8-10-2005

Electrodisinfection of Municipal Wastewater Effluent

Mark Peterson
University of New Orleans

ELECTRODISINFECTION OF MUNICIPAL WASTEWATER EFFLUENT

A Thesis

Submitted to the Graduate Faculty of the
University of New Orleans
in partial fulfillment of the
requirements for the degree of

Master of Science
In
Environmental Engineering

by

Mark R. Peterson

M.S. Metallurgical Engineering
Montana Tech, 1985

August 2005

ACKNOWLEDGEMENTS

The author wishes to gratefully thank the many people who have contributed and supported the work in this thesis.

Dr. Enriqué LaMotta has endured my many questions, delays, and problems without dismay or lack of help and has been the technical foundation of my support and assistance. Dr. Maria Elena Pulido also has endured the long work days and problems with great skill and knowledge to guide us through the complications and obstacles throughout the many turns and changes in the project.

Faculty and staff members at UNO have always responded to their best and provided help with the details beyond my ability on many aspects. Byron Landry and Kenneth McCormack supplied hours of their mechanical talents in assembling and repairing the equipment. Mr. Lucien Wainie helped removed many administrative hurdles without loss of a step when I was pressed for time.

Dr. Paul Schilling and Dileep Simhadri provided the technology and expertise needed for our X-ray diffraction analysis and provided a excellent report to assist us in identifying our problems and materials.

Juana Villavaso, administrative assistant extraordinaire, took care of the many administrative requirements I was unfamiliar with, and generally found out what was urgently required when I was already at the end of my time limit. I will remember her help and reliability always.

Most importantly, my wife Leslie has endured more grumbling, complaining, disturbed slumber and appeals for pity than any woman could without losing faith in me. She has had to be my constant support, sounding board, and editor while trying to work her regular 12 hour per day job and countless other commitments. I have never said thank you enough, reimbursed you for your lost sleep, or loved anyone so dearly.

Finally I thank my mother and family who have done without any help from me while my undivided attention was required here. I required endless errands, excused absences, and special favors from them all only to receive all I asked and more.

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ACRONYMS and ABBREVIATIONS

A – amps, amperes
A/cm^2 – amps per square centimeter
Al – aluminum
°C – degrees Celsius
CD – current Density
CDC – Center for Disease Control
Cl – chlorine (Cl^- = chloride ion, Cl_2 = free chlorine)
cm – centimeter
DT – detention time
e^- - electron
E. coli – Escherichia coli
ℑ - Faraday's Constant = 96,480 coulombs/mole-equivalent
g – grams
H^+ - hydrogen ion, also H_3O^+
H_2O - water
i.d. – inside diameter
kW – kilowatt
$kW\text{-}hr/m^3$ – kilowatt-hour per cubic meter
L – liter
ln – natural logarithm
m^3 – cubic meters
mA – milliamp
mA/cm^2 – milliamp per square centimeter
mg – milligrams
mg/L – milligrams per liter
mL – milliliter
mV - millivolts
nm - nanometer
o.d. – outside diameter
US EPA – United States Environmental Protection Agency
UV - ultraviolet
V – volts
W - watt
μm - micrometer

ABSTRACT

To avoid the spread of disease from sewage treatment effluents, pathogenic microorganisms present must be destroyed by one or a combination of disinfection methods. Chlorine remains the predominant disinfectant used although it consumes considerable amounts of energy and has associated exposure risks from production, transportation and storage of this poisonous gas.

In addition to bacteria and other objectionable microorganisms, color, suspended and colloidal solids also require removal from water for reuse. Aluminum and iron additions have been used to coagulate and remove non-settleable solids. By electrically dissolving aluminum to form solids-bridging aluminum hydroxide, the water itself can also be disinfected by the effects of electrical fields and its reactions to form disinfectant chemicals and direct destruction of microorganisms in the water.

This research investigated the effects of electrical current, time, and chloride concentration on the electrochemical disinfection of sewage treatment plant effluent using aluminum electrodes to substitute for chlorine disinfection.

1. INTRODUCTION

In our closed biosphere of Earth, water is regenerated and reused many times. Most water uses require treatment to remove impurities imparted to the water through the variety of uses needed by man. In particular, it is critical for even sewage water to be treated not only for biological solids and chemical removal, but disinfection to destroy a large variety of microorganisms to prevent sickness and spread of extremely dangerous contagious diseases.

