The Effect of Processed Kimberlite Effluent From the Ekati™ Diamond Mine on Freshwater Zooplankton

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PREFACE

This thesis has been organized as a series of two manuscripts (Chapters 2 and 3) which will be submitted for publication in scientific journals after minor reformatting. Some repetition of introductory material is unavoidable.

ABSTRACT

Broken Hills Proprietary Company Ltd. (BHP; now BHP Billiton Inc.,) Ekati™ Diamond Mine, located approximately 300 km northeast of Yellowknife in Canada's Northwest Territories, began mining operations in the fall of 1998. This diamond mine has a life expectancy of at least 25 years, as more than five diamond bearing kimberlite pipes have been identified on BHP's claim block. Diamonds are extracted from kimberlite using chemical free processes that reduce the ore to fine particles (≤ 0.5 mm). Processed kimberlite effluent (PKE), which consists of these fine particles, water used during the extraction process, coagulants, flocculants, and treated sewage effluent, is disposed of in the designated containment facility, Long Lake. The Long Lake Containment Facility (LLCF) has been subdivided into five cells, of which the first four cells were designed to receive PKE and the last cell would act as a final monitoring stage before water was discharged to the downstream aquatic environment. The magnitude of effects PKE had on the ecosystem of the LLCF was evaluated through field assessments of chemistry and zooplankton community structure from August 1998 to August 2000. The toxic components of PKE were determined through a series of baseline toxicity tests and toxicity identification evaluations (TIE) using *Ceriodaphnia dubia*.

The loss of aquatic habitat, higher turbidity and significantly altered water chemistry resulting from PKE addition reduced zooplankton abundance in the receiving cell of the LLCF to less than 10% of values prior to PKE depositions. From August 1998, prior to the addition of PKE, to August 2000 total water hardness, alkalinity, total dissolved solids, pH, and total nitrogen significantly increased in all cells of the LLCF.

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In the cell farthest from PKE deposition, the concentrations of the following metals increased: barium from 6.6 to 24.3 μg/L; molybdenum from below detection (1.0) to 6.3 μg/L: strontium from 8.1 to 35.0 μg/L, and nickel from below detection (1.0) to 1.2 μg/L. Significantly greater increases in the concentration of these metals were noted in the cell directly downstream of PKE deposition.

Processed kimberlite effluent was chronically (7-d reproductive inhibition) toxic to *C. dubia* at concentrations as low as 12.5% effluent. The following Phase I TIE manipulations did not reduce the toxicity of PKE: ethylenediaminetetra-acetic acid (EDTA) and sodium thiosulfate addition tests, aeration, and solid phase extraction (C-18 column). Toxicity of PKE was significantly reduced with major pH (to 3 or 11) adjustments followed by filtration (0.45 μm). It is believed that the cationic polymer (Magna Floc®) was the toxic component since the estimated final concentration of the polymer in PKE was 1,000 times higher than the estimated 7-day EC_{50} to *C. dubia* of 0.014 mg/L. It is postulated that major pH adjustments altered the behaviour and electrostatic charges of the kimberlite minerals in solution in such a manner that excess cationic polymer became bound to the minerals and was no longer able to elicit a toxic response.

ACKNOWLEDGMENTS

DEDICATION

This thesis is dedicated to my parents, Pat and Sue Crocquet de Rosemond.

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ABBREVIATIONS

BHP	Broken Hills Proprietary Company Ltd. (now BHP Billiton Inc.,)
ANOVA	Analysis of Variance
CCME	Canadian Council of Ministers for the Environment
EC_{50}	Median Effects Concentration
EDTA	Ethylenediaminetetraacetic Acid
ICP-MS	Inductive coupled plasma-mass spectrometry
LC_{50}	Median Lethal Concentration
LLCF	Long Lake Containment Faclity
MPW	Mill Processed Water
NWT	Northwest Territories (also NT)
PKE	Processed Kimberlite Effluent
TIE	Toxicity Identification Evaluation
TSS	Total Suspended Solids
US EPA	United States Environmental Protection Agency

CHAPTER 1

INTRODUCTION

1.1   The Ekati™ Diamond Mine

In the fall of 1998, Broken Hills Proprietary's (BHP; now BHP Billiton Inc.) Ekati™ Diamond Mine, the first diamond mine in Canada's Northwest Territories, began mining operations. BHP's claim block for the mine is situated above the tree line, approximately 300 km north east of Yellowknife, NT, and is part of the Lac de Gras drainage basin (NWT Diamonds Project 1995). This pristine northern environment is characterized by numerous lakes, continuous permafrost, and vegetation and wildlife typical of the Canadian tundra. Initially, the life expectancy of the mine was projected to be about 25 years as five diamond rich kimberlite pipes (geological formations), Panda, Koala, Misery, Fox and Leslie, were identified on the claim block. Presently, BHP has identified at least another 130 kimberlite pipes; however, not all are diamond bearing nor may it be economically beneficial to mine some of the pipes. During the lifetime of the mine, Ekati™ will produce three to five million carats of industrial and gem quality diamonds per year, which amounts to approximately four percent of the current world diamond production by weight and six percent by value (http://www.ekati.ca).

The kimberlite pipes found on BHP's claim block are covered by small lakes.

These lakes are drained, then drilling and blasting are used to remove the overburden rock, mostly granites, that surround and cover the kimberlite pipes. Once the overburden is removed, open pit mining strips the kimberlite rock which is transported to a six-story processing plant.

At the time this project was initiated (October 14, 1998), BHP had just begun processing operations to extract diamonds from the kimberlite pipe known as the Panda pipe. Presently, the Ekati™ Diamond Mine processes approximately 9,000 tonnes of kimberlite per day, however, the plant is capable of processing 18,000 tonnes per day. Kimberlite is a relatively soft rock and only mechanical processes are required to extract diamonds (Howe 1997). Manual crushing is used to reduce the ore, which is then washed and separated based on size. Particles of 0.5 to 8.0 mm eventually end up at a coarse tailings collection site, while the processed kimberlite fines (particles ≤0.5 mm) are pumped, along with water used during the extraction process, to the designated containment facility, Long Lake (NWT Diamonds Project 1995).

The Ekati™ Diamond Mine was granted permission to sacrifice Long Lake for fine tailings containment purposes as part of its' Type "A" Water Licence (N7L2-1616). Processed kimberlite tailings will not be released into the aquatic environment outside the containment facility. However, the study of changes within Long Lake is highly relevant to the understanding of the toxicity of kimberlite and for the appropriate management of the processed kimberlite tailings. The release of potentially toxic kimberlite effluent (Harrison *et al.* 1995, 1997; Enviro-Test Laboratories 1998) to fragile aquatic ecosystems like those found in northern climates (O'Brien *et al.* 1992) may produce long-term effects that are detrimental to exposed aquatic organisms.

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Furthermore, there is concern that potentially toxic components of the processed kimberlite effluent may leach into the aquatic environment outside of the tailings containment facility. In essence, Long Lake is a large-scale bioassay that offers a unique opportunity to assess the effects that prolonged exposure to kimberlite tailings will have on aquatic organisms as well the sequence of ecosystem changes that may result from the addition of processed kimberlite to aquatic environments.

1.2   The Long Lake Containment Facility

Long Lake is used as a containment and settling facility for the kimberlite fines produced by the Ekati™ Diamond Mine processing plant. The aquatic ecosystem of Long Lake will unavoidably be destroyed as processed kimberlite tailings gradually fill the lake basin with solids over the life of the mine. The Long Lake Containment Facility (LLCF) will ultimately be reclaimed as a wetland upon final abandonment (NWT Diamonds Project 1995).

Long Lake was a typical oligotrophic tundra lake located centrally in the Koala Watershed, which forms a portion of the Lac de Gras watershed basin. A complete description of dyke construction and pertinent hydrology of the LLCF is given in the NWT Diamonds Project (1995). In summary, four semi-permeable rock dams have been used to subdivide Long Lake into a series of five cells, A through E (Figure 1.1). These intermediate cells are designed to confine the fine tailings and a high portion of the discharge water. Long Lake has undergone a de-watering program to reduce the water level and make room for the addition of effluent. Initially it was proposed that the basin of Long Lake be filled progressively from Cells A to D with tailings. However,

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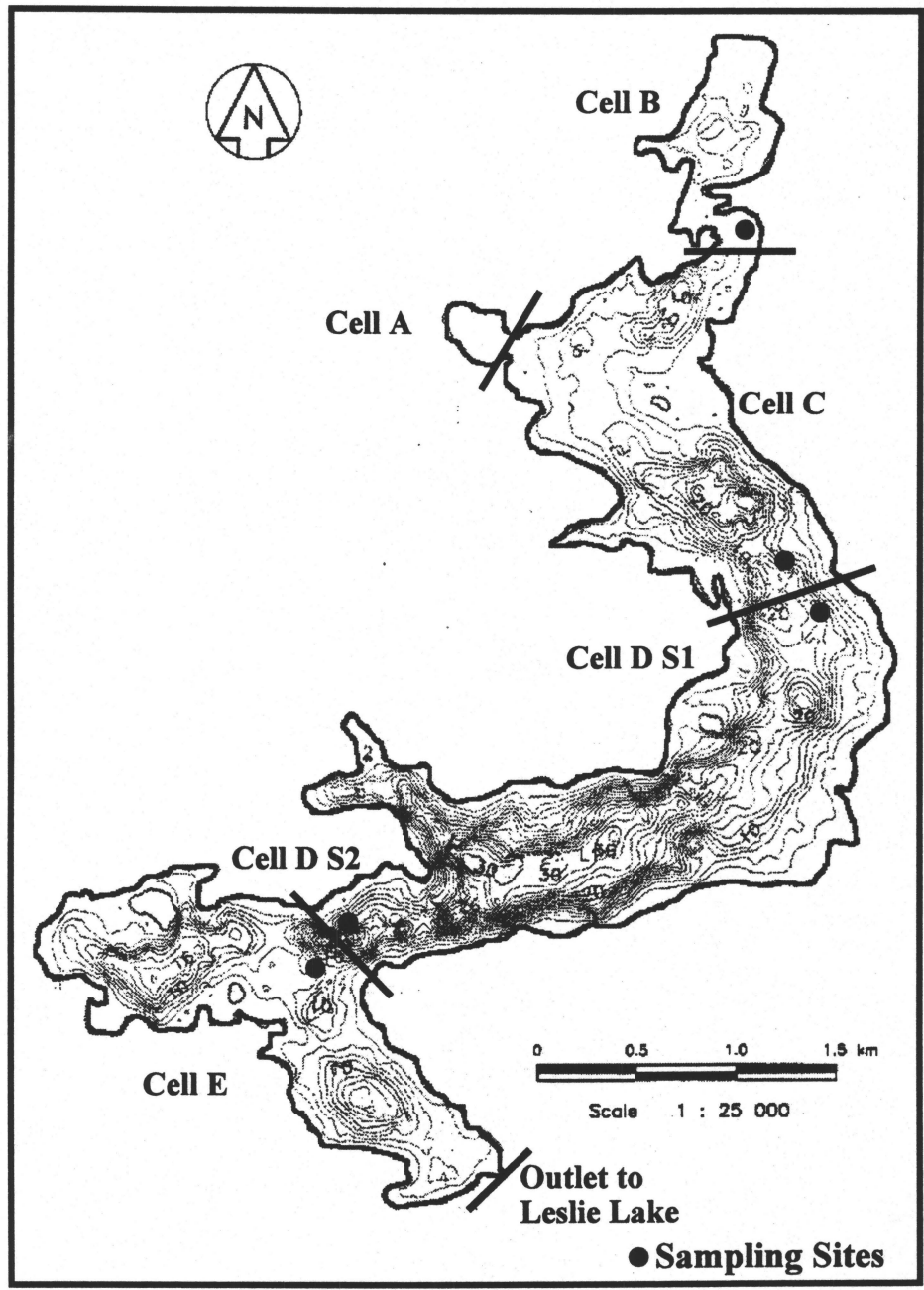
Figure 1.1. The Long Lake Containment Facility at the Ekati™ Diamond Mine.

Cell B has been the recipient of tailings effluent since the beginning of processing

operations in the fall of 1998. Sewage has been added to Cell B since the spring of

1999, when it was found that the proposed sewage facility, Kodiak Lake, was

undergoing eutrophication (Rescan 2000a). The outlet of Long Lake to Leslie Lake, the

next downstream lake, is located at the end of Cell E. Tailings effluent will not be

deposited in Cell E, rather this cell will be used to monitor water quality and to control

water volumes throughout the operations phase. An impermeable frozen core dam has

been constructed at the end of Cell E which will provide containment of the entire

tailings facility (NWT Diamonds Project 1995).

It is believed that the fines in the tailings effluent will settle on the lake bottom,

leaving the tailings water as a supernatant. This supernatant is expected to seep through

the semi-permeable dams, and presumably some of the toxicants that may be associated

with the effluent will be filtered out, degraded, or transformed as the waste water moves

through the various dams and cells. These dams are expected to provide full

containment of the tailings, however, when required, waste water levels in the tailings

recipient cell will be controlled by pumping water into the cell downstream. A gradient

of ecosystem effects should, therefore, be observable in the cells downstream of tailings

deposition. It is believed that all water leaving Cell E will meet water quality guidelines

set by the Canadian Council of Ministers for the Environment (CCME 1999) for

wastewater disposed into aqueous environments. If not, Cell E will act as a clarifier

pond, and water will be treated prior to discharge (NWT Diamonds Project 1995).

Cell C is unique from the other cells and may not reflect the progressive changes

in the LLCF that may occur as a result of the addition of tailings effluent to the upstream

cell, Cell B. Water from Koala Lake, which was being drained for mining purposes, as well as run off water from around the mining camp, were initially pumped to Cell C. The raw intake water for the processing plant and other mining operations will be located at Cell C for the first 5 years of operation, and then moved to Cells D and E as the upstream cells become filled with tailings (NWT Diamonds Project 1995). Furthermore, throughout this study, tailings effluent was sporadically pumped to Cell C (Ekati™ Diamond Mine 2000).

1.3   Processed Kimberlite Effluent

The fine tailings effluent that is pumped to the LLCF will be referred to as processed kimberlite effluent (PKE) for the duration of this thesis. PKE consists of: fine kimberlite tailings that have passed through the processing plant; coagulating and flocculating polymers; wastewater that has been used during the diamond extraction process; and treated sewage effluent. This section contains a description of the following: the mechanical processes that are used to extract diamonds from kimberlite; the geological formation of kimberlite pipes and the components of kimberlite ore; the use and types of coagulating and flocculating polymers; and the type of treated sewage effluent the Ekati™ Diamond Mine produces.

1.3.1   Production of Processed Kimberlite Effluent

The recovery of diamonds from kimberlite is a chemical-free process that can be divided into three basic steps: crushing and washing, primary concentration, and secondary concentration. The following summary of diamond extraction from

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kimberlite is taken from the Ekati™ Diamond Mine (2000) report. First, crushing and washing reduce the ore to particles that are either coarse (0.5 to 8 mm) or fine (≤ 0.5 mm). During these processes, the smaller (≤ 0.5 mm) particles are continuously removed to the deep cone thickener. Prior to entry to the deep cone thickener, coagulating and then flocculating polymers are added to the fines to facilitate particle aggregation. The deep cone thickener has two purposes: to condense the fine particles into a sludge, which will facilitate settling of the fines in the LLCF, and to clarify water used during the extraction processes thus reducing waste by recycling the water used in the plant. In the deep cone thickener, particles flocculate until a critical mass is reached at which they are no longer held in suspension and will sink to the bottom. Self-weight compaction decreases the water content of the sludge as it is drawn from the bottom of the cone. This sludge is the main component of PKE and is eventually pumped to the LLCF.

Once the kimberlite has been reduced, primary separation of the diamonds from the ore is accomplished through heavy medium separation. Most of the particles entering the heavy medium separator are between 1 and 25 mm. In this process, gravity is used to separate the high density particles, such as the diamonds, from the lower density kimberlite particles. A slurry of water and ferro-silicon is used to facilitate separation of particles with different densities. The heavy medium separator uses centrifugation to create a gravity field powerful enough to separate the high density particles, and the ferro-silicon medium, from the lower density particles. The lower density particles are divided into two groups; those which end up at the coarse tailings site, >0.5 to 8.0 mm, and those >8 mm, which are recycled through the processing plant

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for possible diamond recovery. The ferro-silicon slurry is recovered on a magnetic separator and recycled for use in the heavy medium separator.

Secondary concentration is used as the final stage in the diamond extraction process. The separation of diamonds from the rejected mineral material occurs through wet high density magnetic separation in conjunction with an attrition mill. The attrition mill will break down heavy minerals without damaging the diamonds, while the magnetic separator safely removes all diamond-like material, including garnets. Finally, diamonds are luminescent when irradiated with X-rays. This unique property of diamonds is exploited in the last step of diamond extraction from the kimberlite ore.

The last two steps involving concentration processes do not generate fine tailings to the extent that the washing and crushing processes do, however, recycling of the ≤ 8 mm particles does generate some fine tailings. It is noted that approximately 70% of the kimberlite entering the processing plant exits as fine tailings in the form of PKE. Processed kimberlite effluent is comprised of a ratio of approximately 40% fine tailings to 60% wastewater (Ekati™ Diamond Mine 2000).

1.3.2   Kimberlite Ore

Kimberlite is a rare volcanic rock that originates 150 km or more beneath the earth's surface. As magma ascends to the surface it brings up diamonds which are found at similar depths. The formation of diamonds is not contingent upon the presence of kimberlite; rather the magma serves as a vehicle which conveys diamonds to the earth surface (Kjarsgaard 1996).

Kimberlite is a highly reactive, ultrabasic rock that is thought to form as a result

of partial melting of carbonate-enriched peridotitic rock at high pressure (Kjarsgaard 1996). Common minerals found in peridotite are magnesium-rich olivine (Mg_2SiO_4), calcium-rich clinopyroxene ($CaMgSi_2O_6$) and magnesium-rich orthopyroxene ($MgSiO_3$). Olivine is the most common and characteristic mineral of kimberlite. Other indicator minerals of kimberlite include spinels, magnesium-rich ilmenite, chromium-rich diopside, calcite, phlogophite, serpentine, monticellite, apatite, and perovskite (Mitchell 1986). During ascent to the earth's surface, the kimberlite magma may encounter different mantle rocks that may become incorporated, but not assimilated, into the ore. These foreign rock fragments, also called xenoliths, may not survive to earth's surface, however, individual mineral grains, or xenocrysts, may be present in the kimberlite ground mass. The presence of different xenoliths, xenocrysts and/or macrocrysts (a non-genetic term for large crystals of unknown origin, 1-20 cm) in kimberlite magma forms kimberlite pipes with unique mineralogical content (Mitchell1986; Kjarsgaard 1996). Specific diamond indicator minerals such as garnet, ilmenite, chromite, and pyrope are present due to xenolith incorporation into ascending kimberlite magma (Kjarsgaard 1996).

At the earth's surface, the kimberlite will undergo chemical and geological processes which alter the minerals present in the ore. During the late stages of magna consolidation, kimberlite may undergo deuteric and metasomic alterations. Olivine may undergo deuteric alterations to serpentine, which, in turn, may metamorph to calcite, phlogopite and magnetite. Metasomatic reactions such as serpentization, carbonation, and chlorization may occur during and after pipe emplacement (Mitchell 1986). Kimberlite alterations and weathering at the surface produce more than 70 secondary

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mineral products, including serpentine, talc, chlorite, saponite, dolomite, micas, brucite, diopside, monticellite, quartz, barite, pyrite, biotite, and clay minerals such as kaolinite, illite, and montmorillonite (Mitchell 1986; Hodgson and Dudenay 1988).

Each kimberlite pipe present at the Ekati™ Diamond Mine is mineralogically unique (NWT Diamonds Project 1996). The dominant minerals identified in Panda, Fox, Leslie, Koala and Misery Pipes include olivine, phlogopite, garnet, diopside, calcite, quartz, feldspar, serpentine, montmorillonite, and clinochlore (Howe 1997). The distribution of minerals within and among the pipes can vary greatly; for example olivine content in individual pipes ranged from 26 to 87% (Howe 1997). Panda Pit, in mining operations for the duration of this project, contained the highest concentration of olivines, up to 81%, while other minerals were present in concentrations of 5% or less of the total mineralogical content of the kimberlite (Howe 1997).

Olivines are well known for their acid neutralizing capability and their potential to neutralize acid mine waste (Schuiling et al. 1986; van Herk et al. 1989). In slightly acidic aquatic environments kimberlite minerals will dissolve into the free magnesium ion (Mg^{2+}), calcium ion (Ca^{2+}), hydroxide ion (OH^+) and silica (SiO_2) (Hodgson and Dudenay 1988). In acidic environments, free magnesium ion will combine with hydroxide and SiO_2 to form serpentine [$H_2Mg_3Si_2O_9$]. The dissolution of olivine will drive the pH of the water up as free H^+ ions are consumed. In conjunction, the formation of calcium and magnesium carbonates will remove all carbonate species present in solution, thus causing an increase in free hydroxide (Hodgson and Dudenay 1988; van Herk et al. 1989; Wogelius and Walther 1991; Howe 1997). In more alkaline environments, the magnesium cation will combine with hydroxide to form brucite

[Mg(OH)$_2$] (van Herk *et al.* 1989; Wogelius and Walther 1991) .

Experiments were conducted by Howe (1997) to determine the acid neutralizing properties of the kimbelite ore from the Ekati™ Diamond Mine site. The dissolution of olivines in crushed kimberlite will cause a steady pH increase from 6.2 to 8.7 in distilled water over a 96 h period. Values of up to pH 9.4 can be accounted for by the dissolution of olivines in the kimberlite ore in an aqueous environment. The increased alkalinity, pH >10, is assumed to be the result of the formation of calcium hydroxide after the magnesium ion has precipitated out of solution as magnesium hydroxide (Mg(OH)$_2$). Howe (1997) postulated that the deposition of PKE into the LLCF would raise the pH of the water from slightly acid to a more alkaline pH as the kimberlite minerals undergo hydroysis.

There was also concern that the dissolution of kimberlite minerals could cause an increase in the concentration of metals in the LLCF to levels that would eventually exceed water quality guidelines. Chemical leach tests indicated that Mg^{2+} and Ca^{2+} readily leached into solution from kimberlite minerals from the Ekati™ Diamond Mine, although not all pipes leached these metals at the same rate (Howe 1997). Furthermore, it was postulated that recycling water through the processing plant during the extraction processes would only add to the concentration of metals in PKE (Howe 1997).

The processing of kimberlite produces fines that are remarkably stable mineral suspensions presenting a serious challenge for waste removal treatment (Howe 1997). In solution, very small kimberlite fines, also known as colloidal particles, have a negative charge which makes these fines resistant to settling (Howe 1997; O'Gorman and Kitchener 1974). Colloidal particles are so small that Brownian Motion and

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thermal convection currents tend to keep them dispersed, thus preventing removal

through settling (O'Gorman and Kitchener 1974; Penniman 1981). Furthermore, charge

repulsion effects are noticeable with very small particles since the ratio of surface

area:mass, and therefore charge:mass, is greatest. In aqueous environments with pH

>4.0, colloids tend to have a net negative charge and are surrounded by a cloud of

oppositely charged ions. This ionic cloud, also called an electric double layer, has an

inner region which may include adsorbed ions and a diffuse region in which ions are

distributed according to electrical forces and thermal motion. Electrostatic forces bind

the absorbed ions to the surface of the particle, while the diffuse layer is more likely to

move within the solution. The plane of separation between the attached and diffuse

layer of ions creates the surface of shear. At the surface of shear the zeta potential can

be measured. The zeta potential is the measure, in millivolts, of the electrostatic

charges of particles suspended in liquid. The more negative the zeta potential, the

stronger the electrostatic forces that cause these particles to repel one another (Penniman

1981). Electrostatic attraction of these ions keep the particles in constant motion which

will retard settling as well as prevents natural particle attraction through Van der Waal's

forces (O'Gorman and Kitchener 1974).

The kimberlite fines produced by the Ekati™ Diamond Mine have a zeta

potential between -15 and -35 mV (Howe 1997). Research conducted by Howe (1997)

indicated that the kimberlite fines would form stable suspensions in the LLCF and that

water discharged from Cell E of the LLCF may not meet guidelines without treatment to

enhance particle settling. Furthermore, without the addition of polymers, metal cations

are more likely to leach from the kimberlite tailings and will, in all probability, exceed

water quality guidelines (Howe 1997; CCME 1999). The application of synthetic polymers to PKE will increase the efficiency of solid-liquid separation, thus reducing turbidity and facilitating settling of fines in the LLCF (Howe 1997).

1.3.3 Coagulating and Flocculating Polymers

Coagulating and flocculating polymers are added to wastewater to facilitate settling of suspended solids (O'Gorman and Kitchener 1974; Siyam 1997). These polymers, also called polyelectrolytes by water treatment facilities, destabilize suspensions by overcoming the repulsive electrostatic forces between particles which allows the particles to adhere more readily to each other through Van der Waal forces. The polymers used in treating wastewater from mining industries have a two-fold purpose: the condensation of suspended solids, thus a reduction in waste treatment space (O'Gorman and Kitchener 1974), and the recycling of water within the industry, thus a reduction in water consumption (Siyam 1997).

Polyelectrolytes used in water treatment facilities fall into three groups: non-ionic, cationic, and anionic, depending upon the nature of the residual charge on the polymer in aqueous solution (Siyam 1997). The most versatile and useful polymers are cationic polyelectrolytes which function primarily as coagulants. Non-ionic and anionic polyectrolytes are either referred to as coagulant aids or flocculants. Polymers used in water treatment facilities have been developed based on the charge along the polymer chains and the resultant water solubility (Siyam 1997). The wide range of uses for polymers in a number of different waste treatment facilities is due to the development of different polymers, with different ionic nature and proportion of ionic charge density

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(Siyam 1997).

Coagulation is the process by which the electrostatic surface charges of colloids are neutralized through the adsorption of the polymer to the surface of these particles. In order to achieve coagulation in most systems the zeta potential must be reduced to less than -10 mV (Schlauch 1981). Cationic polymers are added to colloid suspensions to neutralize the repulsive electrostatic charges which prevent these particles from coming in contact with one another. Once colloids are close enough for contact to occur chemically attractive forces, such as Van der Waal forces, will bind the particles together.

Anionic or nonionic polymers are added to colloidal suspensions to induce Van der Waal attraction of particles which results in increased efficiency of flocculum formation. Flocculation describes the process by which high molecular weight polymers physically form a bridge between two or more particles into a random, three-dimensional structure which is loose and porous. Flocculation occurs as the polymer is adsorbed by ionic bonding to the particle to form interparticle bridges that draw particles together into aggregates of higher molecular weight that rapidly settle out (Schwoyer 1981).

Experiments conducted by Howe (1997) on crushed kimberlite particles demonstrated that the ore from each pipe required different types and concentrations of both flocculants and coagulants to facilitate particle settling. Though other coagulating and flocculating polymers were tested for use, Percol® 368 and 156 were the best suited for settling kimberlite fines at Ekati™ (Howe 1997). Percol® 368, a cationic polyelectrolyte, adequately cleared suspensions of Panda kimberlite fines at a rate of 23

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g/t whereas Misery fines required the addition of 525 g/t. Generally, it was found that Percol® 156, an anionic co-polymer, removed suspended material at a rate of approximately 50 g/t, with the exception of Misery kimberlite fines which required 125 g/t.

Information regarding the addition of coagulating and flocculating polymers to the kimberlite tailings at the Ekati™ Diamond Mine was supplied through a personal communication with the Plant Metallurgist, Curtis Mohns (2000). Percol® 156 and 368 are the same chemicals as MagnaFloc® 156 and 368 (Ciba Speciality Chemical, Inc., Suffock, VA, USA). MagnaFloc® 368 is dispensed at a rate of 35 g/t upstream of the flocculant to kimberlite fines through a tailings lauder. MagnaFloc® 156 is added to the kimberlite fines at a rate of 55 g/t upstream of the deep cone thickener. These rates are an approximation of the actual dosages. According to the Ekati™ Diamond Mine report (2000), dosages may vary according to the type of ore being processed, as well as to optimize thickening performance and achieve the desired solids content. The kimberlite ore from Panda pit has been separated into "Typical" Panda and "Fox-like" Panda. The estimated dosage rates for MagnaFloc® 368 are 35 g/t for "Typical" Panda, and 90 g/t for "Fox-like" Panda.

1.3.4   Sewage

The Ekati™ Diamond Mine disposes of sewage produced by the camp into the LLCF. The volume of sewage produced by the Ekati™ camp per year is estimated to be 40,000 m^3, with an estimated biochemical oxygen demand (BOD) of 600 mg/L. The sewage is subjected to both primary and secondary levels of treatment in a sewage

treatment facility that was built in 1997. With the designated sewage treatment, sewage effluent discharged to the LLCF should have a BOD ≤40 mg/L and total suspended solids (TSS) ≤60 mg/L. The treated sewage effluent is pumped to the processing plant where it is mixed with processed kimberlite fines that are being pumped to the LLCF (Ekati™ Diamond Mine 2000).

1.4   Potential Toxicity of Processed Kimberlite Effluent to Aquatic Organisms

The existence of potentially toxic constituents in kimberlite solutions (Harrison *et al.* 1995, 1997; Enviro-Test Laboratories 1998) led to the initiation of this research project. There was concern that PKE could potentially contain toxic components and that these toxicants would filter through the semi-permeable rock dams and affect aquatic ecosystems downstream of the LLCF. Although assessments have been conducted to evaluate the toxicity of individual components of PKE, at the time this project was initiated this was the only study underway to investigate the toxicity of whole PKE to aquatic organisms. Initial investigations indicated that both the kimberlite ore in solution (Harrison *et al.* 1995, 1997; Enviro-Test Laboratories 1998) and the cationic polymer (Murgatroyd *et al.* 1996; HydroQual Laboratories Ltd. 1995) were toxic to aquatic organisms.

1.4.1   Toxicity of Kimberlite Solutions to Aquatic Organisms

Preliminary toxicity tests conducted by the Department of Fisheries and Oceans (DFO), Winnipeg, MB, determined that toxicity of kimberlite solutions to fish was dependent upon the pH and the concentration of total suspended solids (TSS) (Harrison

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et al. 1995). Further investigations indicated that nickel and pH-dependent toxicants such as ammonia and aluminum could be the primary toxic constituents in the kimberlite solutions (Harrison *et al.* 1997). Toxicity tests conducted by Enviro-Test Laboratories (1998) confirmed that the toxicity of kimberlite solutions to freshwater organisms could be reduced through the elimination of TSS. These kimberlite solutions were not true representations of PKE as neither the polymers, recycled process water, nor the treated sewage effluent were present. However, evaluating the toxicity associated with these kimberlite solutions gave a framework from which PKE toxicity could be evaluated.

Using kimberlite from two different sources, DFO created two kimberlite solutions called Misery and Fox, named after the respective kimberlite pipes from which the ore was extracted. These solutions were thought to effectively mimic the kimberlite tailings effluent that later would be produced by the Ekati™ Diamond Mine processing plant (Harrison *et al.* 1995). The Fox solution was tested as "whole" Fox (unmanipulated), "filtered" Fox (0.2 μm filter) and "clarified" Fox (TSS were settled out). The Misery solution consisted of water and fine solids from the return flow drilling of Misery Lake kimberlite. Total and dissolved metal concentrations were determined for both Fox and Misery solutions (Table 1.1). Total metal concentrations were higher than dissolved, filtered and clarified metal concentrations by orders of magnitude (Table 1.1). Compared to Winnipeg tap water, Fox solution contained high concentrations of total ammonia, nitrite, nitrate, total dissolved nitrogen, dissolved inorganic carbon, and had high conductivity (Table 1.2) (Harrison *et al.* 1995).

Acute toxicity tests were conducted using fingerling fish of the following

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Table 1.1. Total and dissolved metal concentrations in Fox and Misery kimberlite solutions[a].

Metal	Fox Solution (Total)	Fox Solution (Dissolved)	Fox Solution (Filtered)	Fox Solution (Clarified)	Misery Solution (Total)	Misery Solution (Dissolved)
Aluminum (μg/L)	14800	2090	84	27	3116	122
Barium (μg/L)	1390	<1	30	37	2260	298
Calcium (mg/L)	49.7	6.4	3.59	8.49	119.5	7.4
Cadmium (μg/L)	0.55	<0.05	0.1	<0.05	0.98	0.25
Cobalt (μg/L)	70.1	19.1	0.4	<1.0	75.2	0.7
Chromium (μg/L)	69.6	34.3	1.7	1.1	154.6	1.8
Copper (μg/L)	1.1	1.4	111.9	3.4	39.3	4.4
Iron (μg/L)	34500	4100	<40	48	18400	260
Potassium (mg/L)	32.7	22.7	22.2	20.74	12.9	6.4
Magnesium (mg/L)	50.7	11.9	0.71	1.88	73.72	17.3
Manganese (μg/L)	1620	67	<20	<20	1687	29
Sodium (mg/L)	1265	926	129	130.2	45.7	2.97
Nickel (μg/L)	26.32	231	14	22	1317	15.4
Lead (μg/L)	0.2	<0.1	0.8	0.6	2.3	<0.1
Zinc (μg/L)	171	14	<5	<5	103	<5

[a]Taken from Harrison et al. 1995. An aliquot of kimberlite solution was centrifuged to remove suspended solids before acidification to give *dissolved* metal concentrations, while another aliquot was centrifuged after acidification to give *total* metal concentrations. Whole Fox kimberlite solution was *filtered* through 0.2 μm filters, or *clarified* in the Koala mill to remove suspended solids. Analysis was completed with either flame or graphite furnace atomic absorption or plasma emissions spectroscopies depending upon metal concentrations present in the material.

Table 1.2. Non-metal constituents of Fox and Misery kimberlite solutions and Winnipeg tap water[a].

Non-metal Constituents	Winnipeg Tap Water[b]	Whole Fox Solution	Filtered Fox Solution	Whole Misery Solution
Nitrate (μg/L)	193	3678	4100	2
Nitrite (μg/L)	<1	567	700	2
Total Ammonia (μg/L)	10	830	575	337
Total Dissolved Nitrogen (μg/L)	330	5670	5175	620
Dissolved Inorganic Carbon (μm/L)	1680	4525	5250	1317
Dissolved Organic Carbon (μm/L)	550	928	890	300
Chloride (mg/L)	4.81	7.67	1.99	1.7
Sulphate (mg/L)	3.21	15.6	4.57	38.1
Conductivity (μs/cm)	183	650	646	211
Total Suspended Solids (mg/L)	<1	2761	12	183
Hardness (mg $CaCO_3$/L calcium)	90	na[c]	12	na
Alkalinity (μep/L)	1599	na	4897	1274

[a]Taken from Harrison et al. (1995). Whole fox solution was produced by processing kimberlite ore in the Koala mill. Whole Misery solution consisted of water and fine solids from the return flow drilling of Misery Lake kimberlite. Filtered fox solution is whole Fox kimberlite solution filtered through 0.2 μm filters.
[b]Winnipeg tap water represents a sample taken January 5, 1995.
[c]Not available.

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species: rainbow trout (*Oncorhynchus mykiss*), lake trout (*Salvelinus namaycush*), and lake whitefish (*Coregonus clupeaformis*), the latter two were native to the Lac de Gras region (Harrison *et al.* 1995). Whole Fox solution was the most acutely toxic to all three fish species tested; the median survival time (MST) of the most sensitive species, lake whitefish, was 2.5 h. Misery solution was not acutely toxic to rainbow trout exposed for 48 h, however, whitefish had a MST of 3.9 h. Neither clarified nor filtered Fox solution were acutely lethal to rainbow trout. Reducing the pH from ~9.5 to 7.9 for whole Fox solution significantly reduced toxicity to exposed rainbow trout. Histological examination of gill filaments from all three fish species exposed to whole Fox solution indicated that concentrations of aluminum, barium, chromium, iron, nickel, cobalt, magnesium, and calcium averaged between 6 and 85 times higher than controls. Gill filaments from rainbow trout exposed for 48 h to clarified or filtered whole Fox solution did not have an increase in metal concentrations. These results indicated that toxicity of kimberlite solutions to fish was pH dependent, and could be reduced by removing suspended solids; filtration reduced the pH of Fox solution (Harrison *et al.* 1995). Harrison *et al.* (1997) concluded that both ammonia and aluminum were present in high enough concentrations in whole Fox solutions to be partially responsible for the acute toxicity observed to fish exposed at pH 9.2. Filtration reduced the pH of Fox solution to pH 8, and thereby reduced the toxicity of the pH-dependent toxicants, aluminum and ammonia. Lastly, there was concern that nickel was present at concentrations which could contribute significant stress to lake trout over longer periods of exposure (Harrison *et al.* 1997).

Enviro-Test Laboratories (1998) also determined that the toxicity of kimberlite

solutions could be significantly reduced through the removal of TSS. A stable suspension of TSS was created using kimberlite from both Panda and Fox pipes. At the highest achievable concentration of TSS in Panda solution, 200 mg/L, acute toxicity to rainbow trout, *Daphnia magna*, and *Selenastrum capricornutum* was not observed. Conversely, Fox solution was acutely toxic to rainbow trout (96-h LC_{50}: 667 mg/L), *D. magna* (48-h LC_{50}: 7,305 mg/L) and *S. capricornutum* (72-h LC_{50}: 6,050 mg/L). The toxicity of Fox solution to these organisms was attributed to the high concentration of stable TSS that was used for toxicity testing purposes. Both Panda and Fox solutions were chronically toxic to *Ceriodaphnia dubia* (7-d reproductive study); the respective IC_{50}s were 81 and 55 mg/L. Enviro-Test Laboratories (1998) confirmed through a series of Toxicity Identification Evaluations that acute toxicity of these kimberlite solutions could be significantly reduced by eliminating the TSS.

1.4.2 Polymer Toxicity to Aquatic Organisms

The chemistry, molecular weight and charge density of synthetic polymers vary significantly from compound to compound, making it difficult at best for publically owned treatment works to determine non-toxic concentrations. Moreover, the majority of the information on polymers is proprietary and, therefore, very little to no toxicity data exist for many of these compounds (Rowland *et al.* 2000). Generally, cationic polymers are more toxic to aquatic organism than anionic and non-ionic polymers (Goodrich *et al.* 1991; Cary *et al.* 1987; Biesinger and Stokes 1986; Biesinger *et al.* 1976). Murgatroyd *et al.* (1996), realizing that toxicity of these polymers was dependent upon test species, exposure durations, endpoints, and tests conditions,

calculated median EC_{50}/LC_{50} estimates based on available data to evaluate polymer toxicity to aquatic organisms. The median EC_{50}/LC_{50} values for fish and invertebrates exposed to cationic polymers were 0.89 and 3.9 mg/L, respectively, whereas the values for anionic polymers were 37.2 and 345 mg/L, respectively (Murgatroyd *et al.* 1996).

