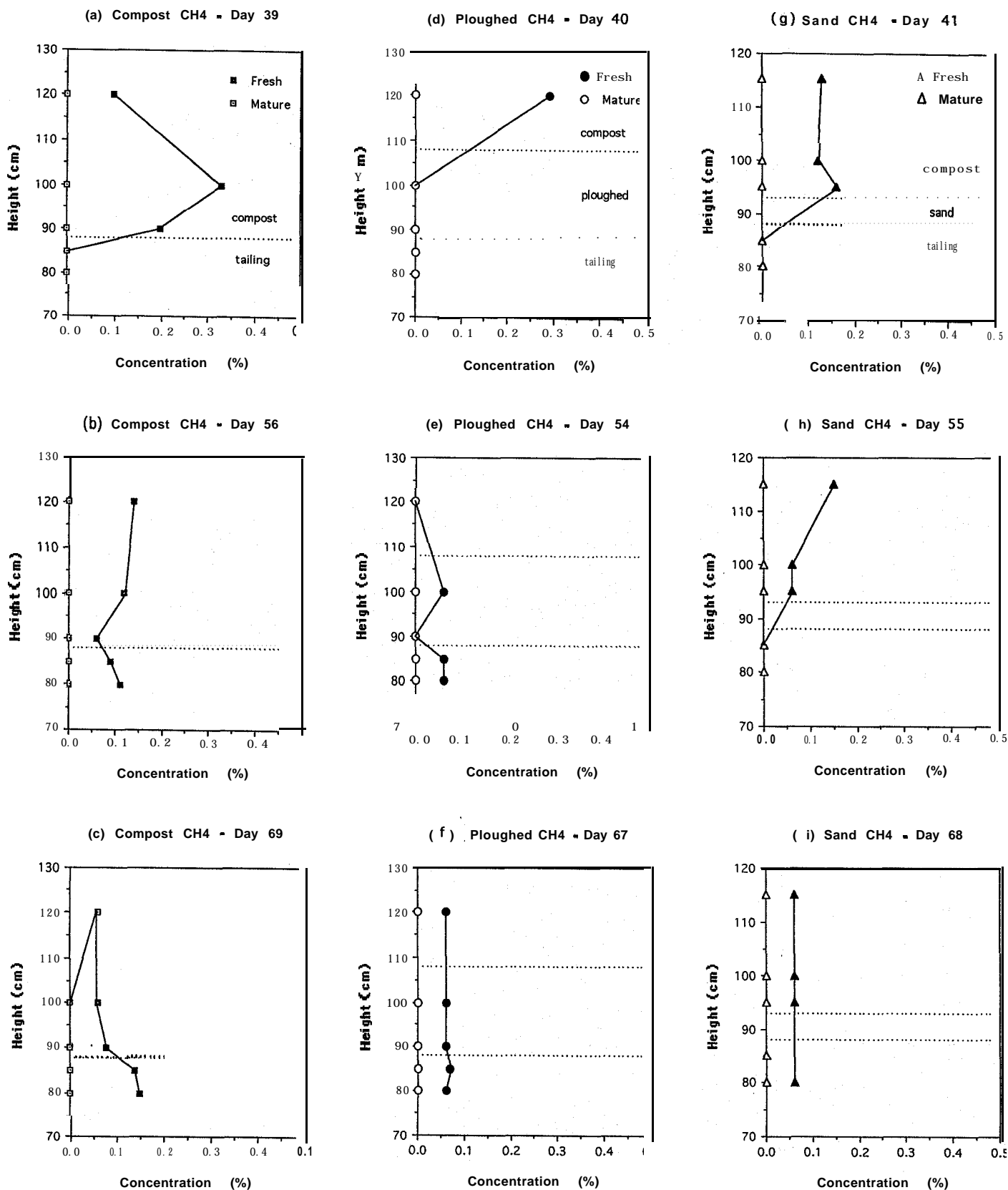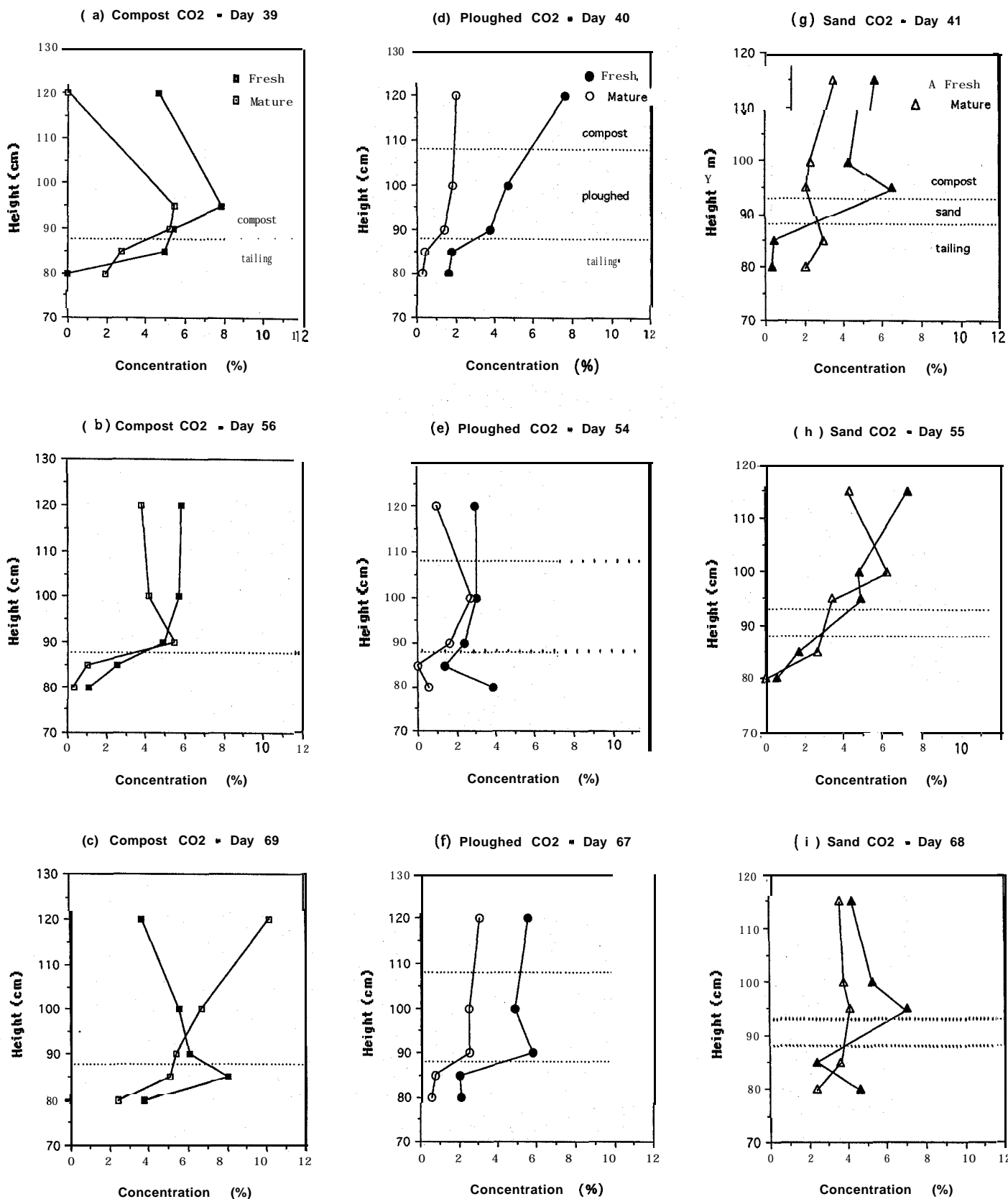
Figure 17. Profiles of carbon dioxide (CO2) in the Compost, Ploughed and Sand model columns during the first four sampling periods of the simulation experiment. (Fresh compost - solid symbols, mature compost - open symbols. Tailing layer O-88 cm, ploughed layer 88-108 cm, sand layer 88-93 cm).

In all three cover layer models, (1) methane and carbon dioxide production showed an increase with time in the tailing region of the profile. (2) However, methane was virtually absent in the Mature compost treatments of all cover layer models, but was found in both the compost and tailing layers in the Fresh compost treatments. These patterns are consistent with the decrease in E_h in the tailing layer (Figure 14) and the establishment of sufficiently strong reducing conditions necessary for methane generation. The relationships between tailing methane concentration and relative E_h (Figure 18), and between carbon dioxide concentration and relative E_h (Figure 19) show an inverse trend of higher gas concentrations at lower Eh, especially in the Fresh compost treatments. (3) Carbon dioxide concentrations were greater in 80% of the fresh compost samples than in the matching treatment Mature compost samples. This is the result of greater rates of anaerobic decomposition in the fresh compost which may be described in their simplest form as the breakdown of carbohydrate (CH_2O) into methane (CH_4) and carbon dioxide (CO_2) :

$$(\quad CH_2O)_2 \quad ====> \quad CH_4 \ (g) \ + \ CO_2 \ (g)$$

There was no significant difference or pattern in CH4 and CO_2 production noted among the three cover layer models for the fresh and mature compost treatments.

Figure 18. Methane (CH4) production in compost cover layer and tailing as a function of relative redox potential (E_h) during the first four sampling periods of the simulation. (Fresh compost - solid symbols, Mature compost - open symbols, Compost model - squares, Ploughed model - diamonds, Sand model - triangles, PFP - ploughed layer fresh compost, PMP - ploughed layer mature compost).

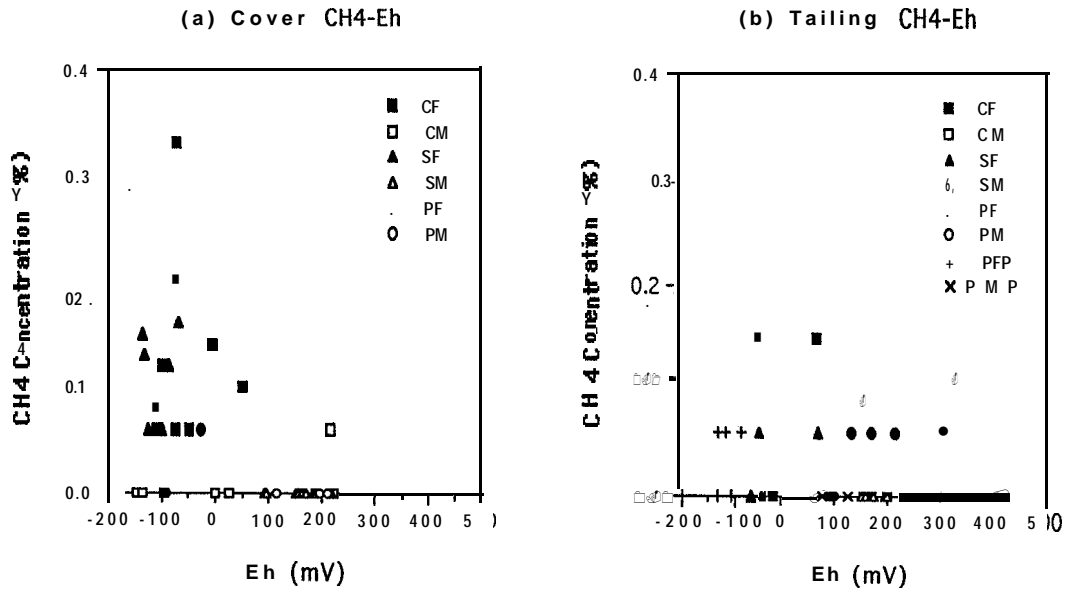**Figure 19.** Carbon dioxide (CO_2) production in compost cover layer and tailing as a function of relative redox potential (E_h) during the first four sampling periods of the simulation. (Fresh compost - solid symbols, Mature compost - open symbols, Compost model - squares, Ploughed model - diamonds, Sand model - triangles, PFP - ploughed layer fresh compost, PMP - ploughed layer mature compost).
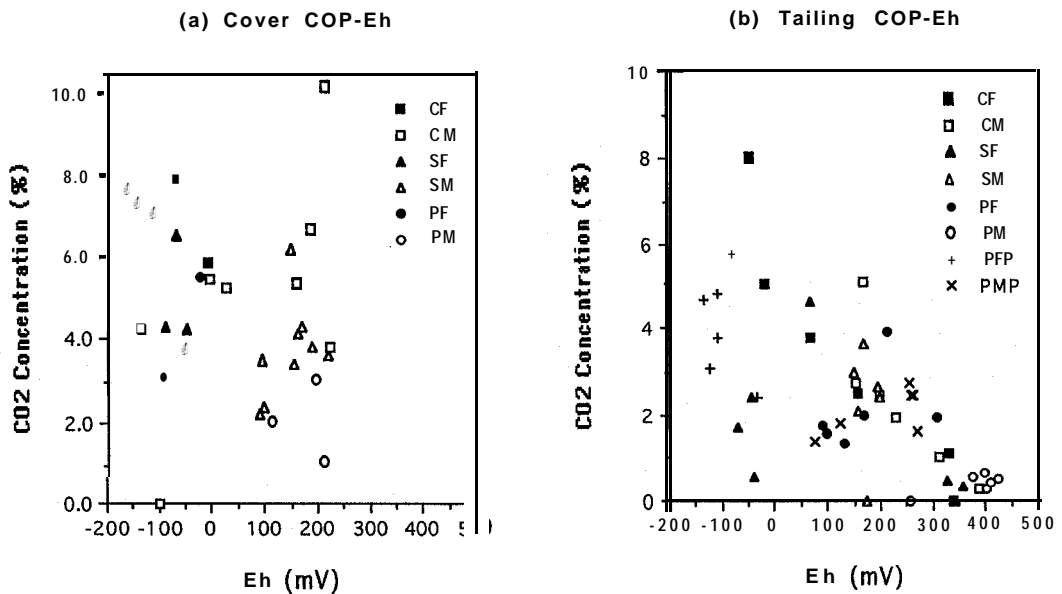
4.4.5 Nitrate, Sulphate and Acidity Analyses

Nitrate and sulphate are two anions which could be expected to show decreases in concentration in a reducing environment (Stumm and Morgan 1981). Sulphate is a major component of the oxidized Nickel Rim tailing (Blowes et al. 199 1), although since the oxidation of pyrite and pyrrhotite has proceeded over several decades, some of the sulphate would have been leached out of the vadose layer. Nitrate and nitrite were present at high concentration in the mature compost stock, but not in the fresh compost where most nitrogen is in an organic form that has not been oxidized (see Table 2).

The profiles of nitrate through the simulation run are shown for the Compost, Ploughed and Sand models (Figure 20). As expected from the compost quality analyses (see Tables 2 & 3), nitrate concentrations were highest in the Mature compost treatments of the Sand and Ploughed models, but not in the Compost model. The lack of nitrate in the Compost Mature replicate columns is very curious. But the E_h values were intermediate to low on day 39 (see Figure 13b), therefore, nitrate may have be reduced already under such strong reducing conditions. The denitrification and the reduction of nitrate would be expected to occur at intermediate E_h levels to produce nitrogen gas or ammonium (Stumm -and Morgan 198 1). The relationship between nitrate and E_h for all samples (Figure 22) appears to support the theory showing an absence of nitrate at low relative redox potentials. The Ploughed and Sand models showed a substantial decline in nitrate from the first to the third sampling period (Figure 20efg, Figure 20ijk). The Ploughed model nitrate samples taken on day 40 (Figure 20e) and 54 (Figure 209 were from the same column, and thus probably had the least amount of variability of all the trends. The Ploughed Mature compost sample on day 54 showed an increase from day 40 in nitrate in the top level of the tailing (85 cm), likely as a result of downward leaching of nitrates. We conclude from these results that nitrates present in a compost cover layer will be reduced quickly under moderately strong reducing conditions. Therefore nitrates should not present a problem in any leachate from a mature or fresh compost cover layer.

Profiles of sulphate concentration showed much more variability than those for nitrate (Figure 2 1). None of the three cover models appeared to show a trend in sulphate concentration through the simulation run. There is no evidence of significant upward leaching of sulphate from the tailing into the compost layer in the Compost model, which does not have a capillary barrier as in the Sand model. The ploughed layer (90 and 100 cm samples) of the Ploughed model has a substantially higher sulphate concentration than the tailing layer in all four Fresh compost profiles. Since the ploughed layer has demonstrated some of the lowest E_h values it is suggested that the processes of reductive dissolution are releasing sulphate ions into solution as the solid

phase minerals bècomes solubilized. The relationship between sulphate reduction and E_h (Figure 23) is unclear because of the presence of high sulphate concentrations at a wide range of relative E_h. It is possible that the reduction of the large amount of sulphate (i.e., 1-4%) in the tailing material requires a much longer time period than the 290 days of the simulation run.

Acidity in the compost-tailing system is a strong function of the oxidation and leaching history of pyrite and pyrrhotite in the tailing at the Nickel Rim site. Sulphate ions and acidity are produced by oxidation of iron sulphides. When sulphate is reduced, acidity is consumed in the formation of hydrogen sulphide, and as a result, the pH increases in a reducing environment as E_h decreases (Rowell 1988). Profiles of relative acidity in the Compost, Ploughed and Sand models during the day 160 and 290 sampling periods are shown in Figure 24. Acidity values in the compost layer of all three models ranged between 7.5 and 8.2. Acidity in the tailing layers was much lower in the Ploughed model (Figure 24cd), but the Compost model showed a strong pH increase to near neutral values from the 80 cm to the 85 cm level. The Sand model exhibited a different pattern with tailing pH near neutral values for both the 80 and 85 cm levels. The cause of these contrasting patterns is not clear. The compost contains a variety of weak organic acids that would act as a buffer to neutralize some acidity through protonation of weak acid functions (e.g., COO- groups). Thus, the buffering role of the compost is clear where the compost is in close contact with the tailing in the case of the Ploughed model mixed layer (Figure 24cd, levels 90 & 100), and in the Compost model near the interface (Figure 24ab, level 85). However, in the Sand model the tailing are physically isolated from the compost, and to be explained by this hypothesis, the near-neutral tailing pH would require a significant downward movement of weak acids through the sand capillary break from the compost cover. The evidence for this process was not quantified in this study.