The disinfectant used in over 90% of disinfection applications is chlorine or a chlorine based chemical to oxidize the water and destroy microorganisms and objectionable odors and tastes. Advances in ability to identify diseases and their sources have accumulated a variety of liabilities associated with chlorine use. The higher levels of chlorine needed for higher levels of organic materials requiring disinfection generate risky levels of disinfection byproducts that have been identified as carcinogens and suspected carcinogenic agents. Residual chlorine in treated sewage effluent can be toxic to fish, algae, crustaceans, and the nutrients they rely on in water causing government to consider a zero-residual for effluent chlorine. The electrochemical production for chlorine consumes high levels of electricity and associated power generation emissions, but now direct mercury emissions during production are also believed to be significant. The increasing loads in contaminated water and a growing population of water demand increases the risks of chemical exposure from accidental releases of the poisonous chlorine gas during production, transportation, and storage of chlorine. This risk must now also be evaluated in the light of targets of opportunity for terrorist attacks.

Other methods of disinfection are being examined ranging from gamma and ultraviolet rays to ozone and electrical pulses. This thesis examines application of electrical charge through water between consumable electrodes made of aluminum. As aluminum dissolves it forms charge ions in solution and solids with the capacity to neutralize dispersed solids that contribute to unwanted color in water and aid in settling problem solids to clarify the water. The electric charge also destroys microorganisms through a variety of methods; some which are known and well-characterized, and some methods that are partially known or unproven. This work attempts to provide more information on the relative significance electrical charge, treatment time, chlorine generated by trace amounts of chloride using electrodisinfection. Added benefit from generating aluminum coagulation during the process will be measured along with microorganism destruction by treating secondary sewage treatment plant effluent.

1.1 Problem Identification

The consequences of tainted water to human health have cursed mankind's history. A Sanskrit document over 4,000 years ago states what may have been the first drinking water standard. It directed people to "heat foul water by boiling and exposing to sunlight and by dipping seven times into a piece of hot copper, then to filter and cool in an earthen vessel." [Hall, E. and Dietrich, 2000]. Applying the mechanisms of ultraviolet radiation and copper toxicity to microbes for water disinfection took another 3,900 years to be verified and understood. The use of alum, an aluminum compound, to remove suspended solids may have first occurred 3,500 years ago in Egypt [EPA 816-R-99-007, 1999].

Initially, affliction from diseases sourced from bacteria and other materials in human wastes was minimal due to low population density, plentiful clean water sources, and man's nomadic migrations. Game fled in efforts for self-preservation spurring man's pursuit and migration. Migration of man followed water, which he needed for food, transportation, washing and waste discharge. Civilization followed establishment of communities, along with development of agriculture, fortifications, commerce, and plagues. The tapestry of mankind's history has been woven with more epidemics of disease than outbreaks of knowledge. Dysentery, typhus, typhoid, and cholera became common as man shared water, waste facilities, food sources, buildings, and commerce. Darwinian adaptation of immune systems developed antibodies that strengthened some immune systems to local microbes allowing survivors to pass on what facts, and superstitions, could be related.

Although in 1684, Antony van Leeuwenhoek published sketches of "wee animalcules," which were forms of common bacteria viewed with his microscope, the link between living organisms in water and disease required another 200 years. Only recently have we developed our knowledge base of sanitation and environmental engineering and decreased the ratio of outbreaks of epidemics to breakthroughs in pollution solutions. A series of worldwide cholera epidemics (pandemics) eventually lead to the linking of water-borne infections spread from bacteria in human wastes.

Cholera initially appeared in Calcutta in 1817, and spread through armies, pilgrimages, steam ships and the urbanization resulting from the Industrial Revolution. Worldwide cholera epidemics eventually spread around the world killing millions [Ponting, 1991]. Although there was a well established relationship between the disease and dirty water and poorly drained sections of towns by 1832, nothing was done to improve sanitation in the cities.

Dr. John Snow carefully observed and tracked cholera victims and water sources in London through the second and third cholera pandemics in 1848 and 1854. The 1854 London outbreak ended when he identified and closed down a pump and well whose water was contaminated by sewage where 500 had died within 250 yards of the neighborhood's water supply pump [BNP Media,1988]. Doubts and reluctance to change persisted, as did cholera, killing over 2,200 in London alone in 1866.

Louis Pasteur established the germ theory of disease from airborne sources in 1878, and Robert Koch, competing against Pasteur, identified and verified the specific bacteria (*vibrio bacter*) that caused cholera in 1884 to link water-borne microorganisms and disease. Unfortunately, elements of government and science still denied that invisible microorganisms could cause the diseases. Finally in 1892, during the fifth cholera pandemic, heeded warnings wrought a clearer lesson. Two adjacent towns used the same water source, but one had a water purification plant. When cholera broke out, it ran down the side of the street dividing the two cities, completely sparing the town with treated water.