The toxic effects of cationic polymers to aquatic organism are attributed to interactions of the polymers with the surfaces of aquatic organisms, rather than with systemic toxicity (Murgatroyd *et al.* 1996). The surface of fish gills, for example, carries a negative charge. Cationic polymers will readily bind to the surface of fish gills as a result of electrostatic attraction, which may cause fusion of gill lamellae and/or interlamellar cell proliferation. It is likely that mortality is a result of suffocation and/or a disruption of internal ionic balance caused by a reduction in oxygen transfer across gill membranes (Biesinger and Stokes 1986). The mechanism of toxicity of cationic polymers to invertebrates is slightly different. It is thought that the polymer binds to the surface of the integument and/or to appendages, thus inhibiting movement and the subsequent uptake of nutrients (Rowland *et al.* 2000). The high molecular weight of most polymers (>10,000) makes it highly unlikely that they will cross biological membranes to elicit toxic effects. Furthermore, the octanol-water partition coefficient of five cationic polymers was found to be less than 1.0, indicating little tendency to partition from the water into lipid phases, even if transfer across biological membranes was possible (Murgatroyd *et al.* 1996).

Toxicity assessments of the polymers Magna Floc® 156 and 368 using *O. mykiss* were conducted by HyrdoQual Laboratories Ltd. (1995). Static, acute toxicity tests indicated that the 96-h LC_{50} was 1,812 mg/L for the anionic polymer, Magna Floc® 156,

and 0.38 mg/L for the cationic polymer, Magna Floc® 368. Polymers of opposite ionic character will bind to each other in solution, and thus reduce toxicity by decreasing the bioavailability of the more toxic cationic polymer (Biesinger and Stokes 1986). Combining the two polymers at a rate of two parts Magna Floc® 156 and to one part Magna Floc® 368 increased the 96-h LC_{50} for Magna Floc® 156 to141 mg/L, and reduced the 96-h LC_{50} for Magna Floc 368 to 71 mg/L (HyrdoQual Laboratories Ltd. 1995).

The contribution of polymer toxicity to the Ekati™ PKE was thought to be negligible. The underlying assumption was that PKE, as a highly turbid solution of approximately 40% solid particles, should bind all the synthetic polymers available in solution. In pure water, polymers are known to be more acutely toxic to aquatic organisms than in waters with a high degree on suspended solids (Goodrich *et al.* 1991; Cary *et al.* 1987; Biesinger and Stokes 1986). Polymers by nature will adsorb strongly and irreversibly onto dissolved organic matter and suspended solids in solution and, therefore, should not be available to affect aquatic organisms (Goodrich *et al.* 1991; Cary *et al.* 1987).

1.5   Assessment of Effluent Impact on Aquatic Ecosystems

There are two main types of endpoints that can be used to evaluate the effects that contaminants may have on aquatic ecosystems (Cairns *et al.* 1995). The first is structural endpoints, which include changes in diversity, evenness, species richness, species abundance, and community similarity. The second is functional endpoints which measure the rate of biomass production, community respiration, nutrient

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uptake/regeneration, decomposition, and recovery after stress (Cairns *et al.* 1995).

In aquatic ecosystems, one of the earliest, and most sensitive, indicators of anthropogenic stress is changes in species composition of small, rapidly reproducing organisms, such as zooplankton (Schindler 1987). Furthermore, zooplankton assemblages provide useful information for assessing water quality (Gannon and Stemberger 1978). Community level studies provide information about the biological integrity of an aquatic system, and allow individual evaluation of a species within the ecosystem (Linthurst *et al.* 1995). Analysis of zooplankton species richness, diversity, abundance and community structure are all structural endpoints that should help evaluate the magnitude of effects contaminants will have on aquatic organisms (APHA 1998). Chlorophyll *a* concentration is a good measurement of the degree of primary production in an aquatic ecosystem (Cairns *et al.* 1995).

1.6   Methods to Assess Effluent Toxicity

The US EPA (1991) recommends that a series of baseline toxicity tests using *C. dubia* be used to determine the toxic nature of effluents to freshwater zooplankton. Establishment of either acute or chronic toxicity of an effluent often leads to the initiation of Toxicity Identification Evaluations (TIEs) procedures. TIEs were designed to determine the class(es) of chemical and physical components of effluents, ambient waters, sediments pore and elutriate waters, and leachates that may be responsible for the observed adverse effects to aquatic organisms. The TIE is divided into three phases: Phase I uses a series of manipulations designed to determine the class of toxicant present; Phase II aims to determine the identity of the toxicant; and Phase III confirms

that the toxicant was responsible for the observed toxicity (US EPA 1991).

Phase I of TIEs is a series of chemical and physical manipulations which are used to characterize whether toxic constituents are volatile, chelatable, filtratable, reducible, non-polar, or pH sensitive (US EPA 1991). This initial toxicity characterization is a two-tiered approach. Tier 1 consists of filtration, aeration and minor pH adjustments, or uses additives to chelate or reduce toxicants, or the uses separation techniques such as solid phase extraction with C-18 resin columns (US EPA 1991). Tier 2 uses aeration, filtration, and C-18 solid phase extraction in conjunction with major pH adjustments (to pH 3 or 10) (US EPA 1991). The following Phase I procedures are established methods used to characterize the class of toxic component(s). Filtration identifies toxicants that may be associated with suspended solids or removed as particle-bound toxicants, but provides little specific information on nature of the toxic compounds. The addition of ethylenediaminetetra-acetic acid (EDTA) will identify if toxicity is caused by certain cationic metals (e.g. nickel, zinc, aluminum^{3+}, lead, copper, manganese^{2+}, and iron). The addition of sodium thiosulfate will reduce the toxicity of oxidative compounds, such as chlorine, bromine, iodine, and chlorine dioxide (US EPA 1991). Sodium thiosulfate is also effective at reducing toxicity of common metals such as Ag, Se(VI), Cu, Hg, Mn, and Cd (Hockett and Mount 1996). Toxic compounds that are volatile, spargeable, sublatable and oxidizable can be removed through aeration. Graduated pH adjustments, or minor pH adjustments, will identify the presence of pH-dependent toxicants (e.g. aluminum and ammonia). A reduction in toxicity through solid phase extraction using C-18 columns suggests that non-polar organics may an important component of the toxic fraction (US EPA 1991).

Characterization of the class(es) of toxicant(s) leads to the initiation of Phase II and III of the TIE. In Phase II, further toxicity testing and analytical methods are used to identify the specific compound(s) and their concentrations in the samples tested. In Phase III, the identified toxicant is removed from the effluent sample and subsequently added back to confirm that toxicity was induced by the specific compound(s) (US EPA 1991).

The Phase I TIE test protocols for characterization of toxic constituents in effluents that display acute and chronic toxicity are the essentially the same; they differ only in test duration and endpoint (US EPA 1991). Acute toxicity refers to tests that run for 96 h or less and utilize lethality as an endpoint, whereas exposure for chronic toxicity tests is longer (>96 h) and uses survival, growth and fecundity as endpoints (US EPA 1991). The two most common species used in TIE investigations are the waterflea (*C. dubia*) and the fathead minnow (*Pimephales promelas*).

1.6.2 *Ceriodaphnia dubia* as a Laboratory Test Organism

The daphnid, *C. dubia,* is routinely used in laboratory toxicity testing to establish baseline toxicity (Environment Canada 1992) and to determine toxic constituents in complex effluents (US EPA 1991). The short duration of the standardized chronic test (7 ± 1-d) and the small volume (<100 mL) of sample needed make this test a cost efficient tool for assessing toxicity (Environment Canada 1992). The three brood 7-d chronic toxicity test utilizes two endpoints, survival and fecundity, which provides a broader indication of the degree of effluent toxicity. Furthermore, the use of daphnids in toxicity testing is appropriate because they are: (1) broadly distributed in Canadian

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freshwater bodies and found in a wide range of habitats; (2) an important link in many

aquatic food chains; (3) sensitive to a broad range of aquatic contaminants; (4) relatively

short-lived, with short life-cycles; and (5) relatively easy to culture (Environment

Canada 1992).

1.7   Research Goals and Objectives

1.7.1   Goal

The overall goal of this project was to evaluate the impact of PKE from the

Ekati™ Diamond Mine on the water chemistry and zooplankton of the LLCF. Two

approaches were used to meet this goal. The first was to determine the magnitude of

possible effects on native zooplankton community populations from the disposal of

PKE in the LLCF, and to monitor the water chemistry and other limnological changes

that occurred in the various cells of the lake over time. The second was to evaluate the

magnitude of PKE toxicity to aquatic organisms through a series of baseline toxicity

tests, followed by TIE procedures to determine the nature of possible toxic components.

The combination of these two approaches would indicate if aquatic organisms living

downstream of the LLCF may potentially be stressed by toxic components of diluted

PKE.

1.7.2   Objectives

The specific objectives of this research project were:

1.   To evaluate the ecosystem changes that could occur in the LLCF between the

fall of 1998, prior to PKE addition, and the fall of 2000, two years after the

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addition of PKE, by collecting and analyzing the following data from Cells B

through E in the spring (July) and fall (September) of each year:

(i)   zooplankton species composition, abundance, and community diversity;

(ii)   phytoplankton biomass through analysis of chlorophyl *a* concentrations;

        and

(iii)   water quality.

Null Hypothesis: the addition of PKE to the LLCF will not alter zooplankton

populations, primary production, or the water quality of the LLCF.

2.   To determine the baseline toxicity of PKE through a series of tests using *C.*

dubia to estimate the type and degree of adverse effects that may occur to

exposed aquatic organisms and to characterize the class(es) of toxic components

that may be present in PKE through a series of Phase I and II TIE manipulations.

Null Hypothesis: PKE does not contain components that affect the survival

and/or fecundity of *C. dubia*.

CHAPTER 2

IMPACT OF PROCESSED KIMBERLITE EFFLUENT ON
ZOOPLANKTON AND WATER CHEMISTRY
IN THE LONG LAKE CONTAINMENT FACILITY

2.1   Introduction

Broken Hills Proprietary's (BHP; now BHP Billiton Inc.) Ekati™ Diamond

Mine, Northwest Territories, began mining operations in October 1998. Diamonds are

found in kimberlite deposits and extracted mechanically using chemical-free processes

that reduce the ore to fine tailings (≤ 0.5 mm) and coarse tailings (≥ 0.5 to 8.0 mm).

Processed kimberlite effluent (PKE) consists of fine tailings, coagulants, flocculants,

water used during kimberlite processing, and secondary treated sewage effluent from the

Ekati™ camp. The coarse tailings are disposed of at a tailings collection site, while

PKE is released to the Long Lake Containment Facility (LLCF) (NWT Diamonds

Project 1995). Permission to sacrifice Long Lake for containment of the kimberlite

fines was granted as part of BHP's Type "A" Water Licence (N7L2-1616).

Long Lake, centrally located in the Koala watershed and at the headwaters of the

Coppermine River drainage, was a typical tundra oligotrophic lake (NWT Diamonds

Project 1995). Four permeable rock dams have been constructed to divide Long Lake

into a series of five cells, A through E. An impermeable frozen-core dam has been built

at the end of Cell E, the outflow of the LLCF, to provide containment of the tailings

site. Water is pumped from Cell E into the downstream lake, Leslie Lake. Since

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October 1998, PKE has predominantly been pumped to Cell B and sporadically to Cell C of the LLCF (Ekati™ Diamond Mine 2000). As the basin of each cell fills with fine tailings, PKE will be deposited in the consecutive cell. Cell E will not be used as a tailings collections site, but rather will be used to monitor the quality of the water exiting the LLCF (NWT Diamonds Project 1995).

The LLCF offers a unique opportunity to study the sequence of ecosystem changes that will result from the addition of PKE. In essence, Long Lake has become a large scale bioassay from which alterations in biological, physical and chemical variables can be monitored and assessed. The division of the LLCF into five cells may create a gradient of ecosystem effects that should be observable in the cells downstream of tailings deposition.

Previous research conducted by the Department of Fisheries and Oceans (DFO) Winnipeg, MB, identified aluminum and ammonia, in combination with high pH, as the primary toxic components of crushed kimberlite solutions to fish (Harrison *et al.* 1995, 1997). The toxicity of these solutions was reduced with filtration, which lowered the concentration of total suspended solids (TSS) and pH (Harrison *et al.* 1995, 1997; Enviro-Test Laboratories 1998). It was postulated that the lower pH influenced the toxicity of the pH-dependent toxicants ammonia and aluminum (Harrison *et al.* 1995; 1997).

Laboratory evaluations cannot always adequately determine the fate and behaviour of pollutants in aquatic ecosystems, or properly determine the effects to native aquatic organisms (Rand *et al.* 1995). Furthermore, assessing the impact of anthropogenic contaminants on ecosystems requires monitoring of both the biotic and

abiotic elements. Chemical monitoring alone has its limitations, such as difficulties associated with temporal and spatial sampling, potential synergistic effects and the possibility of accumulation within ecosystems (Spellerberg 1991). Furthermore, measuring physical and chemical components provides only information on the concentration of pollutants in the ecosystem, but does not provide information on the degree of toxicity to native organisms. Effective monitoring of the biological, physical and chemical components of water provides information on the concentration, the behaviour and the toxicity of pollutants in aquatic ecosystems (Rand *et al.* 1995).

Two types of endpoints are commonly used to measure the biotic response of an ecosystem: structure and function (Cairns *et al.* 1995). Structural endpoints assess changes in measures such as species richness, species abundance, and community similarity. Changes in community composition of small, rapidly reproducing organisms such as zooplankton are one of the earliest and most sensitive indicators of anthropogenic stress (Gannon and Stemberger 1978; Sladecek 1983; Schindler 1987). Community level studies of zooplankton provide useful information about the biological integrity of an aquatic system (Linthurst *et al.* 1995), and may identify more sensitive indicators of aquatic toxicity resulting from exposure to contaminants (Hellawell 1986). Functional endpoints include measures such as the rate of primary production, community respiration, nutrient uptake and regeneration, decomposition and recovery after stress (Cairns *et al.* 1995). Primary production is often evaluated in order to investigate the level of trophic effects. For example, direct alterations in primary production could indirectly lead to changes in the higher trophic levels (Moss 1998).

The effect of PKE on the aquatic ecosystem of the LLCF was evaluated by

studying the changes in zooplankton, water chemistry and lake limnology. Samples of zooplankton were collected from the cells of the LLCF to determine the magnitude of effects that the addition of PKE had on abundance, diversity and community structure. Alterations to water chemistry and limnological characteristics were used to analyze the effects of PKE to water quality within the LLCF. By observing changes to zooplankton, water chemistry and lake limnology in the cell farthest from the site of PKE deposition it may be possible to estimate the effects PKE may have on aquatic environments downstream of the LLCF.

2.2   Materials and Methods

2.2.1   Sampling Site

The LLCF at the Ekati Diamond Mine, NT, located at Long Lake (64° 40' N, 110° 40' W), was originally up to 32 m deep and had a surface area of 6.14 km². It had a watershed area of 45 km² and drained directly into Leslie Lake. The ice-free season was from mid-June to September (NWT Diamonds Project 1995).

The following sampling sites were located on the LLCF: one each in cells B, C and E, and two in D, designated as D S1 and D S2 (Figure 1.1). Cell A was not sampled because it was not a true a true lake nor not downstream of PKE deposition. Each sampling site was located approximately 30 m away from the closest dam. Samples were not collected from Cell C in August 1998 and Cell B in 2000. Sampling commenced in late August (27th to 31st) 1998, conducted by BHP employees, and was repeated in the 2nd week of July (10th to 14th) and the last week of August (28th to September 2nd) in 1999 and 2000. At each sampling time, samples were collected for

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water quality analysis, determination of chlorophyll *a* concentration and zooplankton abundance, and the limnological characteristics of the lake, such as dissolved oxygen (DO) levels, water temperature profiles, and Secchi depth, were measured.

In accordance with its water licence agreement, the Ekati™ Diamond Mine conducted a fish-out program on the LLCF from 1997 to 1999. Details on the dates, numbers and species of fish removed are provided in Appendix A. In 1997, prior to dividing Long Lake into individual cells, 6,533 fish were removed (Rescan 1998a). In 1998, after dike construction, 7,564 fish were removed from Cell C and, in 1999, 3,633 fish were removed from Cell D (Rescan 1999, 2000b).

2.2.2 Dissolved Oxygen/Temperature Profiles and Secchi Depth

A YSI Model 58 DO meter equipped with a 50 m cable and an *in situ* probe was used to obtain DO and temperature profiles for each sampling site. The meter was air calibrated in the field immediately prior to use. The probe was lowered to ~1.0 m from the bottom of the lake and allowed to stabilize for 15 min. Readings were taken at half meter intervals as the probe was raised; the probe remained at the desired sampling depth for 20 sec to acquire a stable reading. The Secchi depth was obtained by lowering a Secchi disk over the shady side of the boat and recording the depth at which it was no longer visible (not available for August 1998 field season).

2.2.3 Collecting Samples for Water Quality Analysis

Samples for water quality, chlorophyll *a,* nutrient and elemental analysis were collected in triplicate 1 m below the surface and 1m above the bottom of Long Lake

using a non-metallic 10-L Van Dorn water sampler. Sample bottles were rinsed three times with Long Lake water. Samples for water quality and nutrient analysis were stored in 500-mL polycarbonate bottles. Water quality samples were preserved with 2 mL of 20% H_2SO_4. Samples for elemental analysis were placed in 250-mL P.E.T.E. wide mouthed jars and were preserved with 5 mL of 20% HNO_3. Preservation was conducted in the Ekati™ environment laboratory and occurred within 12 h of sample collection. Samples for chlorophyl *a* determination were filtered in the Ekati™ environment laboratory to concentrate phytoplankton for chlorophyl *a* analysis. To concentrate phytoplankton 300 to 750 mL of sampled water were concentrated on a 47 mm Supor-450® membrane filter (0.45 μm pore size) using vacuum filtration. Filters were folded in half, wrapped in aluminum foil, frozen and later analyzed at the University of Saskatchewan.

Water quality, nutrient and elemental analyses were performed by Taiga Environmental Laboratory, Yellowknife, NT, in accordance with standard procedures. For each measurement, the mean and standard deviation was calculated by combining the values of the three replicates collected at either 1 m from the surface or 1m from the bottom of the lake at each site. Data for July 1999 are not available.

2.2.4 Extraction and Analysis of Chlorophyll *a*

The APHA (1998) method (10200 H) was used to extract and analyze chlorophyll *a* concentrations. In subdued light, frozen filters of concentrated phytoplankton biomass were placed in ~2 mL of a 90% acetone and 10% saturated magnesium carbonate solution and broken apart. In 1999, filters and phytoplankton

cells were broken apart using a mortar and pestle, followed by 1 h in an ultrasonic

shaker in the dark at 4°C. In 2000, filters and phytoplankton cells were macerated for

~1 min using a mechanical tissue grinder. Samples were placed in calibrated 15-mL

polypropylene screw-cap test tubes, brought to ~10 mL volume with the 90% acetone

and 10% saturated magnesium carbonate solution and held in the dark at 4°C for ~3 h.

To clarify the chlorophyll *a* solution of remaining filter and cell particles, the extract

solutions were centrifuged for 20 min at ~500 *g*. The volume of the extract was

recorded for each sample.

A spectrophotometer was used to measure chlorophyll *a* concentrations in the

extracts. In 1999, a Beckman UV/Vis spectrophotometer was used (with *vis* absorption

reading capability to 0.001) and in 2000 a Cary 100 UV/Vis scanning spectrophotometer

was used (with *vis* absorption reading capability to 0.0001). The spectral band width

was set at 2.0 nm and calibrated with 90% acetone (10% Milli Q® water) at wavelengths

750, 664 and 665 nm. Three mL of the extract was placed into a cuvette with a 1-cm

path length. Optical density (OD) of the extract was measured at 750 and 664 nm. The

extract was acidified with 0.1 mL of 0.1N HCl, and 90 sec later OD was read at 750

and 665 nm. To calculate chlorophyll *a* concentrations, the 750 nm OD value from

before acidification was subtracted from the OD 664 nm reading, and after acidification

from the OD 665 nm reading. Using these values, the concentration of chlorophyll *a*

per m³ of water was calculated (Equation 2.1):

$$\text{Chlorophyll } a \text{ (mg/m}^3) = \frac{26.7 \ (664_b\text{-}665_a) \times V_1}{V_2 \times L} \qquad [2.1]$$

where V_1 = volume of extract (L), V_2 = volume of sample (m³), L = light path length or

width of cuvette (cm), and 664_b and 665_a are the optical densities of the 90% acetone chlorophyll a extract before and after acidification, respectively. The detection limit, which was dependent upon the volume of sample, volume of extract and reading capabilities of each machine, ranged from 0.02 to 0.45 mg/m^3.

2.2.5 Zooplankton Sampling, Identification and Enumeration

Zooplankton samples were collected in triplicate by pulling a plankton net vertically through the water. Sampling depth was determined by subtracting 2 m from the maximum depth of the sample site. The net was raised at a constant speed of ~0.5 m/s. In 1998 and 1999 a simple conical plankton tow-net (118 μm mesh size), rigged for vertical tows, with 30-cm diameter was used to collect zooplankton samples, and in 2000 a Wisconsin plankton net (80 μm mesh size) with a reducing cone forward of the mouth and a 30-cm diameter was used. Zooplankton samples were narcotized with carbonated water and preserved with 5% sugar buffered formalin (APHA 1998).

To enumerate zooplankton, samples were brought to 100 mL and a 1-mL subsample was taken and counted on a Sedgewick-Rafter counting cell. If 300 organisms were not counted in one subsample, then up to 3 subsamples or 3 mL were enumerated. Estimates of zooplankton abundance per m^3 for each replicate of a specific site were calculated using Equation 2.2:

$$\text{Abundance}/m^3 = \frac{C \times V_1}{V_2 \times V_3} \qquad [2.2]$$

where C = number of organisms of a specific taxon, V_1 = volume of the concentrated sample (100 mL), V_2 = volume counted (mL), and V_3 = volume of water sampled (m^3)

(APHA 1998). The volume of water sampled was calculated using Equation 2.3:

$$V = \pi r^2 \times d \qquad [2.3]$$

where r = the radius of the net mouth (m) and d = depth of water sampled (m). To

obtain the mean (± SD) abundance per m³, the three replicates for each site were

averaged.

Rotifera, Cladocera and Copepoda were generally identified to species with the

aid of taxonomic keys (Ward and Wipple 1963; Pennak 1989). If an individual of a

species was not counted in a replicate from a specific site, but one or two of the other

replicates contained this species, then a value of 0.5 times (organisms/m³) the detection

limit was assigned instead of a zero. If an individual of a specific species was not

detected in all three replicate samples from a specific site, then a value of 0.5 times the

detection limit was used to estimate abundance and a value of zero was used to calculate

diversity and proportional similarity of zooplankton communities. The detection limit

ranged from 39 to 472 organisms per m³, depending on the sampling depth and the

number of 1-mL subsamples counted. Zooplankton species and the detection limit for

each sampling site are provided in Appendix B.

2.2.6   Statistical Analysis

Significant changes in the water chemistry variables were determined by

comparing data from August 1998 (prior to PKE addition) to August 2000 for each of

the sampling sites using a Students t-test. One-way ANOVA was used to determine

significant differences in water quality measurements at each sampling site over time

(from July 1999 to August 2000). The mean abundance per m³ of zooplankton was

evaluated by Spearman Rank Order Correlation. Statistical analyses were performed using the computer program SigmaStat® version 2.03 (1992) with a 95% ($\alpha = 0.05$) level of confidence.

The Shannon-Wiener diversity index (H') and the proportional similarity index were used to evaluate changes in zooplankton community composition at the various sampling sites. Shannon-Wiener diversity indices were used to compare changes in each of the specific cells at each sampling time from August 1998, prior to the addition of PKE to August 2000, with the exception of Cell C for which subsequent sampling dates were compared to July 1999 (data for August 1998 were not available). Proportional similarity was used to make spatial comparisons between adjacent cells for specific sampling times and temporal comparison within each sampling site between sampling times. All community analysis were performed using EcoStat 1.1 (1992).

2.3   Results

Estimates of zooplankton abundance per m^3 are presented in Appendix B, detailed water chemistry data are presented in Appendix C, and mean chlorophyll a concentrations are presented in Appendix D. Results for each sampling time are presented on a temporal scale that starts at sampling time '0' (August 1998) (Table 2.1).

Table 2.1. Relation of sampling date to temporal scale used to present results.

Sampling date	August 28 1998	July 14 1999	August 31 1999	July 12 2000	August 31 2000
Temporal date (month)	0	10	12	22	24

2.3.1 Temperature/Oxygen Profiles and Secchi Depth

Sampling site depth, Secchi depth, average DO concentrations and mean temperature at each site in the LLCF are given in Table 2.2. Differences in sampling depths for each site over the course of the study are due to fluctuations in the depth of the lake, and inability to sample at the exact location each sampling time. The DO concentrations in each of the cells generally remained above 9.0 mg/L. There was very little variation among sampling sites, with the exception of Cell C which dropped to 7.8 mg/L in July 2000. In general, water temperatures in the lake were higher in July than in August 1999, ranging from 6.4 to 14.4 °C and 6.4 to 10.8 °C, respectively. There was very little evidence of thermocline development at any sampling time; the temperature generally remained constant (±0.2°C) at each site from the bottom to the surface of the lake, with the exception of sampling sites C and D S1 in July 2000. In Cell C, there was a gradual change in water temperature from 6.1°C at the bottom of the lake (10 m) to 16.1°C at 6 m from the surface, at which point the temperature remained constant. In Cell D site 1, the temperature rose gradually from 7.0 to 12.9 °C from bottom to the top of the lake. At each sampling date, the Secchi depth was low in both Cell B and C (≤2.75 m) relative to the other cells (≥4.2 m). Cell E, 2000, the secchi depth was equal to sampling depth.

2.3.2 Water Quality Analysis

Only the data for water quality, nutrient and elemental analyses collected 1 m below the water surface are presented and discussed in this chapter. There was very little difference between results obtained for water samples collected 1 m below the

Table 2.2. Lake depth, Secchi depth, dissolved oxygen concentration and temperature at each sampling site in the LLCF from July 1999 to August 2000.

Sampling site and time	Lake Depth (m)	Secchi Depth (m)	DO[a] (mg/L)	Temperature[a] (°C)
Cell B				
July 1999	3.0	1.5	9.1±0.0	12.4±0.0
August 1999	3.0	0.9	11.5±0.1	6.4±0.0
Cell C				
July 1999	13.5	2.5	10.1±0.0	11.2±0.0
August 1999	11.5	2.5	11.4±0.0	8.6±0.0
July 2000	11.0	2.0	7.8±0.5	13.4±3.8
August 2000	9.0	2.75	10.4±0.1	8.0±0.0
Cell D S1				
July 1999	13.0	4.3	11.1±0.1	6.4±0.2
August 1999	10.0	7.0	10.6±0.0	9.5±0.1
July 2000	12.0	5.0	9.8±0.4	10.0±2.1
August 2000	11.8	7.5	11.4±0.4	8.8±0.0
Cell D S2				
July 1999	10.0	4.2	10.8±0.1	9.0±0.1
August 1999	8.0	7.0	10.8±0.1	10.8±0.1
July 2000	7.5	5.0	9.4±0.0	14.4±0.0
August 2000	9.75	7.75	9.8±0.1	9.3±0.0
Cell E				
July 1999	9.0	6.4	10.0±0.2	9.6±0.1
August 1999	8.0	6.5	11.0±0.1	8.6±0.0
July 2000	4.25	4.25	9.3±0.2	14.4±1.0
August 2000	4.0	4.0	10.9±0.0	7.6±0.0

[a] Values are mean ± SD from the bottom to the top of the lake measured at 1.0 m intervals.

surface of the lake and those collected 1 m above the bottom of the lake. All data for water quality, nutrient and elemental analysis are presented in Appendix C.

The addition of PKE to Cell B, and sporadically to Cell C, had a pronounced effect on the water chemistry of the LLCF. Prior to the addition of PKE in August 1998, all cells of the LLCF had low alkalinity (<6.2 mg/L as $CaCO_3$), low total hardness (<6.8 mg/L as $CaCO_3$), and a slightly acidic pH (<6.75) (Figure 2.1). In Cell B, from August 1998 to August 1999, alkalinity and total hardness increased to >62 mg/L and >190 mg/L as $CaCO_3$, respectively, and the pH become slightly alkaline (>8.05). By August 2000, significant increases in pH, total hardness and alkalinity were also noted in Cell E; from August 1998 to August 2000 alkalinity increased from 5.0 to 8.2 mg/L (Figure 2.1a), total hardness increased from 5.49 to 20.93 mg/L (Figure 2.1b), and pH increased from 6.57 to 7.16 (Figure 2.1c).

The increase in water hardness in the LLCF was due to increases in the concentration of calcium (Ca), magnesium (Mg), and, to a limited extent, strontium (Sr) (Figure 2.2). The concentrations of these elements increased in Cell E from August 1998 to August 2000 as follows: Ca from 1.23 to 4.10 mg/L (Figure 2.2a), Mg from 0.59 to 2.59 mg/L (Figure 2.2b), and Sr from 8.1 to 35.0 μg/L (Figure 2.2c). Although data were not collected in August 1998 for conductivity and total dissolved solids (TDS), these variables were five and six times higher, respectively, in Cell E in August 2000 compared to Long Lake in August 1997 (Rescan 1998a). Turbidity and total suspended solids increased in Cell B from August 1998 to August 1999, however, significant differences were not observed in downstream cells.

The only significant nutrient change in the LLCF from August 1998 to August
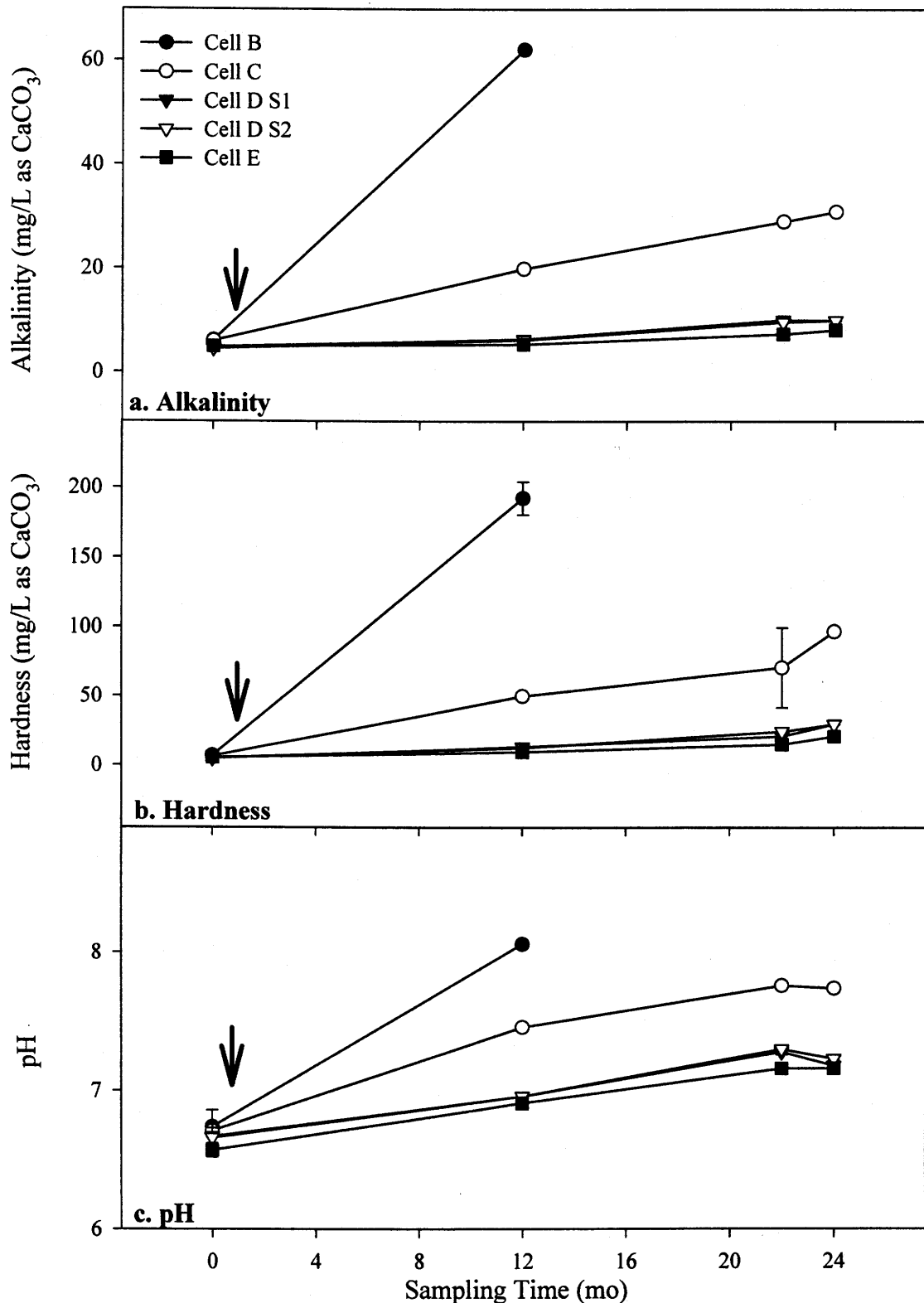
Figure 2.1 The effect of processed kimberlite effluent (PKE) on the mean (±SD) (n=3)
(a) alkalinity, (b) total hardness, and (c) pH in the Long Lake Containment Facility
(LLCF) from August 1998 (0 months) to August 2000 (24 months). Within each cell,
final measurements were always significantly greater than initial measurements (α=0.05).
Arrows indicate initiation of PKE addition to the LLCF.
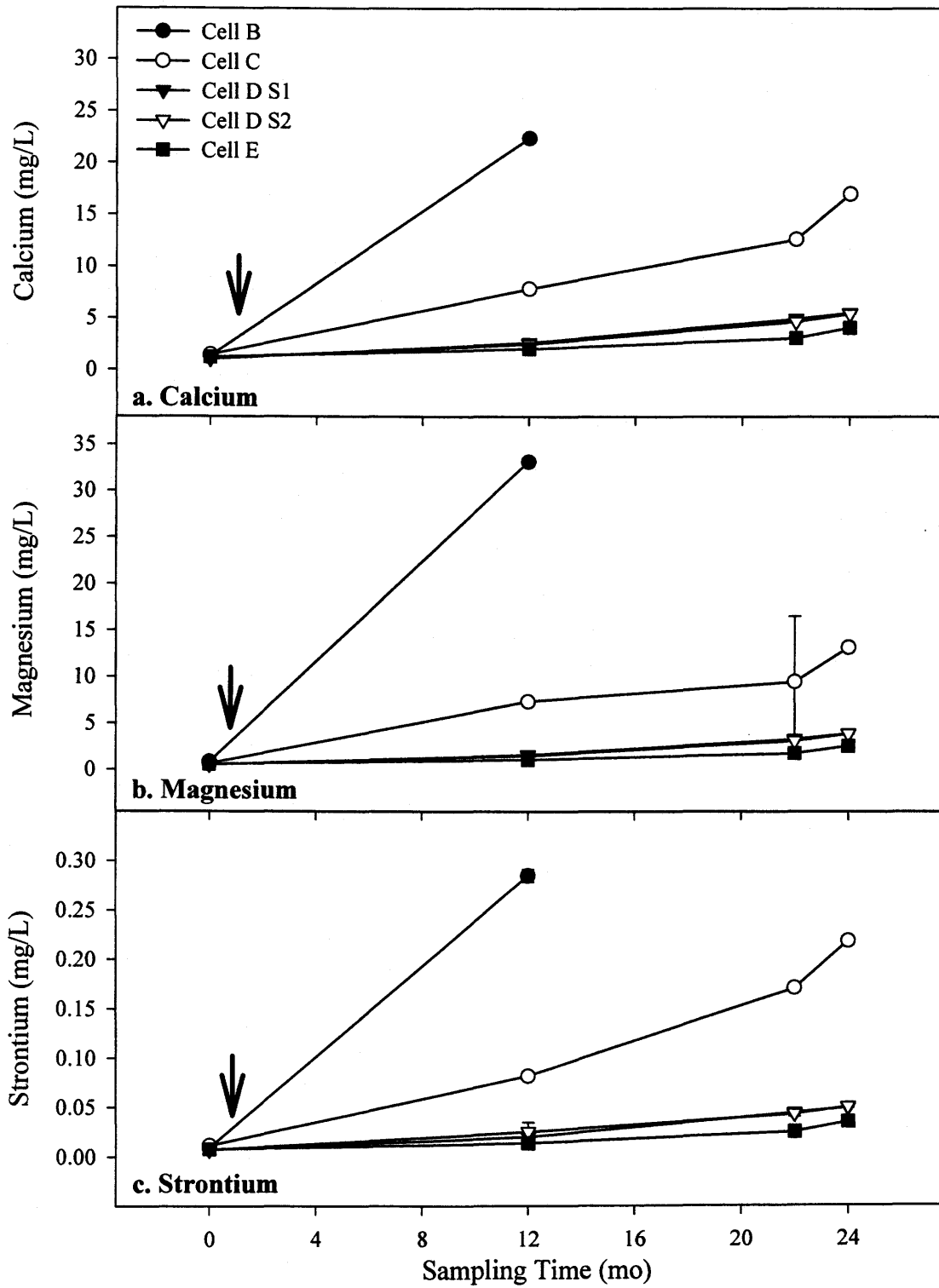
Figure 2.2 The effect of processed kimberlite effluent (PKE) on the mean (±SD) (n=3) concentration of (a) calcium, (b) magnesium, and (c) strontium in the Long Lake Containment Facility (LLCF) from August 1998 (0 months) to August 2000 (24 months). Within each cell, final measurements were always significantly greater than initial measurements (α=0.05). Arrows indicate initiation of PKE addition to the LLCF.

2000 was the concentration of total nitrogen (Figure 2.3a). The concentration of total nitrogen increased significantly in all cells of the LLCF; higher concentrations were noted in cells closer to the site of PKE deposition (Cell B). In Cell E, nitrogen concentrations increased from 0.217 to 1.19 mg/L from August 1998 to August 2000. The concentrations of total phosphorous appeared to fluctuate with sampling date; lower concentrations were generally observed in August (0, 12, and 24 months) than in July (10 and 22 months) (Figure 2.3b). A statistical difference in the concentration of total phosphorous was not discerned between sampling times for a specific site. The ratios of total nitrogen to total phosphorous was greater than 7:1 in all the cells of the LLCF in August 1998; ranging from 7 (Cell B) to 76 (Cell C). By August 2000, N:P ratios ranged from 76 (Cell D site 2) to 529 (Cell C). Although the concentration of ammonia increased in Cell B from 1998 to 1999, other cells in the LLCF have exhibited an overall decrease since 1998.