Examination of the relationship between relative acidity and relative redox potential (Figure 25) demonstrates a linear inverse trend between these variables, especially on day 160. Under anoxic conditions, sulphate-reducing bacteria use small organic molecules as metabolic substrates to reduce sulphate and produce hydrogen sulphide (Stumm and Morgan 1981). Hydrogen sulphide reacts readily with iron and other metals to form sulphide precipitates, and in the process, consumes acidity. Thus, the pH of the tailing should rise as an anaerobic, reducing environment is established in the tailing. This trend was confirmed by pH-Eh inverse relationship shown in Figure 25. This trend suggests that strong reducing conditions (low Eh) in the Sand model tailing layer especially, were causing sulphate reduction and the consumption of acidity. However, the profiles of sulphate for days 68 and 290 (Figure 21 kl) do not clearly show a drop in sulphate concentrations in the tailing layer, but variability in tailing among the replicate treatments may be obscuring any trend.
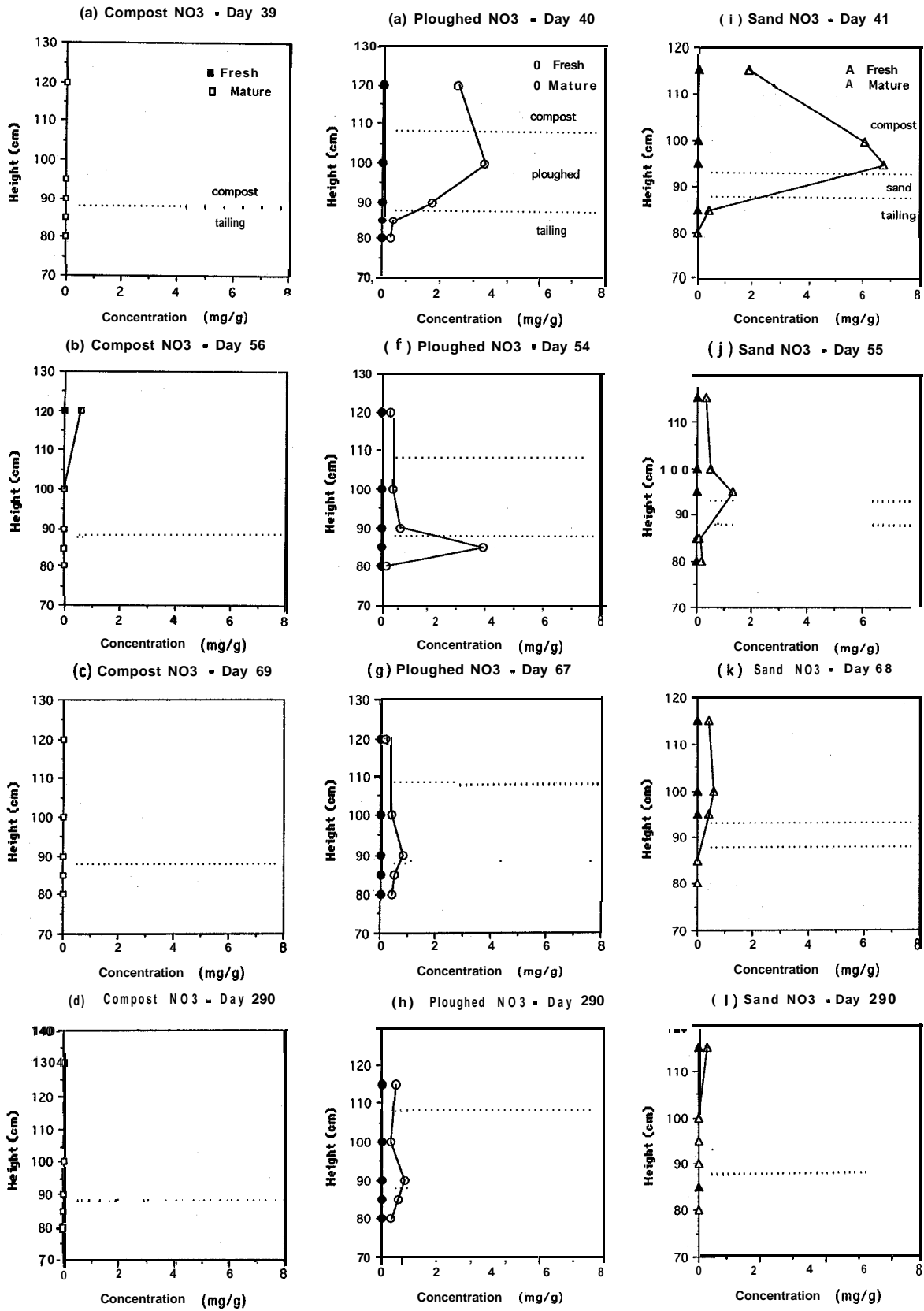
Figure 20. Profiles of nitrate (N03) in the Compost, Ploughed and Sand models during the simulation experiment. (Fresh compost - solid symbols, mature compost - open symbols. Tailing layer O-88 cm, ploughed layer 88-108 cm, sand layer 88-93 cm).
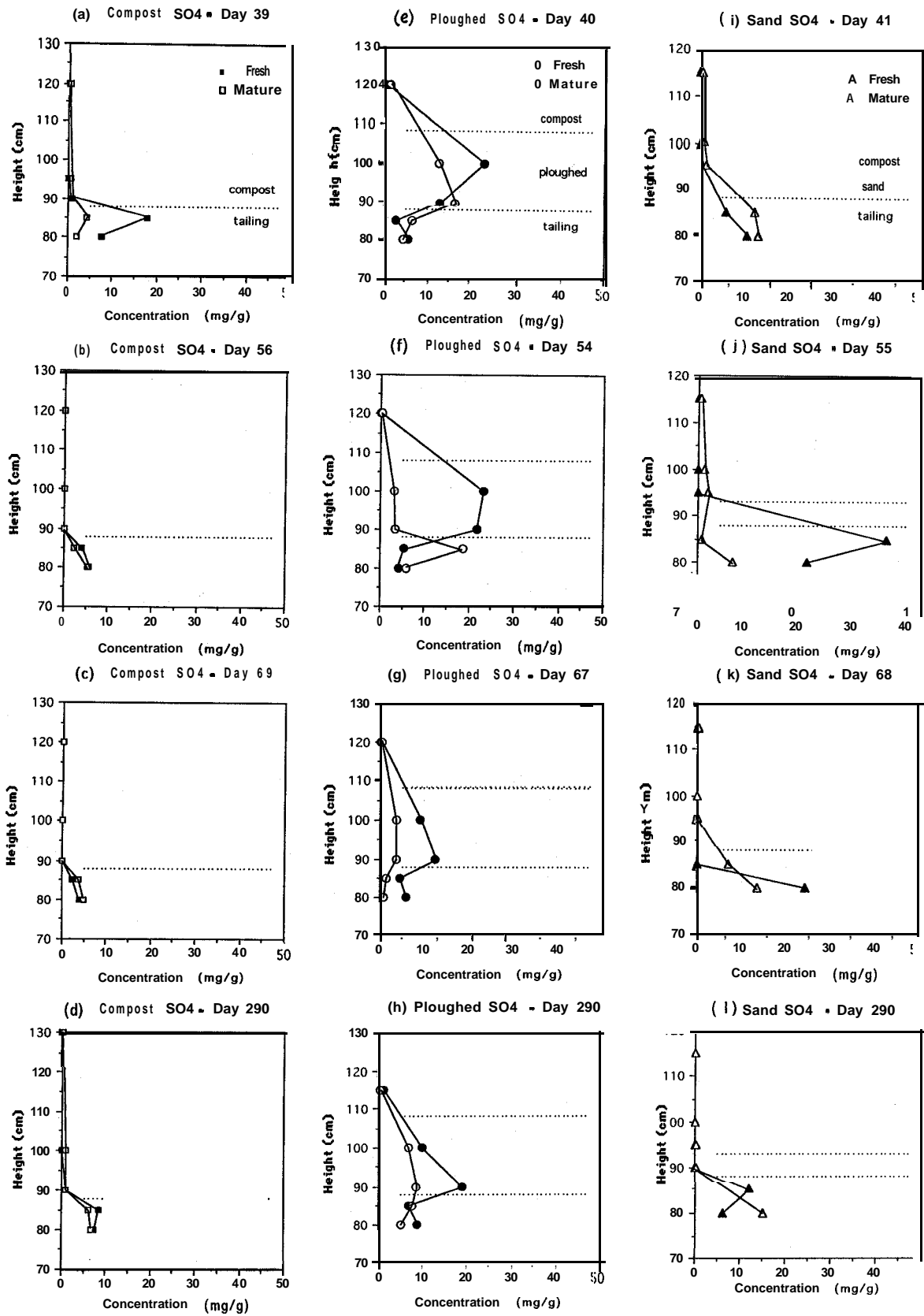
Figure 2 1. Profiles of sulphate (S04) in the Compost, Ploughed and Sand models during the simulation. (Fresh compost ▪ solid symbols, mature compost ▫ open symbols. Tailing layer O-88 cm, ploughed layer 88-108 cm, sand layer 88-93 cm).

(a) Cover NO3-Eh

(b) Tailing NO3-Eh
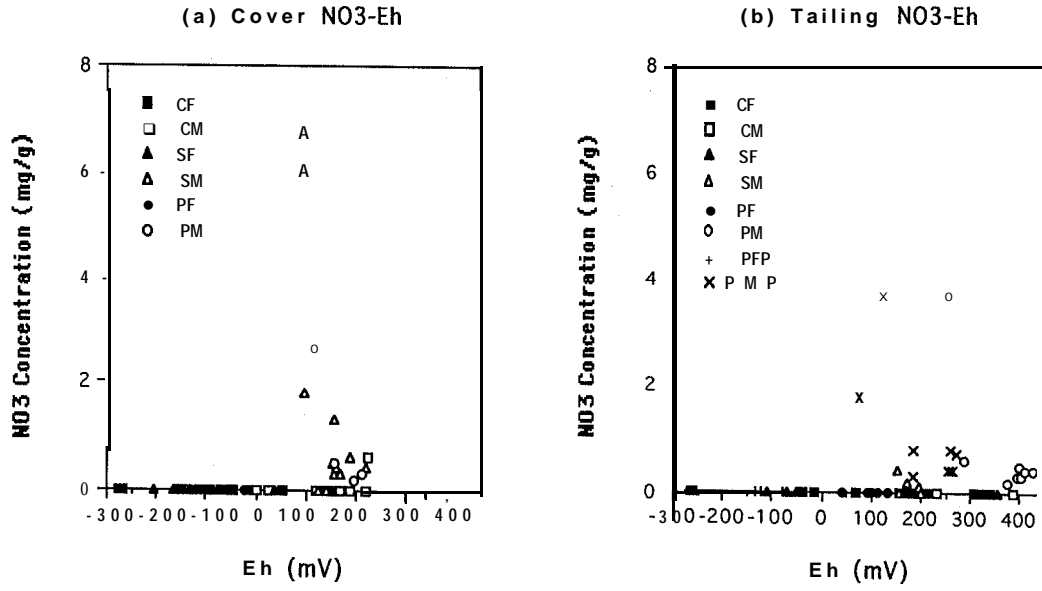
Figure 22. Nitrate concentration (N03) as a function of relative redox potential (Eh) for all samples of the simulation experiment. (Fresh compost ▪ solid symbols, Mature compost ▪ open symbols, Compost cover model ▪ squares, Ploughed cover model ▪ diamonds, Sand cover model ▪ triangles).

(a) Cover SO4-Eh

(b) Tailing SO4-Eh
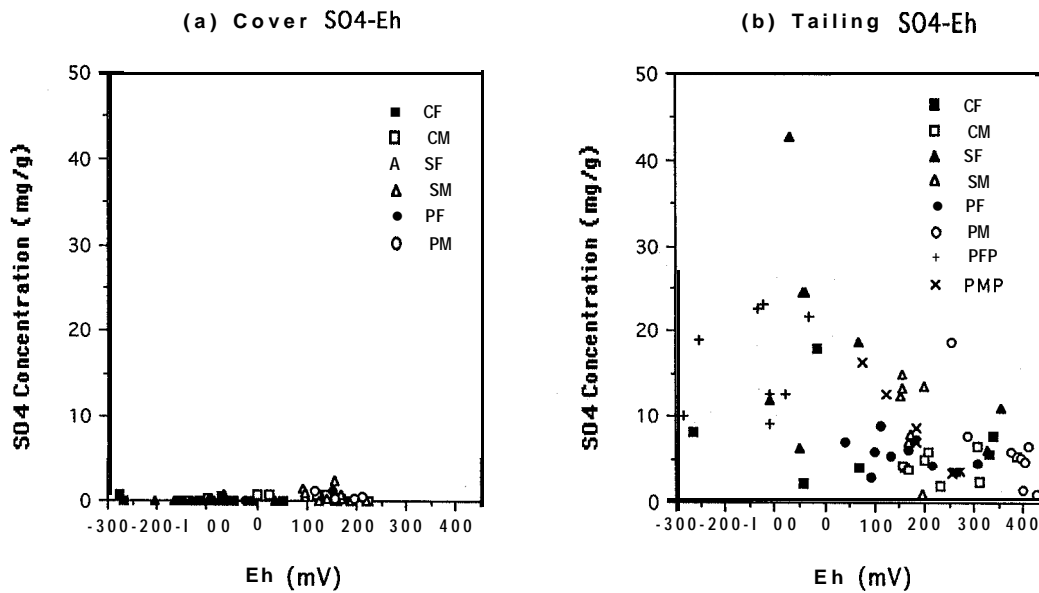
Figure 23. Sulphate concentration (S04) as a function of relative redox potential (Eh) for all samples of the simulation experiment. (Fresh compost ▪ solid symbols, Mature compost ▪ open symbols, Compost cover model ▪ squares, Ploughed cover model ▪ diamonds, Sand cover model ▪ triangles).

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(a) Compost Fresh ▪ Relative pH

(b) Compost Mature ▪ Relative pH

(c) Ploughed Fresh ▪ Relative pH

(d) Ploughed Mature ▪ Relative pH

(e) Sand Fresh ▪ Relative pH

(f) Sand Mature ▪ Relative pH
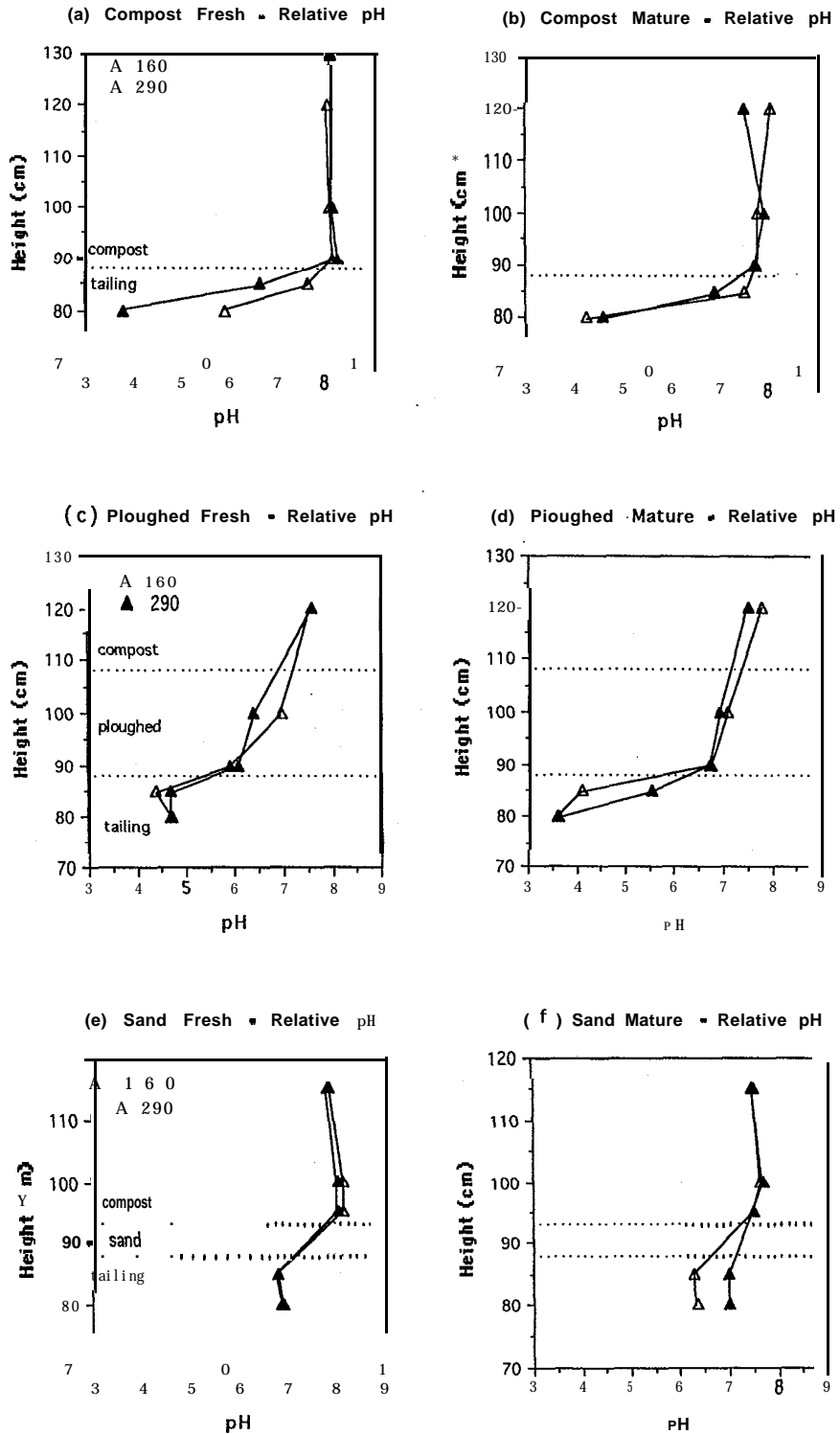
Figure 24. Profiles of relative acidity (pH) in the Compost, Ploughed and Sand model columns during days 160 and 290 of the simulation experiment. (Fresh compost - solid symbols, mature compost - open symbols. Tailing layer O-88 cm, ploughed layer 88-108 cm, sand layer 88-93 cm).