Cholera was one of the first diseases recognized as capable of being waterborne [Lehrer, 1979], but still presents a danger, with a seventh pandemic plaguing South America, India and Africa [CDC, 1992]. In the 1990's a fear of chlorine in Peru caused water treatment to suspend chlorination resulting in over 1 million cases of cholera with over 13,000 deaths in a matter of

months [Schulz, 2004]. Today, the list of potential waterborne diseases due to pathogens has grown considerably larger, including bacterial, viral, and parasitic microorganisms in water. [EPA 815-R-99-014, 1999]

1.2 Microorganisms

Microorganisms commonly occur in nature's water, air, and soils. Before birth, humans are free from microorganisms. Exposure to microorganisms in food, air, and water results in them colonizing and remaining on and in our bodies. Most microorganisms are harmless and will contribute to a number of vital processes in the human body. Pathogenic microorganisms can cause harmful or deadly disease to humans with low or no resistance to the specific microorganism.

Pathogenic microorganisms can be divided up into three types: bacteria, viruses and parasitic protozoa. Bacteria and viruses can exist in both surface water and groundwater, whereas parasitic protozoa (such as those producing malaria, amoebic dysentery, giardiasis) are found mainly in surface water [Lenntech, 1998].

1.2.1 Bacteria

Bacteria, the most abundant type of life form on earth, are single-celled organisms typically ranging in size from 0.1 to 10 micrometers (μm). Bacteria (which can cause salmonella, malaria, and cholera) are active in many biological processes. Some bacteria play an important ecological role by breaking down organic matter, and others assist in the human metabolism. A majority of microorganism-sourced water contamination results from fecal contamination.

Fecal coliforms are bacteria that are associated with human or animal wastes. They usually live in human or animal intestinal tracts, and their presence in drinking water is a strong indication of recent sewage or animal waste contamination. *Escherichia coli* (*E. coli*) is a type of fecal coliform bacteria commonly found in the intestines of animals and humans. The presence of *E. coli* in water is a strong indication of recent sewage or animal waste contamination. Total coliforms present in water are frequently used as an indicator of potential contamination from fecal waste products.

1.2.2 Viruses

Viruses are microorganisms composed of the genetic material deoxyribonucleic acid (DNA) or ribonucleic acid (RNA) and a protective protein coat, typically sized between 0.01 to 0.1 μm, thus defeating most filtration devices. All viruses are strictly parasites, unable to carry out any form of metabolism and completely dependent upon host cells for replication. The virus spreads by contact of secretions (respiration, feces, blood, etc.) from those infected. Viral diseases include influenza, hepatitis, and polio. [EPA 815-R-99-014, 1999].

1.2.3 Protozoa

Protozoa are single-cell eucaryotic microorganisms without cell walls that consume bacteria and other organisms for food. Most protozoa are free-living in nature and can be encountered in water; however, several species are parasitic and live on or in host organisms. Host organisms can vary from simple organisms like algae to complex organisms such as humans. Some

protozoa species use humans hosts, leaving them afflicted with associated diseases), including *cryptosporidium parvum* (cryptosporidiosis), *giardia lamblia* (giardiasis), *entamoeba histolytica* (amoebic dysentery).

1.2.4 Impending Problems

Recent advances of disease identification and epidemiology identified new waterborne pathogenic threats. Outbreaks of these pathogens have caused concern by their increasing occurrences, severity, and resistance to our standard chlorination disinfection.

E. coli

First documented in the 1960s, waterborne *E. coli* outbreaks are claiming lives in increasing numbers. One of hundreds of strains of the bacterium *Escherichia coli, E. coli* O157:H7 is an emerging cause of foodborne and waterborne illness. Most strains of *E. coli* are harmless and live in the intestines of healthy humans and animals, but this strain produces a powerful toxin causing severe illness, especially in immune deficient people [EPA, 2005].

In the USA it is estimated that there are 73,000 cases of *E. coli* infection resulting in 61 deaths each year, mainly from food contamination [CDC, 2004]. Infection from using contaminated water to wash vegetables is included in under food contamination. This waterborne transport of *E.coli* to wash radishes left 12 dead in Sakai, Japan in 1996. In September, 1999 at the New York State Fair, runoff from cow manure after torrential rain was thought to have contaminated a well. The outbreak left 2 dead and affected over 1000. More recently, an outbreak of *E.coli* O157:H7 in Walkerton, Ontario in May 2000 killed 7 and struck at least 900 when a shallow water supply well became contaminated.

Giardia lamblia

Like *E. coli*, *Giardia lamblia* was first identified in the 1960s to be associated with waterborne outbreaks in the United States. *Giardia* has become one of the most commonly identified pathogens responsible for waterborne disease outbreaks. When dormant in its cyst stage, *Giardia* can survive extreme environmental conditions. The cysts are relatively large (8-14 μm) and are amenable to filtration. *Giardia* cysts are relatively resistant to chlorine, especially at higher pH and low temperatures. From 1994 to 1997, over 26,000 cases of giardia per year were reported [CDC, 2000].