The concentrations of many major and trace metals remained below detection limits in the LLCF between August 1998 to August 2000 (Appendix C); only barium (Ba), molybdenum (Mo), and nickel (Ni) showed noteworthy changes (Figure 2.4). Cell B had the greatest increase, with lower increases observed in the cells further downstream. In Cell E, from August 1998 to August 2000, the following increases in concentration were observed: Ba from 6.6 to 24.3 μg/L (Figure 2.4a), Mo from below detection (1.0) to 6.3 μg/L (Figure 2.4b), and Ni from below detection (1.0) to 1.2 μg/L (Figure 2.4c). Manganese, iron and antimony increased slightly, but not to the same extent, nor with the same profile of increase in the downstream cells as the above mentioned metals. The concentration of aluminum increased in Cell B from 34 μg/L
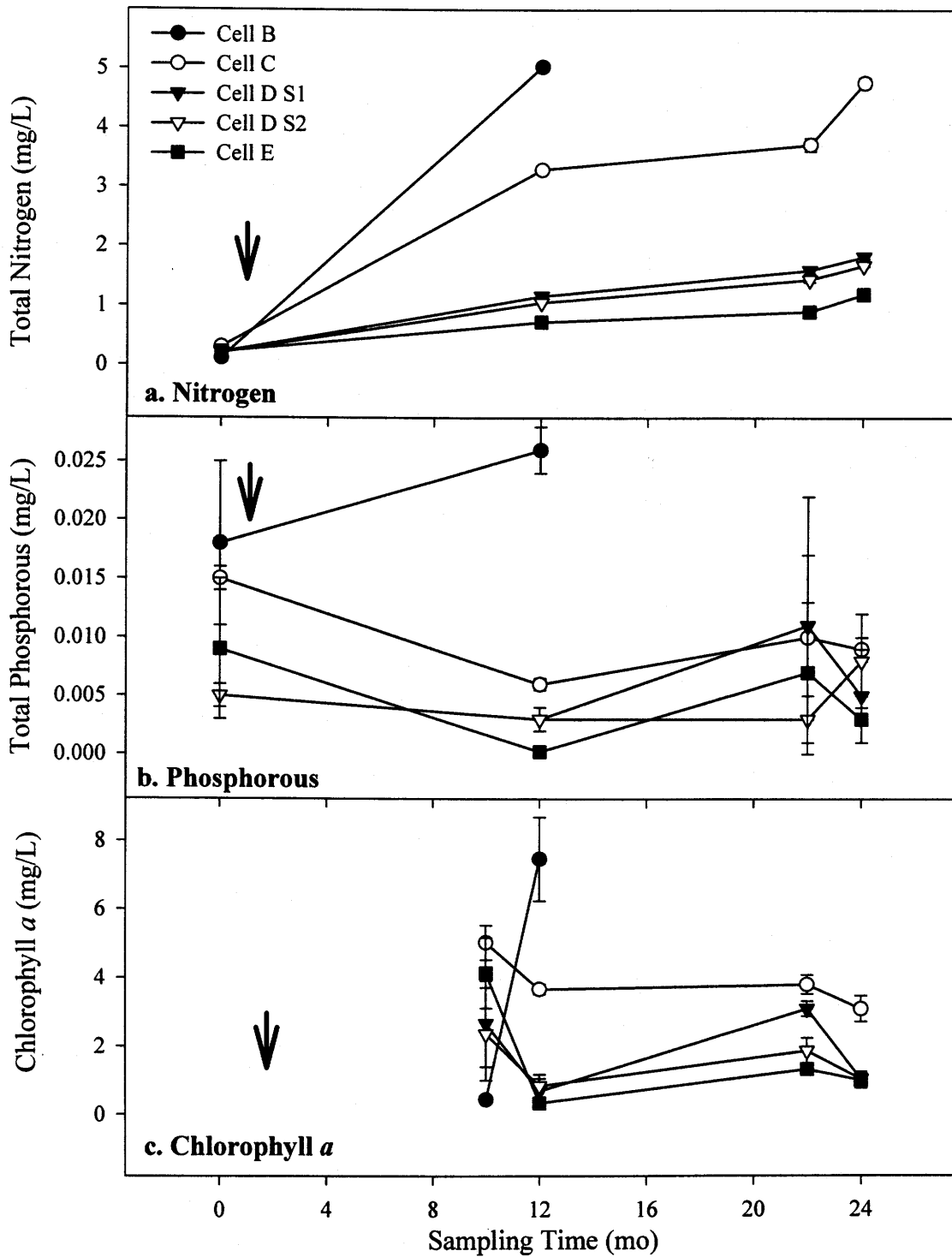
Figure 2.3 The effect of processed kimberlite effluent (PKE) on the mean (±SD) (*n*=3) concentration of (a) total nitrogen, (b) total phosphorous, and (c) chlorophyll *a* in the Long Lake Containment Facility (LLCF) from August 1998 (0 months) to August 2000 (24 months). For graphs a and b, final measurements were always significantly greater than initial measurements (α=0.05) for each cell. Arrows indicate initiation of PKE addition to the LLCF.
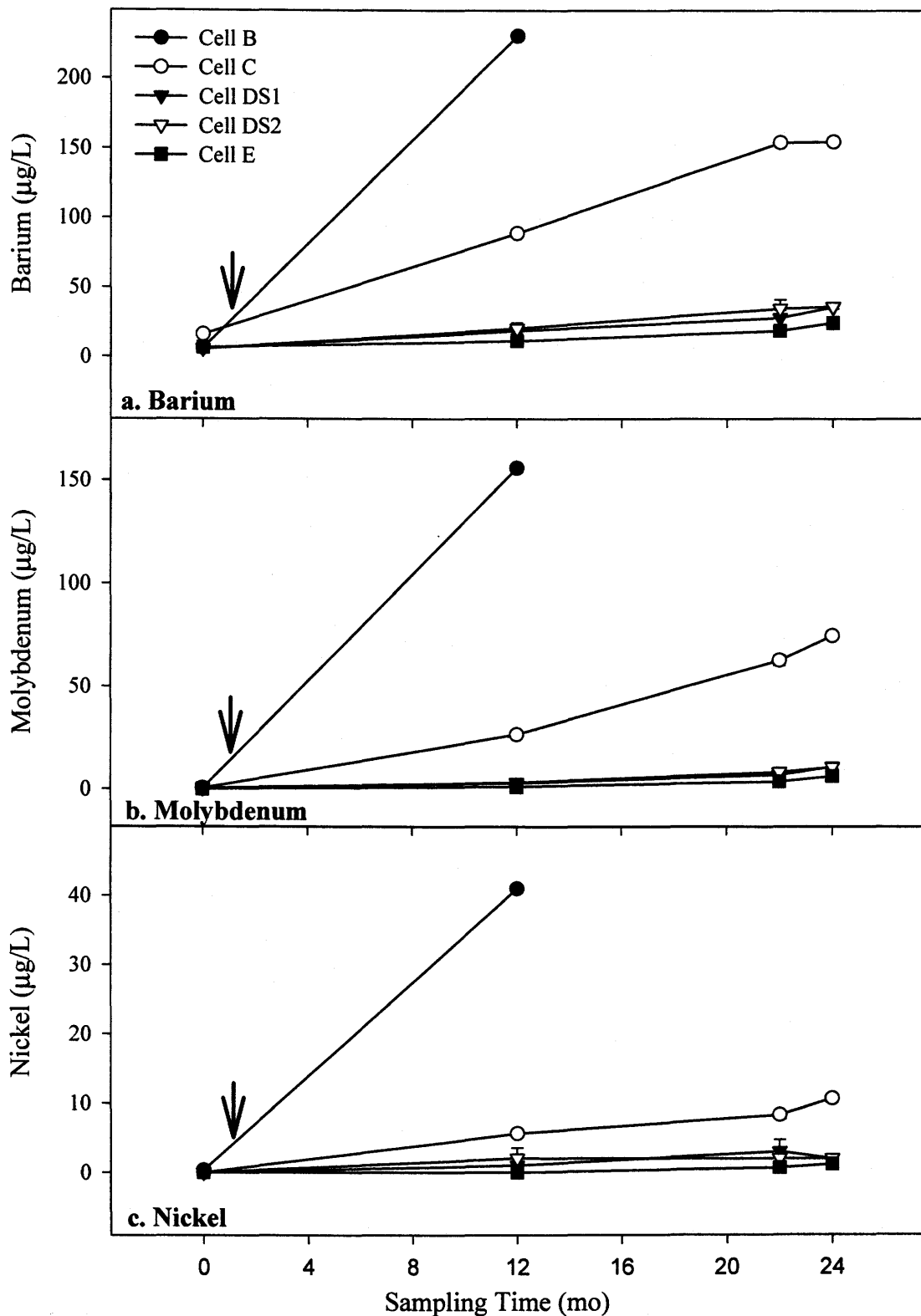
Figure 2.4 The effect of processed kimberlite effluent (PKE) on the mean (±SD) (*n*=3) concentration of (a) barium, (b) molybdenum, and (c) nickel in the Long Lake Containment Facility (LLCF) from August 1998 (0 months) to August 2000 (24 months). Within each cell, final measurements were significantly greater than initial measurements (α=0.05). Arrows indicate initiation of PKE addition to the LLCF.

(August 1998) to 157 μg/L (August 1999), however, concentrations were higher in August 1998 than in August 2000 for the downstream cells.

2.3.3   Chlorophyll *a*

There was very little difference between the concentration of chlorophyll *a* at 1 m below the surface and 1 m above the bottom of the lake. Therefore, only data for samples collected at 1 m below the lake surface are presented (Figure 2.3c). Concentrations of chlorophyll *a* in Cell B ranged from 1.34 to 7.46 mg/m^3. The other cells showed a more moderate temporal fluctuation in chlorophyll *a* concentrations. In Cells D and E, the concentration of chlorophyll *a* ranged from 0.34 mg/m^3 in August 1999 to 3.56 mg/m^3 in July 1999.

2.3.4   Zooplankton Abundance

The Spearman Rank Order Correlation coefficients for zooplankton abundance in the LLCF are presented in Table 2.3. The overall abundance of zooplankton in Cell B of the LLCF was significantly lower in August 1999 than in August 1998 (prior to the addition of PKE) (Figure 2.5). During this period, mean zooplankton abundance decreased from 219,575 to 12,569 organisms/m^3. In August 1998, the high zooplankton abundance in Cell B was driven by the high numbers of rotifers (>86,000 organisms/m^3) and cyclopoid copepodids (>92,000 organisms/m^3) (Figure 2.6a,b). By August 1999, the abundances of both these taxa had declined to less than 5,000 organisms/m^3, and the abundance of Cladocera and Copepoda, not including nauplii and copepodids, were below detection limits ($\leq$472 organism per m^3).

Table 2.3. Spearman Rank Order Correlation coefficient and corresponding p-value for zooplankton abundance per m^3 in the Long Lake Containment Facility from August 1998 to August 2000.

Location	Rotifera	Cladocera	Copepoda	Total
Cell B	-0.843 $p<0.001$	-0.861 $p<0.001$	-0.635 $p<0.001$	-0.738 $p=0.020$
Cell C	0.648 $p=0.020$	0.398 $p=0.189$	0.864 $p<0.001$	0.648 $p=0.020$
Cell D S1	0.714 $p<0.002$	0.138 $p<0.611$	-0.156 $p=0.556$	0.301 $p=0.269$
Cell D S2	0.840 $p<0.001$	0.229 $p=0.403$	0.316 $p=0.240$	0.687 $p=0.004$
Cell E	0.775 $p<0.001$	-0.426 $p=0.110$	0.589 $p=0.020$	0.546 $p=0.034$
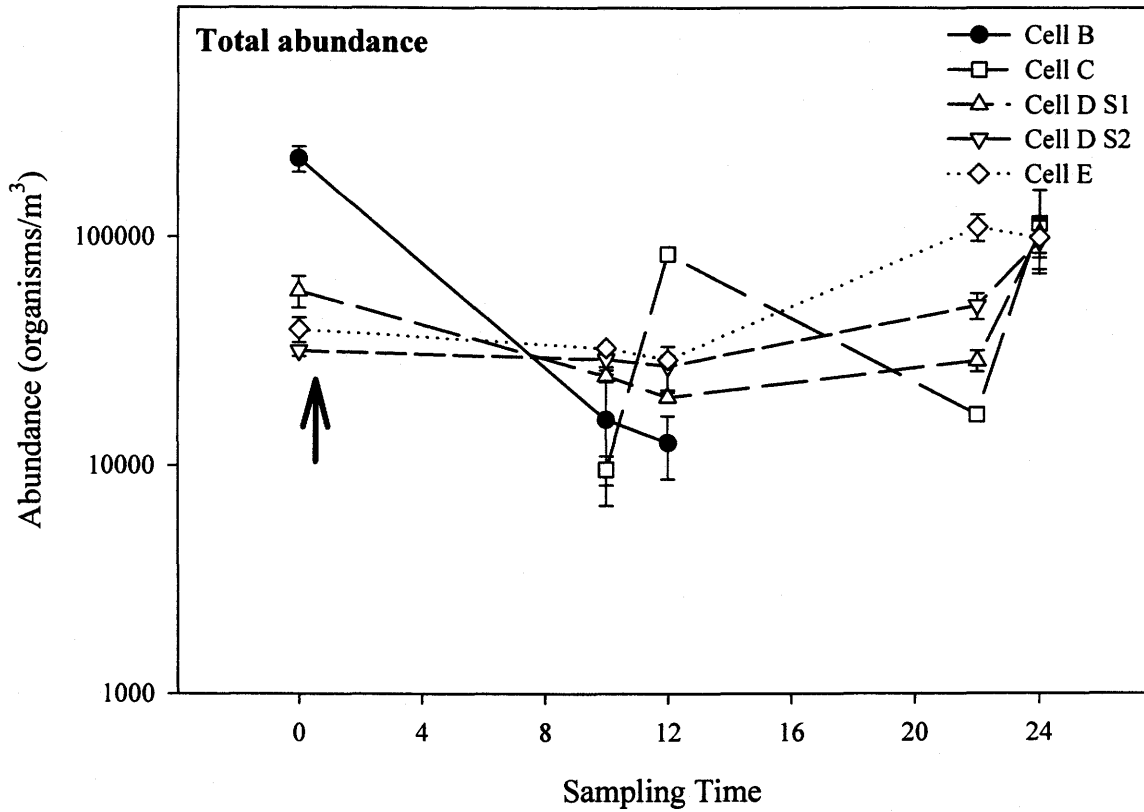
Figure 2.5. Mean (±SD) abundance per m^3 of total zooplankton in Cells B, C, D S1 and S2, and E of the Long Lake Containment Facility from August 1998 (0 months) to August 2000 (24 months). Arrow indicates time of processed kimberlite effluent (PKE) addition. Data were not collected from Cell B in July and August 2000, and from Cell C in August 1998.
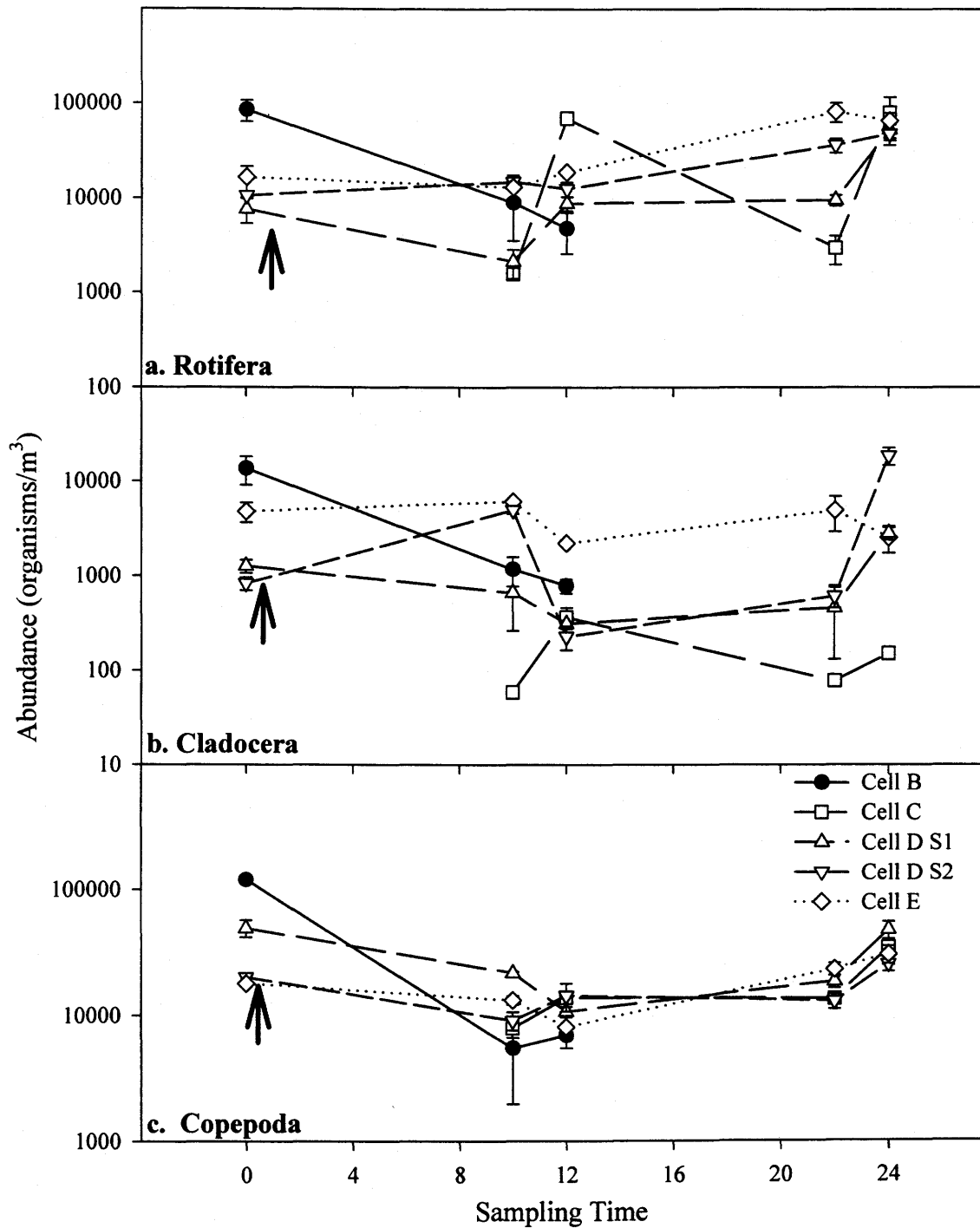
Figure 2.6. Mean (±SD) abundance per m³ of Rotifera, Cladocera and Copepoda in Cells B, C, D S1 and S2, and E of the Long Lake Containment Facility from August 1998 (0 months) to August 2000 (24 months). Arrows indicate time of processed kimberlite effluent (PKE) addition. Data were not collected from Cell B in July and August 2000, and from Cell C in August 1998.

Zooplankton data were not collected from Cell C in August 1998, so a comparison to pre-PKE addition could not be made. The overall abundance of zooplankton was lowest in July compared to August for both sampling years (Figure 2.5). The increases in total zooplankton abundance in August 1999 and 2000 were due to large increases in the abundance of rotifers (Figure 2.6a). Cladocera were not evident (abundance was at or below detection limits; ≤ 157 organisms per m^3) in Cell C for all sampling times (Figure 2.6b). The abundance of Copepoda, which was driven by an increase in nauplii, significantly increased from August 1998 to August 2000 (Figure 2.6c).

Cell D site 1 had a significant increase only in Rotifera abundance from August 1998 to August 1999 (Figures 2.5 & 2.6). Total zooplankton and Rotifera abundance significantly increased in Cell D site 2 and Cell E with the upstream addition of PKE (Figure 2.5 & 2.6). Cell E had a significant increase in Copepoda from August 1998 to August 2000 (Figure 2.6c).

The increases in rotifers in all of the cells of the LLCF were mostly driven by increases in *Kellicotia longispina* (Figure 2.6a). By August 1999 this was the only rotifer enumerated in Cell B. In August 2000, sites C, D S1 and D S2 had an increase in *Keratella cochlearis* from less than 1% to 25% of the total Rotifera abundance. Cell E did not have an increase in this species, but did experience a bloom of Conchilus sp., from <40% for previous sampling times to ~65% of the total rotifer abundance, in July 2000. However, by August 2000, the abundance of Conchilus sp. had decreased to less than 2% of overall zooplankton abundance.

The Spearman Rank Order Correlation coefficients for abundance of the three

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Table 2.4. Spearman Rank Order Correlation coefficient and corresponding p-value for abundance per m^3 for cladoceran species in the Long Lake Containment Facility from August 1998 to August 2000.

Location	Holopedium gibberum	Daphnia middendorffiana	Bosmina longirostris
Cell B	-0.912 $p<0.001$	-0.072 $p=0.844$	-0.845 $p<0.001$
Cell C	-0.40 $p=0.189$	0.398 $p=0.189$	0.400 $p=0.189$
Cell D S1	-0.372 $p=0.167$	0.563 $p=0.0275$	0.251 $p=0.359$
Cell D S2	-0.729 $p=0.002$	0.690 $p=0.004$	0.661 $p=0.007$
Cell E	-0.800 $p<0.001$	0.328 $p=0.224$	0.115 $p=0.676$

identified cladoceran species in the LLCF are presented in Table 2.4. The overall

abundance of Cladocera was lower in August 1999 compared to all other sampling dates

in the cells of the LLCF (Figure 2.6c). It appeared that the abundance of *Holopedium*

gibberum decreased throughout the LLCF; however a significant trend could only be

determined for sites B, D S2 and E (Figure 2.7). The abundance of *Daphnia*

middendorffiana significantly increased in Cell D (S1 and S2) (Figure 2.7). The

abundance of *Bosmina longirostris* fluctuated in all cells except in Cell D site 2, where

a significant increase was observed (Figure 2.7).

2.3.5   Zooplankton Diversity

The Shannon-Wiener diversity indices for zooplankton assemblages in each cell

over time are presented in Figure 2.8. Taxonomic richness and diversity, in Cell B

decreased after the addition of PKE. In August 1998 there were 11 taxa identified in

Cell B, but by August 1999 this was reduced to 4 taxa. The diversity index for Cell C

fluctuated with the presence of rotifers and copepod nauplii. A lower diversity was

observed when these animals dominated the assemblages. Rotifers accounted for 83%

and 70% of the zooplankton assemblage in Cell C in August 1999 and 2000,

respectively.

The zooplankton diversity indices for Cell D sites S1 and S2, and Cell E ranged

between 2.2 and 3.0 in August 1998 (Figure 2.8). Fluctuations in diversity were

generally caused by increases in the abundance of one species in each of these cells.

Decreased diversity at site D S1 in July 1999 was caused by the dominance of cyclopoid

copepodids, which accounted for 73% of the zooplankton assemblage. At Cell D site 2,
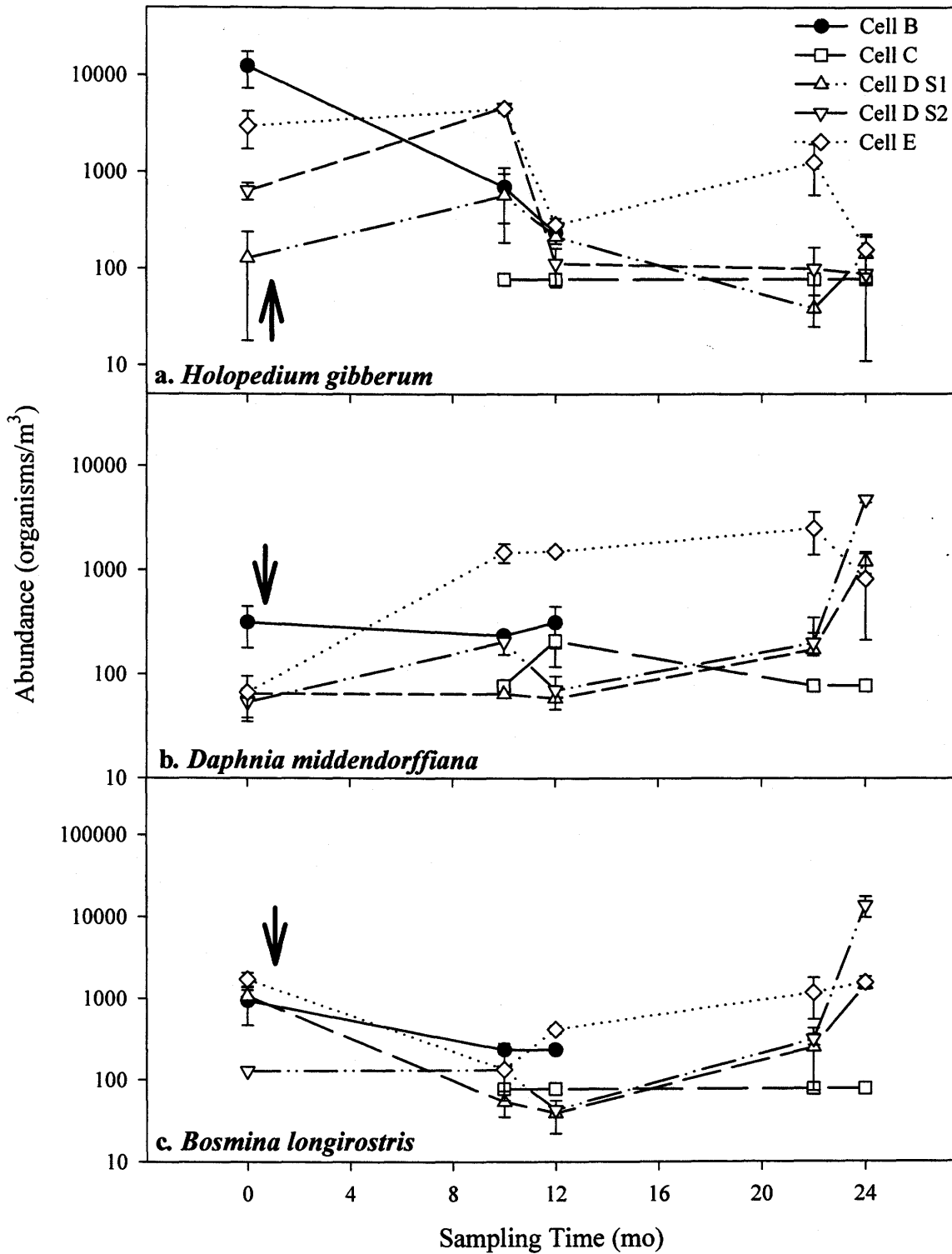
Figure 2.7. Mean (±SD) abundance per m³ of (a) *Holopedium gibberum*, (b) *Daphnia middendorffiana*, and (c) *Bosmina longirostris* in the Long Lake Containment Facility (LLCF) from August 1998 (0 months) to August 2000 (24 months). Arrows indicate initiation of processed kimberlite effluent addition to the LLCF. Data were not collected from Cell B in July and August 2000, and from Cell C in August 1998.
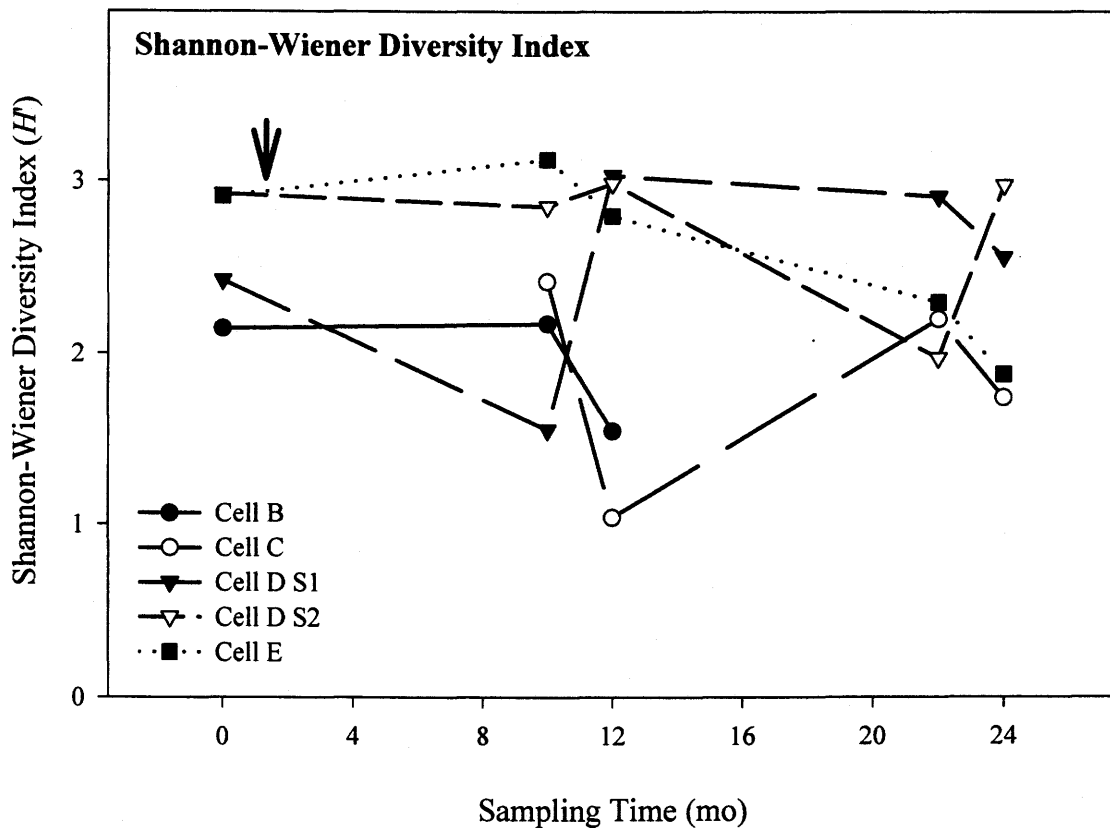
Figure 2.8 Shannon-Wiener Diversity Index (H') for zooplankton in Cells B to E of the Long Lake Containment Facility (LLCF) from August 1998 (0 months) to August 2000 (24 months). Data were not collected from Cell B in July and August 2000, and from Cell C in August 1998. Arrows indicate initiation of PKE addition to the LLCF.

July 2000, and Cell E, August 2000, the decreases in diversity indices, to below 2.0 were due to the numerical dominance of *K. longispina*.

2.3.6. Zooplankton Community Similarity

The proportional similarity of the zooplankton community within each sampling site over time relative to August 1998 is presented in Figure 2.9a. In all cells, zooplankton community similarity decreased relative to the initial sampling date. The greatest decrease was observed in Cell C; from July 1999 onwards there was less than 35% similarity in species assemblages. The proportional similarity within Cell B in July and August 1999 had decreased to 54 and 50%, repectively. In Cell D sites S1 and S2, community similarity fluctuated with sampling date; higher values were seen in August than in July for both 1999 and 2000. Zooplankton community similarity was lower at D S1 than at D S2 for all sampling dates. By August 2000, the similarity of the zooplankton community in Cell E was the lower than previously observed.

Community similarity between adjacent sampling sites over time is presented in Figure 2.9b. Similarity of the zooplankton community did not vary much between Cells C and B despite the drastic alterations in zooplankton abundance within both cells. Initially (August 1998), the proportional similarity between D S1-D S2 and D S2-E were less than 60%, however, by August 2000 similarity was greater than 60%. The same trends of community similarity were observed between Cell E and the other cells over time (Figure 2.9c). The greatest community similarity was observed between Cell E and Cell D sites S1 and S2, with less similarity between Cell E and the sampling sites of Cells B and C.
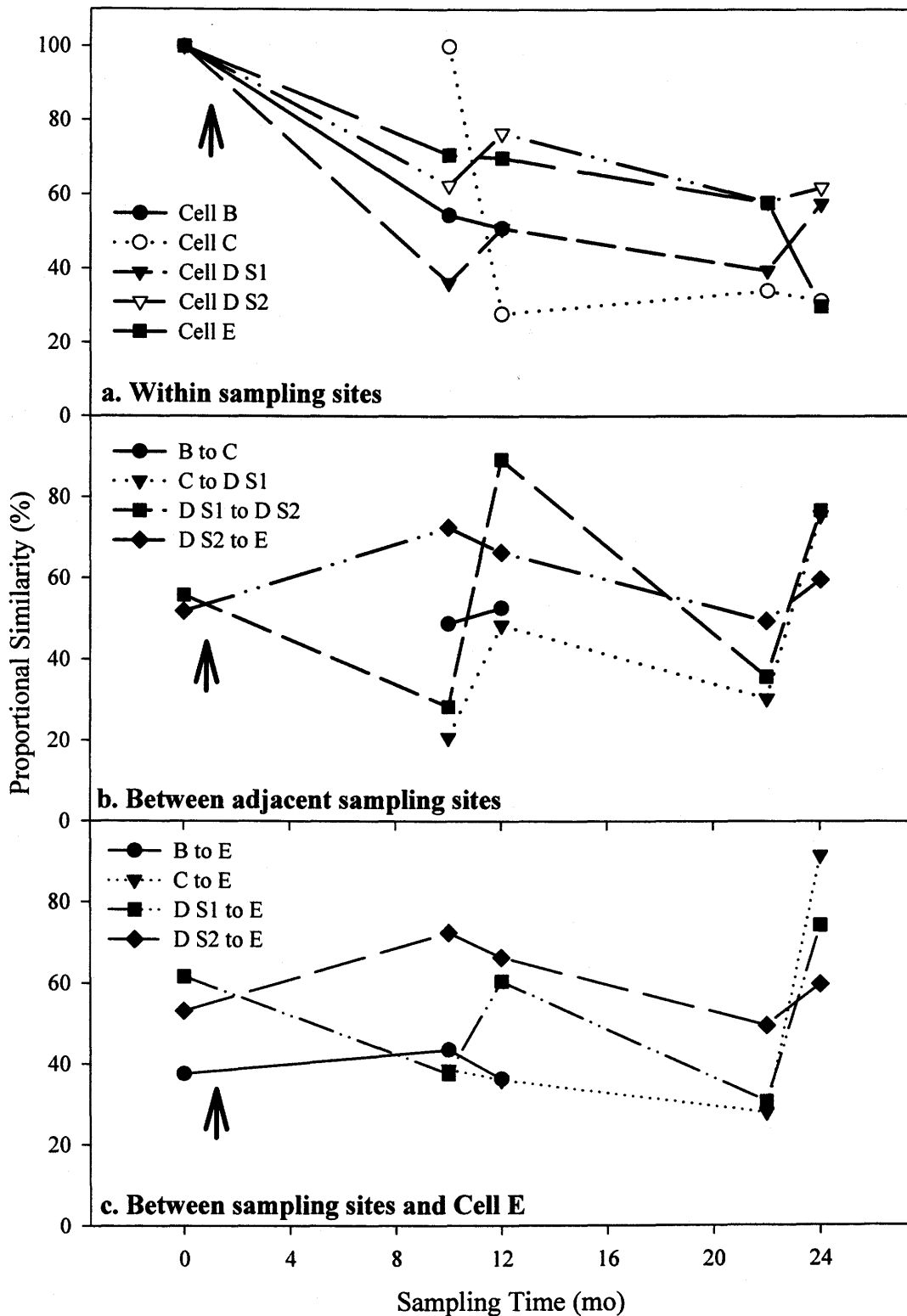
Figure 2.9 Proportional similarity (%) of zooplankton communities (a) within sampling sites over time and (b) between adjacent sampling sites and (c) between Cell E and sampling sites of the other cells over time in the Long Lake Containment Facility (LLCF) from August 1998 (0 months) to August 2000 (24 months). Data were not collected from Cell B in July and August 2000, and Cell C in August 1998. Arrows indicate initiation of PKE addition to the LLCF.

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2.4 Discussion

2.4.1 Water Quality

The addition of PKE and treated sewage effluent to Cell B of the LLCF has altered chemical and physical characteristics of the aquatic environment in the downstream cells. It is assumed that the dams are providing containment of the fine kimberlite tailings, since the turbidity in downstream cells did not change significantly from the beginning to the end of this study. However, the increases in certain water quality variables, concentration of certain metals and nutrients in Cells E indicate that the dams were permeable to some components of PKE and the treated sewage effluent.

The increases in pH, water hardness and alkalinity in the LLCF were most likely caused by the dissolution of kimberlite minerals (Wogelius and Walther 1991, 1992; Howe 1997). Kimberlite is predominantly composed of the magnesium-rich silicate mineral olivine (Mg_2SiO_4) and, to a lesser extent, calcium-rich clinopyroxene ($CaMgSi_2O_6$) and magnesium-rich orthopyroxene ($MgSiO_3$) (Mitchell 1986; Kjarsgaard 1996). In aqueous environments, kimberlite minerals will dissolve into the free magnesium ion (Mg^{2+}), calcium ion (Ca^{2+}), hydroxide ion (OH^+) and silica (SiO_2) (Hodgson and Dudenay 1988). The dissolution of kimberlite minerals in aqueous environments will drive the pH of the water up as free hydrogen (H^+) ions are consumed (Hodgson and Dudenay 1988; Wogelius and Walther 1991; Howe 1997).

Prior to August 1998, the pH of the LLCF was slightly acidic. However, the pH has become slightly alkaline with the addition of PKE. The greatest increases were observed in the receiving cells, followed by the cell directly downstream of deposition. It is expected that the pH of the LLCF, especially in the receiving cells, will continue to

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rise with the deposition of PKE. Howe (1997) demonstrated that the dissolution of kimberlite from the Ekati™ Diamond Mine could result in a pH >9.

The increase in water hardness in the LLCF is a direct result of increases in the divalent cations magnesium and calcium. Other cations, such as strontium and iron are minor contributors to total water hardness (Wetzel and Likens 1991). The slightly alkaline pH in the LLCF in 2000 ensured that magnesium and calcium would remain soluble and, therefore, were able to leach through the dams with downstream flow of water. Magnesium and calcium will remain soluble in moderately alkaline waters (pH<10). Above pH 10, magnesium will begin to precipitate as magnesium carbonate and magnesium hydroxide. Calcium is more soluble than magnesium at alkaline pH, and will not form calcium carbonate precipitates below pH 12 (Cole 1994). It is expected that the water hardness in the LLCF will continue to rise with the deposition of PKE.

Alkalinity, which is governed by the presence of acid neutralizing compounds (Wetzel and Likens 1991), will increase with the liberation of hydroxide ions as kimberlite minerals dissolve (Wogelius and Walther 1991; Howe 1997). Furthermore, in aqueous environments, olivines can be altered by carbonic acid to form silica, carbonate and serpentine ($H_2Mg_3Si_2O_9$) in the following manner:

$$Mg_2SiO_4 + H_2O + CO_2 = H_2Mg_3Si_2O_9 + Mg_2CO_3 + SiO_2$$

The increases of both hydroxides and carbonates are largely responsible for the observed increase in alkalinity in the LLCF.

Conductivity in freshwater lakes is primarily determined by concentrations of the following major ions: calcium, magnesium, sodium, potassium, carbonate, sulphate

and chloride. An increase in conductivity is proportional to an increase in the concentration of total dissolved solids, which is a measure of soluble ions in water (Cole 1994). The increase in the concentration of magnesium and calcium in the LLCF is largely responsible for the observed increase in conductivity.

Barium, strontium, molybdenum and nickel, among other elements, are all found at various concentrations within different xenocryst and macrocystal micas of kimberlite (Mitchell 1986). During ascent to the earth's surface, kimberlite magma may incorporate, but not assimilate, different mantle rocks. Many of these mantle rocks, also called xenoliths, do not survive to the earth's surface, however, individual mineral grains, or xenocrysts, may be present in the kimberlite ground mass. The unique mineralogical content of each kimberlite pipe is determined by the presence of different xenoliths, xenocrysts and/or macrocrysts (a non-genetic term for large crystals of unknown origin, 1-20 cm) (Mitchell 1986; Kjarsgaard 1996). The presence of barium, strontium, molybdenum and nickel in the LLCF indicates that they are part of the mineral make up of the kimberlite pipe that was mined between October 1998 and August 2000. Furthermore, the increases in the concentrations of these elements in Cell E, indicate that they were soluble, or associated with colloidal material that readily leached through the dams.

The addition of PKE and treated sewage effluent to Cell B caused an increase in the concentration of total nitrogen in the LLCF, but not of total phosphorous. Nutrient loading from treated sewage may cause eutrophication of oligotrophic lakes if both phosphorous and nitrogen are available (Freedman 1989). However, according to the Redfield Ratio, nutrient limitations for growth of primary producers may occur if the

ratio of total nitrogen to phosphorous, by weight, deviates from 7:1 (Moss 1998). The large ratios of nitrogen to phosphorous, and the relative low concentrations of total phosphorous, in the cells of the LLCF, indicate that phosphorous is the limiting nutrient for primary production.