(a) Tailing pH-Eh ▪ Day 160

(b) Tailing pH-Eh ▪ Day 290
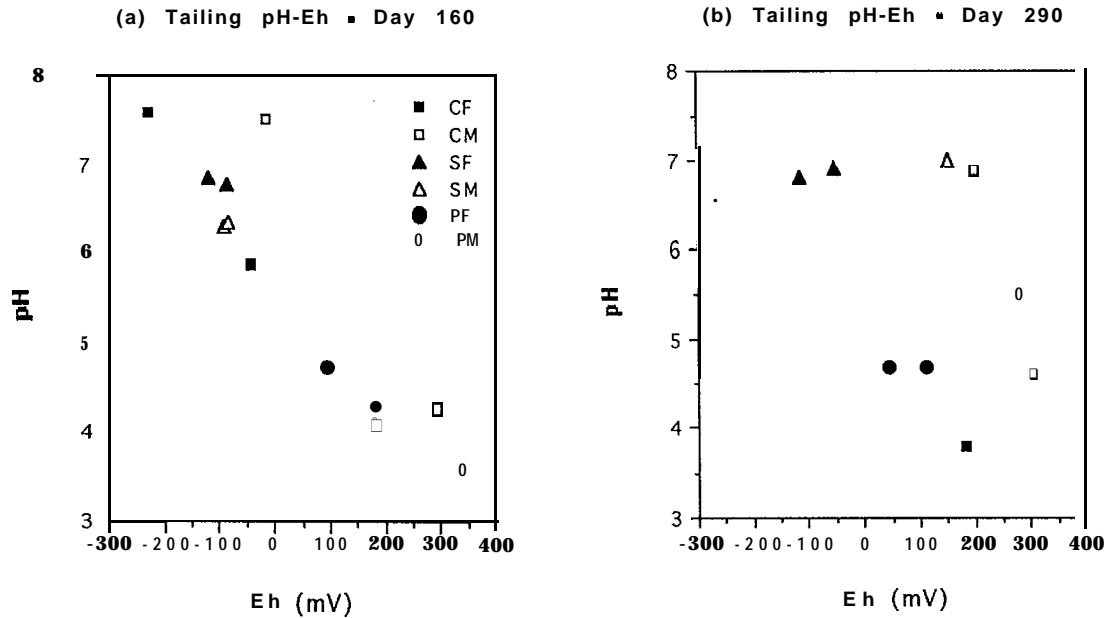
Figure 25.   Relationship between acidity (pH) and relative redox potential (Eh) for ail tailing samples on day 160 and 290 of the simulation experiment. (Fresh compost ▪ solid symbols, Mature compost ▪ open symbols, Compost cover model · squares, Ploughed cover model · circles, Sand cover model · triangles).

4.4.6 Trace Metal Analyses

a) Mobility of Trace Metals

A complete set of analyses for trace metals, Eh, and pH was done on days 160 and 290 of the simulation in order to assess the progress of AMD reversal under the expected increase in pH and the development of reducing conditions. No trends in trace metal mobility were evident between sampling days 160 and 290, so these data are combined as a single data set for our analyses (Figures 26 & 27). (Unfortunately, due to a cleanup mistake in the lab, no other samples from the simulation run were retained for trace metal ICP analysis or relative pH measurement.)

We examined the concentration of mobilized trace metals in relation to the relative E_h (Figure 26) and relative pH (Figure 27) of the sample solution. Fe and other trace metals in tailing samples were most mobile under Fresh compost cover layers at medium to low relative E_h (Figure 26acegik). Fe, Mn and Co concentrations were also high in the sample solutions from the Ploughed Fresh treatment at medium to low E_h (Figure 26aik), and at pH 4 to 6   (Figure

59
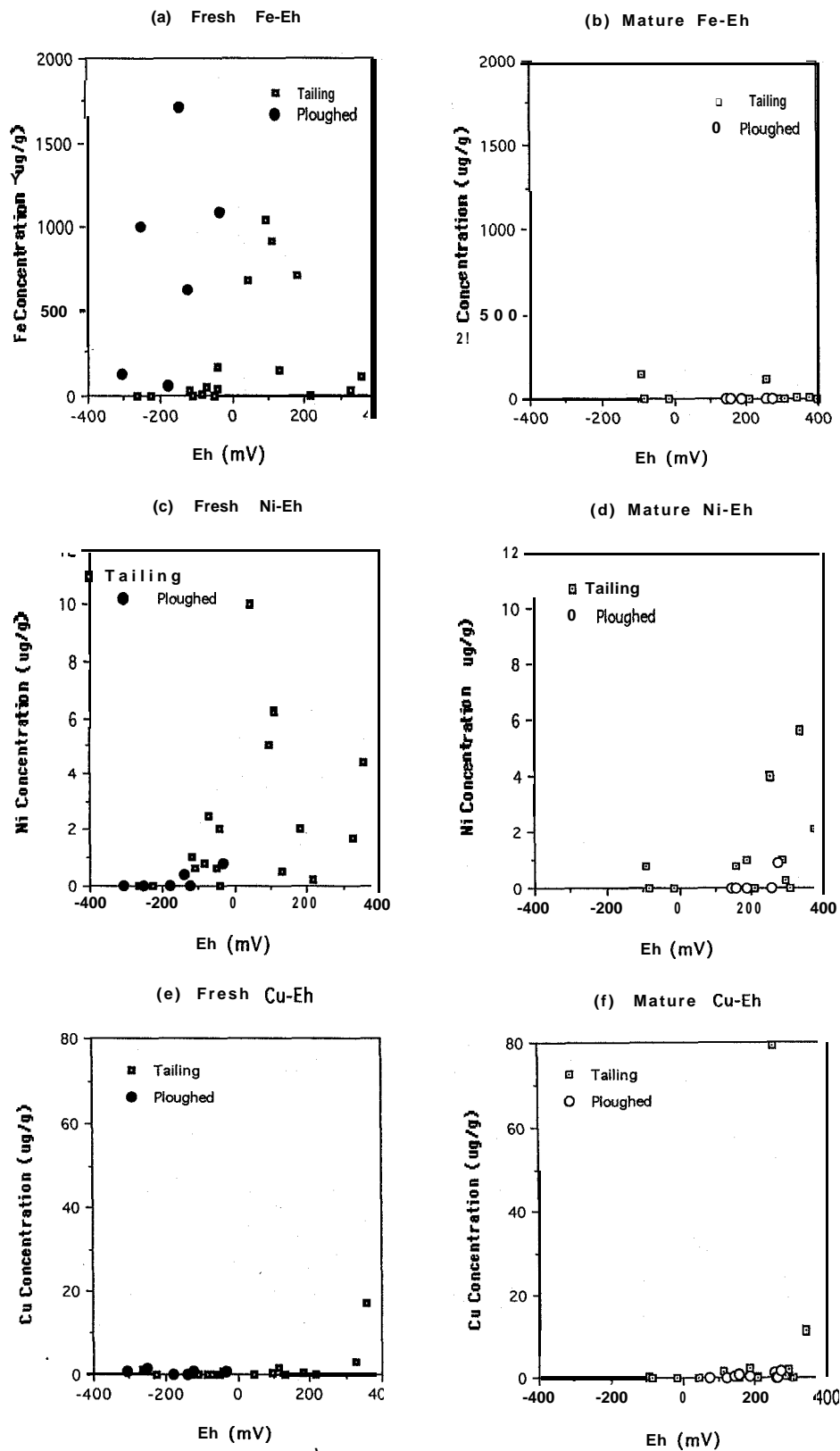
Figure 26. Relationship between relative redox potential (Eh) and the concentrations of the trace metals iron (Fe), nickel (Ni) and copper (Cu) for all tailing layer and ploughed layer samples on days 160 and 290 of the simulation experiment. (Tailing layer = squares, Ploughed layer = circles, Fresh compost = solid symbols, Mature compost = open symbols).
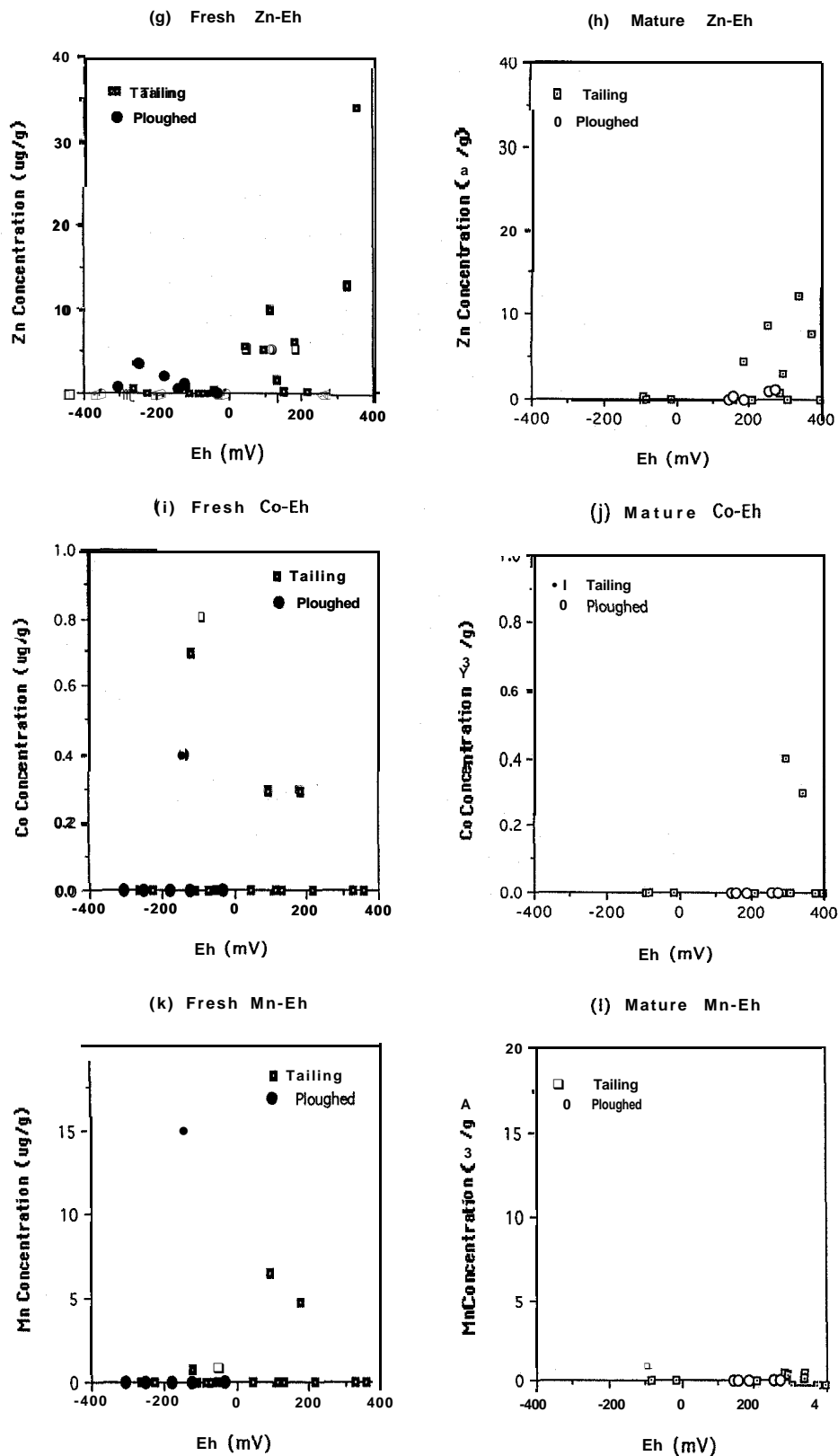
Figure 26 (continued). Relationship between relative redox potential (Eh) and the concentrations of the trace metals zinc (Zn), cobalt (Co) and manganese (Mn) for all tailing layer and ploughed layer samples on days 160 and 290 of the simulation experiment. (Tailing layer - squares, Ploughed layer - circles, Fresh compost - solid symbols, Mature compost - open symbols).
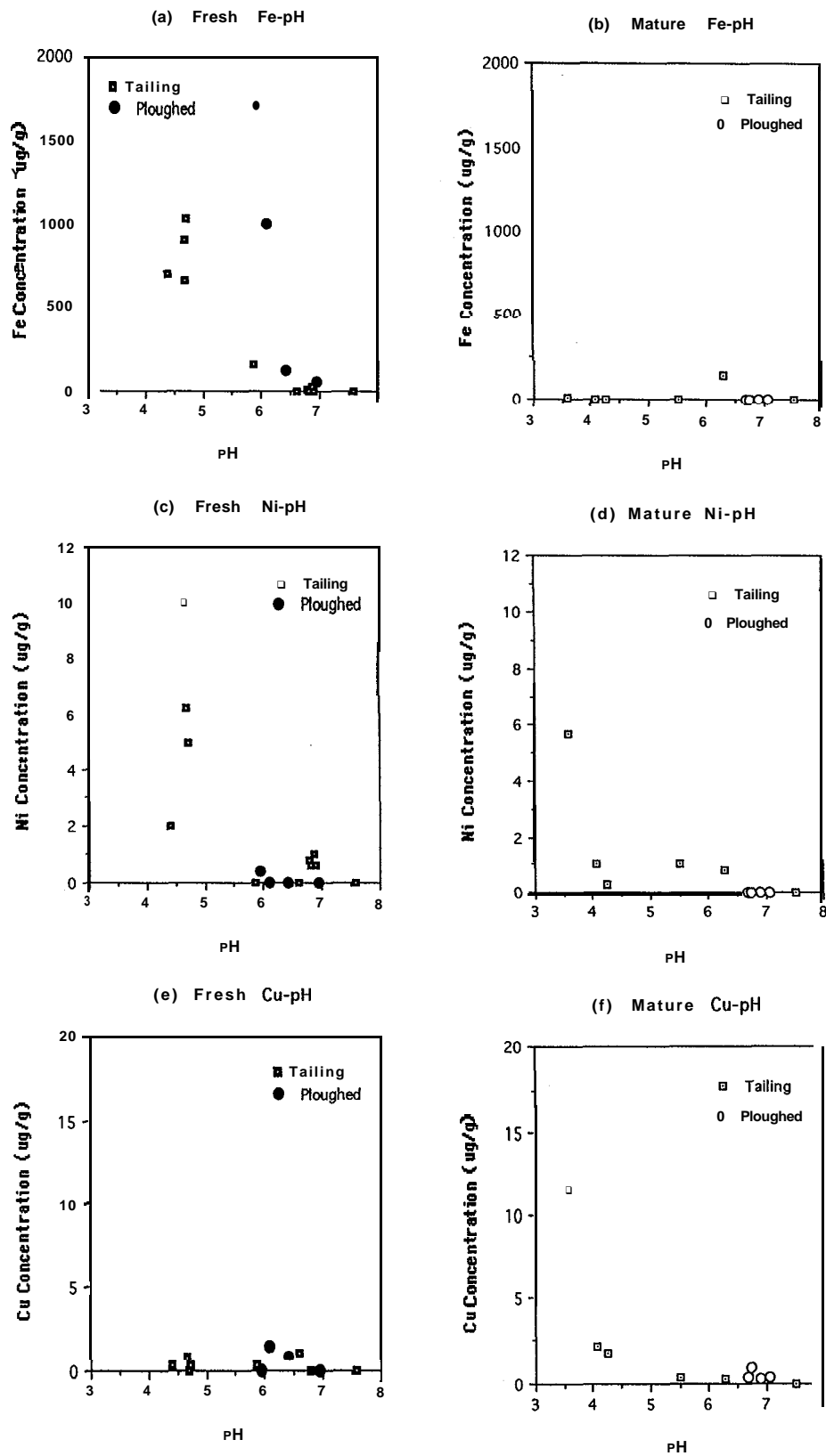
Figure 27. Relationship between relative acidity (pH) and the concentrations of the trace metals iron (Fe), nickel (Ni) and copper (Cu) for all tailing layer and ploughed layer samples on days 160 and 290 of the simulation experiment. (Tailing layer - squares, Ploughed layer - circles, Fresh compost - solid symbols, Mature compost - open symbols).