Cryptosporidium

Cryptosporidium is a protozoan similar to *Giardia*. It forms resilient oocysts as part of its life cycle. The oocysts are smaller than *Giardia* cysts, typically about 4-6 μm in diameter. These oocysts can survive under adverse conditions until ingested by a warm-blooded animal and then continue with excystation.

Due to the increase in the number of outbreaks of cryptosporidiosis, a tremendous amount of research has focused on *Cryptosporidium*. *Cryptosporidum* caused the largest documented waterborne disease outbreak in United States history in Milwaukee in 1993. An estimated 403,000 people became ill, 4,400 people were hospitalized, and 100 people died. The outbreak was associated with a deterioration in raw water quality and a simultaneous decrease in effectiveness of the coagulation-filtration treatment, causing inadequate removal of

Cryptosporidium oocysts. During the past two decades, this pathogen has become recognized as one of the most common causes of waterborne disease within humans in the United States [CDC Factsheet, 2004].

Legionella pneumophila
An outbreak of pneumonia occurred in 1976 at the annual convention of the Pennsylvania American Legion used as its namesake. A total of 221 people were affected by the outbreak, including 35 deaths. A six months investigation by the Center for Disease Control (CDC) finally identified a bacterium which was named *Legionella pneumophila*, which studies have shown enters the body through the respiratory system. *Legionella* can be inhaled in water particles less than 5 μm in size from facilities such as cooling towers, hospital hot water systems, and recreational whirlpools [Witherell et al., 1988].

1.3 Water Disinfection
Disinfection efficiency is measured by the survival ratio, which is the number of microorganisms remaining divided by the original number of microorganisms contained in the sample. Because of the vast number of microscopic creatures in a small volume, and the susceptibility of human infection by relatively few microorganisms, the disinfection removal ratio is unwieldy, so the log of the ratio is used.

According to Chick-Watson's Law [Chick, 1908] the number of organisms destroyed per unit time (rate of kill) is proportional to the organisms remaining at time, t, following first-order reaction kinetics.

$$\log \frac{N}{N_O} = -kt$$

Where k is the decay constant, and a – sign is used assuming a reduction in organisms. Further assuming the reduction in organisms is a direct function of the disinfectant treatment, such as the concentration of chemical, C, the equation would be written:

$$\log \frac{N}{N_O} = -kCt$$

The Surface Water Treatment Rule (SWTR) Surface Water Treatment Rule [54 CFR 27486, June 29, 1989] requires 99.9% (3-log) removal of G*iardia* and 99.99% (4-log) removal of virus from drinking water supplies. Stated mathematically, 3-log removal (99.9%) would be:

$$\log \frac{N}{N_O} = \frac{0.1}{100} = -3$$

Sedimentation and filtration generally form the initial water treatment process and perform a 2.5 log removal (remove 99.68%) of bacteria and viruses. The remaining removal generally relies on chemical disinfection processes. The remaining disinfection following solids removal

processing is a function of disinfectant concentration, activity and time, as stated in Chick-Watson's Law.

1.3.1 Chlorine Disinfectants

Chlorination remains the prevalent method in most countries, including the United States. Chlorine works as a powerful oxidizing agent creating hypochlorous and hydrochloric acids, (HOCl and HCl) which in turn dissociate hydrogen (H^+), chloride (Cl^-) and hypochlorite (OCl^-) ions:

$$Cl_2 + H_2O \rightarrow HOCl + Cl^- + H^+$$

$$HOCl \rightarrow H^+ + OCl^-$$

Other chlorine-based disinfectants include chloramines and chlorine dioxide (ClO_2). Chloramine disinfection uses ammonia addition in conjunction with chlorine to stabilize and extend the time the chlorine remains dissolved in the water and to limit the free chlorine from reactions that form disinfection by-products (DBPs). Chloramine is a weak disinfectant and is less effective against protozoa and viruses than chlorine. In addition, chlorine dioxide (ClO_2) is another chlorine-based disinfectant that has found application in water treatment due to its greater stability.

Non-chlorine Disinfectants

Due to the use of chlorine gas as a chemical warfare agent during World War I, Europe has shown more favor to alternative disinfectants to chlorine. Ozone found favor in Europe due to fewer taste and odor problems. Ozone is generated by passing an electrical (corona) discharge through dry diatomic oxygen gas:

$$3O_2 \rightarrow 2O_3$$

Ozone, responsible for the familiar smell associated with lightning strikes and sparking in overworked electric motors, is a powerful oxidant which engages in oxygen atom transfers.