The initially high concentration of ammonia in the LLCF (August 1998) was caused by explosive residues left on the recently blasted rock that was used to construct the dams (Rescan 1998a). Ammonium nitrate/fuel oil was used as an explosive to create the waste rock. The increase in the concentration of ammonia in Cells B and C from 1998 to 2000 was a direct result of PKE deposition. However, since the concentration of ammonia decreased in the downstream cells, it is assumed that ammonia was transformed as it moved through the LLCF.

By August 2000, the concentrations of the aforementioned variables in Cell E had not exceeded guidelines for the protection of aquatic life set by the Canadian Council of Ministers for the Environment (CCME 1999). However, the increases in pH, water hardness, alkalinity, certain metals and nutrients in the LLCF may have long-term consequences to the naturally soft-water, slightly acidic, oligotrophic ecosystems found on the arctic tundra. It is predicted that the concentrations of magnesium, calcium, molybdenum, nickel, barium and strontium will continue to increase in the cells downstream of PKE deposition as long as these elements remain soluble, or associated with colloidal material (Wetzel and Likens 1991). Furthermore, in 2000, the concentration of molybdenum in Cell B exceeded the guideline limit of 73 μg/L set by the CCME (1999). Already, lakes downstream of the LLCF are exhibiting higher concentrations of nitrogen and certain metals (Rescan 2000c). The rates of increase of

barium, strontium, nickel and molybdenum in Cell C indicate that these metals easily

leached from the cell of deposition into the downstream cells. It is duly noted that Cell

C sporadically received PKE during operations (Ekati™ Diamond Mine 2000), which

could explain the increase in the concentration of these elements in this cell. However,

the increased concentrations of these elemental components of PKE in Cells D and E

indicate that soluble and/or fine colloid fractions of PKE leached through the dams.

2.4.2 Chlorophyll *a*

The low concentrations of chlorophyll *a*, high DO levels and high Secchi depths

in the cells downstream of effluent deposition indicate that eutrophication is not taking

place. In oligotrophic lakes, the mean annual concentration of chlorophyll *a* is generally

<2.5 mg/m^3, with a maximum of <8 mg/m^3 (Freedman 1989). In 1997, the

concentrations of chlorophyll *a* in Long Lake ranged from 0.4 to 2.1 mg/m^3, with the

highest concentration observed in mid-July (Rescan 1998a). The concentrations of

chlorophyll *a* measured in July 2000 were as high as 6.7 mg/m^3 (Cell C) which is

slightly higher than previously measured values (Rescan 1998a), but still within the

normal range for oligotrophic lakes (Freedman 1989). Usually phosphorous is the

limiting nutrient for primary production in oligotrophic lake (Prepas and Trew 1983;

Ostrofsky and Rigler 1987). The observed increase in chlorophyll *a* concentrations in

Cell B are not thought to be due to an increase in the concentration of phosphorous, but

rather a result of a reduction in grazer selective pressure. The addition of PKE to Cell B

resulted in a significant decrease in the abundance of zooplankton, which, indirectly,

would have a positive effect on primary production, and thus, chlorophyll *a*

concentrations. It is predicted that chlorophyll *a* concentrations will not significantly increase in the LLCF as long as the concentration of phosphorous remains low.

2.4.3 Zooplankton

The community structure and abundance of zooplankton in the LLCF prior to the deposition of PKE and treated sewage effluent were generally similar to those of other oligotrophic lakes found in the Koala watershed (NWT Diamond Project 1995; Rescan 1998b). In 1997, zooplankton abundance in Long Lake typically ranged from 20,000 to 40,000 individuals/m^3 and did not vary greatly between sampling stations and sampling times (Rescan 1998a). Fluctuations in the abundance of zooplankton in Cells C, D and E in 1999 and 2000 were similar to those observed in reference lakes in the Koala and Lac de Gras watershed areas (Rescan 2000c). Although zooplankton abundances were higher in the LLCF in 2000 compared to 1998, with exception of Cell B, these observations were consistent with other lakes in the Lac de Gras region (Rescan 2000c).

The observed alterations in zooplankton abundance, diversity and community similarity within the cells of the LLCF may be a result of both biotic and abiotic changes that have occurred since the deposition of PKE and treated sewage effluent into Cell B in October 1998. Zooplankton populations commonly exhibit irregular seasonal patterns in response to alterations in chemical and biological factors in lakes (Moss 1998). In addition to seasonal influences, factors such as pH, turbidity, ionic concentrations, productivity, and predator assemblages influence patterns of zooplankton distribution and abundance (Dodson and Brooks 1965; Herbert and Hann 1986; Shortreed and Stockner 1986; Kirk 1991a; Pinel-Alloul et al. 1990; O'Brien et al.

1979; Galkovskaya and Arapov 1998; Swadling et al. 2000).

The increases in zooplankton abundance in Cells C, D and E were mostly driven by increases in rotifers and copepod nauplii. Increases in rotifers and nauplii are often caused by increased food availability which may be a result of either eutrophication or the removal of larger zooplankton species (Gannon and Stemberger 1978; Pace 1986; Stemberger and Lazorchak 1994). The process of eutrophication in the LLCF does not appear to be occurring at the present time, however, predator assemblages may have been altered through the Long Lake fish-out programs conducted in 1997, 1998 and 1999 (Rescan 1998a, 1999, 2000). Heavy predation by planktivorous fish could lead to zooplankton assemblages dominated by rotifers and small bodied cladocerans and copepods (Hessen 1994; Stemberger and Lazorchak 1994).

In 1997, the LLCF fish-out program removed large numbers of lake trout (*Salvelinus namaycush*), arctic grayling (*Thymallus arcticus*) and round whitefish (*Prosopium cylindraceum*) (Rescan 1998a). While the hatchlings of these species are planktivorous, the adults have more specialized diets. Lake trout are piscivorous, arctic grayling will feed on terrestrial insects, amphipods and aquatic insects, and round whitefish are benthic feeders (Scott and Crossman 1973). Gill netting (mesh size ranged from 1.9 to 10 cm) was adequate to remove some of the above-mentioned fish in 1997 (Rescan 1999). However, the methods used to gill net fish were inadequate for removing large numbers of small planktivorous fish. In 1998, alterations to the fish out methods resulted in the removal large numbers of lake cisco (*Coregonus artedi*) in Cell C that previously had remained undetected in the Koala watershed. Cisco lifestyle strategies and swimming behaviour may have prevented capture and identification of

this species in previous studies (Rescan 1999). Cisco are planktivorous fish and commonly preyed upon by lake trout (Scott and Crossman 1973). The removal of large lake trout in 1997 was presumed to be partially responsible for the large numbers of one and two year-old cisco present in Cell C in 1998 (Rescan 1999). In 1999, gill nets with smaller mesh sizes (3.8, 2.5, 2.2 and 1.9 cm) were eliminated from the fish-out program (Rescan 2000b). This led to greater removal of larger fish over younger fish and smaller species (Rescan 2000b) and resulted in larger populations of planktivorous fish with time.

The lack of cladocerans in Cell C may have been a result of increased predation by planktivorous fish such as the lake cisco. However, if this were so then populations of Cladocera should have returned by 2000 as result of the removal of planktivorous fish in 1998. Similarly, in Cell D, an increase in rotifers should theoretically have coincided with a decrease in the larger Cladocera, *D. middendorffianna,* and an increase in *B. longirostris*, a smaller cladoceran (Dodson and Brooks 1965; Stemberger and Lazorchak 1994). The fluctuations in populations of both these species over the course of this study do not conclusively indicate that alterations in fish populations have affected zooplankton community assemblages in the LLCF.

It is not easy to determine whether alterations in predator-prey relationships have impacted zooplankton abundance, since other factors such as seasonal variability, food availability, competition among species, and the effect of predaceous zooplankton will also alter community assemblages (Lynch 1979; Hessen 1986; Schindler 1987; Vanni 1987; Shuter and Ing 1997). At times, variations in zooplankton abundance and assemblages may be no more complicated than simple ecological variations,

randomness of sampling and sampling techniques, while at other times community level effects can be directly linked to anthropogenic stress (De Bernardi 1984; Schindler 1987; Shuter and Ing 1997).

The decrease in abundance of zooplankton in Cell B was attributed to the deposition of PKE. The most significant effect was the loss of aquatic habitat as the basin of Cell B gradually filled with fine tailings during the period of this study. Although some species, mainly copepod nauplii, and copepodids and one species of rotifer, were still present in 1999, their low abundances and the absence of cladocerans and mature copepods indicates that components of PKE affected zooplankton community structure. In addition to the loss of habitat, the physical addition of PKE resulted in higher turbidity and significantly altered water chemistry within Cell B which may have decreased the ability of certain zooplankton species to survive.

The increase in turbidity may partially explain the loss of cladocerans in Cell B. Cladocerans are particularly sensitive to turbidity, as suspended clay particles alter feeding patterns and their ability to efficiently ingest phytoplankton (Hart 1990; Kirk 1991a, b). An increase in turbidity above 10 NTU is deleterious to the survival of cladocerans (Hart 1990). Furthermore, smaller cladocerans are less selective filter feeders than larger cladocerans and, therefore, generally are more affected by suspended particles (Kirk 1991a). The decrease in cladocerans can lead to an exploitive increase in rotifers which are better selective feeders, and therefore better equipped to out-compete cladocerans in turbid environments (Kirk 1991a; Pollard et al. 1998). The disappearance of cladocerans in Cell B should have led to higher abundances of rotifers in 1999, however, overall abundances of both groups were significantly lower compared

to 1998, indicating that other factors related to the addition of PKE affected zooplankton survival.

The pattern of competitive exploitation by rotifers in the absence of cladocerans was observed in Cell C. Although Cell C did not exhibit alterations in turbidity at the time of sampling, sporadic pumping of kimberlite fines and sump water from the mine camp area into this cell would have had periodic effects on turbidity. Predator-prey relations in Cell C suggest that the removal of large numbers of plantivorous cisco in 1997 should have led to the emergence of larger bodied zooplankton in the following years (Dodson and Brooks 1965; Scott and Crossman 1973; Rescan 1998b). Therefore, the observed lack of cladocerans and increase in rotifers in Cell C in 1999 and 2000 were more likely to be a direct result of mining activities increasing water turbidity.

Alterations in the physical and chemical properties of water may have more subtle and long-term effects on zooplankton assemblages in the future. The increases in pH, water hardness, and the concentration of certain metals may eventually lead to the disappearance of certain species and the emergence of others (Hellawell 1986; Cole 1994). At the present time, it is thought that the increase in water hardness will have the most profound effect on zooplankton assemblages in the cells downstream of PKE addition.

Certain species of zooplankton cannot live in soft water due to the lack of calcium needed to build calciferous exoskeletons, while other species are intolerant of higher water hardness (Cole 1994). The increase in water hardness may lead to the emergence of calciphilic (calcium loving) species, and the decrease in calciphobes (calcium hating) (Cole 1994). The species *H. gibberum* is generally found in soft waters

with low pH and low ionic strength (Kling *et al.* 1992; Cole 1994; Hessen *et al.* 1995). The disappearance of this species in hard waters may be a direct response to physiological stress, or an indirect result of competitive interference (Cole 1994). Other cladocerans that have higher calcium requirements may become more abundant with increased calcium concentrations and thus out-compete *H. gibberum* (Hessen *et al.* 1995; Cole 1994). It is postulated that *H. gibberum* may disappear from the LLCF in the next few years as a result of increasing water hardness and pH.

Alterations in pH occur naturally in most freshwater aquatic environments in response to increased photosynthesis rates in midsummer (Cole 1994). In highly calciferous waters, the pH is more likely to increase as $CaCO_3$ is removed as a precipitate (Cole 1994). The pH in the LLCF has shifted from slightly acidic to slightly alkaline since the addition of PKE, and may experience greater pH fluctuations in the summer months as the rate of photosynthesis is naturally higher during this season. At the present time, it is difficult to predict the effects of higher pH on aquatic organisms in the LLCF, since many of the identified species are present in a diverse array of aquatic environments (Gannon and Stemberger 1978).

The relatively small increases in the already low concentrations of metals in Cell E since the addition of PKE indicate that there is little concern for metal toxicity to aquatic life downstream of the LLCF at the present time. Furthermore, the increase in water hardness and pH may partially protect aquatic organism from the deleterious affects of certain metal, since metal speciation, and therefore bioavailability and toxicity, is affected by both water hardness (Brown 1968; Hellawell 1986) and pH (Campbell and Stokes, 1985).

2.5 Conclusion

This study only evaluated the changes in the water chemistry in the LLCF over a two year period. During that time, the water chemistry changes in Cells B and C were significantly greater than those in Cells D and E. Although Cell C periodically received direct PKE discharge, changes in water chemistry were also as a result of being directly downstream of Cell B. Eventually, Cell D will be the recipient of PKE, and Cell E, therefore, will be the only buffer for aquatic environments downstream of the LLCF. It is most likely, given the effects of Cell B on the water chemistry of Cell C, that once PKE is deposited into Cell D, the water quality of Cell E may not meet CCME (1999) guidelines for waster water discharged into aquatic ecosystems. Furthermore, given the fragile nature of aquatic ecosystems in the Canadian tundra above the tree-line, the requirements for the disposal into aquatic ecosystems may have to become more stringent than the present CCME guidelines (1999) in order to protect all aquatic life downstream of the LLCF.

Over the last two years, from 1998 to 2000, the addition of PKE and treated sewage effluent to Cell B has altered both the physical and chemical characteristics of the water in the downstream cells. The addition of the treated sewage effluent has increased the concentration of total nitrogen in the LLCF and in downstream lakes, however, phosphorous remains limiting and thus eutrophication was not apparent in the LLCF in 2000. At the present time, it appears that overall zooplankton abundance is increasing in the cells downstream of PKE deposition, however, this increase was mostly driven by smaller zooplankton taxa (e.g. rotifers) with rapid generation times, and by copepod nauplii. The effects of increased water hardness, pH and concentrations

of certain metals on aquatic organisms may become apparent with time. Lastly, inherent variability and previously mentioned confounding factors make it difficult discern the effects that the Long Lake fish-out program had on overall zooplankton abundance and community structure in Cells C, D and E.

The disappearance of certain zooplankton species, such as *H. gibberum*, may occur with time as other species better suited to the changing physical and chemical environment of the LLCF establish themselves. It is predicted that species that have higher calcium requirements will eventually emerge in the LLCF. Whether they form a dominant portion of the zooplankton assemblage will depend on their ability to compete with already established species.

CHAPTER 3

DETERMINATION OF TOXIC COMPONENTS IN PROCESSED KIMBERLITE EFFLUENT FROM THE EKATI™ DIAMOND MINE USING TOXICITY IDENTIFICATION EVALUATIONS

3.1   Introduction

Broken Hills Proprietary's (BHP; now BHP Billiton Inc.) Ekati™ Diamond Mine, located in Canada's Northwest Territories, uses chemical free processes to extract diamonds from their native ore, kimberlite. Mechanical crushing is used to reduce kimberlite to fine particles (≤ 0.5 mm) which are discharged, along with the water employed during the extraction process and treated sewage effluent, to the designated containment facility, Long Lake. The Long Lake Containment Facility (LLCF) is designed to provide full containment of the fine kimberlite tailings, however, there is concern that processed kimberlite effluent (PKE) may contain components that could potentially be toxic to aquatic organisms within the receiving environment and downstream of discharge.

Earlier studies performed on crushed kimberlite solutions determined that toxicity to aquatic organisms was dependent upon the concentration of total suspended solids (TSS) and pH (Harrison *et al.* 1995; Enviro-Test Laboratories 1998). Further investigations indicated that the concentrations of ammonia and aluminum were sufficient to cause acute toxicity at the pH of the kimberlite solution. A series of

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toxicities test confirmed that the elimination of TSS, through filtration, resulted in kimberlite solutions with lower pH and significant reductions in toxicity to aquatic organisms (Harrison *et al.* 1995, 1997; Envrio-Test Laboratories 1998). It was also noted that nickel was present at concentrations which could cause significant stress and potentially be lethal to lake fish over longer periods of exposure (Harrison *et al.* 1997).

These crushed kimberlite solutions were thought to effectively mimic processed kimberlite tailings effluent (PKE) that would later be produced during the diamond extraction processes. However, PKE that is discharged to the LLCF also contains synthetic coagulating and flocculating polymers, as well as treated sewage effluent. The sewage is subjected to both primary and secondary levels of treatment, and should have a biochemical oxygen demand ≤ 40 mg/L and a TSS concentration of ≤ 60 mg/L (Ekati™ Diamond Mine 2000). The coagulating and flocculating polymers are added to the kimberlite fines within the processing plant to clarify water used during the extraction processes and to facilitate fines settling within the LLCF. These polymers will destabilize colloidal suspension by reducing electrostatic charges which accelerate particle flocculation and subsequent removal through settling.

Many cationic polymers used in water treatment today contain multiple nitrogen bearing functional groups that provide a net positive charge. These positively charged nitrogen ions will readily bind to the negatively charged surfaces of colloids, thus neutralizing the electrostatic charges that prevent these particles from binding together and settling out of solution. This is essentially the process of particle coagulation (Siyam 1997). In addition to the use of cationic polymers, anionic polymers are used as flocculating aids. These high molecular weight polymers physically form inter-particle

bridges that draw colloids into aggregates that rapidly settle out of suspension.

Cationic polymers are acutely toxic to daphnids. The 48-h LC_{50} estimates for many cationic polymers are <1 mg/L (Biesinger *et al.* 1976; Biesinger and Stokes 1986; Cary *et al.* 1987; Goodrich *et al.* 1991; Fort and Stover 1995). Although molecular weight does not appear to influence the toxicity of cationic polymers to aquatic organisms, charge density and polymer chemistry are factors controlling polymer toxicity to fathead minnows (*Pimephales promelas*) and daphnids (*Daphnia pulex*) (Hall and Mirenda 1991). Anionic polymers tend to be far less toxic to aquatic invertebrates than cationic polymers (Murgatroyd *et al.* 1996; Biesinger *et al.* 1976).

The goal of this study was to determine the magnitude and cause of PKE toxicity through a series of baseline toxicity tests and toxicity identification evaluations (TIEs) manipulations using the laboratory test species, *Ceriodaphnia dubia*. *C. dubia* was chosen as the test species because they are commonly used in effluent toxicity tests required by regulatory agencies and in TIEs, and because they are widely distributed in North American waters and generally sensitive to contaminants. Toxicity identification evaluations are a cheap and efficient mechanism to characterize, identify, and confirm the identity of toxic components in effluents, ambient waters, sediments pore and elutriate waters, and leachates (US EPA 1991). Phase I TIEs are used to characterize whether toxic constituents are volatile, chelatable, filtratable, reducible, non-polar, or pH sensitive (US EPA 1991). Characterization of the class(es) of the toxicant(s) leads to the initiation of Phase II TIEs which use analytical methods, in conjunction with further toxicity testings, to identify the specific toxic compound(s) and its'/their concentration in the sample tested.

3.2 Materials and Methods

3.2.1 Study Organism

C. dubia were obtained from the Canada Center for Inland Waters (Burlington, ON) and cultured in a Coldmatic Model W.I.C. environmental chamber (Refrigeration of Canada Ltd., Toronto, ON) with a set photoperiod of 16:8 h light:dark and an air temperature of 24.5 ± 0.5 °C. *C. dubia*, maintained according to culturing protocols outlined in Environment Canada (1992), were fed daily with a mixed diet of YCT (yeast, Cerophyll™ and trout chow) and *Selenastrum capricornutum* UTCC 37 (University of Toronto Culture Collection of Algae and Cyanobacteria, Toronto, ON). The final concentrations of YCT and algae in culturing beakers were ~10 mg/L and ~1 mg/L, respectively.

Moderately hard reconstituted water, prepared according to the protocol outlined in Environment Canada (1992), was used for *C. dubia* culturing and toxicity testing. Water quality variables were as follows: temperature 22.4 ± 0.9 °C; dissolved oxygen (DO) 7.8 ± 0.3 mg/L; pH 8.0 ± 0.4; hardness 114 ± 4 mg/L as $CaCO_3$; and alkalinity 76 ± 8 mg/L as $CaCO_3$. Prior to use, water was aerated for a minimum of 24 h and 2 μg/L of selenium (as Na_2SeO_4) and 2 μg/L of vitamin B_{12} (as cyanocobalamin) were added.

S. capricornutum were cultured in a Forma Model 3940M environmental chamber (Forma Scientific Inc., Mariette, OH, USA) with an air temperature of 25 ± 0.5 °C, and a photoperiod of 16:8 h light:dark. Algal cultures were maintained in 1-L Erlenmeyer flasks filled with approximately 500 mL of sterile Freshwater Medium (Environment Canada 1992) and placed on a shaker table in the environmental chamber.

Aliquots of algae were transferred to fresh sterile medium weekly to maintain optimal conditions for exponential growth.

3.2.2 Toxicity Tests Procedures

Acute 48-h *C. dubia* toxicity tests were conducted according to Environment Canada (1990) test methods for *D. magna*, with minor modifications. Five neonates ≤24 h old were randomly placed in 25 mL of either control or test solution in 30-mL glass beakers. For each control or test solution series there were ten replicates. Test beakers were randomly placed on a test board and covered with glass plates to reduce evaporation. *C. dubia* mortality was recorded at 48 h. A toxicity test was considered acceptable if at least 80% of the control organisms survived.

Chronic 7-d *C. dubia* toxicity tests were conducted according to the Environment Canada (1992) test method. *C. dubia* neonates, ≤24 h old and within 8 h of the same age, were placed individually in 20 mL of either control or test solution in 30-mL glass beakers. For each control or test solution there were ten replicates. Test and control solutions were renewed every 24 h and mortality and number of neonates per replicate recorded. Chronic toxicity tests were terminated when 60% of the control organisms had produced their third brood, or at the end of 8 d, whichever occurred first. For a test to be acceptable, 80% of the control organism had to survive and 60% of surviving adults had to produce at least three broods with a total of more than 15 neonates per adult.

Acute and chronic toxicity tests were performed in the same environmental chamber used for culturing, and fed as described under culturing. Toxicity tests were

conducted in 30-mL glass beakers, unless otherwise stated, and covered with glass plates.

Water temperature and DO concentrations were measured with an Orion Dissolved Oxygen Meter Model 835 (Orion Research, Beverly, MA, USA) and pH with an Orion PerpHecT LogR Meter Model 370 (Orion Research, Beverly, MA, USA). Water hardness and alkalinity were measured with a Hach Digital Titrator Model 16900 (Hach Company, Loveland, Co, USA). Ammonia was measured with an Orion Aquafast II Ammonia Photometer (Orion Research, Beverly, MA, USA) and conductivity was measured with an Orion Conductivity Meter Model 170 ATI (Orion Research, Beverly, MA, USA).

3.2.3. Processed Kimberlite Effluent

Processed kimberlite effluent was collected outside the Ekati™ processing plant or at the tailings outlet pipe at Cell B of the LLCF and shipped from the Ekati™ Diamond Mine to Saskatoon, SK, by air at ~4°C in 20-L plastic pails. Upon arrival, 72 to 96 hr after collection, PKE was stored at ~4°C for a maximum of 6 weeks; toxicity tests were initiated anywhere from 3 d to 6 weeks after PKE collection. Appendix E lists the date each batch of PKE was collected, numbered in chronological order of arrival, and the type of toxicity test that was conducted.

Processed kimberlite effluent was prepared for toxicity testing in the following manner: baseline toxicity tests were conducted with whole, supernatant, and/or filtered PKE. Toxicity identification evaluation manipulations were conducted with either supernatant or filtered PKE. Whole PKE consisted of effluent that had been stirred to

resuspend all settled kimberlite fines; the effluent was vigorously shaken prior to being placed in test beakers. Processed kimberlite effluent supernatant was collected from 20-L pails that had remained undisturbed for at least 48 h. PKE filtrate consisted of supernatant that had been filtered through #5 Whatman® paper filters (2 μm pore size) (Whatman International Ltd., Maidstone, England) followed by filtration through Gelman Supor®-450 membrane filters (0.45 μm pore size) (Pallman Laboratories, Ann Arbor, MI, USA) at a rate of 25 to 50 mL/min. Prior to filtering PKE, all filters were rinsed with Milli-Q® water (Millipore Corporation, Bedford, MA, USA). Reconstituted water was drawn through the filters and put aside to serve as control water.

Control and test solutions for each set of experiments were prepared up to 96 h prior to test initiation and stored in 1-L Nalgene® bottles at 4°C. Prior to use, both control and test solutions were slowly warmed in a water bath to approximately 25°C. The mean (±SD) measurements of the physical and chemical characteristics of the reconstituted water and 100% PKE supernatant and filtrate used in the various toxicity tests are provided in Table 3.1.

3.2.4 Baseline Toxicity Tests

Acute toxicity tests were conducted using 100% whole, supernatant, or filtered PKE. Baseline experiments were used to determine the concentration of PKE that significantly decreased fecundity of *C. dubia* in 7-d chronic tests. Chronic toxicity tests were conducted using 100% supernatant or filtered PKE. Chronic toxicity tests, using a 0.5 dilution series with filtered PKE from 100 to 6.25% effluent, were used to determine the lowest observed effect concentration (LOEC) and to establish an effective testing

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Table 3.1. Physical and chemical characteristics of reconstituted water, PKE supernatant, and filtered PKE used in the various toxicity tests and TIEs (mean ± SD).

Characteristic		Reconstituted Water	PKE Supernatant[a]	Filtered PKE[b]
Temperature (°C)	(Initial)[c]	22.4 ± 0.9	22.7 ± 2.0	22.2 ± 1.4
	(Final)[d]	22.5 ± 0.9	n/a	22.2 ± 1.7
Dissolved Oxygen	(Initial)	7.7 ± 0.3	7.2 ± 0.5	7.3 ± 0.7
(mg/L)	(Final)	7.2 ± 0.5	7.5 ± 0.1	6.5 ± 1.0
pH	(Initial)	8.0 ± 0.4	7.8 ± 0.5	8.1 ± 0.7
	(Final)	8.0 ± 0.2	8.3 ± 0.1	7.9 ± 0.8
Alkalinity (mg/L as $CaCO_3$)		76.4 ± 8.1	73.4 ± 16.8	59.4 ± 13.6
Hardness (mg/L as $CaCO_3$)		114.6 ± 3.8	130.4 ± 21.7	170.4 ± 46.3
Conductivity (μS/cm)		406.8 ± 17.5	n/a	486.8 ± 107.4
Ammonia (mg/L)		0.01 ± 0.01	n/a	0.9 ± 0.14

[a]Only includes values for 2 batches of processed kimberlite effluent (PKE).
[b]Includes water chemistry for all batches of PKE tested (see Appendix E for details).
[c]Initial measurements taken at the beginning of each tests for acute toxicity tests and at each water renewal period for chronic toxicity tests.
[d]Final measurements taken at the end of each 48 h period for acute toxicity tests and 24 h period for chronic toxicity tests.
n/a = not available.

concentration for TIE manipulations of PKE. Dilution series were made with reconstituted water.

Tailings effluent that did not contain the treated sewage effluent was obtained from the Ekati™ Diamond Mine in April 2000. This effluent, designated mill processed water (MPW), contained the processed kimberlite fines, synthetic polymers and water employed during the diamond extraction process. A chronic toxicity test, using a 0.5 dilution series, was conducted to determine the LOEC of MPW. The Ekati™ Diamond Mine processing plant utilizes water from Cell C of the LLCF during its' diamond extraction processes. A sample of water from Cell C was obtained in April 2000 and tested for chronic toxicity to *C. dubia*.

3.2.5   Phase I Toxicity Identification Evaluations Manipulations

Phase I TIE manipulations were conducted according to the US EPA (1991) and Environment Canada (1992) test methods. The objective of Phase I TIE manipulations was to characterize the class(es) of compounds responsible for effluent toxicity. All TIE manipulations were conducted as chronic (7-d) toxicity tests using filtered 100%, and sometimes 50%, PKE. Filtered PKE which was not manipulated and used as the baseline was termed PKE (unmanipulated). Effluent which was altered according to the TIE manipulation was designated PKE (manipulated). All TIE manipulations of PKE were duplicated with reconstituted water to ensure that the observed toxic effects were in response to components of PKE and not experimental manipulations. Manipulated reconstituted water was the designated control for each TIE manipulation; unmanipulated reconstituted water was used as a blank control.

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3.2.5.1 Graduated pH Test

Graduated pH tests were conducted to determine if PKE contained toxicants which were pH-dependent, such as ammonia or aluminum. Graduated pH tests were performed with 100% and 50% filtered PKE adjusted to pH 6.5 using 0.1N HCl. Routine measurements of pH at the end of each 24 h period indicated that the pH of PKE was increasing from ~6.5 to ~7.9 between each water renewal period (Figure 3.1). Therefore, to help maintain a relatively constant pH of test solutions, graduated pH bioassays were performed in covered, 30-mL plastic vials with no head-space (Figure 3.1).

3.2.5.2 EDTA Addition Test

The ethylenediaminetetraacetic acid (EDTA) addition test was used to determine if chelatible cationic metals were responsible for the observed toxicity of PKE. EDTA (2.5 g/L) was added to the appropriate test solution daily at each water renewal to yield a final EDTA concentration of 0.5 and 3 mg/L; no more than 5% dilution of test solutions occurred with the addition of EDTA. A minimum of 2 h elapsed before addition of organisms. The first EDTA addition test was conducted using 50 and 100% PKE, the second test was conducted using only 100% PKE.

3.2.5.3 Sodium Thiosulfate Addition Test

The sodium thiosulfate addition test was used to determine if oxidative compounds, such as chlorine, were responsible for PKE toxicity. Sodium thiosulfate (2.5 g/L) was added to the appropriate test solution daily at each water renewal to yield
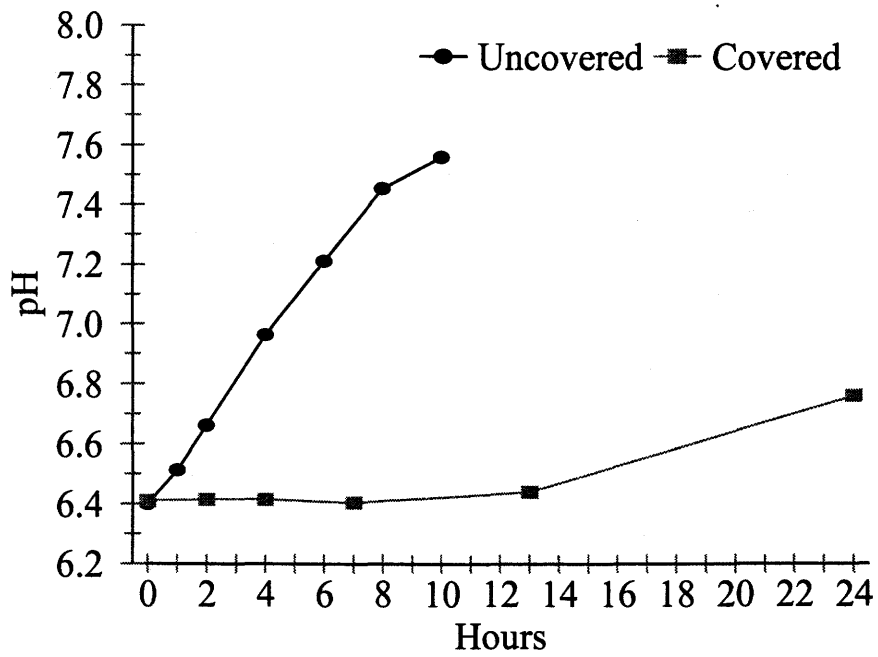
Figure 3.1. Alterations in pH of processed kimberlite effluent adjusted to pH 6.5 with time in uncovered and covered test vessels.

a final sodium thiosulfate concentration of 1, 5 and 10 mg/L; no more than 5% dilution of test solutions occurred with the addition of sodium thiosulfate. Solutions were allowed to equilibrate for 2 h prior to addition of organisms. The sodium thiosulfate addition test was conducted with only 100% PKE.

3.2.5.4 Aeration Test

Aeration was used to determine if PKE contained toxic compounds which were volatile, spargeable, or oxidizable. Filtered PKE was aerated for 60 min in 2-L beakers using an Elite 799™ (Mansfield, MA, USA) pump.

3.2.5.5 Post C-18 Solid Phase Extraction Column Test

Solid phase extraction with C-18 disks was used to determine if non-polar organics were a component of the toxic fraction of PKE. 3M Empore® high performance C-18 disks (3M, St. Paul, MN, USA) were used for SPE of PKE. For this set of TIE manipulations, SPE was conducted at a rate of 100 mL/min using previously filtered PKE. All C-18 SPE disks were conditioned with methanol and Milli-Q® water as described elsewhere (US EPA 1991). Post-SPE PKE was collected and tested for toxicity.

3.2.5.6 pH Adjustments

Major pH adjustments (to pH 3 or 11) were used to determine if components of PKE were altered in such a way that the toxicants could be removed with subsequent filtration or filtration/SPE. Initial pH adjustments were conducted with filtered PKE.

The supernatant of PKE was filtered, adjusted to pH 3, left for 2 h, and then filtered through 3M Empore® high performance C-18 SPE disks at a rate of 10 mL/min. Following this experiment, all pH adjustments were made to the supernatant of PKE. Effluent was adjusted to pH 3 or 11 with either $0.1N$ HCl or $0.5N$ NaOH, left for ~12 h (experiment with batch 9) or for ~2 h (experiment with batch 10), and then filtered through #5 Whatman® paper filters (2 μm pore size) and followed by filtration through Gelman Supor®-450 membrane filters (0.45 μm pore size). For the experiment with PKE batch 9, one aliquot of each manipulation was put aside directly for toxicity testing purposes, the other was filtered through 3M Empore® SPE disks at a rate of 10 mL/min. Toxicity testing for these two TIE manipulations were performed concurrently. Batch 10 was subjected to pH adjustments/filtration and tested to confirm results from batch 9. Prior to toxicity testing all solutions were adjusted back to the initial pH (~8.2).

3.2.5.7 Anionic Resin, Cationic Resin and Activated Charcoal Tests

The anionic resin used in these TIE manipulations was AG® 1-X8 Resin, 20-50 mesh, hydroxide form, and the cationic resin was AG® 50W-X8, 20-50 mesh size, hydrogen form; both resins were supplied by Bio-Rad Laboratories (Hercules, CA, USA). PKE was manipulated by adding 1.25 g/L of each resin, or 2.5 g/L of activated charcoal (J.T. Baker Inc., Phillipsburg, NJ, USA), to 2 L aliquots of 100% filtered PKE. Solutions were stirred for 2 h, then held at 4°C. The resins and activated charcoal were removed 12 h later by filtration through Gelman Supor®-450 membrane filters (0.45 μm pore size). The addition of both resins altered the pH of PKE; the cationic resin decreased the pH to 3.05, and the anionic resin increased the pH to 10.95. Adjustments

back to pH*i* (~8.2) were made with either 0.1*N* HCl or 0.5*N* NaOH prior to toxicity testing.

3.2.5.8 Kaolinite and Bentonite Addition Tests

Kaolinite and bentonite are anionic colloidal particles that should bind to positively charged molecules such as the cationic polymers. These clays were used, in conjunction with filtration, in an attempt to reduce the toxicity of PKE. Kaolinite and bentonite (Sigma® Chemical Co., St. Louis, MO, USA) were added to PKE supernatant at concentrations of 0, 1 and 5 g/L. Solutions were stirred for 2 h and then allowed to settle for 18 h at 4°C. Filtration through #5 Whatman® paper filters (2 μm pore size) and followed by filtration through Gelman Supor®-450 membrane filters (0.45 μm pore size) removed kaolinite and bentonite from each solution.

3.2.6   Phase II Toxicity Identification Evaluation

3.2.6.1 Metals and Ammonia Analyses

Inductive coupled plasma-mass spectrometry (ICP-MS) analysis was performed by the University of Saskatchewan Geological Sciences Department to determine the concentration of metals present in both the supernatant and filtrate of PKE. The concentration of ammonia in PKE was determined using an Orion Aquafast II Ammonia Photometer (Orion Research, Beverly, MA, USA).

3.2.6.2 Alterations in Water Quality Variables

To ensure that neither ammonia nor increased water hardness were affecting the

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fecundity of *C. dubia*, 7-d chronic tests were conducted for both of these variables. The total hardness of reconstituted water was adjusted to 220 mg/L as $CaCO_3$, the highest water hardness of PKE measured to date (December 1999). Since ammonia is a pH dependent toxicant, the reconstituted water used in these experiments was made to resemble, as close as possible, the water characteristics of PKE at that time (July 2000); the mean water hardness was 160 mg/L as $CaCO_3$, alkalinity was 60 mg/L as $CaCO_3$, and pH was 8.3. Two 7-d chronic experiments were conducted to determine the concentration of ammonia that inhibited fecundity of *C. dubia*; test concentrations ranged from 0.1 to 80 mg/L. A 1,000 mg/L standard solution (Orion Research, Beverly, MA, USA) was used to generate the ammonia concentration of each test solution.

3.2.6.3 Toxicity Testing of MagnaFloc® 156 and 368

The toxicity of the two polymers, MagnaFloc® 156 and 368 (Ciba Speciality Chemical, Inc., Suffock, VA, USA), used at the Ekati™ Diamond Mine to *C. dubia* was determined through a series acute (48-h) and chronic (7-d) tests in reconstituted water. Test concentrations were chosen based on the 48-h LC_{50} estimates for *D. magna* listed in the material safety data sheets (MSDS) for each polymer.

Acute 48-h toxicity tests with both polymers were performed with pH adjustment tests to determine if pH altered the chemistry of the polymers in a manner that would affect toxicity to *C. dubia*. Adjustments to pH 3 were made with 0.1 *N* HCl; solutions were allowed 2 h to equilibrate, and then readjusted to pH*i* (8.0 ± 0.4). Further toxicity testing of MagnaFloc® 156 was not performed as the final concentration of this polymer in PKE was estimated to be approximately 10 times lower than the

observed 48-h LC_{50} estimate for *C. dubia*. Adjustments to pH 11, in conjunction with filtration, were conducted with MagnaFloc® 368; solutions were adjusted to pH 11 with $0.5N$ NaOH and allowed to equilibrate for 24 h. One aliquot was adjusted back to pH*i* with $0.1N$ HCl, while the other was filtered through Gelman Supor®-450 membrane filters (0.45 μm pore size) and then adjusted back to pH*i* with $0.1N$ HCl.

3.2.7   Statistical Analysis

Fecundity, or the mean number of neonates per replicate, was the endpoint used to determine the LOEC for the chronic serial dilution studies and significant differences between different concentrations of TIE manipulated PKE and controls. Significant differences in fecundity were determined using one-way analysis of variance (ANOVA) followed by a Bonferroni *t*-test. Where the test for normality failed, the Kruskal-Wallis one-way ANOVA on ranks was run followed by Dunn's Test. Student's *t*-test was used to determine significant differences between the control and PKE for each TIE manipulation; if the test for normality failed, differences were determined using the Mann-Whitney Rank Sum Test. Statistical analysis was performed using the computer program SigmaStat® Software Version 2.03 (1992) with a 95% ($\alpha = 0.05$) level of confidence.