(g) Fresh Zn-pH

(h) Mature Zn-pH

(i) Fresh Co-pH

(j) Mature Co-pH

(k) Fresh Mn-pH

(l) Mature Mn-pH

Figure 27 (continued). Relationship between relative acidity (pH) and the concentrations of the trace metals zinc (Zn), cobalt (Co) and manganese (Mn) for all tailing layer and ploughed layer samples on days 160 and 290 of the simulation experiment. (Tailing layer - squares, Ploughed layer - circles, Fresh compost - solid symbols, Mature compost - open symbols).
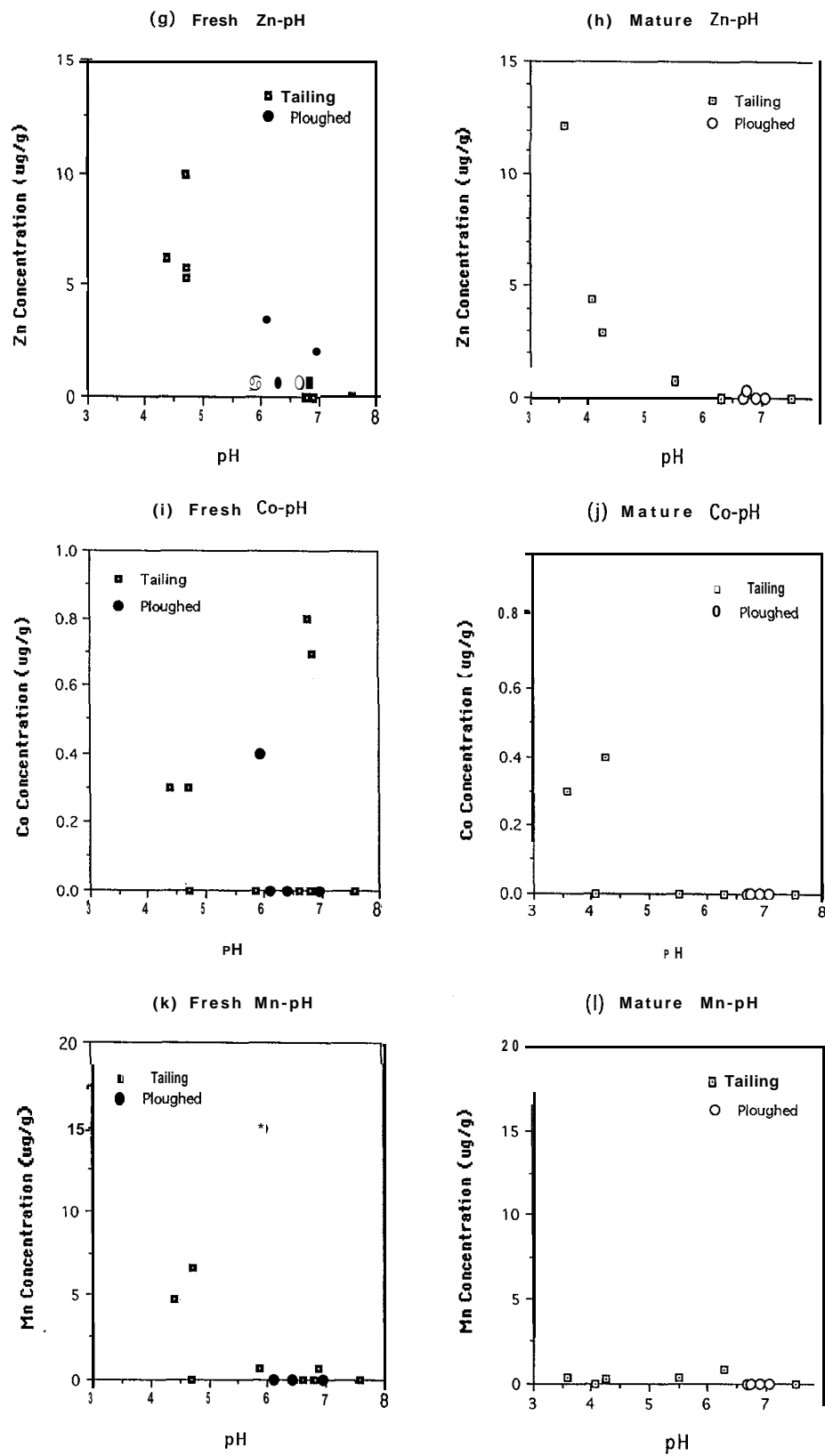
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(Figure 27aik). The highest Fe concentration was found in the 90 cm sample of the ploughed layer (50% tailing-50% compost), but curiously, the 100 cm ploughed layer showed only 3% as much iron in solution. The amount of mobilized Fe in the Ploughed Fresh treatment was one to two orders of magnitude greater than in the Compost and Sand models. The same general pattern of higher concentrations at low E_h was shown by sulphate for the ploughed layer samples (see Figure 23b). The mobilities of Ni, Cu, Zn and Co were very low at low E_h (Figures 26cdefghij) and high pH (Figures 27cdefghij).

We conclude from these patterns that decomposition products in the fresh compost were fuelling a high rate of sulphate and iron reduction by sulphate- and iron-reducing bacteria. Since the oxidized tailing are rich in iron, the reductive processes will reflect a dominante by Fe(III) reduction until the ion is nearly all gone from solution (Rowell 1988). In an environment with limited organic substrates, sulphate-reducing bacteria are often competitively inhibited by the activity of iron-reducing bacteria (Hedin et al. 1989). We think that the close contact between tailing oxides and organic molecules in the ploughed layer enhanced the rate of reductive dissolution of the oxides, causing the release of iron and sulphate into solution, even at low E_h and high pH. The formation of iron sulphide precipitate seen in the region of the tailing-compost interface (see Section 4.4.1 b) also indicates that reductive dissolution of iron oxides and sulphate compounds was proceeding from approximately day 40 of the simulation experiment in the Fresh compost treatments.

Trace metals other than iron were found only in low concentration in the six cover layer treatments and were mobile predominantly under acidic conditions and at high redox potentials. The magnitudes of these relative pH and relative E_h measurements cannot be directly applied to the in situ conditions in the column, but it seems clear that, as reducing conditions become stronger and pH rises under a compost cover layer, we expect a further decrease in the concentrations of these trace metals in the tailing pore water as reductive dissolution shifts to reductive precipitation as sulphides and complexed with organic compounds.

b) Trace Metal Sequential Extraction Analyses

Sequential analyses were done to understand how the trace metals were distributed between the various compounds in the tailing and how the distribution had changed during the cover simulation experiment. Each major trace metal that was detected in the tailing was analyzed from: (1) an original oxidized sample, (2) an original unoxidized sample, and (3) for the tailing samples of each treatment on day 290, the end of the simulation experîment. The results

of ICP analyses of the sequential extraction fractions of oxidized tailing are presented in Table 6. The interpretation of the solubilized fraction at each step of the sequential extraction may be summarized as:

Extracted Fraction

	Step 1	elements weakly bound and highly exchangeable (mobile)
decreasing	Step 2	elements bound to carbonates
mobility	Step 3	elements bound to iron-manganese oxides
	Step 4	elements bound to organic matter and sulphides
	Step 5	residual fraction with elements non-exchangeable (immobile)

In general, Table 6 shows that lower concentrations of most trace metals were found in the oxidized compared to the unoxidized tailing. Exceptions to this pattern were oxides of Fe, Mn, Ni and Cu (Table 6abcd, fraction 3). This pattern is consistent with the fact that the oxidized tailing samples have been weathered for several decades at the Nickel Rim site. As expected, the oxidized tailing contained a higher proportion of trace metals as oxides (fraction 3) and a smaller proportion as sulphides (fraction 4), in contrast to the unoxidized tailing. In oxidized tailing under oxidizing conditions, iron and other trace metals in sulphides are mobilized by reaction with ferric sulphate and sulphuric acid. Only a small portion of the trace metals were weakly bound (fraction 1) or associated with carbonates (fraction 2) in both the unoxidized and oxidized tailing.

Fraction 3 contained high concentrations of Fe, Mn and Ni that are typically associated with amorphous or recently-formed oxyhydroxides (Table 6abc). These elements will be released from the solid phase under reducing conditions since iron hydroxides can be dissolved and thus release Fe(II) and trace metals adsorbed on them. A significant portion of Fe, Cu and Zn came off in fraction 4, indicating an association probably with sulphide solids and possibly with organic matter (Table 6adf). Fe, Mn, Cu and Zn showed the largest concentration in fraction 5, the residual component (Table 6abdf), and thus these metals would not be expected to mobilize under oxidizing or reducing conditions. Other metals, such as Cr, Co and Pb, were extracted only in very low concentrations and are significant only in the residual fraction (Table 6gh).

The sequential extraction results for the day 290 samples of the simulation are more difficult to interpret. Comparisons of these treated samples to the oxidized and unoxidized tailing samples suggests that some chemical changes have taken place in the trace metal distribution in the experimental oxidized tailing, especially under the Fresh compost cover treatments.

Table 6(a). Sequential extraction analyses of iron (Fe) from samples of original oxidized, unoxidized tailing, and the tailing layers on day 290 of the simulation experiment. All concentrations are $\mu g/g$, except Fractions 3 and 5 are g/100g (%).

Sample	Eh mV	Fraction 1 weakly bound	Fraction 2 carbonates	Fraction 3 Fe-Mn oxides	Fraction 4 sulphides, organic	Fraction 5 residual
Oxidized	na		?	1.6	1000	5.8
Non-oxidized	na	450	8	1.12	48700	9.5
CM 85	206	90.7	32.3	3.72	64.6	6.46
CM 80	306	45.8	27.2	3.57	121.3	6.93
CF 85	-263	56.1	38.7	1.87	102.3	7.49
CF 80	182	322.6	29.8	2.09	22.3	6.95
PM 85	286	298	39.7	2.65	5	5.33
PM 80	394	419	39.4	2.13	34.5	6.16
PF 85	43	49.7	33.6	1.8	74.7	5.73
PF 80	113	24.7	23.4	2.56	81.4	6.42
SM 85	206		35.9	1.96	24.7	4.96
SM 80	156		31.3	2.13	12.5	5.25
SF 85	-111		51.4	1.63	5	6.76
SF 80	-51		40	1.38	92.5	4.5

Table 6(b). Sequential extraction analyses of manganese (Mn) from samples of original oxidized, unoxidized tailing, and the tailing layers on day 290 of the simulation experiment. All concentrations are $\mu g/g$.

Sample	Eh mV	Fraction 1 weakly bound	Fraction 2 carbonates	Fraction 3 Fe-Mn oxides	Fraction 4 sulphides, organic	Fraction 5 residual
Oxidized	na	-	?	139	65	1060
Non-oxidized	na	5	-	17	12	265
CM 85	206	5	-	16.2	-	124.3
CM 80	306	2.5	-	13.6	-	153.5
CF 85	-263	2.5	8.7	11.2	-	134.7
CF 80	182	2.5	9.9	13.7	-	141.4
PM 85	286	2.5	-	9.9	-	114.2
PM 80	394	2.5	-	9.8	-	147.9
PF 85	43	3.7	-	12.4	-	102
PF 80	113	3.7	-	6.2	-	101.1
SM 85	206	2.5	7.4	40.8	-	86.6
SM 80	156	2.5	3.8	46.3	-	97.6
SF 85	-111	3.8	-	25.1	-	122.8
SF 80	-51	5	-	43.8	-	87.6

Table 6(c). Sequential extraction analyses of nickel (Ni) from samples of original oxidized, unoxidized tailing, and the tailing layers on day 290 of the simulation experiment. All concentrations are μg/g.

Sample	Eh mV	Fraction 1 weakly bound	Fraction 2 carbonates	Fraction 3 Fe-Mn oxides	Fraction 4 sulphides, organic	Fraction 5 residual
Oxidized	na	6	?	155	24	134
lon-oxidized	na	300	21	55	3715	1000
CM 85	206	2.5	-	19.9		94.4
CM 80	306	2.5	-	27.2		104
CF 85	-263	5	-	23.7	22.5	104.8
CF 80	182	7.4	-	32.3	39.7	99.3
PM 85	286	3.7	-	23.6		84.4
PM 80	394	2.5	-	21		86.3
PF 85	43	14.9	21.2	38.6	14.9	87.1
PF 80	113	7.4	2.5	17.3	14.8	96.2
SM 85	206	2.5	71.8	383.7	9.9	118.8
SM 80	156	3.8	80	412.7	7.5	127.6
SF 85	-111	5	109	338.2	15	137.8
SF 80	-51	11.3	97.5	987.5	22.5	240

Table 6(d). Sequential extraction analyses of copper (Cu) from samples of original oxidized, unoxidized tailing, and the tailing layers on day 290 of the simulation experiment. All concentrations are μg/g.

Sample	Eh mV	Fraction 1 weakly bound	Fraction 2 carbonates	Fraction 3 Fe-Mn oxides	Fraction 4 sulphides, organic	Fraction 5 residual
Oxidized	na	8	?	29	48	110
Non-oxidized	na	182	-	10	410	585
CM 85	206	65.9	-	51.2	72.1	59.6
CM 80	306	50.8	-	54.1	91.6	64.4
CF 85	-263	33.7	-	32.8	89.8	62.4
CF 80	182	59.5	-	34.1	111.7	62
PM 85	286	16.1	-	48.5	69.5	49.6
PM 80	394	34.5	-	37.8	78.9	54.2
PF 85	43	31.1	-	37.6	67.2	47.3
PF 80	113	8.6	-	52.9	78.9	61.6
SM 85	206	-	-	71	49.5	39.6
SM 80	156	-	-	74.9	55	45
SF 85	-111	-	-	94.8	105.2	52.6
SF 80	-51	-	-	94.3	77.5	50

Table 6(e). Sequential extraction analyses of cadmium (Cd) from samples of original oxidized, unoxidized tailing, and the tailing layers on day 290 of the simulation experiment. All concentrations are μg/g.

Sample	Eh mV	Fraction 1 weakly bound	Fraction 2 carbonates	Fraction 3 Fe-Mn oxides	Fraction 4 sulphides, organic	Fraction 5 residual
Oxidized	na	-	?	5	-	-
Non-oxidized	na	-	-	5	5	12
CM 85	206	-	-	-	-	5
CM 80	306	-	-	6.2	-	5
CF 85	-263	-	-	2.5	-	5
CF 80	182	-	-	12.4	-	5
PM 85	286	-	-	3.7	-	5
PM 80	394	-	-	-	-	7.4
PF 85	43			2.5		2.5
PF 80	113			2.5		4.9
SM 85	206		19.8			2.5
SM 80	156		7.5			2.5
SF 85	-111			2.5		2.5
SF 80	-51			2.5	-	4.9

Table 6(f). Sequential extraction analyses of zinc (Zn) from samples of original oxidized, unoxidized tailing, and the tailing layers on day 290 of the simulation experiment. All concentrations are μg/g.

Sample	Eh mV	Fraction 1 weakly bound	Fraction 2 carbonates	Fraction 3 Fe-Mn oxides	Fraction 4 sulphides, organic	Fraction 5 residual
Oxidized	na	-	?	24	74	435
Non-oxidized	na	8	14	14	42	15000
CM 85	206	19.9	-	29.3	-	27.3
CM 80	306	13.6	-	21	-	27.2
CF 85	-263	18.7	-	16.5	-	25
CF 80	182	19.9	-	38.7	-	29.8
PM 85	286	2.5	-	14.1	-	17.4
PM 80	394	9.9	-	20.6	-	24.6
PF 85	43	8.7	-	12.9	-	22.4
PF 80	113	7.4	-	14.2	-	73.9
SM 85	206	-	5	40	-	37.1
SM 80	156	3.8	8.8	42.3	-	27.5
SF 85	-111	-	7.5	31.8	-	30.1
SF 80	-51	3.8	23.8	42.5	-	35

Table 6(g). Sequential extraction analyses of chromium (Cr) from samples of original oxidized, unoxidized tailing, and the tailing layers on day 290 of the simulation experiment. All concentrations are μg/g.

Sample	Eh mV	Fraction 1 weakly bound	Fraction 2 carbonates	Fraction 3 Fe-Mn oxides	Fraction 4 sulphides, organic	Fraction 5 residual
Oxidized	na	-	?	9	6	114
Non-oxidized	na	-	-	24	-	200
CM 85	206	-	-	27.3	9.9	101.9
CM 80	306	-	-	33.4	7.4	128.8
CF 85	-263	-	-	26.2	-	112.3
CF 80	182	-	-	21.1	12.4	124.1
PM 85	286	-	-	24.8	5	89.4
PM 80	394	-	-	19.7	9.9	118.3
PF 85	43	-	-	24.9	5	74.7
PF 80	113	-	-	24.7	5	78.9
SM 85	206	-	-	19.8	-	44.6
SM 80	156	-	-	16.3	-	60
SF 85	-111	-	-	18.8	-	90.2
SF 80	-51	-	-	11.2	-	47.5

Table 6(h). Sequential extraction analyses of cobalt (Co) from samples of original oxidized, unoxidized tailing, and the tailing layers on day 290 of the simulation experiment. All concentrations are μg/g.

Sample	Eh mV	Fraction 1 weakly bound	Fraction 2 carbonates	Fraction 3 Fe-Mn oxides	Fraction 4 sulphides, organic	Fraction 5 residual
Oxidized	na	-	?	5	-	6
Non-oxidized	na	15	-	-	43	28
CM 85	206	-	-	-	-	7.5
CM 80	306	-	-	-	-	7.4
CF 85	-263	-	-	-	-	7.5
CF 80	182	-	-	-	-	7.4
PM 85	286	-	-	-	-	7.5
PM 80	394	-	-	-	-	7.4
PF 85	43	-	-	-	-	7.5
PF 80	113	-	-	-	-	7.4
SM 85	206	-	-	9.9	-	9.9
SM 80	156	-	-	11.3	-	7.5
SF 85	-111	-	-	7.5	-	5
SF 80	-51	-	3.8	20	-	7.5

For iron (Table 6a), the Fe oxide concentrations were higher, while the sulphide/organic Fe concentrations were lower by an order of magnitude, than in the original oxidized sample. This may be explained as an experimental artifact of the different handling of the original sample and day 290 tailing samples. Original oxidized and unoxidized tailing were stored wet in sealed plastic containers so the rate of oxidation by atmospheric oxygen would have been slow. Some Fe sulphide in the oxidized sample still had not been oxidized and was detected by the first sequential extraction analysis (Table 6a). In contrast, the day 290 samples were small and not protected from atmospheric oxygen that would have oxidized any sulphides that had formed during the simulation experiment. We know from visual observation that a significant amount of black iron sulphide precipitate was being formed in many of the experimental columns (see Section 4.4.1, Photo 4), but our sampling method did not allow us to measure this Fe sulphide in the sequential extraction analysis of day 290 samples. An anoxic sample handling procedure would have been necessary to preserve the Fe sulphide precipitate from oxidation for more than a few minutes.

There was a substantial amount of weakly bound Fe in the Compost and Ploughed treatments, but none in the Sand treatment where the tailing does not corne into direct contact with the compost cover layer or its leachates. Therefore, the weakly bound Fe may have become associated with organic molecules originating in the compost layer. The same pattern of weakly bound trace elements in the Compost and Ploughed but not in the Sand treatments was also seen for Cu and Zn (Table 6df). The partitioning of Fe between oxides (fraction 3) and sulphides/organics (fraction 4) did not show a clear difference between cover treatments, although there is some indication that Fe oxides became less concentrated under the Fresh compost treatments, while Fe sulphides/organics were more concentrated.