Ultraviolet (UV) radiation, alone and in combination with ozone, is expanding in water disinfection applications. UV disinfection was abandoned in favor of chlorination in the early 1900s due to high operating costs and low quality technology. Advances in electronics and materials improving the reliability of this method, coupled with concerns over chlorine DBPs have added a revival to UV disinfection. As with ozone, UV disinfection also works using indirectly formed disinfectant species.

Finally, and to the point of this work, electrochemical disinfection of water uses electrical energy to create chemical disinfectants from water decomposition products and by-product interactions. The oxidizing disinfecting species initiate mainly at the anode with the formation of oxygen according to the reaction:

$$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$$

Other oxidized disinfecting species such as ozone, chlorine, and various short-lived, highly reactive radicals are also reported to provide disinfecting properties. These reactions and mechanisms are discussed in the following sections.

Direct destruction of microorganisms by applied electrical fields is also cited as a disinfection method. Some investigators report a combination of direct electrical field combined with incidental production of chemical disinfecting species providing microorganism destruction.

Additionally, any combination of the disinfection methods and agents mentioned are applied in combinations yielding advantages and synergistic effects over using the individual disinfectants. Peroxide and ozone (Peroxone), UV and peroxide and/or ozone, and combinations with chlorine-based disinfectants have been reported. [EPA 815-R-99-014, 1999]

Table 1 summarizes the disinfectant usage by method for the United States. Some of the reporting systems use combinations of disinfectants, including all systems using ozone.

Table 1. Summary of U.S. Drinking Water Disinfectant Method [EPA-815-R-97-001a, b, 1997]

Type of Disinfectant	Number of Systems
Chlorine	22,307
Chlorine dioxide	313
Chloramines	135
Ozone	30
Potassium permanganate	1,122

1.3.3 Disinfection Mechanisms

Chlorine-based disinfectants

It is believed that chlorine (Cl_2^0) and hypochlorite (Cl^{+1}) compounds work primarily by denaturing enzymes or proteins, thereby inactivating microorganisms. In some cases, physical disruption of cell membranes may also contribute [EPA 600-R-01-110, 2001]. Research has shown that chlorine can produce lethal events at or near the cell membrane and affect DNA. Chlorine adversely affects bacteria cell respiration causing an immediate drop in oxygen use, damages the cell wall membrane, promotes leakage through the cell membrane, and produces lower levels of DNA synthesis for *Escherichia coli* and some other bacteria.[Haas and Engelbrecht, 1980]

The chemistry of chlorine has practical considerations in this regard: The chlorine(+1)-cation transfer step means that chlorine and hypochlorous acid both undergo 2-electron reductions. This 2-electron transfer provides a higher energy for reactions than single-electron transfer, allowing more energy to destroy organisms and overcome energy barriers. If a reducing agent does not provide 2 electrons, reactions are generally impeded by mass-transfer limitations. The 2-electron reduction can be expressed as follows:

$HOCl + H^+ + 2e^- \rightarrow Cl^- + H_2O$

$Cl_2 + 2e^- \rightarrow 2Cl^-$

Non-chlorine Disinfectants

Most disinfectants are manufactured using electrolytic methods. Ozone and hydrogen peroxide can also be generated in minor amounts during electrolysis of water. The primary products of water electrolysis are hydrogen and oxygen, but due to variations in potential available at the electrode surfaces and in solution, competing reactions, and concentration effects, other species are formed by the electrical energy input.

In addition to the direct oxidation of O_3, ozone can react with water molecules, producing hydroxyl radical (OH•), especially above pH 7.5. Hydroxyl radicals act as a strong oxidizing disinfectant, but short-lived due to its instability and reactivity, and are termed an indirect disinfectant. The contribution of each oxidant, direct ozone and indirect hydroxyl, is highly dependent on the source water quality because various chemicals, such as the ubiquitous carbonate from alkalinity and hardness, tend to deactivate the hydroxyl pathways. Table 2 lists the various oxidants that can be produced electrolytically along with their Standard Reduction Potential (E^O) in volts.

Ozone and hydroxyl radical attack a variety of sites in organic molecules. The fact that ozone is more effective than hypochlorite or chlorine for inactivating *Cryptosporidium* oocysts contributes to the growing number of disinfection systems using ozone. At the concentrations normally used for disinfecting drinking water, chlorination does not affect cryptosporidians significantly, but ozone does [EPA/600/R-01/110]. Ozone ruptures cell walls in many bacteria and viruses, and even permeates into G*iardia* cysts to attack the cell membranes inside.