Median lethal concentration (LC_{50}) estimates and associated confidence intervals were calculated using the trimmed Spearman-Karber method (Version 1.5, US EPA 1990). Chronic EC_{50} and EC_{20} estimates for ammonia and MagnaFloc® 368, using fecundity as an endpoint, were determined using the Inhibition Concentration (ICp) approach, which is a linear interpolation method for estimating sub-lethal toxicity

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(Version 2.0, US EPA 1993). Confidence intervals were calculated by a bootstrap procedure that subsamples the original data set 999 times to obtain a robust estimate of the standard error.

3.3   Results

3.3.1   Baseline Toxicity Tests

Survival of *C. dubia* exposed to 100% PKE supernatant or filtrate was, in both cases, greater than 80% (data not shown). Whole 100% PKE proved to be acutely toxic to *C. dubia*; survival of exposed organisms for the 48-h test period was 44%.

Chronic exposure of *C. dubia* to 100% PKE filtrate and supernatant had a variable effect on survival which appeared to be independent of filtration and the batch of PKE tested (Figure 3.2a). A significant reduction in fecundity was observed in organisms exposed to both 100% PKE supernatant and filtrate. Control organisms had an average of 12.5 ± 5.8 and 21.9 ± 7.5 neonates per replicate, while organisms exposed to 100% PKE averaged 0.5 ± 1.0 and 4.3 ± 3.7 neonates per replicate (supernatant), and 3.6 ± 3.8 and 3.4 ± 1.3 neonates per replicate (filtrate) (Figure 3.2b).

Seven-d LC_{50} estimates for PKE (Batch 2) and mill processed water (MPW), calculated from serial dilution experiments, are present in Table 3.2. Estimates for PKE (Batch 3) could not be calculated since mortality was not observed during this test. Data on the survival of *C. dubia* exposed to 100% PKE filtrate (Batches 2 and 3) and MPW are presented in Figure 3.3a. Survival of *C. dubia* was ≥90% for PKE and MPW concentrations <25% effluent, but variable at ≥25%. The LOEC for PKE, using fecundity as the endpoint, was 12.5% or 25% effluent, depending on the batch tested

Figure 3.2. Percent survival (a) and mean (±SD) fecundity (b) of *C. dubia* exposed to 100% processed kimberlite effluent (PKE) supernatant and filtrate. *Denotes a significant difference between control organisms and 100% PKE supernatant or filtrate as determined by one-way ANOVA ($p<0.05$).

Table 3.2. Seven-d LC_{50} estimates for *C. dubia* exposed to processed kimberlite effluent (PKE) and mill processed water (MPW).

Type of Effluent	LC_{50} (%)	95% L.C.L.[a] (%)	95% U.C.L.[b] (%)	TSK Trim[c] (%)
PKE Batch 2	46.7	24.7	88	30
MPW	37.5	18.1	77.4	30

[a]95% lower confidence limit.
[b]95% upper confidence limit.
[c]Trimmed Spearman-Karber trim.

Figure 3.3. Survival (a) and mean (±SD) fecundity (b) of *C. dubia* exposed to a dilution series of processed kimberlite effluent (PKE) (Batch 2 & 3) and mill processed water (MPW). MPW did not contain treated sewage effluent. *Denotes that a significant difference exists between respective control organisms and those in different concentrations of PKE or MPW as determined by one-way ANOVA ($p<0.05$).

(Figure 3.3b). MPW appeared to be of comparable toxicity to PKE; the LOEC was 25% MPW (Figure 3.3b).

These chronic serial dilution toxicity tests were initiated three weeks after Batch 2 was collected and 96 h after Batch 3 was collected (Appendix E). For both batches fecundity of organisms exposed to 100% filtered PKE was less than four neonates per replicate (Figure 3.3b). These results suggest that the toxicity of PKE likely did not decrease significantly with 21 d storage at 4°C in 20-L plastic pails.

C. dubia exposed to raw Cell C water averaged 15.3 ± 7.3 neonates per replicate which was not significantly different from controls which averaged 19.7 ± 5.0 neonates per replicate. The raw water from Cell C was collected and stored for 5 weeks in the same 20-L plastic pails used for storage of PKE before toxicity tests were initiated. If storage in plastic pails contributed to the toxicity of PKE, then Cell C water should have elicited a toxic response. These results, therefore, suggest that storage of PKE in the plastic pails did not contribute to toxicity.

3.3.2 Phase I TIE Manipulations

The EDTA addition (Figure 3.4), sodium thiosulfate addition (Figure 3.5) and aeration (Figure 3.6) tests failed to reduce the toxicity of PKE to chronically exposed *C. dubia*. Fecundity of *C. dubia* exposed for 8-d to PKE altered by these TIE manipulations was not significantly different from those in unmanipulated PKE.

Graduated pH adjustment, to pH 6.5, partially reduced the toxicity of 50% and 100% PKE to *C. dubia* (Figure 3.7a,c). Survival of *C. dubia* was variable, but appeared to be greater in pH adjusted PKE compared to pH unadjusted PKE (Figure 3.7b,d)

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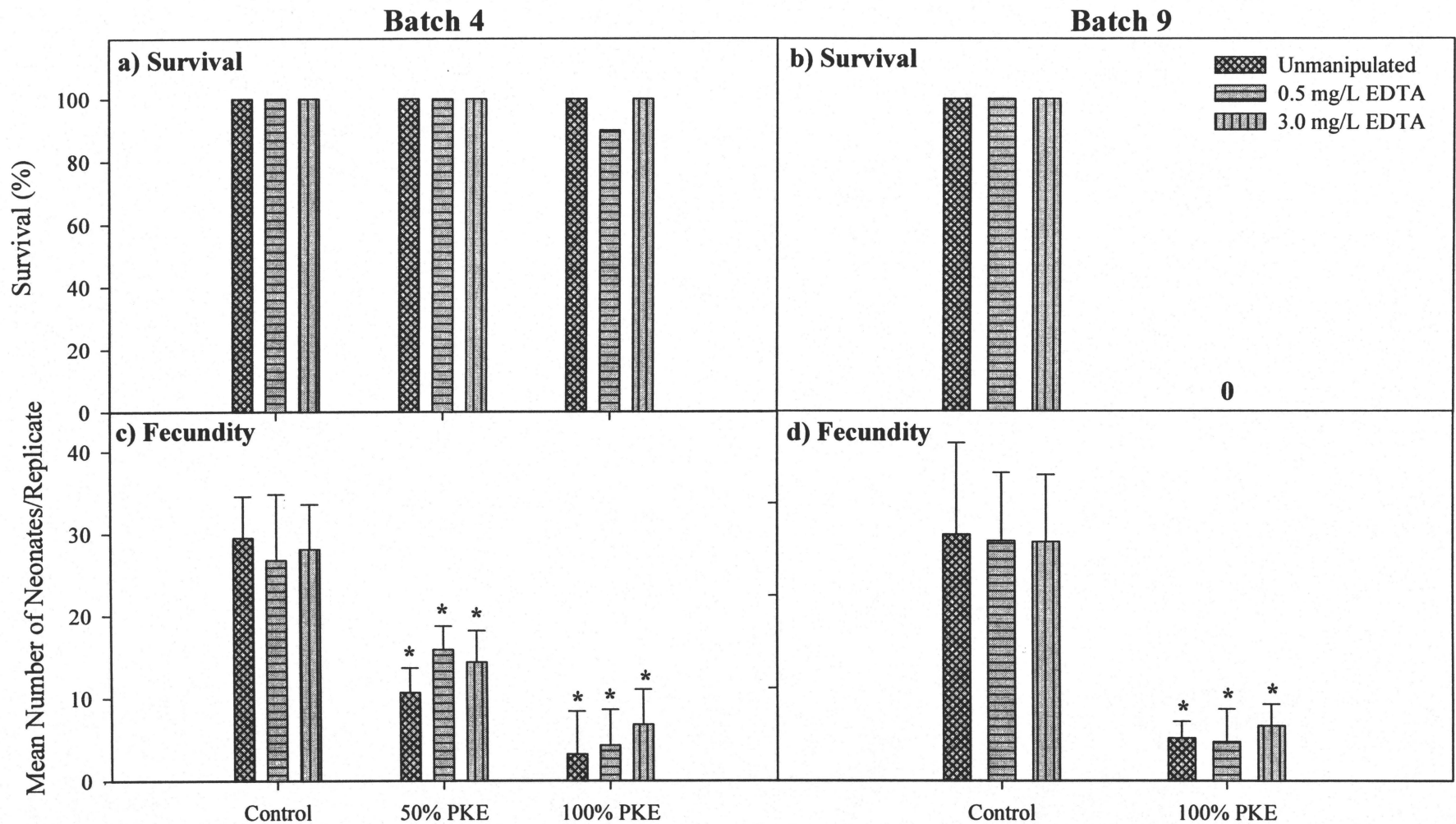
Figure 3.4. Survival (a & b) and mean (±SD) fecundity (c & d) of *C. dubia* exposed to processed kimberlite effluent (PKE) manipulated with different concentrations of EDTA; 50% PKE was not tested for Batch 9. *Denotes that a significant difference exists between organisms in controls and 100 or 50% PKE as determined by one-way ANOVA ($p < 0.05$). There was no significant difference between PKE manipulated with different concentrations of EDTA.
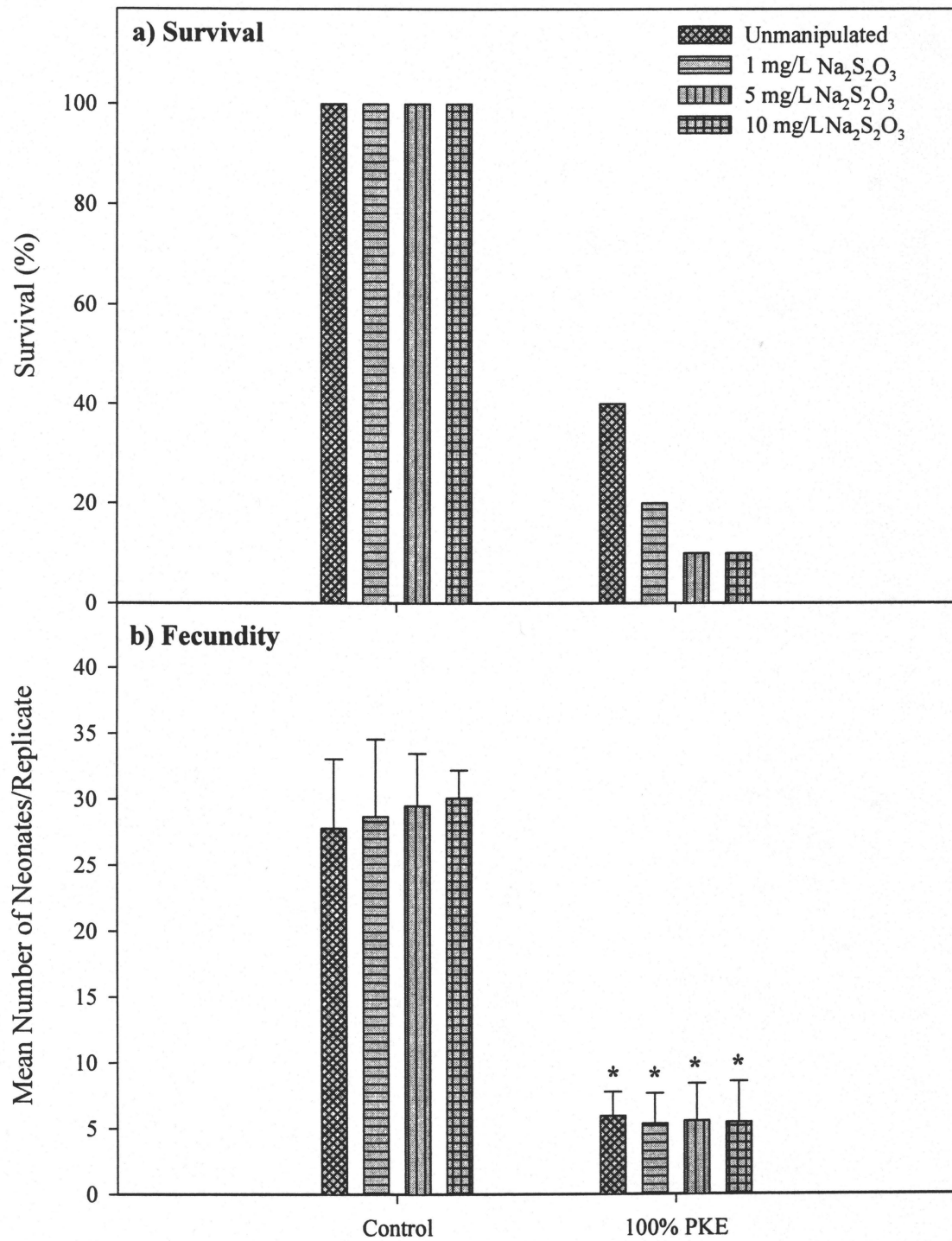
Figure 3.5. Survival (a) and mean ($\pm$SD) fecundity (b) of *C. dubia* exposed to processed kimberlite effluent (PKE) manipulated with different concentrations of sodium thiosulfate. *Denotes that a significant difference exists between organisms in controls and 100% PKE as determined by one-way ANOVA ($p < 0.05$).
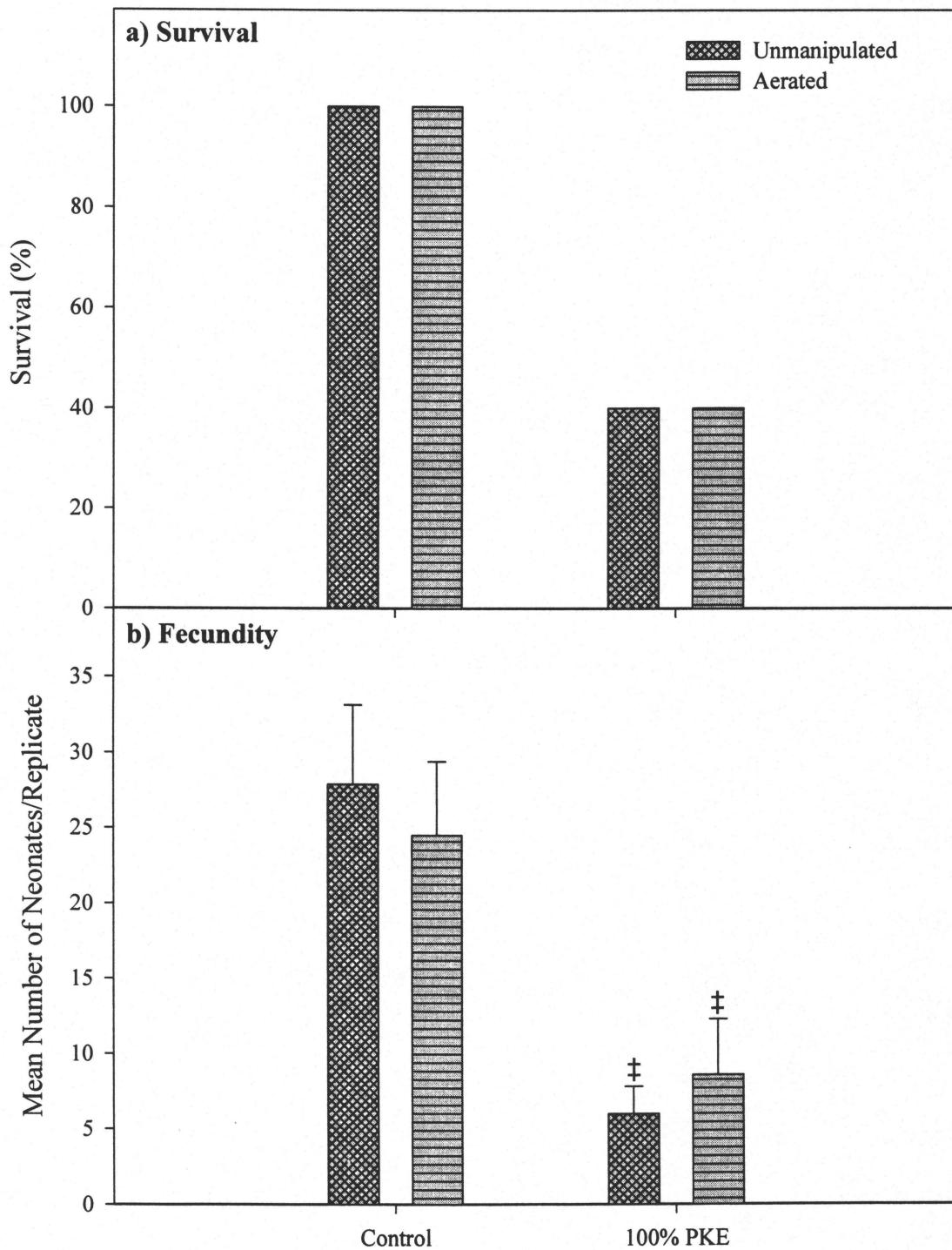
Figure 3.6. Survival (a) and mean (±SD) fecundity (b) of *C. dubia* exposed to 100% processed kimberlite effluent (PKE) that was either not aerated (unmanipulated) or aerated for 1 h. ‡Denotes that a significant difference exists between either the unmanipulated or aerated control and the respective 100% PKE treatment as determined by a Students *t*-test (*p*<0.05).
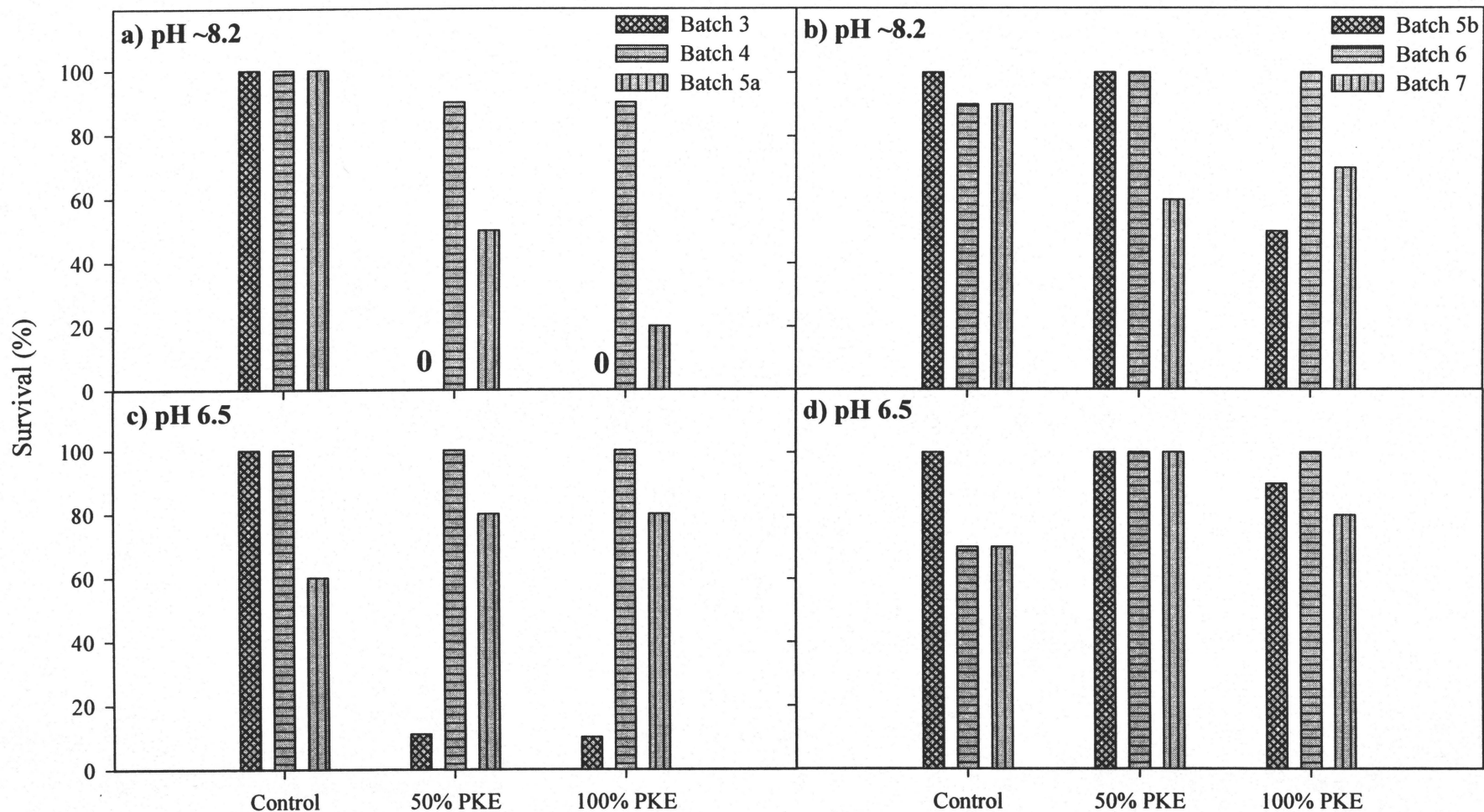
Figure 3.7. Survival of *C. dubia* exposed to 100 and 50% processed kimberlite effluent (PKE) at the initial pH of ~8.2 (a & b) and the adjusted pH of 6.5 (c & d). The adjusted pH of Batch 3 was not properly maintained at 6.5 for the 24 h period between each solution renewal. Batch 5 was tested twice, hence 5a and 5b.

Fecundity was significantly increased with exposure to PKE that was adjusted to pH 6.5

compared to unadjusted PKE (Figure 3.8a,c). However, *C. dubia* exposed to pH

adjusted PKE had significantly fewer neonates per replicate than controls which

indicated that toxicity was only partially removed with graduated pH adjustment.

Graduated pH tests run on Batches 5a, 6 and 7 failed to meet the US EPA (1991)

survival and reproductive criteria for a successful toxicity test. However, close

examination of the results from these experiments revealed that there was still a trend of

reduced toxicity.

Solid phase extraction at initial pH, pH~8.2, did not reduce the toxicity of PKE

to *C. dubia* (Figure 3.9a & c). Although survival was greater, fecundity of organisms

exposed to post-SPE PKE was not significantly different from fecundity of organisms

exposed to unmanipulated PKE. Solid phase extraction of filtered PKE at pH 3 showed

a greater reduction in toxicity; survival of *C. dubia* was greater and there was a

significant increase in fecundity compared to unmanipulated PKE (Figure 3.9c & d).

However, SPE at pH 3 did not completely remove toxicity of PKE that had been filtered

prior to pH adjustments; fecundity was still significantly lower in post-SPE PKE

compared to controls (Figure 3.9d).

The toxic fraction of PKE was removed with major pH adjustments in

conjunction with filtration (Figure 3.10). Adjustment of PKE to pH 3 followed by

filtration eliminated toxicity, whereas adjustment to pH 11, followed by filtration,

significantly reduced toxicity. Major pH adjustments in conjunction with filtration

(Figure 3.10) or SPE (Figure 3.11) were conducted concurrently, therefore statistical

comparisons could be made between filtered PKE and post-SPE. Solid phase extraction
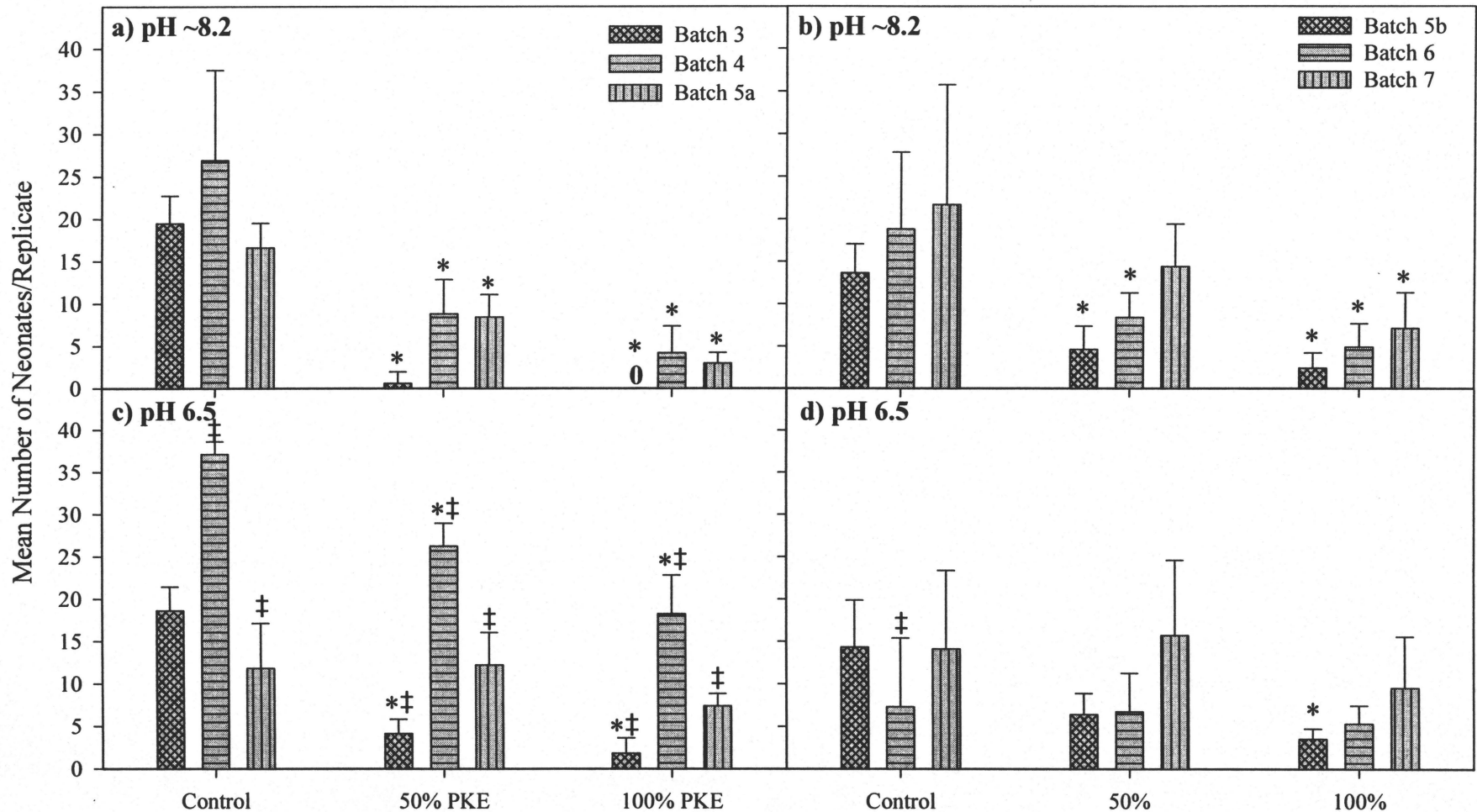
Figure 3.8. Mean (±SD) fecundity of *C. dubia* exposed to 100 and 50% processed kimberlite effluent (PKE) at pH*i* ~8.2 (a & b) and pH 6.5 (c & d). *Denotes a significant difference between fecundity in control and 100% or 50% PKE as determined by one-way ANOVA (*p*<0.05). ‡Denotes that there was a significant difference between concentrations of PKE that were either unmanipulated or adjusted to pH 6.5 as determined by a Student's *t*-test (*p*<0.05). The adjusted pH of Batch 3 was not properly maintained at 6.5 for the 24 h period between each solution renewal. Batch 5 was tested twice, hence 5a and 5b.
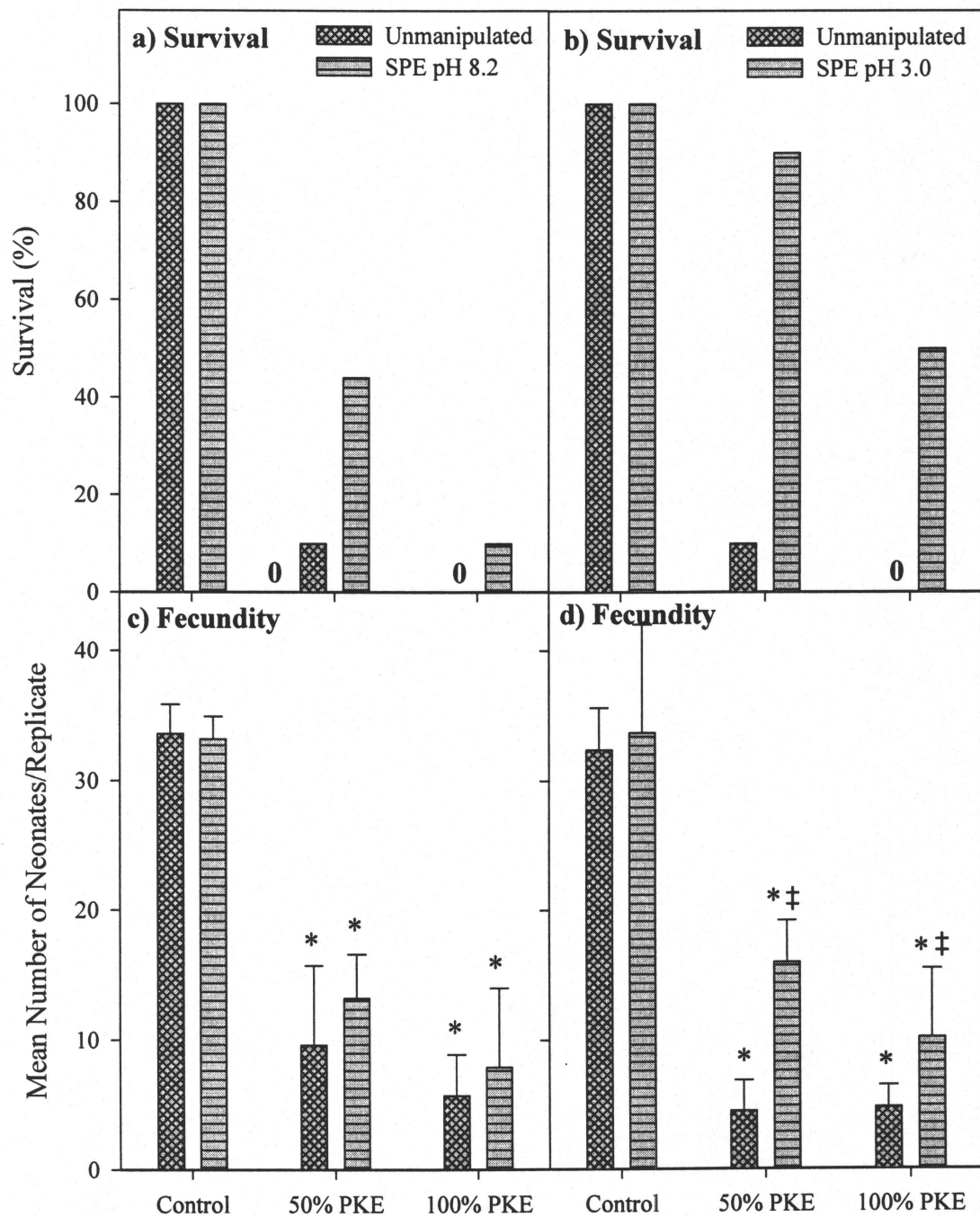
Figure 3.9. Survival (a & b) and mean (±SD) fecundity (c & d) of *C. dubia* exposed to processed kimberlite effluent (PKE) at pH*i* ~8.2 (a & c) and adjusted to pH 3 (b & d) prior to solid phase extraction (SPE); pH adjustments were made on filtered PKE. *Denotes a significant difference between organisms in controls and 100% or 50% PKE as determined by one-way ANOVA ($p<0.05$). ‡Denotes a significant difference between unmanipulated PKE and post-SPE PKE as determined by Student's *t*-test ($p<0.05$).
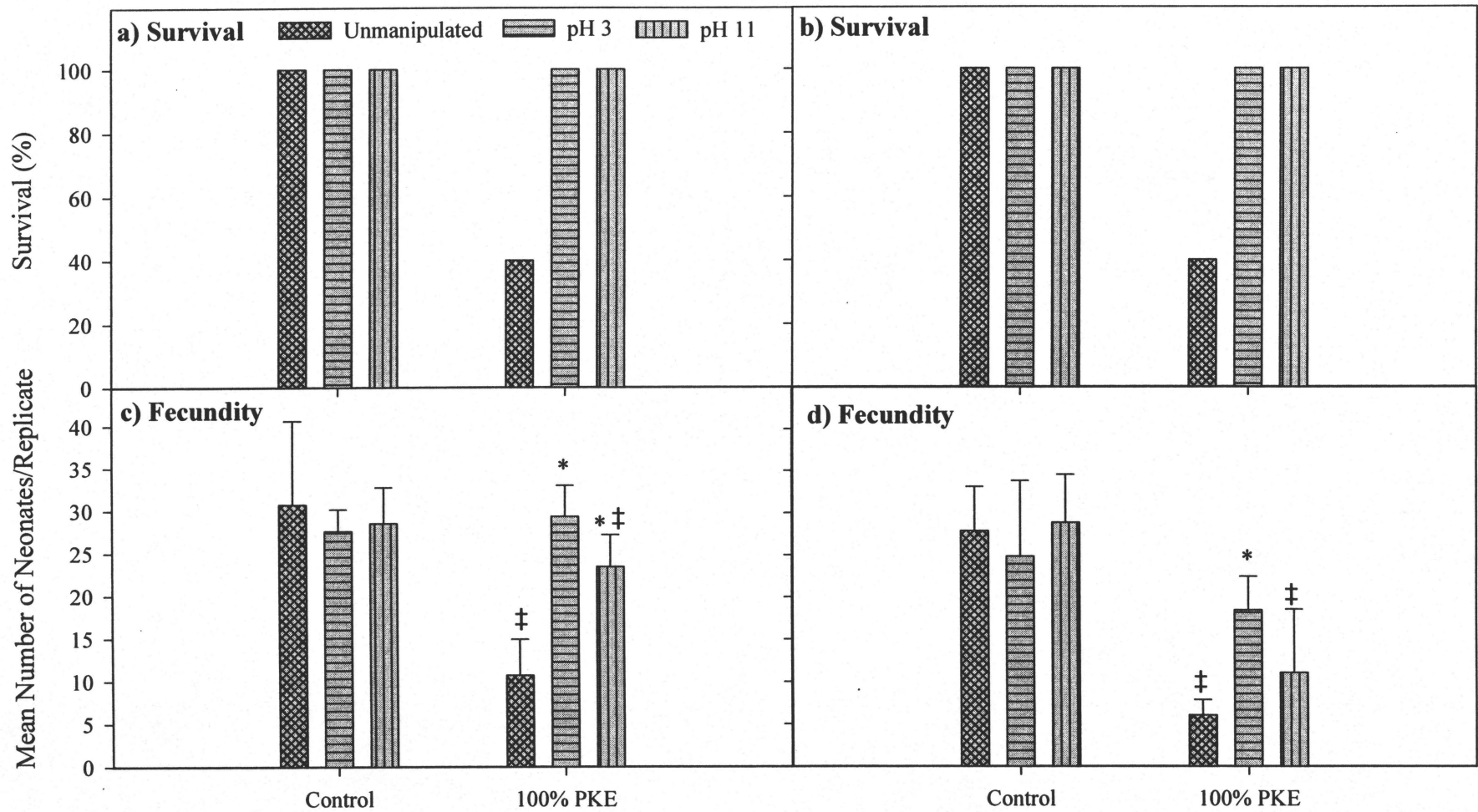
Figure 3.10. Survival (a & b) and mean (±SD) fecundity (c & d) of *C. dubia* exposed to 100% processed kimberlite effluent (PKE) adjusted to pH 3, ~8.2 (unmanipulated), or 11 followed by filtration. *Denotes a significant difference between organisms in controls and PKE at pH 8.2 and pH 3 or 11 as determined by one-way ANOVA ($p<0.05$). ‡Denotes a significant difference between organisms in controls and PKE at each pH adjustment as determined by a Student's *t*-test ($p<0.05$).
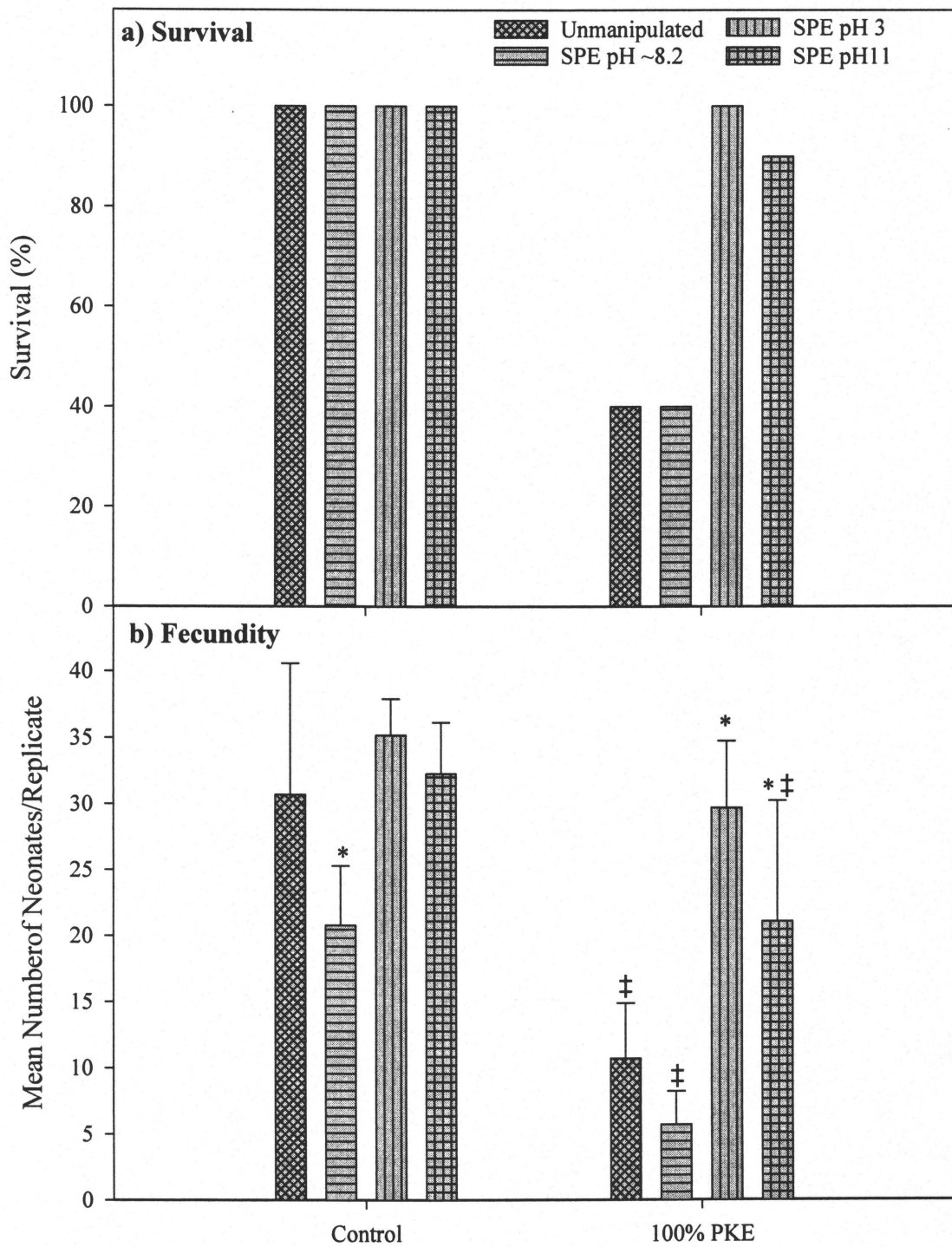
Figure 3.11. Survival (a) and mean (±SD) fecundity (b) of *C. dubia* exposed to post-solid phase extraction (SPE) processed kimberlite effluent (PKE). SPE was performed using 100% PKE supernatant at pH 3, ~8.2, or 11. *Denotes a significant difference among treatments for organisms in controls or those exposed to PKE as determined by one-way ANOVA ($p<0.05$). ‡Denotes a significant difference between organisms in controls and PKE for each manipulation as determined by a Student's *t*-test ($p<0.05$).

with pH adjustments proved to be a redundant step; there was no significant difference in survival or fecundity between the filtered PKE and the filtered/SPE PKE for each pH adjustment. Survival of *C. dubia* was greater for organism exposed to pH adjusted PKE compared to unmanipulated PKE (Figure 3.10a,b & 3.11a). Fecundity in controls and PKE that was adjusted to pH 3 prior to filtration was not significantly different (Figures 3.10c & 3.11b). Fecundity of PKE adjusted to pH 11 for both batches 9 and 10 was significantly reduced compared to the respective controls, indicating adjustments to pH 11 only partially reduced toxicity (Figure 3.10c & d). There was a significant increase in fecundity for *C. dubia* exposed to PKE adjusted to pH 11 prior to filtration compared to unmanipulated PKE (Figure 3.10c). However, this trend is not observed in Figure 3.10d; there is no significant difference between unmanipulated PKE and PKE adjusted to pH 11 prior to filtration.