This interpretation would be consistent with the hypothesis of greater reductive dissolution of oxides under the Fresh compost cover layer which created stronger reducing conditions. There would be a corresponding increase in metal sulphide concentrations as Fe(II) and other metals precipitate under reducing conditions, but the evidence for this process may be only weak for sulphides of Ni and Cu on day 290 (Table 6cd). The especially high concentrations of Ni oxides in the Sand model treatments, compared to oxidized tailing, are also difficult to explain in the light of this suggestion. Unfortunately, there is a great deal of variability evident in the data, and the lack of treatment and sample replication in the experiment makes it impossible to do an analysis of statistical significance of the results.

5. CONCLUSIONS AND RECOMMENDATIONS

The experimental work has shown that a cover layer of MSW compost can be of benefit in the reclamation of oxidized tailing. However, the conclusions that we reached were based on a relatively short-term laboratory experiment that lacked natural environmental conditions and variability. This study should be seen as a preliminary look at compost cover layers and their design as a tailing AMD solution. Comprehensive, long-term field work or large lysimeter pilot studies should now be done to follow up on the potential benefits that were identified, and also those benefits that may have been insufficiently assessed by the current study.

The Compost and the Sand cover layer models appeared to be substantially better than the Ploughed model as a cover layer design for suppressing tailing AMD. Compaction of the compost cover layer appeared to be of little benefit. Despite the substantial vertical pressure applied to the Ploughed and Sand model compost layers, there was not a great deal of compaction observed and the compost layers did not significantly change their physical characteristics. Although there was little reduction in porosity and no large increase water saturation, oxygen concentrations were greatly depressed below atmospheric levels in both the compost layer and the tailing layer of all three cover layer models.

The major contrast in simulation results was between treatments that used fresh (4 week old) compost and treatments that used mature (14 week old) compost. Fresh compost treatments showed much greater water contents, much lower hydraulic conductivity, and lower redox potentials in the compost-tailing system than did the mature compost treatments. The early formation of a black iron sulphide precipitate in the region of the fresh compost-tailing interface indicated that anoxia was present that lead to the development of strong reducing conditions. Profiles of Eh, methane and carbon dioxide showed that low redox potentials and higher gas concentrations developed in the tailing layers at a later time than in the compost layers. Nitrate, which is present in the mature compost but not in the fresh compost, decreased in concentration through the simulation run, presumably as a result of nitrate reduction in the reducing conditions. Sulphate appeared to be mobile even at low redox potentials, but this may be a transient condition as the large amount of sulphate in the tailing would take some time to become reduced.

Chemical analyses of the Nickel Rim oxidized tailing revealed that significant amount of trace metals were present and may become mobilized under reducing conditions. A large portion of metals were retained in highly bound form in the oxidized tailing. Sampling done on day 160 and day 290 of the simulation run showed that trace metals in solution were present in low

concentration. Iron showed high concentrations with the same pattern as sulphate. The greatest suiphate levels occurred in the compost-tailing mixed layer of the Ploughed model under a fresh compost cover layer. Organic compounds from the decomposition of fresh compost were likely serving as substrates for both the reductive dissolution of iron oxides by iron-reducing bacteria, and the activity of sulphate-reducing bacteria.

Compost background and leachate quality were examined in samples of mature and fresh compost. If taken by themselves, the concentrations of nitrogen compounds, manganese and iron might be of concern, however, we suggest that the quality aspects of a compost cover layer must be considered within the context of the compost cover layer-tailing system. The development of reducing conditions appears to cause the reduction or denitrification of compost nitrates. In a cover layer of fresh compost, the hydraulic conductivity would be extremely low, and this property would seal off the compost layer and tailing from the infiltration of precipitation and surface water flow. Therefore, while any nitrogen compounds remained they would not be quickly leached from the compost layer and the acidity and trace metals in the tailing would also not be transported through the tailing by water flow from the cover layer.

Conclusions

From the results of this preliminary study, we have concluded:

- There is strong evidence of a reversal of AMD processes in the oxidized tailing as shown by the formation of iron sulphide precipitate near the cover layer-tailing interface. The mobilities and distribution of trace metals indicate that reductive dissolution of tailing oxides was still in progress in the tailing layer at the end of the 290 day simulation;

- The mobilization of iron and sulphate seen was enhanced by the establishment of strong reducing conditions and the availability of organic substrates for the reductive dissolution of iron oxides by reducing bacteria. This may be a transient condition until iron and sulphur become immobilized as less soluble precipitates, such as sulphides. Trace metals became less mobilized with the development of strong reducing conditions and low acidity;

- The Compost and Sand cover layer models were more effective at maintaining anoxic or other ameliorative conditions in the tailing than was the Ploughed model during the nine month simulation;

- Fresh compost treatments are much more effective than mature compost in maintaining high water content and strong reducing conditions at the compost-tailing interface, and this pattern was also shown by the formation of iron sulphide precipitate and methane;

- Fresh compost cover layers showed a great resistance to the conduction of water that, along with high water content, would seal off the tailing from infiltration of atmospheric oxygen, precipitation and surface water, thus forming a physical oxygen barrier;

- Compost quality tests showed that leachates from mature and fresh compost present a low environmental risk for use on mine lands. The cover layer simulation results indicates that in the compost-tailing system nitrates and metals will be quickly reduced. Additionally, the very low hydraulic conductivity of the fresh compost cover layer will inhibit vertical water flow through the compost layer, and thus reduce the leaching of nitrogen compounds, acids, and metals from the compost-tailing system.

Recommendations

- The simplest, least expensive and thus most beneficial cover layer model to implement is the Compost model, using fresh compost for the cover layer. This model should be tested under field conditions or in a lysimeter pilot study to determine if it beneficial characteristics will be maintained or perhaps can be improved over the long term.

- Long term compaction and decomposition of fresh compost must be examined to see if its favourable hydraulic characteristics and anaerobic conditions will be maintained under field conditions. In particular, the field situation would provide natural wetting-drying cycles, freeze-thaw cycles, and a seasonal variability in the temperature regime.

- The relative importance of the physical oxygen barrier and biological oxygen consumption benefits of the fresh compost needs to be determined, since decomposition of the compost layer will eventually cease.

- The progress of reductive dissolution and precipitation of Fe(II) and sulphur must be monitored to assess if the anaerobic conditions will eventually lead to the immobilization of iron, sulphate and trace metals.

- Leachate and runoff quality from the compost cover layer should be monitored, especially for nitrogen compounds, although water quality problems are not anticipated.

6. SUMMARY

Three compost cover layer models were examined that might be beneficial in the suppression of tailing oxidation and acidic mine drainage. Laboratory studies were done to assess the potential compost cover layer benefits, and to facilitate the design of field tests. Two grades of compost were compared: immature (4 week old) and mature (14 week old) municipal compost. Analyses of the leachate quality from compost cover layers and tailing were done since MSW compost may release trace metals, organic chemicals and pathogens into the watershed outside the tailing area. A laboratory simulation experiment to compare the three cover layer models and two grades of compost was conducted in plexiglass columns over 290 days.

The Compost and the Sand cover layer models appeared to be substantially better than the Ploughed model as a cover layer designs for suppressing tailing AMD. Oxygen concentrations were greatly depressed below atmospheric concentration in the compost and tailing layers in all three cover models. Substantial compaction pressure on the compost cover layer appeared to be of little benefit. Medium to strong reducing conditions developed, especially under an immature compost cover layer, and this enhanced the dissolution of oxides and increased the mobilization of iron, sulphate, and several trace metals. The formation of black iron sulphide precipitate and methane at the compost-tailing interface showed that AMD processes and acidity were being reversed under the immature compost cover. However, reductive dissolution of tailing oxides was still proceeding in the tailing layer at the end of the nine month simulation. This may be a transient condition until iron and sulphur become immobilized as less soluble precipitates such as sulphides.

Compost quality tests showed that leachates from mature and fresh MSW compost present a low environmental risk for use on mine lands. Furthermore, very low hydraulic conductivity of the fresh compost cover layer will inhibit vertical water flow through the compost layer, and thus reduce the leaching of acids, metals and nitrogen compounds from the compost-tailing system.

From this preliminary study it is concluded that the Compost model, using immature MSW compost, appears to be the simplest and most cost-effective tailing cover layer solution. Long term field tests or large lysimeter pilot experiments are needed to determine whether the beneficial characteristics of the compost cover layer can be maintained. Long term compaction and decomposition of fresh compost must be examined to see if its favourable hydraulic characteristics and anaerobic conditions will be maintained under natural conditions. In particular, the relative importance of the physical oxygen barrier and biological oxygen consumption benefits of different grades of MSW compost needs to be determined.

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APPENDICES:

Appendix A: Proposal for Laboratory Studies

Appendix B: Lakefield Research Ltd. Analytical Methods

Appendix C: Lakefield Research Ltd. Analytical Results

Appendix D: X-Ray Diffraction Analyses of Tailing

Appendix A: Proposai for Laboratory Studies

RECLAMATION OF SULPHIDE TAILINGS

USING MUNICIPAL SOLID WASTE COMPOST:

PROPOSAL FOR LABORATORY STUDIES

Centre in Mining and Mineral Exploration Research
Laurentian University
Sudbury, Ontario

A Proposal Submitted to:

Falconbridge Ltd.

October 2, 1992

1. INTRODUCTION

A literature review of what is known about the physical and chemical characteristics of municipal solid waste (MSW) compost and other organic materials (Pierce 1992), revealed that a compost layer on sulphide tailings could be beneficial in the suppression of tailings oxidation and acidic mine drainage, in five ways:

(1) *Physical* oxygen *barrier* - the compost would be saturated with water over at least part of its depth so that the limiting factor in oxygen diffusion would be the diffusivity of oxygen in water;

(2) *Oxygen-consuming barrier* - the continued decomposition of compost would create a large biological oxygen demand that acts as a sink for atmospheric oxygen or dissolved oxygen;

(3) *Chemical* inhibition - compounds and decomposition products in the MSW compost that leach into the tailings inhibit the growth and metabolism of sulphate-producing bacteria;

(4) *Chemical amelioration* - organic constituents in the MSW compost can cause the reductive dissolution of ferric oxides and prevent indirect ferric sulphide oxidation and acid generation;

(5) *Reduced* water infiltration - reduced hydraulic conductivity of compacted, decomposing compost may prevent infiltration of precipitation, thus decreasing tailings ground water flow.

Three compost cover layer models are proposed to produce and maintain these functions (Figure 1). Two models consist of a layer of compost that is compacted by an overburden layer, such as sand and gravel (Figure 1 a & 1 b). In model A, compost is ploughed into the upper layer of tailings before the compacted compost is placed, so that there is close chemical contact between the compost and the oxidized portion of the tailings. In model B, the bottom of the compost layer is separated from the tailings by another coarse layer to isolate it hydraulically from the tailings. The main purpose of the overburden layer is to keep the compost layer permanently compacted so that air-filled pore space is minimized and incoming precipitation can produce a high degree of saturation. The overburden will also be a protective layer against erosion, evaporation and runoff which could destroy the compost layer as an oxygen barrier. The third and most inexpensive compost model consists of a deep layer of compost placed directly on the tailings (Figure 1c). Revegetation requirements have not been considered in these models.

(a)	(b)	(c)
overburden	overburden	
compacted compost	compacted compost	compost
ploughed-in compost	coarse sand	
tailings	tailings	tailings
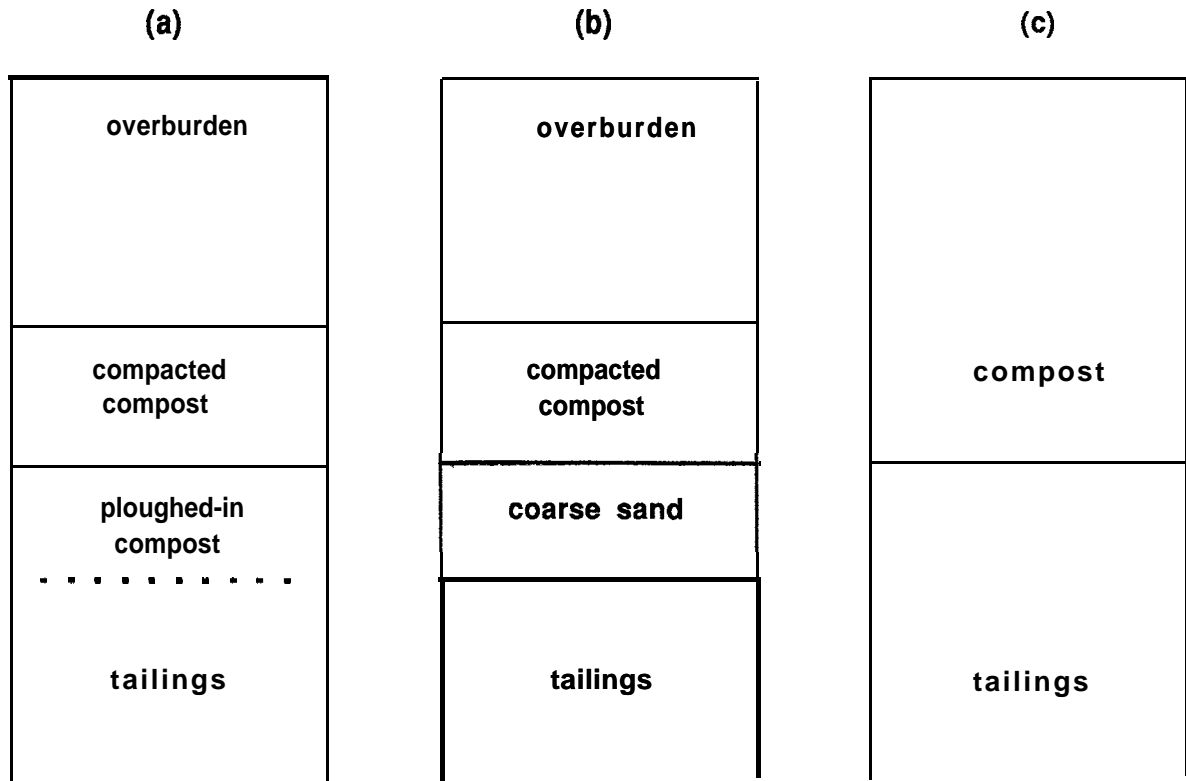
Figure 1. a) Ploughed compost cover model A; b) Sand compost cover model B; c) Compost cover model C.

Experimental investigations in the laboratory are required to assess whether the compost cover layer models will function as expected, in the field. Other waste materials, such as sawdust, can also be incorporated into the compost cover layer to investigate how they alter its physical and chemical properties. Analyses are also required of the leachates that corne out of the compost cover layers and tailings, since MSW compost may release heavy metals, organic chemicals and pathogens into the watershed outside the tailings area.

The risks of unacceptable levels of contaminants are largely a function of the quality of the compost feedstocks and the optimization of the composting process. For MSW compost, separation of organic waste from non-biodegradable garbage and hazardous chemicals is essential for producing uncontaminated, high-quality compost. Sewage sludge compost may also become a high-quality material if pathogens are killed and if the wastewater stream is uncontaminated by industrial effluents. Clean, uncured compost offers advantages over mature, agricultural-grade compost in tailings reclamation because of its high oxygen-consuming demand and the presence of a wide variety of organic compounds that could help in chemical amelioration of acidic mine drainage (AMD). Immature compost could also be diverted much earlier in the composting process and would be much cheaper for a municipality to produce.

Project Goal

The goal of this project is to obtain answers to technical questions about the optimal cover layer design and safety of using a cover of MSW compost as a permanent solution to tailings AMD. Sufficient laboratory work must be completed during this phase to allow the design of summer field studies at the Nickel Rim tailings site. Further laboratory work may be recommended to investigate the use of other organic wastes and to complement the field experiments.

Project Objectives

A. Physical Aspects and Optimization of Compost Cover Layers

> a) Study of the effects of various depths of overburden and compost on compost compaction and its physical characteristics, including bulk density, porosity, degree of saturation, hydraulic conductivity and compost moisture characteristic
>
> b) Measurement of oxygen concentrations within the compost-tailings column to determine the extent of anoxic conditions
>
> c) Determination of the water balance and pattern of saturation of the compost cover system for a multi-week simulation of wetting/drying cycles
>
> Principal Researcher - Glenn Pierce

B. Compost Leachate Composition from Compost of Varying Degrees of Maturity

> a) Determination of the degree of compost maturity
>
> b) Regulation 309 leaching test (Environmental Protection Act)
>
> c) Heavy metal and organic chemical spiking of compost to determine the degree of retention
>
> d) Simulated acid rain effects on leaching of toxic compounds
>
> Principal Researcher - Keith Winterhalder

C. Chemical Processes and Changes Within the Compost and Tailings Layers

> a) Characterization of the crystallinity of oxidized tailings and the distribution of adsorbed trace elements within the tailings
>
> b) Effects of redox potential and acidity changes on solid phase chemical speciation and the composition of tailings pore water and leachate

Principal Researcher - Nelson Belzile

Experimental Design Overview

Four concurrent lines of investigation will address the basic questions concerning the optimal compost cover layer characteristics, compost quality, the redox chemistry of the tailings and the leaching column methodology to be used in a final compost-tailings simulation experiment (Figure 2):

    A. Physical Aspects and Optimization of Compost Cover Layers

    B. Leachate Composition from Compost of Varying Degrees of Maturity

    C. Chemical Processes and Changes Within the Compost and Tailings

    D. Leaching Column Design and Sampling Methodology

The simulation experiment, described as part E, will follow the preliminary experiments.

    E. Compost Cover Layer-Tailings Simulation Run

The optimal compost-tailings models will be treated to wetting/drying cycles for approximately 10 weeks. Periodic analyses on pore water and gases will be done to follow the .progress of chemical change in thecompost .and tailings... Final column sectioning and analyses will be done at the end of the experiment.

Exeerimental Desian Details

A. Physical Aspects and Optimization of Compost Cover Layers

Experiment 1. Effect of overburden pressure on compost compaction. and physical characteristics

Rationale: The hydraulic characteristics of the compost layer are largely a function of its pore size distribution. When an overburden -pressure is applied to a layer of fibrous -- material such as compost, the total porosity will decrease and pore size distribution will change, primarily through the loss of the larger pores. As greater overburden pressures are imposed, smaller pores will be more affected. A cost-effective implementation of a compost cover layer must minimize the depth of overburden required, while creating a compost layer with the desired physical and hydraulic characteristics.