Table 2. Standard Reduction Potential of Disinfectant Chemicals and Radicals compared to the Standard Hydrogen Electrode (SHE)[Rajeshwar K. and Ibanez, 1997] [CRC, 1974]

Oxidant	Formula	Standard Reduction Potential (volts)
Hydroxyl radical	$HO\cdot$	2.80
Monatomic oxygen	O	2.42
Ozone	O_3	2.07
Hydrogen peroxide	H_2O_2	1.78
Perhydroxyl radical	$HO_2\cdot$	1.70
Hypochlorous acid	HOCl	1.49
Chlorine	Cl_2	1.36
Diatomic oxygen	O_2	1.23
Hypochlorite	OCl^-	0.90

Ozone has been shown to be effective for DBP precursor reduction at low pHs. However, at higher pH values (i.e., above 7.5), ozone may actually increase the amount of chlorination byproduct precursors [EPA 815-R-99-014, 1999]. The increase in DBPs occurs in systems that also contain elevated levels of alkalinity. This is because alkalinity scavenges any hydroxyl free radicals formed during ozonation, leaving ozone as the only oxidant, and a lower oxidation potential than the hydroxyl free radical, and is thus less effective [EPA 815-R-99-014, 1999].

UV radiation near the 260 nanometers (nm) wavelength destroys the cell material (nucleic acids) killing the microorganisms. In addition, other UV wavelengths react with water forming highly oxidizing radicals including hydroxyl radicals which contribute to chemical disinfection. The radicals formed interact with water, other radicals or their decomposition byproducts causing further disinfection.

Direct destruction of microorganisms is reported to take place from applying electric potential to water in addition to the disinfection from electrochemically formed radicals [Porta and Kulhanek, 1986]. Applying high voltage electric field pulses (HVP) causing electroporation to kill microorganisms has been reported. In this method a direct current (DC) pulse disrupts the cell membrane, and reports chemical species also are involved in some of the microorganism deaths produced by this method. This method is also used to transfer inoculated DNA into cells in genetic research [Ulrich, 1997][Ghayeni and Coster, 2000]. Other investigations cite electrochemical oxidation of intracellular enzyme using low voltage [Li et al., 2004]. This investigation employed other experimental methods which reportedly excludes bactericidal effects from chemical disinfecting byproducts such as peroxide or free radicals, allowing only direct electrode contact cause disinfection [Matsunaga et al., 1992].

Evaluation of direct destruction of microorganisms by electroporation or molecular or genetic mutation was beyond the scope or measurement of this investigation. Destruction of the microorganisms in this thesis was not distinguished as electrical or electrochemically generated disinfectant sourced.

Drawbacks of Chlorination

Drawbacks noted using chlorination includes (objectionable) taste and odor problems and a variety of undesirable environmental liabilities. Chlorine disinfection can produce carcinogenic disinfection byproducts, and chlorine itself is a hazardous material. Further concerns include material handling, corrosion, and of recent concern, community risk to terrorist sabotage or attacks. In addition parasites such as *cryptosporidium* and *giardia* present in sewage effluent often survive conventional treatment processes using disinfectants such as chlorine, especially when in their oocyst stage. [EPA/815/R-99/014, 1999]

Chlorine generated industrially frequently contains other process contaminants unacceptable for use in drinking water of discharge into watersheds, prohibiting byproduct chlorine use in water treatment. As a result, chlorine generated for water treatment creates undesirable environmental offsets in addition to the objectionable amounts presently released to the environment. In 1981 in the USA alone, chlorination used in the treatment of water and wastewater consumed some 600,000 tons of chlorine and this involved a consumption of 28 million gigajoules of energy (7.8 gigawatt-hours [White, 1986]. Chlorine consumption by the water industry accounts for 4% of the 12,975,000 tons (10.4 million pounds) produced in the in 2002 [Chemical Market Reporter, 2003]. Concerns on mercury emissions from some power generating stations become more alarming the tons of mercury emissions each year from 9 outdated mercury cell chlor-alkali plants and controlling the large amounts of mercury inventory they require here in the U.S.A [Clayton, M., 2005].

Chlorine disinfection can generate many halogenated organic compounds as disinfection by-products (DBPs) from contact with natural organic matter (NOM). A number of DBPs (including trihalomethanes such as chloroform ($CHCl_3$), haloacetates, and chlorophenols) are probable or suspected carcinogens, or associated with exposure and causing cancer. Health risks associated with the DBPs produced by chlorination disinfection resulted in the Disinfection and Disinfectants Byproducts Rule promulgated by the EPA in 1998 [USEPA, 1998]. To reduce chlorine consumption and DBP formation, filtration removal of bulk organic material is usually required for water treatment. Additional oxidation of organic material using potassium permanganate is sometimes required, especially with surface water treatment, to assure final chlorination requirements do not exceed the maximum allowable chlorine or DBP levels.