The toxic fraction of PKE was completely removed with the addition of cationic and anionic resins; neither survival nor fecundity were significantly different from controls (Figure 3.12a). The toxicity of PKE was partially reduced by the addition of activated charcoal; there was greater survival and the mean number of neonates per replicate was significantly greater than in the unmanipulated PKE (Figure 3.12b). However, fecundity of *C. dubia* exposed to PKE with activated charcoal was significantly lower than the controls for this manipulation.

The toxicity test with bentonite and kaolinite addition failed to meet criteria for a valid test; 60% of controls did not have 3 broods with 15 neonates or more in 7(± 1)-d. However, there was 100% survival for all controls in all treatment groups. The survival data revealed some important trends: 100% PKE was toxic to *C. dubia* since there was

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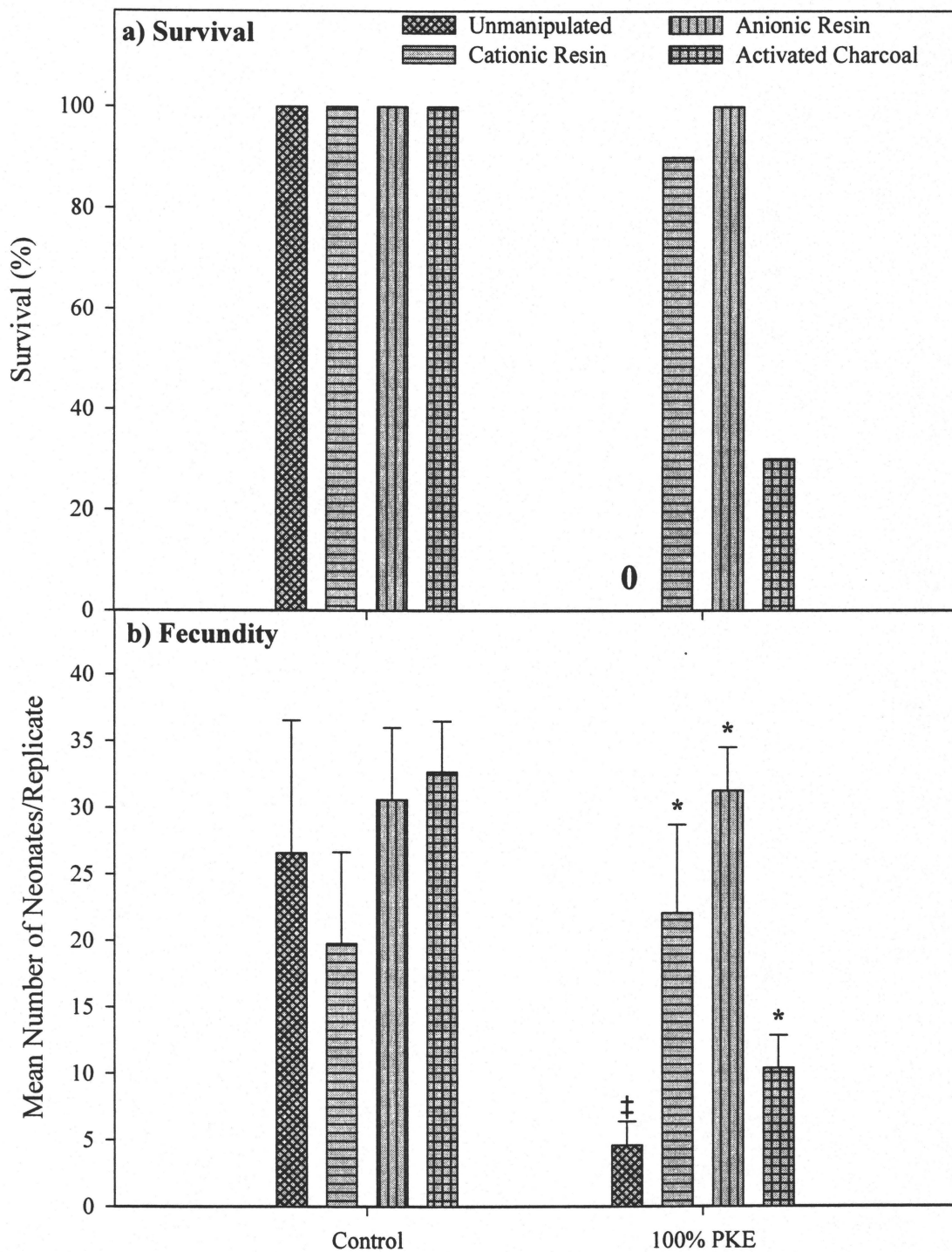
Figure 3.12. Survival (a) and mean (±SD) fecundity (b) of *C. dubia* exposed to 100% processed kimberlite effluent (PKE) manipulated with anionic and cationic resins, or activated charcoal. *Denotes a significant difference between unmanipulated control organisms and those in PKE or the TIE manipulated control and PKE, as determined by one-way ANOVA ($p < 0.05$). ‡Denotes a significant differences between organisms in controls and PKE for each manipulation as determined by a Student's t-test ($p < 0.05$).

no survival; exposure to PKE with either 1 g/L of bentonite or kaolinite increased survival to 20 and 60%, respectively; and exposure to 5 g/L of bentonite or kaolinite increased survival to 80% (Figure 3.13a). Fecundity data for this toxicity test were difficult to interpret since the toxicity test did not meet US EPA (1991) criteria for a successful test (Figure 3.13b). Statistically, a significant difference in fecundity was not observed between unmanipulated PKE and PKE treated with either bentonite or kaolinite at both concentrations tested.

3.3.3   Phase II TIE Tests

3.3.3.1 Metals and Ammonia Analyses

Analysis of PKE by ICP-MS determined that metals were not present at concentrations that should be toxic to *C. dubia*, assuming comparable toxicity to *D. magna* (Table 3.3). The ammonia concentrations in filtered PKE ranged from 0.61 to 1.12 mg/L; the mean concentration was 0.9 ± 0.14 mg/L (Table 3.1)

3.3.3.2 Hardness and Ammonia Tests

Neither survival nor fecundity of *C. dubia* were affected by increasing the total hardness of reconstituted water from 115 mg/L to 220 mg/L as $CaCO_3$. The toxicity test with ammonia (0 to 80 mg/L) produced a 7-d LC_{50} estimate of 14.01 ± 4.44 mg/L and a 7-d EC_{50} estimate, using fecundity as the endpoint, of 4.89 ± 1.38 mg/L.

3.3.3.3 Toxicity Tests with MagnaFloc® 156 and 368

Table 3.4 contains the 48-h LC_{50} estimates for MagnaFloc® 156 and 368 toxicity
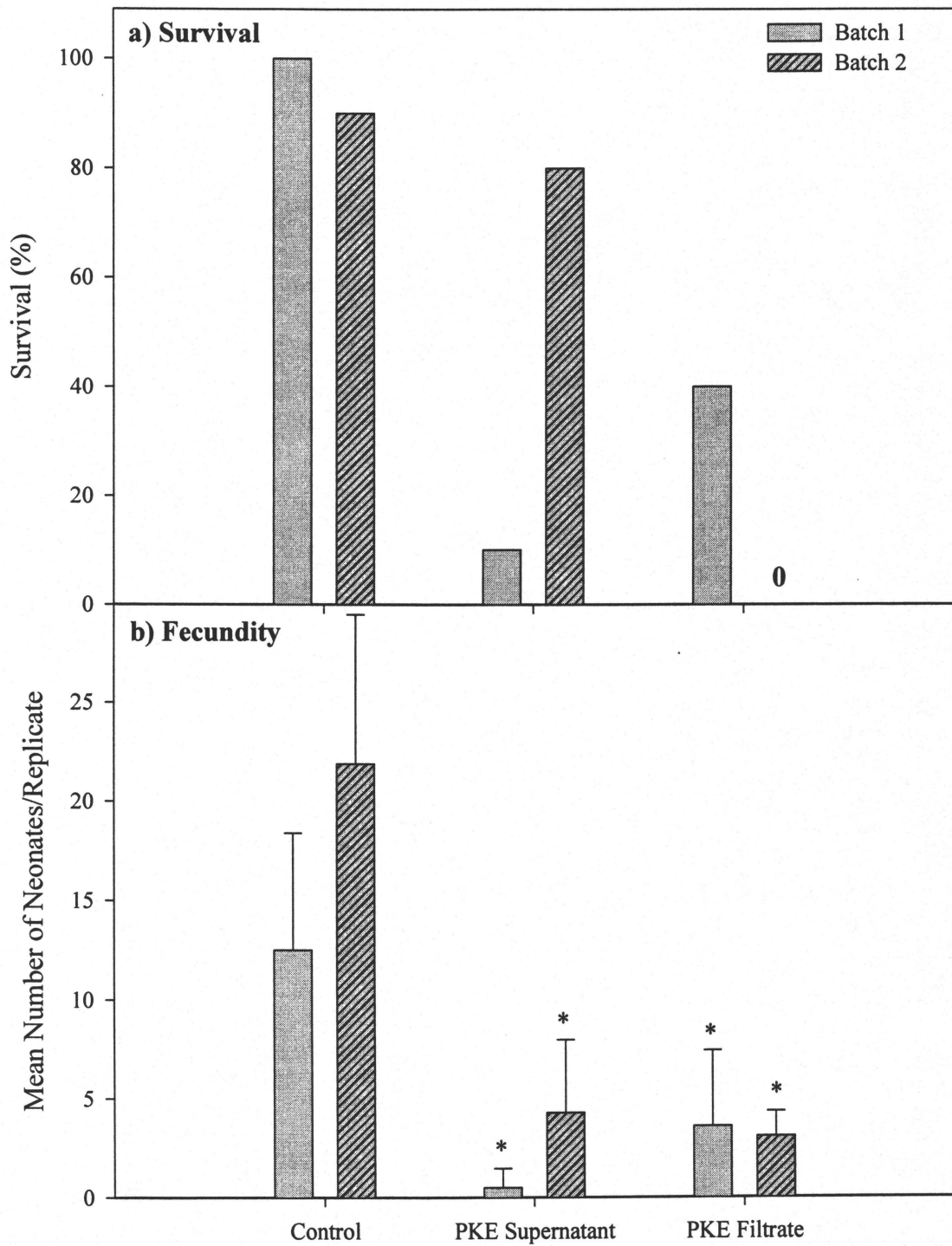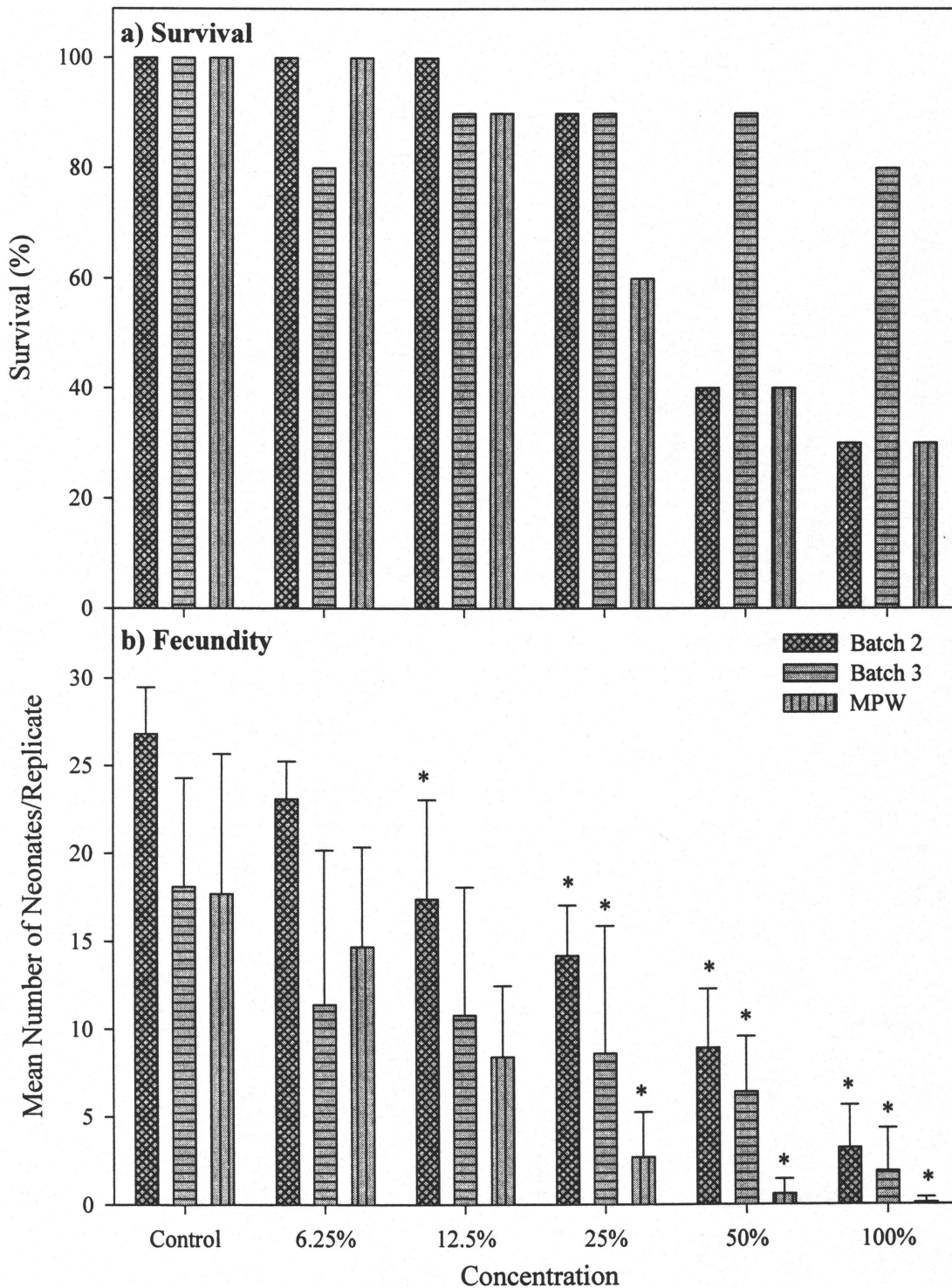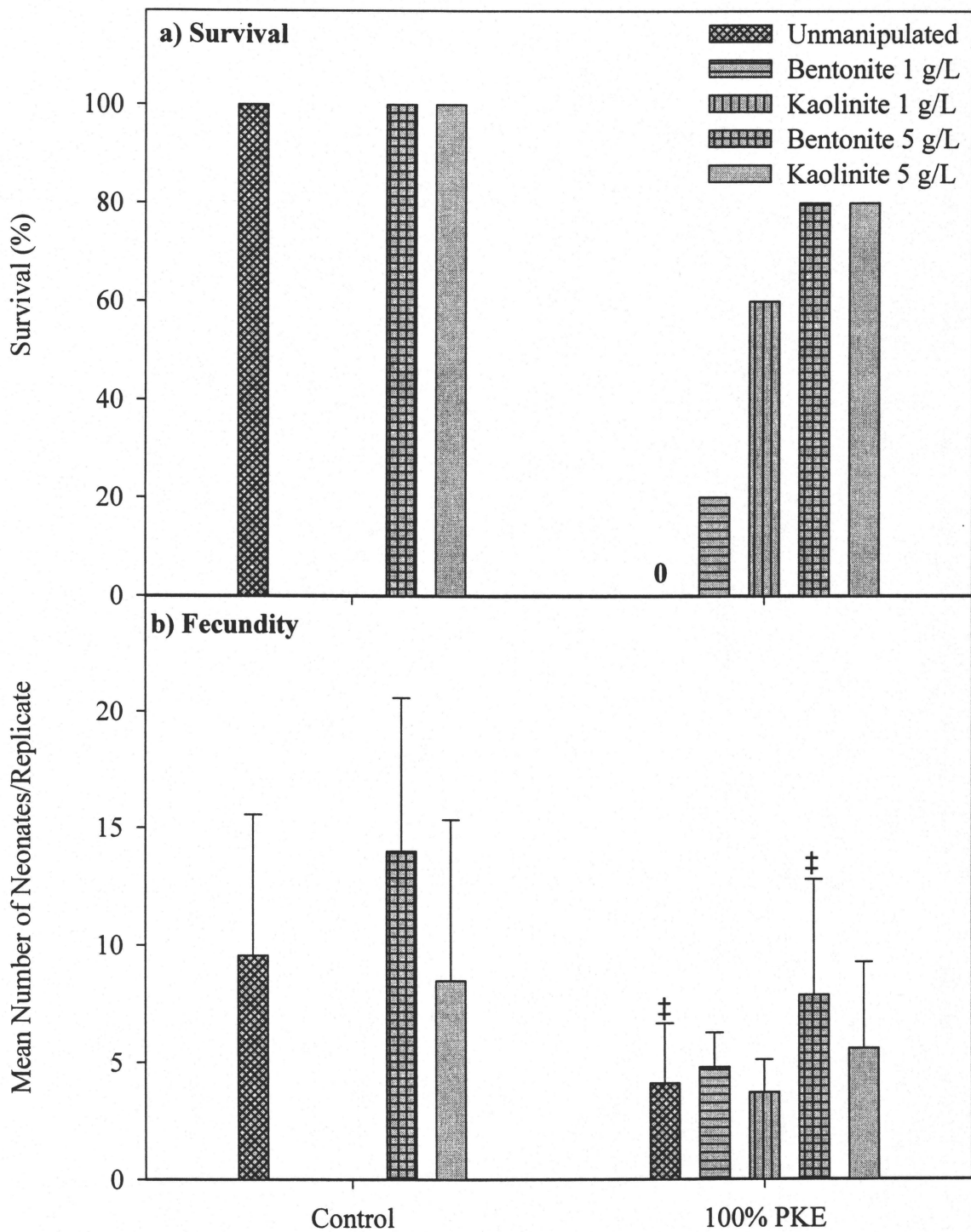
Figure 3.13. Survival (a) and mean (±SD) fecundity (b) of *C. dubia* exposed to 100% processed kimberlite effluent (PKE) treated with different concentrations of bentonite or kaolinite. There was no significant difference in fecundity among organisms in the various controls and those exposed to PKE as determined by one-way ANOVA ($p<0.05$). ‡Denotes a significant differences between organisms in controls and PKE for each manipulation as determined by a Student's *t*-test ($p<0.05$). 100% PKE treated with 1 g/L of either bentonite or kaolinite was compared to controls treated with 5 g/L of each respective clay.

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Table 3.3. Mean concentration of quantifiable trace elements in the supernatant and filtrate of processed kimberlite effluent (PKE), and their relationship to published 21-d IC_{50} estimates for *D. magna*.

Element[a]	Supernatant[b] (μg/L)	Filtrate[c] (μg/L)	*D. magna* 21-d IC_{50}[d] (μg/L)
Aluminum	12.72[e]	0.1 - 3.1	680
Arsenic	0.02 - 0.04	0.02 - 0.05	1,400
Barium	1.94 - 15.65	4.9 - 7.0	3,200
Copper	1.01 - 1.78	0.27 - 0.85	35
Iron	0.09 - 1.27	0.1 - 0.3	5,200
Molybdenum	14.45 - 15.78	14.43 - 16.46	797,000[f]
Nickel	0.32 - 0.76	0.15 - 0.51	95
Lead	0.09 - 0.10	0.0 - 0.1	100
Strontium	321.2 - 373.6	204.9 - 361.1	60,000

[a]The following elements were below their detection limit (≤ 0.01 μg/L): Ag, B, Be, Bi, Cd, Ce, Co, Cr, Mn, Se, Sb, Sn, Zn, U.
[b]Mean concentration ranges from analysis of PKE batch 1 and 2.
[c]Mean concentration ranges from analysis of PKE batch 1 ,2 and 4.
[d]Reproductive inhibition (from Biesinger and Christensen 1973).
[e]Aluminum concentration was determined for only one sample.
[f]Reproductive inhibition (from Naddy *et al.* 1995).

Table 3.4. 48-h LC$_{50}$ estimates for *C. dubia* exposed to MagnaFloc® 156 and 368.

MagnaFloc® Compound	pH Adjustment	LC$_{50}$ (mg/L)	95% L.C.L.[a] (mg/L)	95% U.C.L.[b] (mg/L)	TSK Trim[c] (%)
156	pH*i* (~8)	218.1	181.2	262.4	0
156	pH 3	93.0	75.5	114.6	0
368	pH*i* (~8)	0.32	0.27	0.39	0
368	pH 3	0.54	0.51	0.56	0
368	pH 11	0.51	0.47	0.55	0
368	pH 11 with filtration[d]	0.77	0.61	0.98	11.54

[a]95% lower confidence limit.
[b]95% upper confidence limit.
[c]Trimmed Spearman-Karber trim.
[d]MagnaFloc® 368 was adjusted pH 11 and filtered through 0.45 μm Gelman Supor®-450 membrane filters prior to readjustment to pH*i* (~8.2).

Table 3.5. Seven-d EC$_{50}$ and EC$_{20}$ estimates of MagnaFloc® 368 to *C. dubia*.

	7-d Estimates (mg/L)	95% L.C.L.[a] (mg/L)	95% U.C.L.[b] (mg/L)
EC$_{50}$	0.014	0.0047	0.0233
EC$_{20}$	0.0042	0.0011	0.0128

[a]95% lower confidence limit.
[b]95% upper confidence limit.

to *C. dubia*. Adjusting MagnaFloc® 368 to pH 3 slightly decreased the toxicity of this compound to *C. dubia*, whereas the toxicity of MagnaFloc® 156 was slightly increased with adjustments to pH 3. Adjusting MagnaFloc® 368 to pH 11 appeared to slightly reduce the toxicity to *C. dubia*. The 7-d EC_{50} and EC_{20} estimates of MagnaFloc® 368 toxicity to *C. dubia* were 0.014 and 0.0042 mg/L, respectively (Table 3.5).

3.4   Discussion

Phase I of the TIE study provided very little information on the specific nature of the toxicant(s) in PKE. Earlier research conducted by DFO demonstrated that the toxicity of crushed kimberlite in solution could be reduced with the removal of TSS (Harrison *et al.* 1995, 1997; Enviro-Test Laboratories 1998). The persistence of toxicity in this study, despite filtration, indicated that PKE contained toxic components which were not identified and/or present when toxicity testing was conducted on the crushed kimberlite solutions. Furthermore, the toxicity of PKE could not be reduced with EDTA and sodium thiosulfate addition tests, nor with aeration and SPE. Although these tests do not provide information on the specific group of toxicants present, the failure of these tests to reduce the toxicity of PKE indicates that certain toxicants were not present at significant concentrations.

Failure of the EDTA and sodium thiosulfate addition tests to reduce the toxicity of PKE indicated that certain cationic metals and oxidative compounds, respectively, were not present at concentrations sufficient to cause the observed toxicity (US EPA 1991). EDTA will strongly chelate with the following divalent cationic metals: Zn, Ni, Pb, Mn, Cu, Hg, Fe, and Cd, and weakly chelate with Ba, Ca, Co, Mg, Sr, and Tl (US

EPA 1991; Hockett and Mount 1996). Sodium thiosulfate, in addition to reducing the toxicity of oxidants such as chlorine, bromine, iodine, and ozone (US EPA 1991), is also effective at reducing the toxicity of common metals such as Ag, Se(VI), Cu, Hg, and Cd (Hockett and Mount 1996). The results from these two TIE procedures indicated that neither metals nor oxidative compounds were likely to be responsible for PKE toxicity. Furthermore, ICP-MS analysis of PKE confirmed that metals were not present at sufficient concentrations to be chronically toxic to *C. dubia* (Biesinger and Christensen 1973).

The persistence of chronic toxicity of PKE after aeration implies that toxicity was not caused by volatile compounds. Aeration will reduce the toxicity of compounds which are oxidizable, spargeable, or sublatable (US EPA 1991). In addition, if non-polar organics were responsible for the observed toxicity of PKE, then SPE with C-18 disks should have reduced toxicity (US EPA 1991).

Partial reduction of PKE toxicity with minor pH adjustment to pH 6.5, indicated that pH-dependent toxicants, such as ammonia or aluminum, may have been present in PKE (US EPA 1991; Anderson and Buckley 1998). Previous research conducted by DFO demonstrated that ammonia and aluminum, in combination with high concentration of TSS and high pH, were possible toxicants of crushed kimberlite in solution (Harrison *et al.* 1995; Enviro-Test Laboratories 1998). However, analysis of PKE during this study established that neither aluminum (Biesinger and Christensen 1973) nor ammonia were present at concentrations high enough to be chronically toxic to *C. dubia*.

The toxicity of ammonia to aquatic organisms is largely attributed to the un-

ionized (NH_3) form (Emerson et al. 1975; Erickson 1985). The concentration of NH_3 relative to the ionized form (NH_4^+) is dependent upon pH, temperature, and the total concentration of ammonia present. At pH 8, the estimated 7-d EC_{50} of total ammonia to *C. dubia* was 9 mg/L (Cowgill and Milazzo 1991) which corresponds to approximately 1 mg/L of NH_3 (Anderson and Buckley 1998). The total concentration of ammonia in PKE was approximately 10 times lower than the value reported by Cowgill and Milazzo (1991) and five times lower than the 7-d EC_{50} estimate established with in-house toxicity testing.

The complete removal of the toxic fraction of PKE with major pH adjustments in conjunction with filtration, provided little specific information on the nature of the toxicant(s) present in PKE. The results from this experiment indicated that components of PKE were being chemically and/or structurally modified by pH such that the toxicant(s) were destroyed, or sorbed to particulate matter and removed via filtration.

A limited weight-of-evidence approach was used to identify the toxic components of PKE. The results of the aforementioned TIE manipulations and the previous research conducted by Harrison (1995; 1997) and Enviro-Test Laboratories (1998) suggested that crushed kimberlite fines were not responsible for the observed toxic effects of PKE to *C. dubia*. The only known components of PKE that were not in the previously tested crushed kimberlite solutions were the treated sewage effluent, the synthetic polymers, and the waste water from the diamond extraction processes. The possibility that the treated sewage effluent contributed to toxicity was eliminated through toxicity testing of MPW; MPW was essentially PKE without the treated sewage. The persistent toxicity of MPW was comparable to that of PKE indicating that

the treated sewage was not the source of the toxic components. Furthermore, the water

from Cell C, which was used during the diamond extraction processes, was not toxic,

suggesting that the toxicity of PKE was generated within the processing plant.

The only known toxic components added to PKE during processing were the

cationic (MagnaFloc® 368) and anionic (MagnaFloc® 156) polymers. MagnaFloc® 368

and 156 were added to kimberlite fines at rates of 35 g/t and 55 g/t, respectively

(personal communication, Curtis Mohns, Ekati™ Plant Metallurgist). It was difficult to

estimate the final concentration of these polymers in PKE because water from the

diamond extraction processes is recycled within the processing plant. However, given

that approximately 40% of PKE was crushed kimberlite and approximately 60% water

(Ekati™ Diamond Mine 2000), the final concentration of MagnaFloc® 368 and

MagnaFloc® 156 in PKE should be approximately 40% of the original dosage to the

kimberlite fines, or approximately 14 and 22 mg/kg, respectively (approximately 14 and

22 mg/L assuming a PKE density of 1.0).

Anionic polymers tend to be relatively non-toxic to aquatic invertebrates

(Biesinger *et al.* 1976; Murgatroyd *et al.* 1996). In-house toxicity testing of

MagnaFloc® 156 determined that the 48-h LC_{50} estimate of 218 mg/L to *C. dubia* was

comparable to the reported 48-h EC_{50} estimate of 212 mg/L to *D. magma* listed in the

MagnaFloc® 156 MSDS. It is, therefore, unlikely that the anionic polymer was

responsible for the toxicity of PKE since the estimated concentration of MagnaFloc®

156 in PKE was approximately 10 times lower than concentration which was acutely

toxic to daphnids. It is not suggested that anionic polymers are non-toxic, only that it

appears that the contribution of MagnaFloc® 156 to PKE toxicity was minor compared

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to that of the cationic polymer.

Cationic polymers, on the other hand, are very toxic to several cladoceran species and fathead minnows (Biesinger *et al.* 1976; Biesinger and Stokes 1986; Cary *et al.* 1987; Goodrich *et al.* 1991; Fort and Stover 1995). The mechanism of toxic action of cationic polymers to aquatic organisms is through surface membrane interactions. Biesinger and Stokes (1986) found that electrostatic forces bound the cationic polymer to negatively charged sites on fish gills and induced lamellar cell proliferation which eventually destroyed the lamellae structure. Increased lamellar cell proliferation decreased respiration efficiency as the distance between oxygen in the water and red blood cells in the gill epithelium increased. It is likely that fish mortality was a result of suffocation and reduced ability to maintain internal ionic balance (Biesinger and Stokes 1986). The mechanism of toxicity of cationic polymers to cladocerans is slightly different. Here, the polymer will bind to the surface of the integument and/or to appendages, thus inhibiting movement and the subsequent uptake of nutrients. Smaller daphnids tend to be more susceptible to the physical effects of cationic polymers due to greater surface area in relation to size (Rowland *et al.* 2000).

The 48-h LC_{50} estimate for the cationic polymer, MagnaFloc® 368, to *D. magma* has been reported as 0.33 mg/L (listed in the MSDS), which was comparable to the 48-h LC_{50} estimate of 0.32 mg/L established here with *C. dubia*. The 7-d EC_{50} estimate of 0.014 mg/L for MagnaFloc® 368 was 1000 times lower than the estimated MagnaFloc® 368 concentration in PKE. Therefore, less than 0.1% of this polymer would have to be free in solution to elicit the observed toxic effect of PKE to *C. dubia*. Polymers are known to be less toxic to aquatic organisms in waters with a high degree of suspended

solids (Biesinger and Stokes 1986; Cary *et al.* 1987; Hall and Mirenda 1991; Goodrich *et al.* 1991; Fort and Stover 1995). For example, the 48-h LC_{50} estimates for one group of cationic polymers to *C. dubia* ranged from 0.07 to 0.12 mg/L in reconstituted water and from 2.74 to 7.06 mg/L in solutions with 1,350 mg/L and 2,300, respectively, of suspended solids (Fort and Stover 1995).

Unfortunately, a complete weight of evidence approach could not be used to confirm that excess cationic polymer was responsible for the toxicity of PKE since it was impossible to obtain effluent samples without the cationic polymer for toxicity testing purposes. It is, however, possible that the cationic polymer is added in excess to kimberlite fines within the processing plant. The identification of the cationic polymer as the toxic component of PKE is therefore based on the high toxicity of this compound to *C. dubia*, evidence from the TIE manipulations performed, and the behaviour of kimberlite minerals in solution. Furthermore, the failure of the EDTA addition, aeration and SPE TIE manipulations to remove toxicity of PKE is good evidence that the cationic polymer may be the toxic component. Dissolved cationic polymers are not volatile, chelatable, or extensively filterable, and they are extremely polar (Rowland *et al.* 2000).

Coagulants and flocculant aids are frequently overused in wastewater treatment facilities (Fort and Stover 1995; Rowland *et al.* 2000; Hamilton and Sutcliffe 1997). Limited knowledge of the effectiveness of polymers, in combination with in-plant fluctuations in the concentration and nature of total suspended solids, make it difficult to gauge the concentration of polymer needed to effectively settle suspended particles. Often trial and error is used to determine the concentration and type of polymer, or

combination of polymers, that are most effective at settling colloidal particles (Penniman 1981, Howe 1997). There are many factors that affect the effectiveness of polymers in wastewater including: solution pH, particle size, type of suspended solids, dosage rates, molecular weight and charge density of polymer, mixing techniques, and temperature (Howe 1997; Siyam 1997).

The reduction of PKE toxicity with the addition of both the anionic and cationic resins was mostly to be a result of the interaction of cationic polymer with the charged resins. The cationic resin, in solution, will carry a net negative charge and should bind to positively charged particles such as the cationic polymer, which would subsequently be removed during the filtration process. The change in the pH of PKE with the addition of the cationic and anionic resin may have also altered toxicity. The addition of the cationic resin caused the pH of PKE decrease to pH 3, while the anionic resin increased the pH to 11. It is possible that the decrease in pH of PKE with the addition of the cationic resin could have caused the reduction in toxicity in the same way that adjustments to pH 3 in conjunction with filtration removed the toxic fraction of PKE. However, it is noted that both resins were added to PKE post filtration, while major pH adjustments only altered the toxicity of PKE that was adjusted as supernatant. Furthermore, the removal of PKE toxicity with the addition of the anionic resin is unlikely since both the anionic resin and cationic polymer carry positive charges. At this time, it is not possible to determine how the toxicity of PKE was removed with the addition of the anionic resin.

Originally, it was thought that the loss of PKE toxicity with major pH adjustments was due to chemical alterations of the cationic polymer rendering it non-

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toxic to aquatic organisms. Saponkanaporn and Gehr (1989) demonstrated that the degradation of cationic ester-type polyacrylamides was pH dependent. These polymers degraded very slowly at pH 3 (very little polymer had degraded after 30 days), however, rapid hydrolysis (within hours) occurred under alkaline conditions, (pH >9). Ester hydrolysis will cleave the cationically charged functional groups from the polymer backbone to form low molecular weight alcohols. Meanwhile, the backbone of the polymer would essentially become insoluble with the loss of the cationic moiety and thereby rendered non-bioavailable (Murgatroyd *et al*. 1996). Under alkaline conditions, hydrolysis of ester-type polymers will be an important mechanism by which the toxicity of these polymers in the receiving environment can be significantly reduced. However, Murgatroyd (1996) cautioned that degradation through hydrolysis applies only to ester type polymers. It is believed that the polymers used at Ekati™ are not ester-type polymers. The persistence of polymer toxicity with adjustments to pH 3 (both polymers) and pH 11 (cationic polymer) prior to toxicity testing, indicated that pH did not alter the chemistry of either polymer in a manner which would render them non-toxic to aquatic organisms.

Since the reduction in PKE toxicity with pH adjustments is not believed to be due to polymer degradation, it is thought that modifications to the minerals found in kimberlite may have helped to alter toxicity. Understanding the behaviour of kimberlite minerals in aqueous environments is an integral part of understanding the reduction of PKE toxicity with pH adjustments followed by filtration. Major pH adjustments of PKE may have altered the chemistry of the minerals, or affected the electrostatic surface charges of suspended colloids, in such a manner that the cationic polymer was tied up

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and no longer available to elicit a toxic response.

The Ekati™ Diamond Mine kimberlites are predominantly composed of olivine, specifically the magnesium rich, foresterite (Mg_2SiO_4) (Howe 1997). The kimberlite found in Panda, the pit in mining operations for the duration of this project, consisted of approximately 60% olivine, with some samples comprised of greater than 80% olivine (Howe 1997). Magnesium-rich silicate minerals, such as olivine, are extremely sensitive to the processes of chemical weathering (Loughnan 1969). In acidic aqueous environments, the kimberlites will undergo rapid hydrolysis to silica and metal ions (e.g. Mg^{2+}, Ca^{2+}) (Luce et al. 1972; Hodgson and Dudeney 1988), while in highly alkaline environments, the rate of metal ion leaching from kimberlite decreases rapidly (Howe 1997). While these two reactions seem to be opposite in nature, both will effectively remove magnesium ions from solution. It is postulated that excess magnesium ions in PKE competes with the cationic polymer for the negatively charged sites on the surface of colloidal particles.

Magnesium salt ($MgSO_4$) has been used as an inorganic coagulant in conjunction with an anionic flocculant aid to settle colloidal suspensions of kimberlite fines (O'Gorman and Kitchener 1974). In solutions below pH 10.6, magnesium exists as the divalent cation (Mg^{2+}) (Cole 1994). This cation will form a positive cloud around anionic colloids, which will reduce the electrostatic charges that cause these particles to repel one another. Colloids, in the absence of flocculant aids, will naturally form weak aggregates if the particles can get close enough (<0.01 μm) to one another so that Van der Waals forces can predominate. In order for Van der Waals forces to predominate, the zeta potential, a measure of electrostatic charge, of colloids should be between +10

and -10 mV (Moss and Dymond 1987). Howe (1997) demonstrated that magnesium was effective at reducing the zeta potential of kimberlite suspensions to values between +10 and -15 mV.

It is postulated that the reduction in PKE toxicity at pH 11 was due to the removal of magnesium. Above pH 10.6, magnesium will form magnesium hydroxide and carbonate precipitates (Cole 1994). In addition to the removal of magnesium via precipitation, Howe (1997) demonstrated that at pH 11 the Ekati™ kimberlites leached substantially less metal ions (e.g. Mg^{2+}) than at more neutral pH (pH 7). At pH 11 the concentration of magnesium from crushed kimberlite solutions was 80 times lower than at pH 7. The loss of magnesium in solution may be an important part of the explanation of the reduction in PKE toxicity at pH 11. As explained earlier, only very low concentration of the cationic polymer have to remain free in PKE to cause toxicity to aquatic organisms. The precipitation of magnesium at high pH, in conjunction with decreased leaching of metals from kimberlite, will result in reduced competition for the surface charges on colloids. This should increase the potential for the cationic polymer to bind to the surface of the colloids and create particle aggregates that would subsequently be removed from PKE by filtration.

The reduction in toxicity of PKE at pH 3 is thought to be a result of increased negative surface charges caused by the hydrolysis of kimberlite minerals and the subsequent formation of phyllosilicate clays. The dissolution of olivine with acid hydrolysis is initially very rapid; Mg^{2+} will exchange rapidly with H^+, two hydrogen ions for one magnesium ion. Magnesium will dissociate from the mineral surface first and, at the same time, H^+ will become sorbed to the surface. Extraction of magnesium ions

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and silicon occurs by solid-state diffusion and hydrogen ion exchange, with magnesium being removed at a faster rate than silicon. As dissolution of kimberlite occurs, cavities will form on the surface of mineral particles at points where acid hydrolysis of the individual minerals proceeds at different rates. These cavities were believed to be negatively charged as H^+ tended to accumulate at these sites (Luce et al. 1972; Hodgson and Dudeney 1988). It is thought that the rapid alteration in the surface charges of minerals may attract the cationic polymer, which would subsequently bind to the surface of the colloidal particles. Once bound to the surface, it is likely that the cationic polymer and colloids will form aggregates that will be effectively removed through filtration.