Method: (a) A measured weight of moist compost will be compacted between porous plates within a cylindrical column by a known mechanical pressure (i.e. weight). Bulk density will be determined by the resultant volume of the compacted compost. The experiment will be repeated at various overburden pressures for samples at the same initial water content.

(b) When in .a compacted.state, the saturated hydraulic conductivity of the compost core will be measured by the standard falling head method. These data will show the effect of compaction on the ability of percolating water to flow downward through the compost to the tailings (benefit 5).

(c) Porosity of the compacted compost core will be determined at the end of the experiment while the sample is still under pressure. This measurement will quantify the change in pore size distribution caused by compaction and show the relative benefit of an increasing overburden pressure (benefit 1).

Experiment 2. Compost compaction as function of initial water content and swelling

Rationale: Water will be held by capillary forces more strongly in the smaller pore sizes of the compost matrix. As water content increases, larger pores sizes will become water-filled. Compost compaction occurs at the expense of air-filled pore spaces until the large pores are gone and water may be redistributed to smaller air-filled pores until all pores are water-filled. Since water is essentially incompressible, the water-filled compost matrix will not compact further. Water saturation may also cause the swelling of compost particles as they expand due to colloidal adsorption of water. Therefore, the initial water content of the compost may be an important variable determining the degree of compaction and compost hydraulic characteristics under an overburden pressure (benefits 1 & 5).

Method: The compaction experiment, Experiment I(a), will be repeated using compost samples of different initial water contents, from air dry up to full saturation, for selected overburden pressures. Bulk density will be determined as a function of both initial moisture content and overburden pressure. These trends will indicate to what extent the initial compost water content is important to the degree of compaction that can be achieved.

Experiment 3. Determination of the moisture characteristic for the optimal compaction treatment

Rationale: When compacted, the compost will display an altered relationship between its water content and the tension under which the water is held. This is called the moisture characteristic and is a function of the pore size distribution in the sample. In general, the smaller the pores, the greater the pressure required to remove the pore water. This moisture characteristic relationship is important to model B (Fig. 1 b) where the compacted compost layer must retain high water content at high tensions in the coarse layer.

Method: The compacted compost moisture characteristic will be determined, using a modified pressure plate apparatus, on samples which demonstrate the optimal degree of compaction. A constant volume pressure chamber will be filled with a compost sample of the specific weight needed to create the desired degree of compaction. The sample inside the chamber will be subjected to a range of external air pressures to drive water out of the pores. When water efflux ceases and equilibrium is attained, the sample will be removed and its wet and oven dry weights will be measured. Some development of the pressure chamber design will be necessary.

Experiment 4. Oxygen concentration profile in the compacted compost

Rationale: Oxygen concentration at any point in the compost column will be a function of the rate of diffusion of atmospheric oxygen and the activity of oxygen-consuming organisms. A profile of oxygen concentration with depth in the compacted compost will indicate the efficiency of compost as a physical oxygen diffusion barrier and as a sink for molecular oxygen (benefits 1 & 2).

Method: In a column of compacted compost, the pore water and gas will be sampled at various depths by inserting the needle of a syringe into the compost through a septum in the column wall. The free oxygen concentration in the sample will be measured by gas chromatography. The experiment will continue until a steady-state oxygen profile is achieved. The experiment will be repeated with samples at various degrees of initial saturation and compaction, as suggested by the results of experiments 1 and 2.

B. Compost Compaction from a Degrees of Maturity

Experiment 1. Determination of the degree of compost maturity

Rationale: The rapid decomposition processes in immature compost produce a variety of organic compounds and consume considerable molecular oxygen from the air. Both of these processes are potentially beneficial in tailings reclamation (benefits 2, 3 and 4) but greatly decrease as organic matter becomes humified to form mature compost, a relatively stable product. Thus, it is important to characterize the degree of compost maturity, both at the beginning and end of an experiment, in order to assess the relative capability of immature and mature compost to stop tailings oxidation.

Methocf: Several approaches have been developed to characterize compost-maturity (Inbar *et al.* 1990, Jimenez & Garcia 1989). The most common methods are chemical analyses of cation exchange capacity, soluble organic matter, carbon/nitrogen ratio and total humic substances. Immature and mature compost will be analyzed by several methods to determine the degree of difference and variability between samples.

Experiment 2. Regulation 309 leaching test

Rationale: The Environmental Protection Act 1990 for Ontario specifies Regulation 309 for the standard testing of the acceptable levels of certain contaminants in solid waste. Unless shown by the Regulation 309 test to be safe, MSW compost would have to be treated as a restricted or hazardous waste with potentially harmful effects on the environment.

Method: Mature and immature compost samples will be sent to an outside laboratory for the Regulation 309 leaching test, which involves a dilute acid leaching.

Experiment 3. Heavy metal spiking of compost to determine the degree of heavy metal retention under simulated acid rain leaching

Rationale: One of the roles played by the compost material will be the complexation of metals. It is important to know the capacity of the compost to complex metals and the stability and leachability of the organic matter itself. In the field, the chemical composition of the precipitation falling on the tailings site will be an uncontrolled variable. The pH of the rain or snowmelt may have a significant effect on the leaching process or the effluent chemistry.

Method: a) Mature and immature compost samples will be spiked with metals and 2,4-D herbicide and incubated for one week. The spiked and control samples are then leached in a column ten times with 500 ml distilled water. The effluent is tested immediately for pH and redox potential (Eh) then stored for later metal and organic analyses, acidifying if necessary.

b) The spiking and leaching tests will be repeated using simulated acid rain as the leachant.

C. Chemical Processes and Changes Within the Compost and Tailings

Experiment 1. Characterization of the crystallinity of oxidized tailings

Rationale: Trace metals exist in tailings primarily in an adsorbed state. The sorption capacity of tailings oxides is inversely related to their degree of crystallinity. Thus, it is useful to characterize the initial state of crystallinity as a basis for comparison with the tailings oxides which have been exposed to the effects of compost cover layers.

Method: X-ray diffraction spectrometry will be-give information on the sorption capacities of dry tailings samples for trace metals.

Experiment 2. The distribution and bio-availability of adsorbed trace elements within the tailings

Rationale: Identification of the toxic potential of trace elements in the tailings is an important aspect of AMD prediction. Trace elements are adsorbed to many chemical species in oxidized tailings, particularly iron oxides. If the conditions change as a result of anoxia and chemical amelioration to reducing conditions, metals will become mobile and possibly readsorbed on sulphides in a sequence determined by the changing redox chemistry. This sequence will be unique to the particular tailings components.
Method: The quantities and distribution of trace elements will be determined using proven chemical sequential leaching techniques (e.g. Tessier et al 1979). Trace element concentrations will be measured by Inductively Coupled Plasma technology.

Experiment 3. Effects of redox potential and acidity changes on solid phase chemical speciation and the composition of tailings pore water and leachate

Rationale: In the compost cover layer situation, redox chemistry in the underlying tailings will change as a result of anoxia (benefit 1) and possibly as a result of chemical leachates from the compost (benefit 3 & 4). The effect of redox potential on the reactivity and reduction of tailings oxides and the mobilization of trace elements may be examined in tailings samples.

Method: Laboratory simulations with slurries of tailings will be employed. The redox potential of the pore water can be altered using chemicals or compost and is monitored by electrodes. The effects are measured by the techniques described previously for trace element analysis.

D. Leaching Column Design and Sampling Methodology

Experiment 1. Design requirements to avoid the lateral diffusion or introduction of atmospheric oxygen into leaching columns

8

Rationale: The investigation of the compost-tailings system will be done in the laboratory using columns of compost and tailings layers. As in the expected field situation, oxygen diffusion into the system will be only downwards from the atmosphere through the cover layers -essentially a one-dimensional system. In the laboratory, plexiglas (or some other polymerized material) tubes will be used to enclose the compost and tailings. It will be important that atmospheric oxygen not diffuse into the compost or tailings through the walls of the column or through any sampling ports.

Method: Experimental investigations will be done on designs for enclosures to maintain the leaching columns in a nitrogen atmosphere during simulation runs lasting several weeks or months.

Experiment 2. Design requirements and techniques for the sampling of pore water and gases in compost and tailings layers

Rationale: Samples of pore water and gases should be extracted from the columns without disturbance to the compost-tailings system. All samples must be obtained, handled and analyzed in a manner which ensures that the data are representative of the *in situ* experiment.

Method: A potential source of error in the simulation run will be the introduction of atmospheric oxygen into the column each time it is opened or into the sample before it is analyzed. A device such as a portable, nitrogen-filled glove box will be designed to avoid sampling error. The techniques for using platinum redox electrodes, pH electrodes and syringes will also be examined.

Experiment 3. Design requirements for the simulation of the drainage conditions present in tailings and compost layers in the field

Rationale: In the field situation, the tension (suction or negative hydraulic head) on the pore water in the compost will be the result of a gravitational component (i.e. height above the water table) and a matric component (i.e. due to capillary forces in the pores). A gradient of hydraulic head is the driving force for downward drainage in the compost-tailings system. But drainage is also a strong function of the unsaturated hydraulic conductivity and is thus dependent on the degree of pore water continuity (i.e. inversely related to air-filled porosity) through the profile.

Method: Preparation of tailings material, depth of tailings layer, techniques of packing the leaching columns and initial hydration of the column must all be examined when designing the simulation of the field situation. Tensiometers will be used to monitor the tailings moisture tension and assess the drainage regime.

E. Compost Cover Layer-Tailings Simulation Runs

The last group of experiments will use the results and conclusions of the previous four sections of experimental work to design and run leaching column simulations of the three compost cover layer-tailings models.

Experiment 1. Simulation of the dynamics of field conditions through several wetting-drying cycles

Rationale: To understand the saturation dynamics of the optimal compost cover layer models in the field, a simulation experiment will be devised to investigate the water balance-and redox chemistry of a compacted compost-tailings column under simulated precipitation, evaporation and drainage conditions.

Method: The specific design of the simulation experiments will not be known until preliminary work has be completed. However, the following outline represents the current view of the design.

Columns:　　2 cover layer models & 1 control
　　　　　　　2 compost quality grades
　　　　　　　3 replicates

Sequence of Treatments: 1. Saturate the column
　　　　　　　　　　　　　　2. Incubate 2 weeks
　　　　　　　　　　　　　　3. Leach with 50mm water
　　　　　　　　　　　　　　4. Incubate 2 weeks
　　　　　　　　　　　　　　5. Leach with 50mm water
　　　　　　　　　　　　　　6. Drying cycle 4 weeks
　　　　　　　　　　　　　　7. Leach in excess
　　　　　　　　　　　　　　8. Incubate 2 weeks
　　　　　　　　　　　　　　9. End experiment • section columns

Periodic Analyses: redox potential, pH
　　　　　　　　　　trace metals, major organics
　　　　　　　　　　O_2, CO_2, CH_4, sulphate
　　　　　　　　　　moisture tension

Section Analyses: crystallinity, sulphate, sulphides
　　　　　　　　　compost physical characteristics
　　　　　　　　　compost maturity

Projec Schedule

Nov. 1   Start of experimental work (approximate)

Nov. 10   MEND project plan review

Dec. 15   Progress report

Feb. 15   End of experimental work (approximate)

Mar. 15   Final report

Mar. 30   Deadline for invoices

Personnel

Dr. Nelson Belzile - Assistant Professor, Dept. of Chemistry

Dr. Gerard Courtin - Executive Director, Centre in Mining and Mineral Exploration Research and Associate Professor, Dept. of Biology

Dr. Glenn Pierce - Research Associate, Centre in Mining and Mineral Exploration Research

Prof. Keith Winterhalder - .Associate Professor, Dept. of Biology

Technician

Facilities and Major Equipment

A. Laboratory Space

A wet lab of at least 30 m² floor area, with work benches and thermostat-controlled temperature will be required.

B. Leaching Columns & Enclosures $1300.

Leaching columns will be constructed out of acrylic plastic (plexiglas) tubing of 10 cm outside diameter with 3.2 mm walls. Eighteen tubes, 183 cm long will be required for the simulation experiment. A group of six tubes will be enclosed inside a plywood cabinet containing a nitrogen atmosphere at slight positive pressure to exclude atmospheric oxygen.

C. ICP Analyses $2500.

ICP analysis cost for a range of heavy metals will be about $25. per sample. The expected number of samples will be in the range of 100.

D. Gas Chromatography - Mass Spectrometery $400.

Operating costs for the GC-MS will be about $ 100. per month.

E. Electrode System - E_h and pH $1200.

    Redox potential electrodes (3 @ $300.)
    pH electrodes (3 @ $ 100.)

F. Tensiometers $ 500.

    Soil tensiometers (6 @ $ 62. & calibration kit)

Costs

        Salaries
                        Glenn Pierce $ 13,000.
                        Nelson Belzile $ 1,000.
                        Keith Winterhalder $ 1,000.
                        technician (4 mo.) $ 12,000.
                        CIMMER overhead $11,600.

        Lab development & operation $ 6,000.

        Chemical analyses $ 3,000.

        Travel, administration $ 3,000.

        Total Project Cost - CIMMER portion $50,600.

References

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Pierce, W.G. 1992. **Reclamation of Sulphide Tailings Using Municipal, Solid Waste Compost: Literature Review and Recommendations.** Report prepared for Falconbridge Ltd. by the Centre in Mining and Mineral Exploration Research, Laurentian University, Sudbury, Ontario. July 1992.

Tessier, A., P.G.C. Campbell & M. Bisson 1979. Sequential extraction procedure for the speciation of particulate trace metals. Analyt. Chem. 51: 844-851.

Appendix B: Lakefield Research Analytical Methods

March 17, 1993

Mark Wiseman
Supervisor • Environmental Projects
Falconbridge Sudbury Operation
Falconbridge, Ontario

Dear Mark:

Re: **Analytical Results** of Compost Leachate Testing Programme
Lakefield Research Project No. 7777-024

Following is a summary report of the test work performed at Lakefield Research on the compost samples as received from Laurentian University. The page numbeers referred to in the following discussion refer to the page numbers of the analytical report appended as Appendix A.

Method
Two samples of compost, one raw and one mature, were received at Lakefield Research in December 1992. Both samples were kept frozen until test work was conducted in order to reduce the possibilities of further maturing and change in the compost character during storage.

Prior to initiating the test work, both samples were thawed and homogenized as much as possible. The mature compost sample was riffled to provide a representative portion for background characterization. The raw sample, due to the nature of the material, had to be hand quartered to provide representative samples.

The raw compost was reciuced in particle size accordmg to the requirements of the Ontario Regulation 309 (0. Reg. 309) leachate procedure and the Toxicity Characteristic Leaching Procedure (TCLP) . For both leachate tests the particle size of the samples were reduced to minus 9.5 mm. In order to evaluate the effect of particle size reduction on the raw compost, an 0. Reg. 309 leachate test was conducted on the coarse "uncut" raw compost in both its natral "As Received" state and after spiking with both metal and organic spiking solutions (pages 14 and 15).

The background characterization for metals required that each of the samples be dried and pulverized prior to analysis. As received samples were used for nitrogen, pesticide and PCB analyses.

Method (Continued)

The background characterization (pages 1 and 8) and leachate analyses (pages 2, 3, 6, 7, 11, and 12) were performed for the following analyses (as requested during a telephone conversation Mark Wiseman • Linda Elliott December 17, 1993):   Ni, CU, Fe, Pb, Zn, Cd, Cr, As, Se, Co, Mn, Mo, Hg, N03, N02, Total Kjedhal Nitrogen (TKN) and NH3 + NH4. Herbicides, pesticides and PCB analyses were performed for the parameters listed in Schedule 4 of Ont. Reg. 309.

One hundred grams (dry weight) were cut from each of the raw and mature homogenized compost samples and then spiked with the appropriate analyte quantities. The samples were spiked with 10 times the maximum allowable concentrations for composts (MOE, 1991) and then left for 24 hours before follow-up test work (as requested during a telephone conversation: Linda Elliott • Mark Wiseman, December 9, 1993). The samples were not dried after spiking.

Water soluble compounds were used as spiking solutions for the metal components (page 4). Some of the compounds used are incompatible together in solution (e.g. if solutions of $PbCH_3COOH$ and Cr_2O_3 are mixed they will form highly insoluble precipitates). To avoid this problem, separate spiking solutions were made. However, once combined in the compost samples some precipitation may have occurred.

Compost samples were spiked with the organic analytes (page 9) for subsequent leachate testing. A median pesticide concentration of 0.05 µg/g of dry weight sample was used as the spiking concentration. At this level, the concentration in the compost would be, on average, 10 times greater than those listed in Schedule 4 of Ont. Reg. 309.

Samples were spiked based on dry weight. Leachate procedures require dewatering (but not dried samples), therefore, each sample was spiked with the metal and organic solutions, homogenized and left for 24 hours before leachate tests were initiated. No nitrogen compound spikes were used, as concentrations of these compounds were already at high levels. Samples of the spiked solutions were subjected to thorough background characterization prior to leachate testing (pages 5 and 10).

0. Reg. 309 and TCLP leachate tests were conducted in duplicate for each of the raw and mature, as received and spiked compost samples.

Results

The laboratory analytical results are appended.

Background Content Characterization

Background content characterization of the samples shows the molybdenum concentration is higher than the maximum allowable concentration (2 mg/kg dry weight) for compost in both the raw and mature compost samples (37.8 and 52.6 mg/kg average dry weight, respectively).

The concentration of all other parameters listed in the MOE Compost Quality Specifications - Draft Guideline for the Production and Use of Aerobic Compost in Ontario (1991) are below the recommended maximum allowable concentrations. The other compounds of interest which were analyzed for include Fe, Mn, N03, N02, TKN and NH3 + NH4. Each of these parameters are present at relatively high concentrations in the as received raw and mature, compost samples.

The initial background scan on the compost samples showed that there were no pesticides, herbicides or PCB's present above the analytical method detection limits. Based on these results, 0. Reg. 309 and TCLP leachates on the "as received" compost samples were not analyzed for organic parameters.