Chlorine is a poisonous, corrosive gas requiring special construction materials and a high level of diligence to inspect for corrosion and deterioration and to maintain the structural integrity of the system. Pressurized or high volume vessels increase the risk and rate of accidents and dispersal. The risks of transporting and handling chlorine result in minor and major accidents each year. In 2005 local news, a leak from a failed chlorine tank valve at the Thibodaux, Louisiana water treatment plant evacuated the downtown area for several hours, and a rail accident near Graniteville, South Carolina released 11,500 gallons of chlorine gas, killing 9 and injuring over 500 [Center for Disease Control, MMWR, 2005].

Of the 49,450 events reported to Hazardous Substances Emergency Events Surveillance (HSEES) during 1999--2004, a total of 12,845 (30%) were transportation related; of these, 1,165 (9%) were rail events. Chlorine gas accounted for 11 (0.8%) of the releases reported to HSEES in rail events. Approximately 800,000 shipments of hazardous substances travel daily throughout the United States by ground, rail, air, water, and pipeline; approximately 4,300 shipments of hazardous materials travel each day by rail [US Dept. Transportation, 1998].

Though rail transport handled only 0.8% of chemical transportation shipments (which would include chlorine), these accounted for almost 42% of the tons moved. This reliance points to the reliability and overall good safety attributed to rail transportation. According to the Federal Railroad Administration (FRA) the train accident rate dropped 65% from 1980-2004 measured in accidents per million miles traveled [Association of American Railroads, 2004]. Though perhaps the safest transportation method, the breakdown of the available accident statistics for rail transport by HSEES (which only includes 16 states), the amount and routes still present a cause for concern. Of the 938 (81%) railroad events for which population data were available, 185,801 persons lived within one-quarter mile of the release. Durations of evacuation ranged from less than 1 hour to 13 days. Of the 1,055 (91%) railroad events for which a primary cause was identified, 645 (61%) resulted from equipment failure and 258 (24%) from human error. Elimination of all human error, though nearly impossible, would still result in a significant number of incidents.

1.5 Electrochemistry

1.5.1 Nernst Equation

In addition to the anticipated destruction of bacteria in the samples treated, there are a number of reactions in the aqueous solution competing for energy to complete various types of reactions. A

large variety of chemical and electrical reactions consume the voltage applied across the sample in the electrocoagulation cell (EC). Spontaneity of electrochemical reactions is determined by the free energy rules of thermodynamics, but reaction kinetic factors determine which spontaneous reactions will proceed and their rates.

In electrochemical reactions, applied electrical potential (voltage) provides the energy required for non-spontaneous reactions to occur. Dissolved species in the water undergo chemical oxidation and reduction reactions near the electrode surfaces, as do the electrodes themselves. Oxidation reactions takes place at the anode (positive electrode) and liberate electrons (e^-), while reduction takes place at the cathode (negative electrode) and consume electrons. The electrical charge decreases as the distance from the electrode increases because of increasing resistance and the volume in which the charge is dissipated.

The spontaneity of a reaction can be predicted by the Gibbs free energy change (ΔG) for the reaction. If the change in Gibbs free energy is negative ($\Delta G < 0$) the reaction is thermodynamically favored (occurs spontaneously). If the reaction can be carried out electrochemically, ΔG can be measured directly. The Gibbs free energy can be directly correlated to the voltage (E) of an electrochemical cell reaction:

$$\Delta G = -n \times \Im \times E$$

Where n represents the moles of electrons transferred per mole of reactant, E is the voltage change measured between the electrodes, and $\Im$ represents Faraday's Constant = 96,500 amp-seconds/equivalent weight.

The Vant Hoff isotherm is related to the Nernst equation through the thermodynamic property of Gibbs free energy:

$$\Delta G = \Delta G^o + R \times T \times \ln Q$$

With R = gas law constant; and T = absolute temperature; and Q is the equilibrium constant for the reaction for any generic reaction of the form:

$$aA + bB \rightarrow cC + dD$$

$$Q = \frac{[a_C]^c \times [a_D]^d}{[a_A]^a \times [a_B]^b}$$

a_i = the activity of each respective reactant and product species. The activity of a solution component is defined as the product of the activity coefficient (γ) and the molar concentration [C_i] of the component:

$a_i = \gamma_i \times [C_i]$ for component i.

Frequently it is more convenient to use the molar concentrations rather than the activities of the solution species by factoring out the activity coefficient. After inverting the reactants and products in the term for Q, and combining the activity coefficients term with the constant term for standard potential, E^0, for convenience. The equation then appears in the most used form:

$$E = E^{O'} + \frac{R \times T}{n \times \Im} \times \ln \frac{[C_O]}{[C_R]}$$

with $[C_O]$ = molar concentrations of the oxidized components, and $[C_R]$ = molar concentrations of the reduced components.