Furthermore, magnesium does not appear to accumulate in solution at pH 3. In the presence of dilute hydrochloric acid, olivine type kimberlites will precipitate into magnesium-rich clay-like phases called phyllosilicates. The formation of phyllosilicates will rapidly remove magnesium from solution (Hodgson and Dudeney 1988). Some of the phyllosilicates formed during the dissolution of kimberlite include vermiculite and serpentine. This rapid precipitation of magnesium to clay was thought to increase the rate of kimberlite dissolution, which would eventually result in the complete breakdown of kimberlite minerals (Hodgson and Dudeney 1988).

It is postulated that the formation of clays (e.g. serpentine and vermiculite) from kimberlite minerals during acid hydrolysis (Hodgson and Dudeney 1988) may have facilitated the removal of excess cationic polymer. Clays have a higher cation exchange capacity (CEC) than the parent mineral, olivine (Drever 1988; Manahan 1991). The increased negative surface charges on these particles enhances the chance that the

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positively charged polymer will be bound and removed with filtration at pH 3, since cationic polymers interact with the surface of colloidal particles through cation exchange mechanisms (Siyam 1997). Therefore, the greater the CEC of colloids in solution, the greater the chances that the cationic polymer will be removed. Vermiculite clays have a CEC of 120 to 200 meq/100 g, as opposed to the kimberlite minerals in PKE which have a CEC of 2.79 to 28.72 meq/100 g (Drever 1988; Howe 1997). The very high CEC of the clays (e.g. vermiculite) formed from the dissolution of kimberlite minerals lends support to the theory that the cationic polymer was removed at pH 3 by sorption onto products of kimberlite hydrolysis.

The reduction in the toxicity of PKE with the addition of bentonite or kaolinite also supports the theory that acid hydrolysis of kimberlite into clays reduced the concentration of excess cationic polymer in solution. The addition of clays to pure water solutions containing cationic polymers has been shown to reduce the toxicity of these polymers to daphnids and fathead minnows (Biesinger and Stokes 1986; Cary *et al.* 1987). It was thought that bentonite which has a CEC of 80-150 meq/100 g would be more effective at reducing the toxicity of cationic polymers in solution than kaolinite, which has a CEC of 1-10 meq/100 g (Mitchell 1986; Drever 1988). The near equal reduction in PKE toxicity observed with the addition of both clays in this study may be a result of other interactions occurring. It is possible that Mg^{2+} was interfering with the ability of the cationic polymer to interact with the surface of clays in solution by forming a cloud of cationic particles around the surface of the anionic colloids. Thus, excess cationic polymer would still be available to elicit a toxic response.

The partial removal of PKE toxicity with the addition of activated charcoal may

also be explained by this theory, since Mg^{2+} may also have interfered with the interaction of the cationic polymer with activated charcoal. There may be other possible reasons why activated charcoal did not fully remove PKE toxicity, but it is generally thought that the cationic polymer was not able to bind effectively to the activated charcoal.

3.5   Conclusion

Positive identification of the cationic polymer as the toxic component of PKE can not be achieved unless a method to measure the concentration of the polymer in PKE can be devised. The high molecular weight of polymers makes it nearly impossible to identify and determine the concentration of cationic polymers in effluents using standard methods (personal communication Carolyn Rowland, Wright State University, Dayton, Ohio). Therefore, the determination of the cationic polymer, MagnaFloc® 368, as the toxic component of PKE is based on a weight-of-evidence approach which incorporates the results of the TIE procedures performed, including Phase II tests with MagnaFloc® 368, knowledge of the components of PKE, and the behaviour of kimberlite minerals in solution.

CHAPTER 4

CONCLUSION

4.1   Summary

This research project was initiated to evaluate the potential effects of processed kimberlite effluent (PKE) from the Ekati™ Diamond Mine, Northwest Territories, on aquatic organisms within, and downstream of, the receiving environment of the designated PKE containment facility, Long Lake. Two different approaches were used to evaluate the possible impact of PKE to aquatic organisms. The first approach was to describe the changes to zooplankton populations, water chemistry and lake limnology that would occur with the aqueous deposition of PKE into the Long Lake Containment Facility (LLCF). The second approach was to use the laboratory test organism, *Ceriodahpnia dubia*, as a surrogate for native zooplankton, in order to determine the magnitude of potential effects that PKE could have on aquatic organisms. In addition, the nature of toxic components of PKE could be evaluated with this species using toxicity identification evaluation (TIE) procedures.

4.1.1   The Field Study

The disposal of PKE into the LLCF had two main effects on the receiving environment. The first, and foremost, effect was the loss of aquatic habitat as the

kimberlite fines gradually filled the basin of Cell B throughout the duration of this study. The physical addition of PKE to the receiving cell resulted in higher turbidity and total suspended solids (TSS), and significantly altered water chemistry which may have decreased the ability of certain zooplankton species to survive. The second effect was the changes in water chemistry in the cells downstream of PKE deposition; components of PKE that were soluble, or associated with fine colloidal material, leached through the semi-permeable rock dams.

Monitoring the changes in water chemistry and zooplankton in the cells directly downstream of PKE deposition provided some insight into the long-term alterations that may be expected to occur in the aquatic environments downstream of the LLCF. Prior to the addition of PKE to Cell B in August 1998, all cells of the LLCF had a slightly acidic pH, low total hardness and low alkalinity. As of August 2000, Cell E, which is near the outlet of the LLCF and the cell farthest from PKE deposition, was slightly alkaline, and the total water hardness, alkalinity, conductivity, total dissolved solids, and total nitrogen had all increased. Furthermore, the concentration of certain metals, such as molybdenum, nickel, strontium, barium, magnesium, and calcium, had significantly increased in Cell E. It is likely that the observed alterations in water chemistry of Cell E will eventually be noted downstream of the LLCF with the continued deposition of PKE. These alterations may lead to changes in zooplankton assemblages, as sensitive species, such as *Holopedium gibberum*, could disappear and more tolerant and/or competitive species begin to thrive.

Zooplankton assemblages in the cells of the LLCF have changed according to proximity to PKE deposition. Cell B had a decrease in total zooplankton abundance and

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diversity, whereas Cell C, which sporadically received PKE, had a decrease in species

sensitive to increases in TSS. Cells D and E, which were farther downstream,

experienced seasonal fluctuations in zooplankton abundances and diversity, but a

distinct effect as a result of PKE addition could not be discerned. It can, therefore, be

concluded that changes to water chemistry with the addition of PKE may play, in the

short-term, a less significant role than the physical effects of the solid tailings waste on

the aquatic fauna of the LLCF. However, it is postulated that the continued deposition

of PKE into the LLCF will lead to higher concentrations of metals as well as

significantly altered water chemistry that may adversely affect native organisms in the

cells downstream of PKE deposition, as well as aquatic environments downstream of

the LLCF.

4.1.2   The Laboratory Study

Processed kimberlite effluent contained components that were toxic to *C. dubia*.

Laboratory testing determined that PKE was chronically toxic at concentrations as low

as 12.5% effluent, and that toxicity could not be reduced with the removal of the

secondary treated sewage effluent. It was highly unlikely that the kimberlite minerals

were directly responsible for the toxicity of PKE. Earlier research conducted by the

Department of Fisheries and Ocean (DFO), MN, (Harrison *et al.* 1995; 1997) and

Enviro-Test Laboratories (1998) demonstrated that the removal of TSS significantly

reduced the toxicity of crushed kimberlite in solution to aquatic organisms. Analysis of

PKE by ICP-MS determined that metals were not present at sufficient concentrations to

be chronically toxic to *C. dubia*. A limited weight-of-evidence approach, which

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incorporated the results of the TIE manipulations and supplemental toxicity testing, determined that the cationic polymer, MagnaFloc® 368, was most likely to be the toxic component of PKE.

The persistence of PKE toxicity in the EDTA and sodium thiosulfate addition tests, and with aeration and solid phase extraction with C-18 disks, lends support to the theory that MagnaFloc® 368 was responsibly for the observed toxicity of PKE. Cationic polymers are not filterable, chelatible, or volatile, and they are extremely polar (Rowlands *et al.* 2000) which would explain the inability of the above-mentioned TIE manipulations to reduce the toxicity of PKE. In-house laboratory testing determined that MagnaFloc® 368 had a 7-d EC_{50} estimate of 0.014 mg/L to *C. dubia*. The estimated final concentration of MagnaFloc® 368 in PKE was 14 mg/L, based on the dosage rate and volume of water used within the Ekati™ Diamond Mine processing plant. It is, therefore, possible that excess polymer was present in PKE at concentrations high enough to be chronically toxic to *C. dubia*.

A more thorough weight-of-evidence approach would have tested PKE without the addition of MagnaFloc® 368. Unfortunately, the Ekati™ Diamond Mine processing plant was unable to suspend the addition of the cationic polymer, and therefore an effluent sample without MagnaFloc® 368 was not available for toxicity testing. Finally, confirmation that the cationic polymer was present at toxic concentrations cannot be achieved until analytical methods are available to measure the final concentration of MagnaFloc® 368 in PKE.

4.2   Integration of the Field and Laboratory Assessments

The effect of PKE on freshwater zooplankton differed with the two assessment approaches. In the field, zooplankton appeared to be most affected by the physical effects of PKE; the increase in TSS and the loss of aquatic habitat as the basin of LLCF filled with tailings. The laboratory study gave a more insightful picture as to the toxic nature of PKE. The persistence of toxicity with the removal of the TSS revealed that PKE contained soluble components (<0.45 μm) that were toxic to *C. dubia*. The most likely cause of toxicity was the cationic polymer, MagnaFloc® 368. How, then, did the results from the laboratory assessment relate to what has happened, or could happen in the future, to native zooplankton in the LLCF?

First, it is proposed that the toxic effects of MagnaFloc® 368 to zooplankton will be masked by the physical effect of the fine tailings. Second, if excess MagnaFloc® 368 from PKE is present in the LLCF, it should eventually bind to anionic colloidal particles as well as to algae and zooplankton. This could be detrimental to organisms living within the LLCF and may, eventually, be harmful to aquatic organisms downstream of the LLCF. Cationic polymers have a low octanol-water partition coefficient and a high water solubility (Murgatroyd *et al.* 1996), and, therefore, it is possible that the cationic polymer may leach through the semi-permeable rock dams. Initially, anionic colloidal particles and biofilms within the dams may bind the free polymer. However, eventually some of the polymer may be able to leach through and affect organisms in the cell(s) downstream of PKE deposition. At the present time water must flow through Cells D and E before exiting the LLCF. However, in the future, when Cell D is used for the disposal of PKE, the only buffer zone will be Cell E.

The LLCF was designated as a tailings collection facility for the Ekati™

Diamond Mine and the lake itself will be destroyed over the life of the mine with the deposition of PKE. An additional purpose of the LLCF was to mitigate the effects of PKE to downstream aquatic ecosystems. It was postulated that the construction of the semi-permeable rock dams on the LLCF would minimize the effects of components in PKE on the downstream aquatic environment. However, results from the field study indicated that components of PKE were soluble, or associated with fine colloids, that leached through the dykes. Furthermore, the TIE determined that MagnaFloc® 368 was most likely to be the toxic component in PKE. It is possible that in the future, due to the solubility of components of PKE, that the LLCF may not adequately protect organisms in the downstream aquatic environment.

The Ekati™ Diamond Mine processing plant could be asked to suspend the use of MagnaFloc® 368 in its water treatment process, given that the cationic polymer may be the toxic component of PKE. Suspending the use of MagnaFloc® 368 should reduce the toxicity of PKE, but could result in potentially greater environmental issues. Suspension of these polymers may result in significantly greater alterations to the water chemistry downstream of the LLCF, as well as a significant increase in water use and waste water produced. The synthetic polymers, Magna Floc® 156 and 368, are added to PKE within the processing plant in order to recycle water used during the diamond extraction process, and also to facilitate particle settling within the LLCF through the process of particle flocculation. The use of a cationic polymer allows the processing plant to recycle 80% of the water used during the diamond extraction processes (Ekati™ Diamond Mine 2000). Furthermore, Howe (1997) postulated that the use of these polymers would significantly reduce the concentration of metals leaching from crushed

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kimberlite in the LLCF. Already significant increases in metals have been noted in the final cell of the LLCF.

Despite the toxicity of MagnaFloc® 368, it is not suggested that Ekati™ Diamond Mine suspend the use of the cationic polymer. Rather, efforts should be made to reduce the concentration of the cationic polymer used. However, this may be difficult since the concentration of synthetic polymer needed to facilitate particle agglomeration is dependent upon a number factors including: solution pH, particle size, type of suspended solids, mixing techniques, and temperature. Therefore, it may not be possible to meet the requirements for particle flocculation by decreasing the use of the cationic polymer. An alternative would be to try using a coagulant that is less toxic to aquatic organisms.

The primary goal of the LLCF was to contain the fine kimberlite tailings waste and minimize the impact of PKE to the downstream aquatic environment. To the best of my judgement, the Ekati™ Diamond Mine has a well designed containment facility, and is functioning well, for the present time, with the use of the cationic polymer, MagnaFloc® 368.

4.3   Future Research Consideration

Evaluation of the effect of PKE from the Ekati™ Diamond Mine on zooplankton in the field and laboratory is only one component of a potentially much larger research project. For this project, the field study looked at the effects of PKE on zooplankton within the LLCF, and the laboratory study focused primarily on identifying the toxic component(s) of PKE. However, there is potential for other research initiatives to be

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explored.

This study was not able to determine the magnitude of effects that alterations to water chemistry would have on potentially sensitive zooplankton species, or whether more tolerant or competitive species would begin to thrive within the LLCF. A extension of this research project would be to continue to monitor zooplankton populations and water chemistry within the LLCF to determine if there are species present in the LLCF that are sensitive to the water chemistry changes that are occurring with the addition of PKE. It is proposed that more frequent monitoring of zooplankton populations within the LLCF may tease out seasonal effects and give a better approximation of the magnitude of effects that PKE has on zooplankton.

Another initiative could evaluate the effects of PKE on benthic organisms within the LLCF. Field assessments could be conducted to determine if benthic organisms in the downstream cells were affected by the deposition of PKE in the receiving cells. Other studies could be conducted to evaluate the suitability of the sediment formed by the deposition of fine tailings for colonization by benthic organisms.

With regards to toxicity testing, this project was not able to confirm the presence of the cationic polymer as the toxic fraction of PKE. It would be interesting and relevant to develop TIE manipulations that could be used to positively identify the cationic polymer as the toxicant. As well, it would be necessary to develop analytical techniques that can detect or measure the concentration of synthetic polymers in effluent and environmental samples.

CHAPTER 5

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Table A.1. Number and species of fish removed from different locations within the Long Lake Containment Facility (LLCF) from 1997 to 1999.

Fish Species	July-September 1997[a] LLCF[d]	July-August 1998[b] Cell C	August-September1999[c] Cell D
Arctic grayling (*Thymallus arcticus*)	595	1293	532
Burbot (*Lota lota*)	72	173	89
Longnose sucker (*Catostomus catostmus*)	1	0	0
Lake chub (*Couesius plumbius*)	1	0	3
Lake cisco (*Coregonus artedi*)	0	880	293
Lake trout (*Salvelinus namaycush*)	3710	2562	1590
Round whitefish (*Prosopium cylindraceum*)	2319	2655	1156
Slimy sculpin (*Cottus cognatus*)	3	1	0
Total	**6701**	**7564**	**3633**

[a]From Rescan (1998).
[b]From Rescan (1999).
[c]From Rescan (2000).
[d]Fish out program occurred on the entire Long Lake Containment Facility (LLCF) before the dykes were built.

APPENDIX B

Table B.1. Sampling date, depth, detection limit, and mean (± SD) abundance per m^3 of zooplankton in Cell B of the LLCF[1].

Sampling date	August 1998	July 1999	August 1999	July 2000[3]	August 2000[4]
Sampling depth[2]	3m	1m	1m		
Detection limit (abundance/m^3)	472	472	472		
Zooplankton species (mean/m^3)					
Kellicotia longispina	76887 ± 18708	7856 ± 5213	3614 ± 2227		
Conochilus spp.	8176 ± 3069	314 ± 136	<472		
Keratella cochlearis	<472	314 ± 136	<472		
Keratella quadratus	<472	<472	<472		
Asplanchna	<472	<472	<472		
Polyarthra vulgaris	<472	<472	<472		
Total Rotifera	**86006 ± 21725**	**8955 ± 5436**	**4792 ± 2227**		
Holopedium gibberum	12421 ± 5088	707 ± 408	<472		
Daphnia middendorffiana	314 ± 136	<472	314 ± 136		
Bosmina longirostris	943 ± 472	<472	<472		
Total Caladocera	**13679 ± 4591**	**1178 ± 408**	**786 ± 136**		
Diaptomus pribilofensis	1887 ± 1415	786 ± 952	<472		
Diaptomus sicilis	2516 ± 1187	<472	<472		
Heterocope septentrionalis	314 ± 136	<472	<472		
Calanoida copepodid	314 ± 136	1964 ± 1769	<472		
Cyclops scutifer	11006 ± 1362	471 ± 408	<472		
Diacyclops biscupidatus thomasi	1179 ± 1435	<472	<472		
Cyclopoida copepodid	92138 ± 6010	393 ± 136	1021 ± 828		
Copepoda nauplii	10535 ± 1906	1178 ± 1080	4556 ± 2227		
Total Copepoda	**119890 ± 6250**	**5499 ± 3530**	**6992 ± 1497**		
TOTAL	**219575 ± 27983**	**15868 ± 9195**	**12569 ± 3839**		

[1]LLCF = Long Lake Containment Facility.
[2]Sampling depth was 2 m above the bottom of the lake.
[3,4] Cell B could not be sampled in July and August 2000.

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Table B.2. Sampling date, depth, detection limit, and mean (± SD) abundance per m^3 of zooplankton in Cell C of the LLCF[1].

Sampling date	August 1998[3]	July 1999	August 1999	July 2000	August 2000
Sampling depth[2]		12m	9m	9m	7m
Detection limit (abundance/m^3)		39	157	52	101
Zooplankton species (mean/m^3)					
Kellicotia longispina		1466 ± 159	68774 ± 2123	1113 ± 211	67267 ± 28457
Conochilus spp.		39 ± 34	183 ± 120	426 ± 404	67 ± 29
Keratella cochlearis		<39	734 ± 240	1060 ± 347	11964 ± 8192
Keratella quadratus		<39	<157	148 ± 189	253 ± 182
Asplanchna		26 ± 11	<157	226 ± 109	253 ± 182
Polyarthra vulgaris		<39	<157	<52	135 ± 146
Total Rotifera		**1571 ± 187**	**69927 ± 2282**	**2997 ± 1007**	**79938 ± 37039**
Holopedium gibberum		<39	<157	<52	<101
Daphnia middendorffiana		<39	210 ± 91	<52	<101
Bosmina longirostris		<39	<157	<52	<101
Total Caladocera		**59 ± 0**	**367 ± 91**	**78 ± 0**	**152 ± 0**
Diaptomus pribilofensis		654 ± 23	393 ± 283	748 ± 80	1415 ± 365
Diaptomus sicilis		445 ± 295	524 ± 396	1339 ± 241	2966 ± 1114
Heterocope septentrionalis		340 ± 45	79 ± 0	348 ± 80	67 ± 29
Calanoida copepodid		4673 ± 884	236 ± 136	365 ± 104	438 ± 254
Cyclops scutifer		576 ± 60	524 ± 240	156 ± 52	1281 ± 478
Diacyclops biscupidatus thomasi		288 ± 113	105 ± 45	504 ± 30	<101
Cyclopoida copepodid		137 ± 109	4927 ± 595	43 ± 15	2191 ± 772
Copepoda nauplii		838 ± 327	7129 ± 2259	10135 ± 951	26354 ± 8528
Total Copepoda		**7952 ± 1277**	**13917 ± 1236**	**13638 ± 703**	**34762 ± 9583**
TOTAL		**9601 ± 1396**	**84211 ± 3250**	**16715 ± 591**	**114650 ± 45502**

[1]LLCF = Long Lake Containment Facility.
[2]Sampling depth was 2 m above the bottom of the lake.
[3] Cell C was not sampled in August 1998.

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Table B.3. Sampling date, depth, detection limit, and mean (± SD) abundance per m^3 of zooplankton in Cell D S1 of the LLCF[1].

Sampling date	August 1998	July 1999	August 1999	July 2000	August 2000
Sampling depth[2]	11m	11m	8m	10m	10m
Detection limit (abundance/m^3)	129	64	59	47	71
Zooplankton species (mean/m^3)					
Kellicotia longispina	6090 ± 1949	1414 ± 633	6049 ± 2062	4493 ± 190	36046 ± 7791
Conochilus spp.	1201 ± 324	578 ± 170	1944 ± 312	1320 ± 163	2548 ± 534
Keratella cochlearis	107 ± 37	43 ± 19	196 ± 90	3017 ± 974	13918 ± 4554
Keratella quadratus	<129	<64	<59	377 ± 141	59 ± 20
Asplanchna	<129	54 ± 19	530 ± 412	456 ± 152	<71
Polyarthra vulgaris	<129	<64	<59	<47	<71
Total Rotifera	**7591 ± 2230**	**2121 ± 772**	**8779 ± 1487**	**9968 ± 1257**	**52642 ± 11828**
Holopedium gibberum	129 ± 111	578 ± 391	216 ± 34	39 ± 14	142 ± 71
Daphnia middendorffiana	<129	<64	59 ± 0	173 ± 178	1203 ± 283
Bosmina longirostris	1072 ± 197	54 ± 19	39 ± 17	251 ± 178	1486 ± 142
Total Caladocera	**1265 ± 207**	**664 ± 402**	**314 ± 17**	**463 ± 330**	**2831 ± 430**
Diaptomus pribilofensis	472 ± 197	<64	1100 ± 531	189 ± 170	2477 ± 308
Diaptomus sicilis	2916 ± 2259	75 ± 49	3103 ± 494	566 ± 776	7006 ± 675
Heterocope septentrionalis	86 ± 37	<64	39 ± 17	<47	47 ± 20
Calanoida copepodid	944 ± 414	364 ± 323	324 ± 436	896 ± 170	613 ± 610
Cyclops scutifer	11666 ± 4495	2057 ± 232	1866 ± 741	1068 ± 272	1038 ± 390
Diacyclops biscupidatus thomasi	515 ± 386	471 ± 148	412 ± 118	6143 ± 1155	661 ± 349
Cyclopoida copepodid	7763 ± 6352	18016 ± 207	1768 ± 1183	8845 ± 152	6770 ± 1137
Copepoda nauplii	24919 ± 6285	771 ± 129	2141 ± 778	990 ± 236	29464 ± 6056
Total Copepoda	**49279 ± 7558**	**21819 ± 579**	**10752 ± 1013**	**18720 ± 2192**	**48077 ± 7490**
TOTAL	**58136 ± 9220**	**24636 ± 1466**	**19845 ± 1030**	**28846 ± 3012**	**103550 ± 18686**

[1]LLCF = Long Lake Containment Facility.

[2]Sampling depth was 2 m above the bottom of the lake.

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Table B.4. Sampling date, depth, detection limit, and mean (± SD) abundance per m^3 of zooplankton in Cell D S2 of the LLCF[1].

Sampling date	August 1998	July 1999	August 1999	July 2000	August 2000
Sampling depth[2]	11m	8m	5.5m	5.5m	8m
Detection limit (abundance/m^3)	64	88	86	86	88
Zooplankton species (mean/m^3)					
Kellicotia longispina	8985 ± 1034	9996 ± 3096	7398 ± 2899	32049 ± 6540	31287 ± 8952
Conochilus spp.	1480 ± 561	4807 ± 676	3770 ± 600	2457 ± 431	4364 ± 1208
Keratella cochlearis	64 ± 0	<88	243 ± 310	1343 ± 262	12267 ± 2225
Keratella quadratus	<64	<88	<86	157 ± 108	59 ± 26
Asplanchna	54 ± 19	74 ± 26	1085 ± 216	328 ± 251	<88
Polyarthra vulgaris	<64	<88	<86	<86	<88
Total Rotifera	**10647 ± 531**	**14965 ± 2411**	**12582 ± 2215**	**36376 ± 6237**	**48066 ± 11812**
Holopedium gibberum	643 ± 129	4689 ± 468	114 ± 49	100 ± 65	88 ± 77
Daphnia middendorffiana	54 ± 19	206 ± 51	71 ± 25	200 ± 49	4748 ± 311
Bosmina longirostris	129 ± 0	133 ± 77	<86	314 ± 49	13800 ± 3975
Total Caladocera	**826 ± 130**	**5028 ± 460**	**229 ± 65**	**614 ± 150**	**18636 ± 3930**
Diaptomus pribilofensis	965 ± 232	442 ± 319	1685 ± 216	1400 ± 423	5101 ± 5788
Diaptomus sicilis	3324 ± 651	324 ± 184	3285 ± 1255	200 ± 99	5249 ± 1127
Heterocope septentrionalis	<64	619 ± 177	57 ± 25	114 ± 49	118 ± 51
Calanoida copepodid	2788 ± 644	3008 ± 234	71 ± 25	4999 ± 1085	74 ± 26
Cyclops scutifer	2337 ± 465	1180 ± 135	3285 ± 1514	71 ± 25	1740 ± 135
Diacyclops biscupidatus thomasi	365 ± 186	737 ± 204	286 ± 49	1114 ± 309	1180 ± 223
Cyclopoida copepodid	6155 ± 799	1887 ± 859	2257 ± 643	371 ± 131	5013 ± 652
Copepoda nauplii	4139 ± 1180	944 ± 487	3485 ± 1085	4770 ± 487	10616 ± 1614
Total Copepoda	**20136 ± 1532**	**9141 ± 1513**	**14411 ± 3612**	**13040 ± 1757**	**29090 ± 9328**
TOTAL	**31609 ± 1594**	**29178 ± 2210**	**27208 ± 5877**	**49987 ± 6515**	**95792 ± 23710**

[1]LLCF = Long Lake Containment Facility.
[2]Sampling depth was 2 m above the bottom of the lake.

Table B.5. Sampling date, depth, detection limit, and mean (± SD) abundance per m^3 of zooplankton in Cell E of the LLCF[1].

Sampling date	August 1998	July 1999	August 1999	July 2000	August 2000
Sampling depth[2]	7m	7m	6m	2m	2m
Detection limit (abundance/m³)	101	101	78	236	236
Zooplankton species (mean/m³)					
Kellicotia longispina	3404 ± 1606	4920 ± 1081	5707 ± 1103	27573 ± 4731	58839 ± 13465
Conochilus spp.	12774 ± 3422	7986 ± 708	11650 ± 181	53732 ± 13926	1728 ± 272
Keratella cochlearis	101 ± 0	118 ± 77	157 ± 79	236 ± 0	5106 ± 136
Keratella quadratus	<101	<101	<78	157 ± 68	157 ± 68
Asplanchna	152 ± 134	67 ± 29	1100 ± 157	432 ± 297	<236
Polyarthra vulgaris	67 ± 29	<101	52 ± 23	<236	<236
Total Rotifera	**16549 ± 4805**	**13142 ± 695**	**18705 ± 1267**	**82248 ± 18852**	**66066 ± 13301**
Holopedium gibberum	3000 ± 1256	4549 ± 441	288 ± 45	1257 ± 680	157 ± 68
Daphnia middendorffiana	67 ± 29	1483 ± 309	1518 ± 120	2514 ± 1114	825 ± 612
Bosmina longirostris	1719 ± 350	135 ± 146	419 ± 45	1178 ± 624	1571 ± 272
Total Caladocera	**4786 ± 1131**	**6167 ± 401**	**2225 ± 120**	**4949 ± 2014**	**2553 ± 802**
Diaptomus pribilofensis	404 ± 101	67 ± 29	812 ± 163	5106 ± 2731	1964 ± 593
Diaptomus sicilis	1416 ± 101	1213 ± 267	1309 ± 297	943 ± 236	471 ± 236
Heterocope septentrionalis	<101	135 ± 58	<78	<236	174 ± 159
Calanoida copepodid	270 ± 58	2494 ± 383	52 ± 23	4949 ± 1080	157 ± 68
Cyclops scutifer	6808 ± 688	1954 ± 58	1675 ± 371	1414 ± 236	1571 ± 981
Diacyclops biscupidatus thomasi	607 ± 175	741 ± 58	340 ± 181	1178 ± 0	864 ± 544
Cyclopoida copepodid	3876 ± 409	4448 ± 802	1466 ± 395	864 ± 136	1414 ± 408
Copepoda nauplii	4550 ± 202	2190 ± 765	2408 ± 730	8877 ± 952	24117 ± 3311
Total Copepoda	**17981 ± 1013**	**13243 ± 1044**	**8102 ± 99**	**23449 ± 2624**	**30676 ± 4972**
TOTAL	**39316 ± 4913**	**32602 ± 899**	**29033 ± 1226**	**110529 ± 14856**	**99295 ± 18189**

[1]LLCF = Long Lake Containment Facility.
[2]Sampling depth was 2 m above the bottom of the lake.

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APPENDIX C

Table C.1. Measurements of water quality within Cell B of the LLCF[a]. Water collected 1 m below the surface of the lake.

Measurement	Unit	DL[b]	August 1998	August 1999	July 2000[f]	August 2000[f]
			(mean ± SD of 3 replicate samples)			
pH	pH	0.05	6.74 ±0.12	8.06 ±0.02		
Conductivity	μS/cm	0.3	n/a[c]	387.3 ±2.5		
Alkalinity	mg/L	0.3	6.2 ±0.3	62.1 ±0.2		
Calcium	mg/L	0.05	1.34 ±0.03	22.40 ±0.20		
Magnesium	mg/L	0.01	0.843 ±0.006	33.13 ±2.92		
Tot-Hardness	mg/L	0.17	6.81 ±0.07	192.33 ±11.93		
Turbidity	NTU	0.1	1.5 ±0.1	7.8 ±0.3		
Tot-Suspended Solids	mg/L	3	3 ±3	14 ±3		
Tot-Dissolved Solids	mg/L	10	n/a	287.3 ±3.1		
Tot-Nitrogen	mg/L	0.008	0.114 ±0.007	5.033 ±0.159		
Ammonia	mg/L	0.005	0.018 ±0.004	0.141 ±0.002		
Tot-Phosphorous	mg/L	0.004	0.018 ±0.007	0.026 ±0.002		
Ortho-Phosphorous	mg/L	0.002	BDL[d]	0.011 ±0.002		
Tot-Org. Carbon	mg/L	0.2	n/a	4.0 ±0.1		
Diss-Org. Carbon	mg/L	0.5	n/a	n/a		
Tot-Aluminum	μg/L	30.0	34.1 ±18.9	157.0 ±19.1		
Tot-Antimony	μg/L	0.5	BDL	8.9 ±0.4		
Tot-Arsenic	μg/L	2.0	BDL	2.9 ±0.2		
Tot-Barium	μg/L	1.0	6.4 ±0.8	230.7 ±1.2		
Tot-Beryllium	μg/L	2.0	BDL	1		
Tot-Bismuth	μg/L	10.0	BDL	BDL		
Tot-Cadmium	μg/L	0.3	BDL	BDL		
Tot-Cesium	μg/L	0.4	BDL	BDL		
Tot-Chromium	μg/L	3.0	BDL	BDL		
Tot-Cobalt	μg/L	1.0	BDL	BDL		
Tot-Copper	μg/L	2.0	2.3 ±0.1	1.4 ±0.6		
Tot-Iron	mg/L	0.03	0.15 ±0.03	0.29 ±0.05		
Tot-Lead	μg/L	1.0	0.5 ±0.7	BDL		
Tot-Lithium	μg/L	3.0	1.0 ±0.4	3.7 ±0.4		
Tot-Manganese	μg/L	1.0	9.4 ±0.2	32.8 ±0.3		
Tot-Molybdenum	μg/L	1.0	BDL	156.0 ±2.0		
Tot-Nickel	μg/L	1.0	0.4 ±0.2	41.0 ±0.3		
Tot-Selenium	μg/L	10.0	BDL	BDL		
Tot-Silver	μg/L	0.3	BDL	BDL		
Tot-Strontium	μg/L	1.0	9.3 ±0.2	285.0 ±6.1		
Tot-Thallium	μg/L	0.4	BDL	BDL		
Tot-Titanium	μg/L	3.0	0.9 ±0.8	9.0 ±1.0		
Tot-Uranium	μg/L	0.3	BDL	0.3 ±0.1		
Tot-Vanadium	μg/L	1.0	BDL	1.9 ±0.1		
Tot-Zinc	μg/L	10	8.4 ±5.9	BDL		

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.
[f] Samples were not collected in Cell B in July and August, 2000.

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Table C.2. Measurements of water quality within Cell B of the LLCF[a]. Water collected 1 m above the bottom of the lake.

Measurement	Unit	DL[b]	(mean ± SD of 3 replicate samples)			
			August 1998[f]	August 1999[f]	July 2000[g]	August 2000[g]
pH	pH	0.05	6.66 ± 0.06	8.05 ± 0.01		
Conductivity	μS/cm	0.3	n/a[c]	385.7 ± 1.2		
Alkalinity	mg/L	0.3	6.0 ± 0.1	62.1 ± 0.1		
Calcium	mg/L	0.05	1.33 ± 0.02	22.43 ± 0.06		
Magnesium	mg/L	0.01	0.837 ± 0.006	31.00 ± 1.49		
Tot-Hardness	mg/L	0.17	6.77 ± 0.06	183.67 ± 6.43		
Turbidity	NTU	0.1	1.5 ± 0.2	7.9 ± 0.2		
Tot-Suspended Solids	mg/L	3	6.66 ± 1	13 ± 4		
Tot-Dissolved Solids	mg/L	10	n/a	284.7 ± 9		
Tot-Nitrogen	mg/L	0.008	0.113 ± 0.006	5.117 ± 0.015		
Ammonia	mg/L	0.005	0.018 ± 0.002	0.142 ± 0.003		
Tot-Phosphorous	mg/L	0.004	0.012 ± 0.001	0.027 ± 0.002		
Ortho-Phosphorous	mg/L	0.002	BDL[d]	0.014 ± 0.002		
Tot-Org. Carbon	mg/L	0.2	n/a	4.0 ± 0.0		
Diss-Org. Carbon	mg/L	0.5	n/a	n/a		
Tot-Aluminum	μg/L	30.0	64.2 ± 8.8	163.7 ± 48.0		
Tot-Antimony	μg/L	0.5	0.1 ± 0.1	9.1 ± 0.2		
Tot-Arsenic	μg/L	2.0	0.1 ± 0.1	2.3 ± 1.2		
Tot-Barium	μg/L	1.0	6.1 ± 0.2	232.3 ± 4.0		
Tot-Beryllium	μg/L	2.0	0.1	1		
Tot-Bismuth	μg/L	10.0	BDL	BDL		
Tot-Cadmium	μg/L	0.3	BDL	BDL		
Tot-Cesium	μg/L	0.4	BDL	BDL		
Tot-Chromium	μg/L	3.0	BDL	BDL		
Tot-Cobalt	μg/L	1.0	BDL	BDL		
Tot-Copper	μg/L	2.0	2.1 ± 0.1	2.1 ± 0.1		
Tot-Iron	mg/L	0.03	0.16 ± 0.05	0.26 ± 0.01		
Tot-Lead	μg/L	1.0	BDL	BDL		
Tot-Lithium	μg/L	3.0	1.0 ± 0.2	3.4 ± 0.4		
Tot-Manganese	μg/L	1.0	9.7 ± 0.5	33.0 ± 0.3		
Tot-Molybdenum	μg/L	1.0	BDL	157.3 ± 3.5		
Tot-Nickel	μg/L	1.0	BDL	41.4 ± 1.9		
Tot-Selenium	μg/L	10.0	BDL	BDL		
Tot-Silver	μg/L	0.3	BDL	BDL		
Tot-Strontium	μg/L	1.0	10 ± 0.6	286.7 ± 5.7		
Tot-Thallium	μg/L	0.4	BDL	BDL		
Tot-Titanium	μg/L	3.0	1.2 ± 0.5	BDL		
Tot-Uranium	μg/L	0.3	BDL	0.3 ± 0.0		
Tot-Vanadium	μg/L	1.0	BDL	2.0 ± 0.1		
Tot- Zinc	μg/L	10	BDL	8.2 ± 5.6		

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.
[f] Sampling depth from the lake surface was 4 m in August 1998, and 2 m in August 1999.
[g] Samples were not collected in Cell B in July and August, 2000.

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Table C.3. Measurements of water quality within Cell C of the LLCF[a]. Water collected 1 m below the surface of the lake.

Measurement	Unit	DL[b]	(mean ± SD of 3 replicate samples)			
			August 1998	August 1999	July 2000	August 2000
pH	pH	0.05	6.71 ±0.00	7.46 ±0.01	7.76 ±0.01	7.74 ±0.01
Conductivity	μS/cm	0.3	n/a[c]	127.0 ±1.0	222.0 ±0.0	270.3 ±0.6
Alkalinity	mg/L	0.3	6.1 ±0.1	19.9 ±0.0	29.0 ±0.6	30.9 ±0.0
Calcium	mg/L	0.05	1.48 ±0.07	7.86 ±0.07	12.63 ±0.06	17 ±0.30
Magnesium	mg/L	0.01	0.667 ±0.006	7.34 ±0.06	9.49 ±7.04	13.17 ±0.15
Tot-Hardness	mg/L	0.17	6.44 ±0.16	49.87 ±0.25	70.53 ±28.83	96.63 ±1.30
Turbidity	NTU	0.1	8.9 ±0.7	1.0 ±0.1	1.1 ±0.1	1.7 ±0.1
Tot-Suspended Solids	mg/L	3	9 ±2	2 ±1	7 ±1	5 ±3
Tot-Dissolved Solids	mg/L	10	n/a	97.3 ±4.9	195.0 ±4.6	193.3 ±1.2
Tot-Nitrogen	mg/L	0.008	0.305 ±0.002	3.287 ±0.055	3.717 ±0.112	4.763 ±0.086
Ammonia	mg/L	0.005	0.036 ±0.005	0.099 ±0.003	0.054 ±0.009	0.077 ±0
Tot-Phosphorous	mg/L	0.004	0.015 ±0.001	0.006 ±0.000	0.010 ±0.007	0.009 ±0
Ortho-Phosphorous	mg/L	0.002	BDL[d]	0.007 ±0.005	0.001	0.001
Tot-Org. Carbon	mg/L	0.2	n/a	3.6 ±0.0	3.4 ±0.2	3.4 ±0.1
Diss-Org. Carbon	mg/L	0.5	n/a	n/a	3.5 ±0.3	3.1 ±0.1
Tot-Aluminum	μg/L	30.0	290.0 ±39.9	21.3 ±11.0	15	15
Tot-Antimony	μg/L	0.5	0.1 ±0	2.6 ±0.1	4.3 ±0.4	4.0 ±0.3
Tot-Arsenic	μg/L	2.0	0.1 ±0.1	0.6 ±0.2	3.0 ±0.0	1.7 ±0.6
Tot-Barium	μg/L	1.0	16.2 ±0.1	88.9 ±1.5	154.0 ±1.7	154.7 ±2.5
Tot-Beryllium	μg/L	2.0	0.1	BDL	BDL	BDL
Tot-Bismuth	μg/L	10.0	BDL	BDL	BDL	BDL
Tot-Cadmium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Cesium	μg/L	0.4	BDL	BDL	BDL	BDL
Tot-Chromium	μg/L	3.0	BDL	BDL	BDL	BDL
Tot-Cobalt	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Copper	μg/L	2.0	2.2 ±0.2	2.4 ±0.4	2.0 ±1.7	1
Tot-Iron	mg/L	0.03	0.43 ±0.02	BDL	0.08 ±0.11	0.24 ±0.18
Tot-Lead	μg/L	1.0	0.2 ±0.1	0.5	0.5	0.5
Tot-Lithium	μg/L	3.0	1.5 ±0.4	1.5	BDL	BDL
Tot-Manganese	μg/L	1.0	12.3 ±0.2	6.4 ±0.2	13.3 ±0.6	11.7 ±0.6
Tot-Molybdenum	μg/L	1.0	0.1	26.8 ±0.5	62.7 ±2.5	74.7 ±1.2
Tot-Nickel	μg/L	1.0	BDL	5.7 ±0.2	8.3 ±0.6	10.7 ±0.6
Tot-Selenium	μg/L	10.0	5	5	5.0 ±0.0	7.0 ±3.5
Tot-Silver	μg/L	0.3	BDL	BDL	3.1 ±4.7	0.2
Tot-Strontium	μg/L	1.0	11.7 ±0.3	85.0 ±2.6	171.0 ±3.5	219.0 ±3
Tot-Thallium	μg/L	0.4	0.1	0.2	0.2	0.2
Tot-Titanium	μg/L	3.0	21.9 ±2.7	BDL	BDL	BDL
Tot-Uranium	μg/L	0.3	0.2	0.2	0.2	0.2
Tot-Vanadium	μg/L	1.0	0.9 ±0.3	0.5	0.5	0.5
Tot-Zinc	μg/L	10	BDL	BDL	8.3 ±2.9	6.7 ±2.9

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.