Leachate Test Results for the "As-Received" Compost

O.Reg. 309 Leachate Results For "As Received" Compost Samnles
Mature "As Received" Compost

Analytical results from the mature, as received, compost leachate tests show that the average nitrate + nitrite concentration (>100 mg/l) exceeds the leachate quality criteria set out in 0. Reg. 309 (10 mg/l).

The concentration of organic nitrogen, an aesthetic water quality parameter (N_{org} = TKN - (NH3 + NH4) = approximately 11 mg/l) is elevated with respect to the Ontario Drinking Water Objectives (ODWO) limit of 0.15 mg/l.

The concentration of manganese, also an aesthetic water quality parameter, is similarly elevated above the ODWO limit of 0.05 mg/l in the mature, as received, compost leachate.

Results (Continued)

Raw "As Received" Compost

The concentrations of nitrate + nitrite in the raw compost leachates are within allowable levels according to the requirements of 0. Reg. 309, however, the organic nitrogen content (N_{org} = approximately 33 mg/l) is elevated with respect to the ODWO.

The concentrations of manganese and iron, both aesthetic water quality parameters, are similarly elevated above the ODWO in the raw, as received, compost leachates.

Raw - Natural (uncut) "As-Received" Compost

The results of the O.Reg. 309 leachate test on the natural uncut (non-size reduced) raw compost (page 14) are very similar to those for the size reduced raw compost (page 2).

TCLP Leachate Test Results For "As Received" Compost Samples

Mature "As Received" Compost

Analytical results from the mature, as received, compost leachate tests show that the average nitrate + nitrite concentration (>100 mg/l) exceeds the leachate quality criteria set out in 0. Reg. 309 (10 mg/l), however, there are no equivalent requirements in the EP toxicity list. The concentrations measured are in excess of the ODWO limit of 10 mg/l.

The concentration of organic nitrogen, an aesthetic water quality parameter (N_{org} = TKN - (NH3 + NH4) = approximately 5 mg/l) is elevated with respect to the Ontario Drinking Water Objectives (ODWO) limit of 0.15 mg/l.

Raw "As Received" Comnost
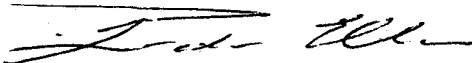
The concentrations of nitrate + nitrite in the raw compost leachates are within allowable levels according to the requirements of 0. Reg. 309, however, the organic nitrogen content (N_{org} = approximately 10 mg/l) is elevated with respect to the ODWO.

Concentrations of manganese and iron, both aesthetic water quality parameters, are similarly elevated above the ODWO in the raw, as received, compost leachates.

Based on the findings of the test work, it was recommended in our preliminary report dated February 9, 1993 that Fe, Mn, N03, N02, NH3 + NH4 and TKN, be included in analyses performed on the leachates generated from the column tests at Laurentian University.

If you have any questions regarding the contents of this report please feel free to call the undersigned.

Respectfully submitted
LAKEFIELD RESEARCH

Linda C.M. Elliott, M.Eng.
Project Manager - Environmental Services

cc.   Dr. Glen Pierce, Laurentian University

encl.

SEND TO Dr. Glen Pierce
Laurentian University

FROM Linda Elliott   **DEPT.** Lakefield Res.   **DATE** March 25

SUBJECT Leachate Test Methods

As per our discussion

TCLP - Extraction Fluid #1 was used
according to 7.2.2 and 7.2.3
∴ 100g sample - used 2 l of extraction fluid

Reg 309 - 50g sample - use 1 l of extraction fluid
the method describes has this is
added

I hope this answers your questions. Call if you

REPLY have any more   Regards

REPLY FROM	REPLY DATE
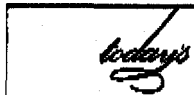
Appendix C: Lakefield Research Analytical Results

APPENDIX B

QUALITY CONTROL QUALITY ASSURANCE

The testwork performed at Lakefield Research was an exercise in quality control and quality assurance.

Duplicate samples were run for the background contant characterization for the organic and inorganic parameters of interest in both the as-received raw compost and the as-received mature compost.

Duplicate samples were run for the background content characterization for the inorganic parameters of interest in both the spiked raw compost and the spiked mature compost.

Duplicate samples were run for the inorganic parameters of interest ,in the Ontario Regulation 309 leachate testing on both the as-received and the spiked raw and mature compost samples.

Duplicate samples were run for the inorganic parameters of interest in the dilute acid rainfall (TCLP) leachate testing on both the as-received and the epiked raw and mature compost samples.

An uncut, non-size reduced raw compost sample was analyzed for Ontario Reg. 309 leachate testing for purposes of comparing results with the size reduced raw compost sample.

Blanks were run for each of the inorganic and organic O.Reg. 309 and TCLP leachate test runs. Results of all blanks analyzed showed less than detection level readings.

LAKEFIELD RESEARCH
A division of **Falconbridge Limited**
Postal Bag 4300,185 Concession Street,
Lakefield, ON KOL 2H0   Phone • 705-652-3341 / Fax • 705-652-6365

Falconbridge Limited	Date: Feb. 10, 1993
Environmental Services	Date Samples Received: **Dec. 22, 1992**
Sudbury, ON POM 1S0	No. of Samples: 2
	Lakefield Receipt No. : 9241469
	Page No. : 1

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

"As received" Compost: Background Content Characterization

Analytes	Unit	Raw 1	Raw2	Raw Average	Mature 1	Mature 2	Mature Average
As	µg/g	5	<2	2.5	7	<2	3.5
Cd	µg/g	1.9	1.8	1.9	1.8	1.5	1.7
CO	µg/g	11.6	8.9	10.3	12.3	8.2	10.3
Cr	µg/g	24.4	31.6	28.0	18.4	24.4	21.4
Cu	µg/g	30.7	31.4	31.1	32.9	34.0	33.5
Fe	µg/g	10900	9990	10445	10900	11300	11100
Hg	µg/g	0.02	0.02	0.02	0.04	0.05	0.05
Mn	µg/g	528	486	507	438	431	435
Mo	µg/g	40.1	35.4	37.8	48.5	56.7	52.6
Ni	µg/g	16.5	16.5	16.5	13.2	12.1	12.7
Pb	µg/g	32	32	32	48	57	53
Se	µg/g	<1	<1	<1	<1	<1	<1
Zn	µg/g	156	153	155	186	172	179
NO3	µg/g	520	519	520	2960	2975	2968
NO2	µg/g	<10	<10	<10	<10	<10	<10
TKN	µg/g	16513	16500	16507	11471	11450	11461
NH4+NH3 as N	µg/g	673	671	672	93	90	92

• All results based on a dry weight

Signed: _____

J. R **Johnston**
Chief Chemist.

LAKEFIELD RESEARCH
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Postal Bag 4300,185 Concession Street,
Lakefield, ON K0L 2H0   Phone - 705-652-3341 / Fax - 705-652-6365

Falconbridge Limited
Environmental Services
Sudbury, ON POM 1S0

Date:	Feb. 10, 1993
Date Samples Received:	Dec. 22, 1992
No. of Samples:	2
Lakefield Receipt No. :	924 1469
Page No. :	2

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

"As reeeived size redneed compost ":Leachate Inorganic Extraction Data - Reg. 309

Analytes	Unit	Raw 1	Raw2	Raw Av.	Mature 1	Mature 2	Mature Av.
As	mg/L	CO.05	<0.05	<0.05	<0.05	<0.05	<0.05
Cd	mg/L	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
CO	mg/L	<0.02	CO.02	CO.02	CO.02	<0.02	<0.02
Cr	mg/L	KO.02	<0.02	<0.02	<0.02	<0.02	<0.02
Cu	mg/L	CO.02	<0.02	<0.02	<0.02	<0.02	<0.02
Fe	mg/L	0.42	0.78	0.60	0.10	0.24	0.17
Hg	mg/L	~0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Mn	mg/L	4.89	5.58	5.24	1.23	1.36	1.29
Mo	mg/L	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Ni	mg/L	<0.02	<0.02	KO.02	<0.02	CO.02	KO.02
Pb	mg/L	CO.05	<0.05	<0.05	<0.05	<0.05	<0.05
se	mg/L	<0.01	KO.01	<0.01	<0.01	<0.01	KO.01
Zn	mg/L	0.13	0.13	0.13	0.09	0.08	0.09
NO3	mg/L	0.35	0.48	0.42	98.3	0.55	49.4
NO2	mg/L	<0.10	1.00	0.55	113	41.5	77.3
TKN	mg/L	47.2	45.1	46.2	12.3	9.80	11.1
NH4+NH3 as N	mg/L	14.0	13.5	14.0	0.13	0.34	0.24

Signed: _____
J. R. Johnston
Chief Chemist.

Falconbridge Limited	Date:	Feb. 10, 1993
Environmental Services	Date Samples Received:	Dec. 22, 1992
Sudbury, ON POM 1S0	No. of Samples:	2
	Lakefield Receipt No. :	9241469
	Page No. :	3

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

"As received **size** reduced **compost":** Leachate **Inorganic** Extraction Data-TCLP

Analytes	Unit	Raw 1	Raw2	Raw Av.	Mature 1	Mature 2	Mature Av.
As	mg/L	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Cd	mg/L	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
CO	mg/L	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Cr	mg/L	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
CU	mg/L	<0.02	<0.02	CO.02	<0.02	KO.02	<0.02
Fe	mg/L	0.80	0.58	1.38	0.40	0.19	0.29
Hg	mg/L	0.002	0.002	0.002	0.001	0.001	0.001
Mn	mg/L	0.12	0.12	0.12	<0.01	<0.01	<0.01
Mo	mg/L	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Ni	mg/L	<0.02	<0.02	KO.02	<0.02	<0.02	<0.02
Pb	mg/L	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
se	mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Zn	mg/L	0.04	0.04	0.04	0.03	0.02	0.03
NO3	mg/L	<0.10	<0.10	<0.10	98	110	104
NO2	mg/L	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
TKN	mg/L	16.3	14.8	15.6	5.50	5.08	5.29
NH4+NH3 as N	mg/L	4.30	6.58	5.49	0.41	0.48	0.45

Signed: _____

J. R Johnston
Chief Chemist.

Falconbridge Limited   Date: Feb. 10, 1993
Environmental Services   Date Samples Received: Dec. 22, 1992
Sudbury, ON POM 1S0   No. of Samples: 2
                                   Lakefield Receipt No. :   9241469
                                             Page No.: 4

Attention: Mr. Mark **Wiseman**

CERTIFICATE OF ANALYSIS

Inorganic Spiking Materials and Quantities Used

Analyte	Compound	Concentration µg/g of sample
As	As2O3	100
c d	CdSO4	30
C O	CoCl	250
Cr	Cr2O3	500
CU	CuSO4	600
Fe	Fe2SO4	10,000
Hg	Hg	2
Mn	MnSO4	500
Mo	Mo	20
Ni	NiSO4	600
Pb	PbCH3COOH	1500
S e	S e	20
zn	ZnCH3COOH	5000

As no limits were given for Fe or Mn, it was decided to use **amounts equal** to the **background** concentration **of both these analytes.**

Signed: _____
        J. R **Johnston**
        Chief Chemist.

LAKEFIELD RESEARCH
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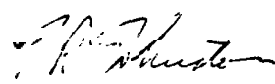
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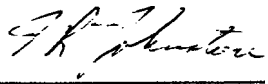<tr><td>Sudbury, ON POM 1S0</td><td>No. of Samples:</td><td>2</td></tr>
<tr><td></td><td>Lakefield Receipt No.:</td><td>9241469</td></tr>
<tr><td></td><td>Page No. :</td><td>5</td></tr>
</table>

Attention: Mr. Mark Wiseman

<u>CERTIFICATE OF ANALYSIS</u>

<u>Spiked Compost Background Characterization</u>

Anvtes	Unit	Raw 1	Raw2	Raw Average	Mature 1	Mature 2	Mature Average
As	µg/g	<12	<12	<12	<12	<12	<12
c d	µg/g	19.9	18.5	19.2	20.1	21.1	20.6
Co	µg/g	168	170	169	166	161	164
Cr	µg/g	340	340	340	317	302	310
CU	µg/g	470	475	473	409	398	404
F e	µg/g	19700	19900	19800	21100	21000	21050
Hg	µg/g	1.0	1.1	1.1	1.2	1.1	1.2
Mn	µg/g	850	840	845	880	850	865
Mo	µg/g	68	72	70	78	72	75
Ni	µg/g	468	480	474	458	456	457
Pb	µg/g	1230	1190	1210	1210	1198	1204
Se	µg/g	20	18	19	21	20	21
Zn	µg/g	3330	3295	3313	3320	3325	3323
*NO3	µg/g	-	-	-		-	-
*NO2	µg/g	-	-	-	▪	-	-
*TKN	µg/g	-	-	-		-	-
*NH4+NH3 as N	µg/g	-	-	-		-	-

*Samples were not spiked for the Nitrogen paramaters due to high background
levels of these paramaters

Results are on a dry weight basis.

Signed: _____
J. R. Johnston
Chief Chemist.

Falconbridge Limited	Date: Feb. 10, 1993
Environmental Services	Date Samples Received: Dec. 22, 1992
Sudbury, ON POM 1S0	No. of Samples: 2
	Lakefield Receipt No. : 924 1469
	Page No.: 6

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

"Size reduced spiked compost":Leachate Inorganic Extraction Data ▪ Reg. 309

Analytes	Unit	Raw 1	Raw 2	Av.	Mature 1	Mature 2	Mature Av.
As	mg/L	0.45	1.32	0.93	0.91	1.07	0.99
Cd	mg/L	0.10	0.12	0.11	0.09	0.14	0.12
Co	mg/L	2.13	3.11	2.62	2.11	3.61	2.86
Cr	mg/L	0.10	0.23	0.17	0.80	1.13	0.97
Cu	mg/L	0.19	0.31	0.25	0.25	0.38	0.32
Fe	mg/L	14.1	14.5	14.3	18.1	15.8	17.0
Hg	mg/L	0.002	0.004	0.003	0.001	0.002	0.001
Mn	mg/L	18.1	17.5	17.8	15.4	15.0	15.2
Mo	mg/L	1.00	1.20	1.10	0.60	0.82	0.71
Ni	mg/L	3.09	4.70	3.90	2.96	5.48	4.22
Pb	mg/L	0.21	0.06	0.12	0.06	0.06	0.06
se	mg/L	0.16	0.36	0.26	0.29	0.43	0.36
Zn	mg/L	55.2	21.8	38.5	37.9	52.3	45.1

Signed: _____

J. R. **Johnston**
Chief Chemist.

Falconbridge Limited	Date:	Feb. 10, 1993
Environmental Services	Date Samples Received:	Dec. 22, 1992
Sudbury, ON POM 1S0	No. of Samples:	2
	Lakefield Receipt No. :	**924 1469**
	Page No.:	7

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

Size reduced Spiked compost ": Leachate Inorganic Extraction Data-TCLP

Analytes	Unit	Raw 1	Raw2	Raw Av.	Mature 1	Mature 2	Mature Av.
As	mg/L	0.67	1.32	1.00	1.06	1.10	1.08
Cd	mg/L	0.093	0.099	0.096	0.10	0.10	0.10
Co	mg/L	2.35	2.13	2.24	2.50	2.36	2.43
Cr	mg/L	0.16	0.10	0.13	0.72	0.68	0.70
Cu	mg/L	0.23	0.19	0.21	0.24	0.26	0.25
Fe	mg/L	410	400	405	432	432	432
Hg	mg/L	0.004	0.002	0.003	0.001	0.002	0.002
Mn	mg/L	18.2	18.0	18.1	19.6	19.0	19.3
Mo	mg/L	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Ni	mg/L	3.51	3.09	3.30	3.60	3.51	3.56
Pb	mg/L	<0.05	0.21	0.13	<0.05	<0.05	<0.05
Se	mg/L	0.23	0.16	0.20	0.34	0.31	0.33
Zn	mg/L	26.5	55.2	40.9	49.7	38.5	44.1

Signed: _____

J. R. Johnston
Chief Chemist.

Falconbridge Limited   Date: Feb. 10, 1993
Enviromnental Services   Date Samples Received: **Dec. 22, 1992**
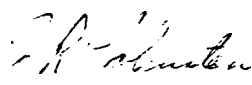Sudbury, ON POM 1S0   No. of Samples: 2
                                  Lakefield Receipt No. :   9241469
                                            Page No.: 8

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

"As receieved compost":Organic Background Content Characterization
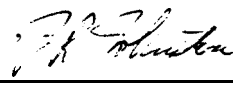
Analytes	Unit	Raw 1	Raw 2	Raw Average	Mature 1	Mature 2	Mature Average
Aldrin	µg/g	ND	ND	ND	ND	ND	ND
Dieldrin	µg/g	ND	ND	ND	ND	ND	ND
Chlordane	µg/g	ND	ND	ND	ND	ND	ND
DDT	µg/g	ND	ND	ND	ND	ND	ND
Endrin	µg/g	ND	ND	ND	ND	ND	ND
Heptachlor epoxide	µg/g	·ND	ND	ND	ND	ND	ND
Methoxychlor	µg/g	ND	ND	ND	ND	ND	ND
Toxaphene	µg/g	ND	ND	ND	ND	ND	ND
2,4-D	µg/g	ND	ND	ND	ND	ND	ND
Silvex	µg/g	ND	ND	ND	N-D	ND	ND
PCB	µg/g	ND	ND	ND	ND	ND	ND

N D   = Not detected

2,4-D
  and silvex = The phenoxy herbicides are being analyzed separately

Pesticide and PCB analysis were conducted using methods outlined by
 the MOE Method PSAOC-E27OA.1

Signed: _____
                    J. R Johnston
                    Chief Chemist.