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Table C.4. Measurements of water quality within Cell C of the LLCF[a]. Water collected 1 m above the bottom of the lake.

Measurement	Unit	DL[b]	(mean ± SD of 3 replicate samples)			
			August 1998[f]	August 1999[f]	July 2000[f]	August 2000[f]
pH	pH	0.05	6.69 ± 0.02	7.49 ± 0.02	7.53 ± 0.03	7.75 ± 0.01
Conductivity	μS/cm	0.3	n/a[c]	127 ± 1.0	215.7 ± 0.6	270.3 ± 1.2
Alkalinity	mg/L	0.3	6.1 ± 0.1	20.2 ± 0.4	27.5 ± 0.1	30.9 ± 0.1
Calcium	mg/L	0.05	1.43 ± 0.09	7.99 ± 0.06	12.50 ± 0.10	17.10 ± 0.20
Magnesium	mg/L	0.01	0.667 ± 0.006	7.33 ± 0.05	13.43 ± 0.45	13.17 ± 0.23
Tot-Hardness	mg/L	0.17	6.32 ± 0.25	50.10 ± 0.36	86.5 ± 2.26	96.87 ± 0.51
Turbidity	NTU	0.1	7.5 ± 0.3	1.0 ± 0.1	1.2 ± 0.2	1.6 ± 0.2
Tot-Suspended Solids	mg/L	3	6 ± 0	3 ± 2	9 ± 1	4 ± 2
Tot-Dissolved Solids	mg/L	10	n/a	86 ± 0.0	197 ± 6.0	124.3 ± 12.1
Tot-Nitrogen	mg/L	0.008	0.312 ± 0.002	3.267 ± 0.032	4.01 ± 0.437	4.747 ± 0.084
Ammonia	mg/L	0.005	0.029 ± 0.002	0.087 ± 0.021	0.020 ± 0.013	0.079 ± 0.001
Tot-Phosphorous	mg/L	0.004	0.017 ± 0.003	0.01 ± 0.001	0.015 ± 0.011	0.012 ± 0.006
Ortho-Phosphorous	mg/L	0.002	BDL[b]	0 ± 0.001	BDL	0.001
Tot-Org. Carbon	mg/L	0.2	n/a	3.6 ± 0.0	3.2 ± 0.3	3.2 ± 0.2
Diss-Org. Carbon	mg/L	0.5	n/a	n/a	3.2 ± 0.1	3.2 ± 0.1
Tot-Aluminum	μg/L	30.0	326 ± 50.8	238 ± 194.0	BDL	22.7 ± 13.3
Tot-Antimony	μg/L	0.5	0.1 ± 0.1	2.9 ± 0.2	3.8 ± 0.1	3.8 ± 0.1
Tot-Arsenic	μg/L	2.0	0.1 ± 0.1	0.6 ± 0.1	. 2 ± 0.0	1.7 ± 0.6
Tot-Barium	μg/L	1.0	17.1 ± 0.7	88.6 ± 2.1	170.3 ± 7.6	156.7 ± 2.1
Tot-Beryllium	μg/L	2.0	BDL	BDL	BDL	BDL
Tot-Bismuth	μg/L	10.0	BDL	BDL	BDL	BDL
Tot-Cadmium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Cesium	μg/L	0.4	BDL	BDL	BDL	BDL
Tot-Chromium	μg/L	3.0	BDL	BDL	BDL	BDL
Tot-Cobalt	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Copper	μg/L	2.0	2.4 ± 0.3	1.7 ± 0.6	BDL	1
Tot-Iron	mg/L	0.03	0.49 ± 0.09	0.02	0.04 ± 0.04	0.15 ± 0.09
Tot-Lead	μg/L	1.0	0.2 ± 0.1	0.5	BDL	1.0 ± 0.9
Tot-Lithium	μg/L	3.0	1.1 ± 0.5	1.5	1.5	1.5
Tot-Manganese	μg/L	1.0	12.8 ± 0.5	6.5 ± 0.1	20 ± 6.2	11.0 ± 0.0
Tot-Molybdenum	μg/L	1.0	0.1	26.1 ± 0.7	53.3 ± 1.5	72.3 ± 0.6
Tot-Nickel	μg/L	1.0	BDL	5.8 ± 0.0	12.3 ± 1.2	10.0 ± 0.0
Tot-Selenium	μg/L	10.0	5	5	BDL	6.7 ± 2.9
Tot-Silver	μg/L	0.3	BDL	BDL	BDL	0.4 ± 0.5
Tot-Strontium	μg/L	1.0	12.1 ± 0.6	85.4 ± 1.4	167.7 ± 7.1	217.7 ± 1.5
Tot-Thallium	μg/L	0.4	0.1	0.2	BDL	0.2
Tot-Titanium	μg/L	3.0	24.3 ± 1.1	0.2	BDL	2 ± 0.9
Tot-Uranium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Vanadium	μg/L	1.0	0.9 ± 0.2	1.4 ± 0.9	BDL	BDL
Tot-Zinc	μg/L	10	BDL	10.4 ± 9.4	BDL	BDL

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.
[f] Sampling depth from the lake surface was 6 m in August 1998; 10 m in August 1999; 10 m in July 2000; and 8 m in August 2000.

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Table C.5. Measurements of water quality within Cell D S1 of the LLCF[a]. Water collected 1 m below the surface of the lake.

Measurement	Unit	DL[b]	(mean ± SD of 3 replicate samples)			
			August 1998	August 1999	July 2000	August 2000
pH	pH	0.05	6.67 ± 0.06	6.96 ± 0.01	7.28 ± 0.02	7.18 ± 0.01
Conductivity	μS/cm	0.3	n/a[c]	37.9 ± 0.2	74.7 ± 0.2	74.5 ± 0.3
Alkalinity	mg/L	0.3	4.9 ± 0.3	6.4 ± 0.1	10.1 ± 0.0	10.0 ± 0.1
Calcium	mg/L	0.05	1.06 ± 0.03	2.63 ± 0.02	4.97 ± 0.01	5.52 ± 0.03
Magnesium	mg/L	0.01	0.55 ± 0.000	1.62 ± 0.02	3.32 ± 0.02	3.91 ± 0.02
Tot-Hardness	mg/L	0.17	4.91 ± 0.09	13.23 ± 0.06	21.1 ± 5.05	29.9 ± 0.1
Turbidity	NTU	0.1	1.2 ± 0.4	0.6 ± 0.1	0.6 ± 0.0	0.4 ± 0.0
Tot-Suspended Solids	mg/L	3	BDL[d]	3 ± 2	3 ± 3	2
Tot-Dissolved Solids	mg/L	10	n/a	30 ± 7.8	28 ± 6.6	66.3 ± 9.1
Tot-Nitrogen	mg/L	0.008	0.214 ± 0.004	1.157 ± 0.006	1.593 ± 0.055	1.820 ± 0.036
Ammonia	mg/L	0.005	0.034 ± 0.003	0.003	BDL	0.013 ± 0.002
Tot-Phosphorous	mg/L	0.004	0.01 ± 0.001	0.003 ± 0.001	0.011 ± 0.011	0.005 ± 0.004
Ortho-Phosphorous	mg/L	0.002	BDL	0.01 ± 0	BDL	0.001
Tot-Org. Carbon	mg/L	0.2	n/a	2.7 ± 0.1	3.3 ± 0.4	3.0 ± 0.1
Diss-Org. Carbon	mg/L	0.5	n/a	n/a	2.7 ± 0	3.1 ± 0.1
Tot-Aluminum	μg/L	30.0	42.4 ± 4.3	15	138.7 ± 214.2	15
Tot-Antimony	μg/L	0.5	0.1 ± 0.1	0.7 ± 0.1	0.7 ± 0.1	0.4 ± 0.3
Tot-Arsenic	μg/L	2.0	0.1	0.2 ± 0.1	BDL	0.5
Tot-Barium	μg/L	1.0	5.8 ± 0.4	18.8 ± 0.4	28.0 ± 13.0	35.7 ± 1.2
Tot-Beryllium	μg/L	2.0	0.1	1	1	BDL
Tot-Bismuth	μg/L	10.0	BDL	BDL	BDL	BDL
Tot-Cadmium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Cesium	μg/L	0.4	BDL	BDL	BDL	BDL
Tot-Chromium	μg/L	3.0	BDL	BDL	BDL	BDL
Tot-Cobalt	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Copper	μg/L	2.0	1.1 ± 0.4	1.8 ± 0.7	3.3 ± 4.0	BDL
Tot-Iron	mg/L	0.03	0.06 ± 0.01	0.02 ± 0.00	0.07 ± 0.05	0.04 ± 0.04
Tot-Lead	μg/L	1.0	0.1	0.8 ± 0.5	0.5	BDL
Tot-Lithium	μg/L	3.0	0.3 ± 0.1	BDL	6.7 ± 8.9	BDL
Tot-Manganese	μg/L	1.0	3.4 ± 0.8	2.1 ± 0.2	24.3 ± 34.4	2 ± 0.0
Tot-Molybdenum	μg/L	1.0	0.1	3.1 ± 0.1	7.0 ± 3.5	11 ± 0.0
Tot-Nickel	μg/L	1.0	BDL	1.1 ± 0.1	3.0 ± 1.7	2 ± 0.0
Tot-Selenium	μg/L	10.0	BDL	BDL	5	BDL
Tot-Silver	μg/L	0.3	BDL	BDL	0.5 ± 0.5	0.2 ± 0.0
Tot-Strontium	μg/L	1.0	7.5 ± 0.3	20.1 ± 0.8	108.7 ± 109.4	49.3 ± 0.6
Tot-Thallium	μg/L	0.4	0.1	BDL	0.2	BDL
Tot-Titanium	μg/L	3	2.3 ± 1.4	BDL	3.0 ± 2.6	BDL
Tot-Uranium	μg/L	0.3	0.1	BDL	0.2 ± 0.1	0.2 ± 0.1
Tot-Vanadium	μg/L	1	BDL	BDL	0.7 ± 0.3	0.7 ± 0.3
Tot-Zinc	μg/L	10	BDL	BDL	BDL	BDL

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.

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Table C.6. Measurements of water quality within Cell D S1 of the LLCF[a]. Water collected 1 m above the bottom of the lake.

Measurement	Unit	DL[b]	(mean ± SD of 3 replicate samples)			
			August 1998[f]	August 1999[f]	July 2000[f]	August 2000[f]
pH	pH	0.05	6.66 ±0.10	6.97 ±0.02	7.32 ±0.01	7.22 ±0.01
Conductivity	μS/cm	0.3	n/a[c]	36.3 ±0.1	71.6 ±0.2	73.1 ±0.3
Alkalinity	mg/L	0.3	4.6 ±0.1	6.3 ±0.1	9.7 ±0.0	10 ±0.0
Calcium	mg/L	0.05	1.08 ±0.02	2.52 ±0.01	4.64 ±0.01	5.4 ±0.03
Magnesium	mg/L	0.01	0.553 ±0.006	1.52 ±0.03	3.13 ±0.01	3.88 ±0.02
Tot-Hardness	mg/L	0.17	4.97 ±0.03	12.53 ±0.12	24.47 ±0.06	29.47 ±0.12
Turbidity	NTU	0.1	1.2 ±0.1	1.3 ±0.2	0.4 ±0.1	0.5 ±0.1
Tot-Suspended Solids	mg/L	3	BDL[d]	4 ±2	6 ±3	7 ±1
Tot-Dissolved Solids	mg/L	10	n/a	30.3 ±6.7	38 ±2.6	67 ±5.2
Tot-Nitrogen	mg/L	0.008	0.206 ±0.005	1.050 ±0.010	1.470 ±0.020	1.72 ±0.040
Ammonia	mg/L	0.005	0.031 ±0.001	0.003	BDL	0.015 ±0.006
Tot-Phosphorous	mg/L	0.004	0.005 ±0.001	0.003 ±0.002	0.016 ±0.024	0.008 ±0.004
Ortho-Phosphorous	mg/L	0.002	BDL	0.003 ±0.001	BDL	0.001
Tot-Org. Carbon	mg/L	0.2	n/a	3 ±0.3	3.5 ±0.4	2.9 ±0.1
Diss-Org. Carbon	mg/L	0.5	n/a	n/a	2.8 ±0.1	3.0 ±0.1
Tot-Aluminum	μg/L	30.0	66.4 ±14.4	45.3 ±31.9	BDL	15
Tot-Antimony	μg/L	0.5	0.2 ±0.1	0.6 ±0.3	BDL	0.3
Tot-Arsenic	μg/L	2.0	BDL	0.6 ±0.8	BDL	0.8
Tot-Barium	μg/L	1.0	5.7 ±0.3	17.4 ±0.5	34.3 ±0.6	34.3 ±0.6
Tot-Beryllium	μg/L	2.0	BDL	BDL	BDL	BDL
Tot-Bismuth	μg/L	10.0	BDL	BDL	BDL	BDL
Tot-Cadmium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Cesium	μg/L	0.4	BDL	BDL	BDL	BDL
Tot-Chromium	μg/L	3.0	BDL	BDL	BDL	BDL
Tot-Cobalt	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Copper	μg/L	2.0	1.5 ±0.4	1.3 ±0.6	1	BDL
Tot-Iron	mg/L	0.03	0.06 ±0.00	BDL	0.02 ±0.01	BDL
Tot-Lead	μg/L	1.0	0.6 ±0.7	BDL	BDL	BDL
Tot-Lithium	μg/L	3.0	0.6 ±0.4	1.5	1.5	BDL
Tot-Manganese	μg/L	1.0	3.1 ±0.2	3.2 ±1.7	4.0 ±0.0	2.3 ±0.6
Tot-Molybdenum	μg/L	1.0	0.1	2.7 ±0.0	8.7 ±0.6	10.3 ±0.6
Tot-Nickel	μg/L	1.0	10.9 ±18.7	1.4 ±0.6	2 ±0.0	1.7 ±0.6
Tot-Selenium	μg/L	10.0	5	5	4.8 ±0.3	4.8 ±0.3
Tot-Silver	μg/L	0.3	0.1	0.2	0.2	BDL
Tot-Strontium	μg/L	1.0	7.5 ±0.1	18.7 ±0.7	42.7 ±0.6	47.3 ±0.6
Tot-Thallium	μg/L	0.4	0.1	0.2	0.2	BDL
Tot-Titanium	μg/L	3.0	2.4 ±0.9	2.5 ±3.0	1.5	BDL
Tot-Uranium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Vanadium	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Zinc	μg/L	10	BDL	BDL	BDL	BDL

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.
[f] Sampling depth from the lake surface was 12 m in August 1998; 9 m in August 1999; 11 m in July 2000; and 11 m in August 2000.

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Table C.7. Measurements of water quality within Cell D S2 of the LLCF[a]. Water collected 1 m below the surface of the lake.

Measurement	Unit	DL[b]	August 1998	August 1999	July 2000	August 2000
			(mean ± SD of 3 replicate samples)			
pH	pH	0.05	6.66 ± 0.1	6.96 ± 0.01	7.3 ± 0.01	7.23 ± 0.01
Conductivity	μS/cm	0.3	n/a[c]	36.6 ± 0.4	71.4 ± 0.2	73.4 ± 0.2
Alkalinity	mg/L	0.3	4.6 ± 0.1	6.2 ± 0.1	9.7 ± 0.0	10 ± 0.0
Calcium	mg/L	0.05	1.08 ± 0.02	2.50 ± 0.01	4.67 ± 0.03	5.45 ± 0.02
Magnesium	mg/L	0.01	0.553 ± 0.006	1.53 ± 0.01	3.14 ± 0.02	3.89 ± 0.02
Tot-Hardness	mg/L	0.17	4.97 ± 0.03	12.50 ± 0.00	24.57 ± 0.15	29.6 ± 0.10
Turbidity	NTU	0.1	1.2 ± 0.1	1.1 ± 0.6	0.6 ± 0.2	0.4 ± 0.1
Tot-Suspended Solids	mg/L	3	BDL[d]	4 ± 2	3 ± 2	BDL
Tot-Dissolved Solids	mg/L	10	n/a	26.0 ± 2.6	33.7 ± 1.5	122.3 ± 14.6
Tot-Nitrogen	mg/L	0.008	0.206 ± 0.005	1.050 ± 0.000	1.44 ± 0.046	1.673 ± 0
Ammonia	mg/L	0.005	0.031 ± 0.001	BDL	0 ± 0.003	0.015 ± 0
Tot-Phosphorous	mg/L	0.004	0.01 ± 0.001	0.003 ± 0.001	0 ± 0.002	0.022 ± 0
Ortho-Phosphorous	mg/L	0.002	BDL	0.003 ± 0.002	BDL	BDL
Tot-Org. Carbon	mg/L	0.2	n/a	3.3 ± 2.5	3.4 ± 0.2	2.8 ± 0.1
Diss-Org. Carbon	mg/L	0.5	n/a	n/a	2.8 ± 0.1	2.8 ± 0.1
Tot-Aluminum	μg/L	30.0	66.4 ± 14.4	85.3 ± 121.8	BDL	BDL
Tot-Antimony	μg/L	0.5	0.2 ± 0.1	0.9 ± 0.4	0.9 ± 0.6	BDL
Tot-Arsenic	μg/L	2.0	BDL	0.1 ± 0.1	.BDL	BDL
Tot-Barium	μg/L	1.0	5.7 ± 0.3	20.5 ± 3.9	34.7 ± 1.2	36.0 ± 1.0
Tot-Beryllium	μg/L	2.0	BDL	BDL	BDL	BDL
Tot-Bismuth	μg/L	10.0	BDL	BDL	3.5 ± 2.6	3.5 ± 2.6
Tot-Cadmium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Cesium	μg/L	0.4	BDL	BDL	BDL	BDL
Tot-Chromium	μg/L	3.0	BDL	BDL	BDL	BDL
Tot-Cobalt	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Copper	μg/L	2.0	1.5 ± 0.4	2.8 ± 1.4	2.3 ± 2.3	1 ± 0.0
Tot-Iron	mg/L	0.03	0.06 ± 0.00	BDL	0.21 ± 0.10	0.21 ± 0.26
Tot-Lead	μg/L	1.0	0.6 ± 0.7	0.8 ± 0.5	BDL	BDL
Tot-Lithium	μg/L	3.0	0.6 ± 0.4	BDL	BDL	BDL
Tot-Manganese	μg/L	1.0	3.1 ± 0.2	2.7 ± 0.8	5.3 ± 1.5	2 ± 0.0
Tot-Molybdenum	μg/L	1.0	BDL	3.2 ± 0.6	8.3 ± 0.6	10.7 ± 0.6
Tot-Nickel	μg/L	1.0	10.9 ± 18.7	2.1 ± 1.5	2.0 ± 0.0	2 ± 0.0
Tot-Selenium	μg/L	10.0	BDL	BDL	BDL	BDL
Tot-Silver	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Strontium	μg/L	1.0	7.5 ± 0.1	25.1 ± 9.5	42.7 ± 0.6	50 ± 1.0
Tot-Thallium	μg/L	0.4	BDL	BDL	BDL	BDL
Tot-Titanium	μg/L	3.0	2.4 ± 0.9	BDL	BDL	BDL
Tot-Uranium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Vanadium	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Zinc	μg/L	10	BDL	BDL	BDL	BDL

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.

Table C.8. Measurements of water quality within Cell D S2 of the LLCF[a]. Water collected 1 m above the bottom of the lake.

Measurement	Unit	DL[b]	(mean ± SD of 3 replicate samples)			
			August 1998[f]	August 1999[f]	July 2000[f]	August 2000[f]
pH	pH	0.05	6.66 ± 0.10	6.97 ± 0.02	7.32 ± 0.01	7.22 ± 0.01
Conductivity	μS/cm	0.3	n/a[c]	36.3 ± 0.1	71.6 ± 0.2	73.1 ± 0.3
Alkalinity	mg/L	0.3	4.6 ± 0.1	6.3 ± 0.1	9.7 ± 0.0	10 ± 0.0
Calcium	mg/L	0.05	1.08 ± 0.02	2.52 ± 0.01	4.64 ± 0.01	5.4 ± 0.03
Magnesium	mg/L	0.01	0.553 ± 0.006	1.52 ± 0.03	3.13 ± 0.01	3.88 ± 0.02
Tot-Hardness	mg/L	0.17	4.97 ± 0.03	12.53 ± 0.12	24.47 ± 0.06	29.47 ± 0.12
Turbidity	NTU	0.1	1.2 ± 0.1	1.3 ± 0.2	0.4 ± 0.1	0.5 ± 0.1
Tot-Suspended Solids	mg/L	3	BDL[d]	4 ± 2	6 ± 3	7 ± 1
Tot-Dissolved Solids	mg/L	10	n/a	30.3 ± 6.7	38 ± 2.6	67 ± 5.2
Tot-Nitrogen	mg/L	0.008	0.206 ± 0.005	1.050 ± 0.010	1.470 ± 0.020	1.72 ± 0.040
Ammonia	mg/L	0.005	0.031 ± 0.001	0.003	BDL	0.015 ± 0.006
Tot-Phosphorous	mg/L	0.004	0.005 ± 0.001	0.003 ± 0.002	0.016 ± 0.024	0.008 ± 0.004
Ortho-Phosphorous	mg/L	0.002	BDL	0.003 ± 0.001	BDL	0.001
Tot-Org. Carbon	mg/L	0.2	n/a	3 ± 0.3	3.5 ± 0.4	2.9 ± 0.1
Diss-Org. Carbon	mg/L	0.5	n/a	n/a	2.8 ± 0.1	3.0 ± 0.1
Tot-Aluminum	μg/L	30.0	66.4 ± 14.4	45.3 ± 31.9	BDL	15
Tot-Antimony	μg/L	0.5	0.2 ± 0.1	0.6 ± 0.3	BDL	0.3
Tot-Arsenic	μg/L	2.0	BDL	0.6 ± 0.8	BDL	0.8
Tot-Barium	μg/L	1.0	5.7 ± 0.3	17.4 ± 0.5	34.3 ± 0.6	34.3 ± 0.6
Tot-Beryllium	μg/L	2.0	BDL	BDL	BDL	BDL
Tot-Bismuth	μg/L	10.0	BDL	BDL	BDL	BDL
Tot-Cadmium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Cesium	μg/L	0.4	BDL	BDL	BDL	BDL
Tot-Chromium	μg/L	3.0	BDL	BDL	BDL	BDL
Tot-Cobalt	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Copper	μg/L	2.0	1.5 ± 0.4	1.3 ± 0.6	1	BDL
Tot-Iron	mg/L	0.03	0.06 ± 0.00	BDL	0.02 ± 0.01	BDL
Tot-Lead	μg/L	1.0	0.6 ± 0.7	BDL	BDL	BDL
Tot-Lithium	μg/L	3.0	0.6 ± 0.4	1.5	1.5	BDL
Tot-Manganese	μg/L	1.0	3.1 ± 0.2	3.2 ± 1.7	4.0 ± 0.0	2.3 ± 0.6
Tot-Molybdenum	μg/L	1.0	0.1	2.7 ± 0.0	8.7 ± 0.6	10.3 ± 0.6
Tot-Nickel	μg/L	1.0	10.9 ± 18.7	1.4 ± 0.6	2 ± 0.0	1.7 ± 0.6
Tot-Selenium	μg/L	10.0	5	5	4.8 ± 0.3	4.8 ± 0.3
Tot-Silver	μg/L	0.3	0.1	0.2	0.2	BDL
Tot-Strontium	μg/L	1.0	7.5 ± 0.1	18.7 ± 0.7	42.7 ± 0.6	47.3 ± 0.6
Tot-Thallium	μg/L	0.4	0.1	0.2	0.2	BDL
Tot-Titanium	μg/L	3.0	2.4 ± 0.9	2.5 ± 3.0	1.5	BDL
Tot-Uranium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Vanadium	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Zinc	μg/L	10	BDL	BDL	BDL	BDL

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.
[f] Sampling depth from the lake surface was 12 m in August 1998; 6 m in August 1999; 6.5 m in July 2000; and 9 m in August 2000.

Table C.9. Measurements of water quality within Cell E of the LLCF[a]. Water collected 1 m below the surface of the lake.

Measurement	Unit	DL[b]	(mean ± SD of 3 replicate samples)			
			August 1998	August 1999	July 2000	August 2000
pH	pH	0.05	6.57 ±0.05	6.91 ±0.02	7.16 ±0.02	7.16 ±0.02
Conductivity	μS/cm	0.3	n/a[c]	28.8 ±0.2	49.8 ±0.2	53.8 ±0.9
Alkalinity	mg/L	0.3	5 ±0.0	5.4 ±0.1	7.4 ±0.0	8.2 ±0.1
Calcium	mg/L	0.05	1.23 ±0.02	2.06 ±0.03	3.09 ±0.03	4.10 ±0.08
Magnesium	mg/L	0.01	0.590 ±0.000	1.08 ±0.01	1.80 ±0.02	2.59 ±0.05
Tot-Hardness	mg/L	0.17	5.49 ±0.04	9.53 ±0.06	15.1 ±0.1	20.93 ±0.4
Turbidity	NTU	0.1	1.0 ±0.2	1.1 ±0.4	0.3 ±0.1	0.4 ±0
Tot-Suspended Solids	mg/L	3	BDL	3 ±3	5 ±1	2 ±1
Tot-Dissolved Solids	mg/L	10	n/a	20 ±3.6	54 ±16.1	60.7 ±7.4
Tot-Nitrogen	mg/L	0.008	0.217 ±0.003	0.725 ±0.002	0.898 ±0.01	1.19 ±0
Ammonia	mg/L	0.005	0.015 ±0.002	0.003 ±0.000	0.006 ±0.007	0.01 ±0.002
Tot-Phosphorous	mg/L	0.004	0.009 ±0.006	BDL	0.007 ±0.006	0 ±0.002
Ortho-Phosphorous	mg/L	0.002	BDL[d]	0.003 ±0.001	BDL	BDL
Tot-Org. Carbon	mg/L	0.2	n/a	3.2 ±0.1	2.8 ±0.0	3.1 ±0.1
Diss-Org. Carbon	mg/L	0.5	n/a	n/a	2.9 ±0.1	3 ±0.1
Tot-Aluminum	μg/L	30.0	43.1 ±14.7	BDL	BDL	24.7 ±16.7
Tot-Antimony	μg/L	0.5	BDL	0.3 ±0.1	BDL	0.4 ±0.3
Tot-Arsenic	μg/L	2.0	BDL	BDL	BDL	BDL
Tot-Barium	μg/L	1.0	6.6 ±0.5	11.5 ±0.1	18.7 ±0.6	24.3 ±3.2
Tot-Beryllium	μg/L	2.0	BDL	BDL	BDL	BDL
Tot-Bismuth	μg/L	10.0	BDL	BDL	BDL	BDL
Tot-Cadmium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Cesium	μg/L	0.4	BDL	BDL	BDL	BDL
Tot-Chromium	μg/L	3.0	BDL	BDL	BDL	2.0 ±0.9
Tot-Cobalt	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Copper	μg/L	2.0	1.6 ±0.2	3.5 ±1.4	1.3 ±0.6	BDL
Tot-Iron	mg/L	0.03	0.06 ±0.00	0.02	0.04 ±0.04	0.03 ±0.01
Tot-Lead	μg/L	1.0	0.1	1.1 ±1.1	0.5	1.0 ±0.9
Tot-Lithium	μg/L	3.0	0.9 ±0.4	1.5	1.5	1.5
Tot-Manganese	μg/L	1.0	4.1 ±0.2	3.1 ±0.1	3.3 ±0.6	2.0 ±0.0
Tot-Molybdenum	μg/L	1.0	0.1	1.2 ±0.1	3.7 ±0.6	6.3 ±0.6
Tot-Nickel	μg/L	1.0	0.1	0.5	0.7 ±0.3	1.2 ±0.8
Tot-Selenium	μg/L	10.0	5	5	5	7.0 ±3.5
Tot-Silver	μg/L	0.3	0.1	0.2	0.2	0.3 ±0.2
Tot-Strontium	μg/L	1.0	8.1 ±0.2	13.7 ±0.2	24.7 ±0.6	35.0 ±3.5
Tot-Thallium	μg/L	0.4	0.1	0.2	0.2	0.2
Tot-Titanium	μg/L	3.0	1.1 ±1.5	BDL	BDL	BDL
Tot-Uranium	μg/L	0.3	0.1	BDL	BDL	BDL
Tot-Vanadium	μg/L	1.0	0.1	BDL	BDL	BDL
Tot-Zinc	μg/L	10	BDL	BDL	BDL	BDL

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.

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Table C.10. Measurements of water quality within Cell E of the LLCF[a]. Water collected 1 m above the bottom of the lake.

Measurement	Unit	DL[b]	(mean ± SD of 3 replicate samples)			
			August 1998[f]	August 1999[f]	July 2000[f]	August 2000[f]
pH	pH	0.05	6.58 ±0.04	6.91 ±0.02	7.17 ±0.03	7.13 ±0.02
Conductivity	μS/cm	0.3	n/a[c]	28.7 ±0.1	51.3 ±0.6	53.3 ±0.3
Alkalinity	mg/L	0.3	5.0 ±0.0	5.4 ±0.0	7.6 ±0.1	8.2 ±0.1
Calcium	mg/L	0.05	1.23 ±0.01	2.06 ±0.02	3.40 ±0.18	4.08 ±0.01
Magnesium	mg/L	0.01	0.590 ±0.000	1.08 ±0.01	1.91 ±0.04	2.57 ±0.01
Tot-Hardness	mg/L	0.17	5.5 ±0.03	9.6 ±0	16.33 ±0.29	20.77 ±0.1
Turbidity	NTU	0.1	1.0 ±0.1	1.7 ±0.7	0.3 ±0.1	0.4 ±0.1
Tot-Suspended Solids	mg/L	3	27 ±45	3 ±1	4 ±2	3 ±2
Tot-Dissolved Solids	mg/L	10	n/a	19 ±2.6	48.7 ±1.5	51.7 ±7.4
Tot-Nitrogen	mg/L	0.008	0.191 ±0.017	0.726 ±0.004	0.949 ±0.016	1.133 ±0.060
Ammonia	mg/L	0.005	0.016 ±0.006	0.003	0.003	0.007 ±0.002
Tot-Phosphorous	mg/L	0.004	0.006 ±0.004	BDL	0.012 ±0.012	0.005 ±0.004
Ortho-Phosphorous	mg/L	0.002	BDL[d]	0.002 ±0.001	0.002 ±0.001	0.001 ±0.001
Tot-Org. Carbon	mg/L	0.2	n/a	3.1 ±0.0	2.8 ±0.1	3.1 ±0.1
Diss-Org. Carbon	mg/L	0.5	n/a	n/a	2.7 ±0.1	3.0 ±0.1
Tot-Aluminum	μg/L	30.0	34.7 ±5.8	15	15	15
Tot-Antimony	μg/L	0.5	0.1	0.5 ±0.2	0.3	0.7 ±0.0
Tot-Arsenic	μg/L	2.0	0.1	0.1	1	0.5
Tot-Barium	μg/L	1.0	6.4 ±0.1	11.7 ±0.1	19.3 ±0.6	24.0 ±0.0
Tot-Beryllium	μg/L	2.0	BDL	BDL	BDL	BDL
Tot-Bismuth	μg/L	10.0	BDL	BDL	BDL	BDL
Tot-Cadmium	μg/L	0.3	BDL	BDL	BDL	BDL
Tot-Cesium	μg/L	0.4	BDL	BDL	BDL	BDL
Tot-Chromium	μg/L	3.0	BDL	BDL	BDL	BDL
Tot-Cobalt	μg/L	1.0	BDL	BDL	BDL	BDL
Tot-Copper	μg/L	2.0	2.0 ±0.6	2.6 ±0.3	1	1
Tot-Iron	mg/L	0.03	0.06 ±0.00	0.02	0.02 ±0.01	0.02
Tot-Lead	μg/L	1.0	0.1	0.8 ±0.5	0.5	0.5
Tot-Lithium	μg/L	3.0	1.0 ±0.3	1.5	1.5	1.5
Tot-Manganese	μg/L	1.0	4.1	3.3 ±0.3	3.0 ±0.0	2.0 ±0.0
Tot-Molybdenum	μg/L	1.0	BDL	1.2 ±0.1	4.3 ±0.6	6.0 ±0.0
Tot-Nickel	μg/L	1.0	BDL	0.5	1.2 ±0.8	1
Tot-Selenium	μg/L	10.0	5	7.9 ±5.0	5	BDL
Tot-Silver	μg/L	0.3	0.1	0.2	0.2	BDL
Tot-Strontium	μg/L	1.0	8.2 ±0.3	13.9 ±0.4	26.3 ±0.6	30.7 ±0.6
Tot-Thallium	μg/L	0.4	0.1	0.2	0.2	0.2
Tot-Titanium	μg/L	3.0	0.8 ±0.3[d]	1.5	1.5	1.5
Tot-Uranium	μg/L	0.3	0.1	0.2	0.2	0.2
Tot-Vanadium	μg/L	1.0	0.1	0.5	0.5	0.5
Tot-Zinc	μg/L	10	BDL	BDL	BDL	BDL

[a] LLCF=Long Lake Containment Facility.
[b] DL=Analytical Detection Limit.
[c] Data not available for that variable and sampling season.
[d] BDL=Below Detection Limit.
[e] Numbers appear below detection limit because a lower DL was used at this time. Values used for DL (column 3) were the highest detection limits used by Taiga Environmental Laboratory during this study.
[f] Sampling depth from the lake surface was 8 m in August 1998; 8 m in August 1999; 3 m in July 2000; and 3 m in August 2000.

Table D.1. Mean ± SD concentration of chlorophyll *a* in water collected 1 m below the surface of the LLCF[1] at each sampling site from July 1999 to August 2000.

Sampling Time	Mean (mg/m³) ± SD chlorophyll *a* concentration				
	Cell B	Cell C	Cell D S1	Cell D S2	Cell E
July 1999	1.34±0.63	5.34±0.89	3.56±0.89	2.23±0.63	2.97±0.51
August 1999	7.46±1.35	3.73±1.08	N/A[3]	1.12±0.55	0.34±0.29
July 2000[2]		6.66±0.34	2.05±0.31	1.85±0.24	1.6±0.26
August 2000[2]		3.25±0.11	1.04±0.24	1.16±0.11	0.89±0.16

[1]LLCF=Long Lake Containment Facility.
[2]Sampling could not be conducted on Cell B in July and August, 2000.
[3]Chlorophyll *a* sample lost.

Table D.2. Mean ± SD concentration of chlorophyll *a* in water collected 1m above the bottom of the LLCF[1] at each sampling site from July 1999 to August 2000.

Sampling Time	Mean (mg/m³) ± SD chlorophyll *a* concentration				
	Cell B	Cell C	Cell D S1	Cell D S2	Cell E
July 1999	0.45±0	5.04±0.51	2.67±1.26	2.37±1.36	4.15±1.26
August 1999	7.48±1.22	3.68±0.15	0.69±0.37	0.85±0.33	0.34±0.12
July 2000[2]		3.81±0.28	3.1±0.22	1.87±0.37	1.33±0.11
August 2000[2]		3.1±0.38	1.04±0	1.04±0.21	1.0±0.23

[1]Long Lake Containment Facility.
[2]Sampling could not be conducted on Cell B in July and August, 2000.

Table E.1. Date processed kimberlite effluent (PKE) was collected from the Ekati™ Diamond Mine, Batch number assigned to PKE, date toxicity tests were initiated, and the type of toxicity test conducted.

Effluent Collection Date	Batch #	Date Toxicity Test Initiated	Type of Toxicity Test Conducted[a]
April 5, 1999	1	April 22, 1999	Acute whole, supernatant, and filtered
	1	May 13, 1999	Chronic 100% supernatant, and filtered
May 28, 1999	2	June 7, 1999	Chronic 100% supernatant, and filtered
	2	June 18, 1999	Serial dilution 6.25 to 100%
July 30, 1999	3	Aug. 1, 1999	Serial dilution 6.25 to 100%
	3	Aug. 13, 1999	Graduated pH (6.5) adjustment
Oct. 21, 1999	4	Nov. 1, 1999	Graduated pH (6.5) adjustment
	4	Nov. 27, 1999	EDTA chellation
Feb. 14, 2000	5	Feb. 22, 2000	Graduated pH (6.5) adjustment
	5	Mar. 7, 2000	Graduated pH (6.5) adjustment
April 6, 2000		May 16, 2000	Intake water from Cell C
April 6, 2000		May 16, 2000	Mill Processed Water; serial dilution
June 9, 2000	6	June 18, 2000	Graduated pH (6.5) adjustment
Aug. 6, 2000	7	Aug. 13, 2000	Graduated pH (6.5) adjustment
Oct. 16, 2000	8	Oct. 23, 2000	Post-SPE with C-18 extraction disks
	8	Oct. 30, 2000	Post-SPE (C-18) @ pH 3
Jan 8, 2001	9	Jan. 15, 2001	Filtered (0.45 μm) @ pH 3 & 11 Post-SPE (C-18) @ pH 3 & 11
	9	Jan. 25, 2001	EDTA chellation Anionic & cationic resin Activated charcoal
Feb. 12, 2001	10	Feb. 23, 2001	Filtered (0.45 μm) @ pH 3 & 11 Sodium thiosulfate Aeration
Oct. 13, 2001	11	Oct. 18, 2001	Bentonite and kaolinite

[a]All tests were 7-d chronic toxicity tests with *C. dubia*, except for the first test initiated April 22, 1999, which was a 48-h acute toxicity test.