Falconbridge Limited	Date: Feb. 10, 1993
Environmental Services	Date Samples Received: Dec. 22. 1993
Sudbury, ON POM **1S0**	No. of Samples: 2
	Lakefield Receipt No. : **9241469**
	Page No.: 9

Attention: **Mr**. Mark Wiseman

CERTIFICATE OF ANALYSIS

Organic Spiking Materials and Quantities Used

Analyte	Concentration **μg/g of sample wt**
Aldrin	**0.5**
Deldrin	0 5
Chlordane	0.5
DDT	0.5
Endrin	0.5
Heptachlor	0.5
Heptachlor Epoxide	0.5
Lindane	0.5
Methoxychlor	0.5
Toxaphene	0.5
2,4-D	0.5
Silvex	0.5
PCB	0.5

Samples were **spiked based** on a dry weight **basis.**

Signed: _J. R. Johnston_

J. R. **Johnston**
Chief Chemist.

LAKEFIELD RESEARCR
A division of Falconbridge Limited
Postal Bag 4300,185 concession Street,
Lakefield, ON K0L 2H0   Phone - 705-652-3341 / Fax - 7054524365

Falconbridge Limited	Date: Feb. 10, 1993
Environmental Services	Date Samples Received: Dec. 22, 1992
Sudbury, ON POM 1S0	No. of Samples: 2
	Lakefield Receipt No. : **9241469**
	Page No.: 10

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

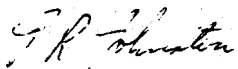## "Spiked compost": Organics Background Content Characterization

Analytes	Units	Raw	Mature
Aldrin	μg/g	0.026	0.12
Dieldrin	μg/g	0.001	< 0.001
Chlordane⸽	μg/g	< 0.6	< 0.6
DDT	μg/g	0.003	0.006
Endrin	μg/g	0.006	0.003
Heptachlor epoxide	μg/g	0.011	0.036
Methoxychlor	μg/g	0.13	0.20
Toxaphene ⸽	μg/g	< 6.0	< 6.0
2.4-D	μg/g	4 . 0	< 1.0
Silvex	μg/g	0.6	0.5
PCB	μg/g	0.38	0.63

⸽ Toxaphene and Chlordane were found to be present, however in this instance they coeluted and could not
be separated and quantified to levels lower than those indicated.

Pesticide and PCB analysis were conducted using methods outlined by
the MOE Method PSAOC-E27OA. 1

Signed: _____
J. R. Johnston
Chief Chemist.

Falconbridge Limited
Environmental Services
Sudbury, ON POM 1S0

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

Size reduced **spiked compost"**: Leachate **Organic parameters** Extraction Data -TCLP

Analytes	Unit	Raw 1 Spike	Mature 1 Spike
Aldrin	µg/L	0.3	0.7
Dieldrin	µg/L	<0.1	<0.1
Chlordane	µg/L	<3.4	<3.4
D D T	µg/L	<0.1	<0.1
Endrin	µg/L	0.4	0.9
Heptachlor epoxide	µg/L	1.0	1.1
Methoxychlor	µg/L	4.0	3.9
Toxaphene	µg/L	<34	<34
2,4-D	µg/L	4.4	9.2
Silvex	µg/L	6.9	11
P c 3	µg/L	14	10

Toxaphene and **Chlordane** were **found** to be **present,** however **in this** instance **they coeluted** and **could not** **be separated and quantified** to **levels lower than those** indicated.

Pesticide and PCB **analysis** were conducted **using** methods **outlined** by
the MOE Method **PSAOC-E27OA.** 1

Signed: _____
J. R. Johnston
Chief Chemist.

LAKEFIELD RESEARCH
A division of **Falconbridge Limited**
Postal Bag 4300, 185 Concession **Street,**
Lakefield, ON KOL **2H0   Phone - 705-W-3341 / Fax - 705-652-6365**

Falconbridge Limited
Environmental Services
Sudbury, ON POM **1S0**

Date: Feb. 10, 1993
Date Samples Received: Dec. **22, 1992**
No. of Samples: 2
Lakefield Receipt No. :   9241469
Page No.: 12

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

Size reduced sdked **compost":** Leachate **Organic parameters** Extraction Data **-Reg** 309

Analytes	Unit	Raw	Mature
Aldrin	µg/L	0.9	6.0
Dieldrin	µg/L	<0.1	<0.1
Chlordane*	µg/L	<3.4	<3.4
DDT	µg/L	a.1	0.2
Endrin	µg/L	0.7	1.2
Heptachlor epoxide	µg/L	1.0	1.3
Methoxychlor	µg/L	7.0	4.0
Toxaphene*	µg/L	<34	<34
2,4-D	µg/L	7.8	9.7
Silvex	µg/L	5.5	7.3
PCB	µg/L	14	13

• Toxaphene **and Chlordane** were **found** to be **present,** however in **this** instance they **coeluted and could not** be separated **and quantified** to levels lower **than those indicated.**

Pesticide and PCB **analysis** were **conducted using methods outlined by** the MOE Method **PSAOC-E270A.** 1

Signed: *J.R. Johnston*

3. R. **Johnston**
Chief **Chemist.**

Falconbridge Limited   Date: Feb. 10, 1993
Environmental Services   Date Samples Received: Dec. 22, 1992
Sudbury, ON POM 1S0   No. of Samples: 2
                                              Lakefield Receipt No.:   9241469
                                                        Page No.:   13
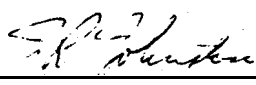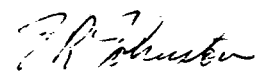
Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

Pesticide **and** PCB **analysis** were conducted using methods outlined by the MOE method
PSAOC-E270A. 1 for soils.

 The PCB standards were dissolved in hexane to **provide** for a **spiking** solution. This was
added in the aforementioned **quantities** to the as received composts and homogenized. The
samples were not dried as this would contradict the leachate protocols.

The PCB extration data indicates recoveries were **in** the **area** of 76% to 126%. This is **very**
acceptable and demonstrates that the method of extraction used works well in this
instance.

All pesticides were dissolved in a solution of methanol and added to the as received
compost. The samples were homogenized and **left** to stand for a 24 hour period . They
were not dried as this would contradict the leachate protocols.

It is **evident** that the pesticide **values** are on the low **side** in terms of recovery. This **can** be
attributed to the **difficult** sample matrix encountered with the compost samples.

 It should be pointed **out** that in most cases the methanol dissolved pesticides and hexane
dissolved **PCB's** do show up in the spiked compost leachate **analysis** data.

Signed:_____
                J. R. **Johnston**
                Chief Chemist.

Falconbridge Limited
Environmental Services
Sudbury, ON POM **1S0**

Date: Feb. 10, 1993
Date Samples Received: Dec. 22, 1992
No. of **Samples**: 2
Lakefield Receipt No.: **9241469**
Page No.: 14

Attention: Mr. Mark Wiseman

CERTIFICATE OF ANALYSIS

"As received compost": Leachate Inorganic Extraction Data - Reg. 309

Analytes	Unit	Raw Uncut	Raw Uncut spiked
As	mg/L	<0.05	<0.05
Cd	mg/L	<0.005	0.24
Co	mg/L	<0.01	4.24
Cr	mg/L	K0.02	0.02
Cu	mg/L	a.02	0.27
Fe	mg/L	0.52	2.47
Hg	mg/L	<0.001	0.004
Mn	mg/L	5.00	19.5
Mo	mg/L	4.05	0.64
Ni	mg/L	a.02	7.43
Pb	mg/L	<0.05	0.17
se	mg/L	<0.01	<0.1
Zn	mg/L	0.13	87.3

Signed: _J. R Johnston_
J. R Johnston
Chief Chemist.

LAKEFIELD RESEARCH
A division of Falconbridge **Limited**
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Lakefield, ON **K0L 2H0   Phone** ▪ **705-652-3341 / Fax** ▪ **705-652-6365**

Falconbridge Limited	Date: Feb. **10,** 1993
Environmental **Services**	Date Samples Received: Dec. 22, 1992
Sudbury, ON POM **1S0**	No. of Samples: 2
	Lakefield Receipt No.: 9241469
	Page No. : 1 5

Attention: Mr. Mark Wiseman

<u>CERTIFICATE OF ANALYSIS</u>

"As received spiked compost":Leachate Organic parameters Extraction Data ▪ Reg. 309

		Raw
<u>**Analytes**</u>	<u>**Unit**</u>	
Aldrin	µg/L	0.6
Dieldrin	µg/L	<0.1
Chlordane*	µg/L	<3.4
DDT	µg/L	<0.1
Endrin	µg/L	0.4
Heptachlor epoxide	µg/L	0.6
Methoxychlor	µg/L	4.0
Toxaphene*	µg/L	<34
2,4-D	µg/L	4.5
Silvex	µg/L	3.6
PCB	µg/L	8.0

* Toxaphene and **Chlordane were found** to be **present,** however in this **instance** they coeluted and **could** not **be** separated **and quantified** to **levels lower than those indicated.**

Pesticide **and** PCB **analysis** were **conducted using** methods outlined by **the** MOE Method **PSAOC-E270A.** 1

Signed: _J. R̓ohnston_

J. R **Johnston**
Chief **Chemist.**

Appendix D: X-Ray Diffraction Analyses of Tailing

Table D. 1 X-ray diffraction analysis results for Nickel Rim oxidized tailing.

Start of Search/Match: 22-SEP-92   22:28:53 Peaks File: FALCOO
k2S
ERROR(DXE TAI 0.300

    250   PHASE(S   3 STRONGEST LINES   DATA BASE: 1   NUMBER OF OBSERVED LINES= 37
                MATCHED   NREQ= 0 NELEM=13   O NA MG AL SI P S K CA TI
RHO-WE-SEARCH

DMAX=   OP CUT OFF IS   320.
                DMIN= 1.541

JCPDS #	PHASE		OP	IC/IOB	PDF/MATCH	%PATTERN
33.1161 SI O2		QUARTZ, SYN	3.15	1.o	13/ 12	3 2
5. 490 SI O2		LOW	4.75	1.o	13/ 12	3 2
2.1280 NI S			8.01	0.0	4/ 3	8
28. 775 CA O			9.9h	071.o	18/ 118	21 21
14.47 FE3 P ONI5						
24.1069 NA2 CA SI O4			13.27	0.0	9/ 8	21
16, 270 NA2 O2			14.49	0.0	5/ 5	13
39, 742 NI .35 AL .30 TI .35			15.91	0.0	18/ 14	38
2. 951 NA2 CA (SI O4) .35			16.74	0.1	8/ 7	18
35. 40 CA3 (AL FE)2 OO			16.99	0.0	6/ 5	13
18: 894 MGFE2NI O4		MAGNESIGFERRITE, O	18.41	0.0	18/ 14	38
10. 170 CA MG SI2 O6		TETRATAENITE	18.91	1.o	16/ 12	32
6, 494 MG TI O3			18.54	0.1	3/ 3	8
26, 316 CA MN SI4 O10		GEIKIELITE, SYN	18.95	0.0	12/ 10	27
5. 582 MG2 P2 O7			19.57	1.o	26/ 20	52
2,1120 TI SI2			. 5	0.3	10.' 8	21
40, 994 K			20.23	0.1	5/ s	13
3, 650 K2 FE O4			21.75	0.0	4/ 4	10
18.1041 K MG (P O3)3			22.35	0.0	21/ 16	43
34. 192 FE AL2 O4			22.95	0.0	23/ 17	45
24, 901 K3 TI8 O17		HERCYNITE; SYN	24.11	0.1	7/ 5	13
38. 781 CA NI5			24.25	0.0	10/ 8	21
30,1221 NA2 MN7 SI10 O28			24.74	0.0	10/ 8	21
			25.45	0.0	10/ 7	
27:770 341 NI3 NA3P S2 O4			25.72	0.0	3/ 3	22
2. 520 (MG FE) 51 O3			2s.72	0.0	16/ 12	24
34, 529 FE		HYPERSTHENE	26.60	0.3	13/ 10	32
23.1364 K TI S2			28.34	0.0	18/ 13	38
			29.39	0.0	33/ 22	55
36, 742 CA2 MG NA) (EMG FE1 AL) SI2 O6		OMPHAC ITE	29.43	0.1	5/ 4	10
27, 522			29.49	0.3	43/ 27	71
1, 509 K			29.58	0.1	3/ 3	8
2, 647 CA2 P2 O7			30.22	0.2	3/ 3	8
30, 903 O2			30.31	0.3	23/ 16	43
27; 343 N I 2 .824 S2		NEPHELINE	31.95	0.0	5/ 4	13
9. 458 NA3 K AL04 SI4 O16		NEPHEL	32.19	0.0	41' 25	10
29. 881 MN2 AL04			32.94	0.3	27. 17	66
3,1069 NI2 SI			33.33	0.1	CL/ 15	45
28, 792 K4 P2 O7			33.46	0.0	20/ 14	40
39, 1 K FF SI O4						o
3, 943 NI2 SI			34.46	0.0		40
32.1124 NA2 MG SI O4			35.15	0.7	22/ 15	66
14, 152 CA4 TI3 O10			35.21	0.0	29/ 24	
30, 795 MG P4			35.53	0.0	9/ 7	18
3111264 NA AL P2 O7						83
40, 995 K		ANORTHITE, SODIAN	35.64	0.3	19/ 11	27
18,1202 (CA NA) (SI AL)4 O8			36.11	0.7	15/ 10	
37, 61 FE3 P O7		PYROXMANGITE	36.40	0.0	40/ 23	62
12, 432 MN SI O3						

kOS

Table D.2 X-ray diffraction analysis results for Nickel Rim non-oxidized tailing.

JCPDS #	PHASE		OP	IC/IOB	PDF/MATCH	%PATTERN
26, 920 K .6 NA .4 CL	/ SYLVITE . SODIAN. S		6.02	0.1	. 4	7
19 . 850 NI TI			2.55	0		7
9, 465 (CA NA) (SI AL)4 08	/ ANORTHITE. SODIAN.		12.89	1 .0	19/ 16	31
18.1202 (CA NA) (SI AL)4 08	/ ANORTHITE. SODIAN.		13.38	0.2	53 34	71
39. 145 CA TI 03			13.45			9
313.1397 FE SI	/ FERSILICITE. SYN [		13.91	0.3	8/ 6	1 1
5, 582 MG2 P2 07			14.05	0.1	11/ 10	19
30.1220 NA2 MN6 SI7 021			15.45	4	7/	11
33, 853 MG AL2 04			15.80	0.1	15/ 12	23
20, 554 NA AL SI3 08	/ ALBITE, ORDERED		16.36	1.0	65/ 43	83
.7, 35 AL CL3			17.30	0.1	2/ 7	13
20, 548 (NA CA) (SI AL)4 08	/ ALBITE, CALCIAN. D		18.47	1.0	66/ 44	83
18, 43 AL NI2 SI			19.16	3.0	7/ 7	13
21.1272 TI 02	/ ANATASE. SYN		19.35	0.	9/ 7	13
10, 359 .62 NA AL SI2 08 . .38 CA AL2 SI/ ANDESINE. LOW			13.66	1.o	21/16	31
10, 360 (CA NA) (SI AL)4 08	/ ANORTHITE , SODIAN ,		19.96	1.0	19/15	28
20, 572 NA AL SI3 08	/ ALBITE, ORDERED		20.06	1.0	70/ 45	3
40.1236 AL50 MN20 SI30			20.18	0.2	4/ 4	7
1, 850 NA			20.35	0 1	. / 4	7
14, 358 NI3-x S2			5 3	0.2	4/ 4	7
26,1069 CA1-x SI 03-x			20.93	0.2	19/1	23
38, 466 CA (MG FE AL) (SI AL)2 06	/ DIOPSIDE, ALUMINIA		21.06	0.1	66/ 43	83
13, 343 CA4 FE14 025			21.09	0.1	24/ 13	74
9, 467 .23 NA AL SI2 08 . .77 CA AL2 SI/ BYTOWNITE, LOW			21.27	1.0	19/ 15	28
1,1234 MN			22.07	0.1	6/ 5	9
19, 629 FE3 04	/ MAGNETITE. SYN		22.14	0.	9/ 7	13
17, 200 FE S	/ PYRRHOTITE 4C		22.18	0.1	64/ 42	8 3 '
21.1130 NA FE TI3 08			22.56	0.1	27/ 18	34
22. 358 FE1 x S	/ PYRRHOTITE 5C		77 on	0.1	8/ 6	11
9, 456 (NA CA) (SI AL)4 08	/ ALBITE. CALCIAN. 0		23.47	1.0	19/14	27
10, 339 NI AL2 04			23.89	0.1	7/ 5	9
33.1206 AL NA SI			24.25	0.0	20/ 15	28
21, 922 FE4 S5 021			24.43	0.1	30/ 21	40
22, 627 FE8 NI8 S16			24.74	0.2	9/ 7	13
38.1423 K AL SI2 06	/ LEUCITE. SYN		25.51	0.	46/ 42	83
18,1035 K MN 02			25.83	0.1	8/ 7	13
35, 479 (NA K CA)8 AL6 SI6 024 (S 04)2/ GIUSEPPETTITE			25.Y?	0.1	68/ 42	83
35, 485 NA3 CA P SI 07	/ CLINOPHOSINAITE		24.43	0.0	26/ 18	34
26.1371 NA CA AL SI2 07			26.61	0.1	33/ 22	41
4, 829 MG 0	/ PERICLASE. SYN		26.81	0.1	2. 3	5
18,1259 N A 3 P S 03			26.99	0.1	25/ 17	32
30 742 NI .35 AL .30 TI .35			27.13	0.3	10 / 6	15
12, 720 MN 02	/ GROUTELLITE. SYN		27.36	0.0	3/ 3	5
15, 460 CA- MG- FE- TI- AL SI 0	/ UNNAMED MINERAL [N		27.37	0.1	11/ 9	17
36, 691 NA (MG (CL 04)3)			27.79	0.1	15/ 12	23
20, 528 (CA NA) (SI AL)4 08	/ ANORTHITE. SODIAN.		27.85	1 .0	89/ 51	100
23, 671 NA2 CA3 SI6 016			27.97	3.1	31/ 20	38
35, 751 MN S 04			28.04	1.0	26/ 19	37
22,1320 SI P2 07			28.50	0.1	9/ 6	41
31,1056 K2 NI 03			28.75	4.0	44/ 11	21