1.5.2 Electrochemical Reactions

In addition to the concentration of dissolved species in the electrochemical cell, the reactions are also strongly dependent on internal resistance. Additional resistance to current flow through the cell by imperfect conduction from low conductivity of the water contributes to inefficient current use by the process, manifested by heat generated raising the water temperature. Conductance increases by about 2% per degree centigrade [Duby, 1976] allowing some recovery of this energy loss by increased current efficiency

Another dominant factor limiting possible electrochemical reactions in aqueous solutions is the stability limits of water, which is the main component in aqueous systems. Water electrolyzes into hydrogen and oxygen at applied potentials above 1.23 volts at the cathode (-).

$2H^+ + 2e^- \rightarrow H_2\uparrow$ $\quad E^o = 0.0V$

$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$ $\quad E^o = -1.23V$

The voltage values cited, called the half-cell reaction potential, are measured in comparison to the standard hydrogen electrode (SHE), which assumes the voltage of hydrogen ions being reduced by electrons to form hydrogen gas is equal to 0 volts at standard reference conditions.

E = cell voltage = $E_{cathode} + E_{anode}$

Where $E_{cathode} + E_{anode}$ is the sum of the half-cell voltages for all the reactions, known and unknown, occurring in the system at the anode (oxidation) and cathode (reduction).

Additional electrochemical calculations used in this thesis include:
Quantity of electricity (coulomb, or amp-second) = $I \times t$

1.5.3 Polarization

The theoretical chemical yield cited for a reaction voltage will not be attained because of the non-ideal nature of reality. The rate of an electrochemical reaction is limited by numerous physical and chemical influences. These effects "polarize" or interfere with and slow down the reaction. Polarization results from reaction sequence effects or concentration effects, so the two types of polarization are called activation polarization and concentration polarization.

Polarization consumes part of the energy supplied by the electrodes as the reactions overcome these barriers. Each surface contact or change in phase (including conversion of reactants or

products from one phase to another) presents a resistance, an energy barrier, or a required addition of activation energy to achieving the desired reaction. Surface conditions also strongly affect the reactions with orientation and type of crystal structure, smoothness, residual mechanical stress, impurities, or coatings. The more complicated the system is, the more overpotentials required before sufficient energy has been added to allow the reactions to finally proceed.

Overpotential **(η)** is the shift in potential in the opposite direction to the applied current. The shift is away from the equilibrium value, increases with increasing current, is nonlinear, acts as an electrode resistance, varies with system (surface condition, distance, conductivity, materials, ions, solvent, crystallography, temperature, …) and is irreversible.

Overpotential types include:

Activation (or charge transfer) overpotential is an energy barrier to reactants and products on the reaction path (activated complex). Activation polarization occurs when multiple steps are required for a net reaction to complete. Reactions that require adsorption of an ion to the electrode surface, then the transfer electrons, followed by combination of two molecules to form an elemental gas bubble provide the best example. Production of hydrogen and oxygen at the electrodes is controlled by the slowest step in the process and is usually limited by activation polarization. Activation overpotential can be reduced by increasing the potential gradient across the double layer surrounding the electrode.

Concentration (or mass transfer or diffusion) overpotential is caused by slow diffusion of ions to or away from the electrode (J=D*dc/dx). Concentration polarization results when the concentration of oppositely charged counterions around the electrode becomes low enough that the reaction rate slows down while waiting for more counterions to diffuse to the electrode to be reacted.

Reaction overpotential results from slow reaction rate of ions supplied to the electrode which generates the species reacting at the electrode.

Crystallization overpotential is produced by a slow rate of inserting ions into the electrode crystal lattice resistance (ohmic), or from porous layers or films around electrodes during reduction reactions [Twidwell, 1976].

The final actual cell voltage required can then be written:

$$E = E^{o} + IR + \eta - \frac{RT}{n\Im} \times \ln Q \frac{oxidized}{reduced}$$

1.6 Statistical Analyses

1.6.1 Factorial Design Tests

In factorial designed tests the variables examined are tested at a high and low level. With 3 variables, as in this study, this results in a test space resembling a cube with 4 points on each corner representing a data point with 3 coordinates corresponding to the 3 levels of the variables

at that point. Each variable is evaluated alone at its high test level at 3 of the corner points. Each variable is involved with one other variable at their high test level at 3 other corners. One corner has all 3 variables at their high levels and the final corner has all 3 variables at their low test level. An additional point may be used in the center of the cube to check for nonlinearity in the responses between the high and low test levels. This point is also frequently used as the replicate point to test for data scatter for the test space from simple noise not due to changing variable test levels [Anderson and Whitcomb, 2000]. This idea is portrayed in Figure 1 with the coordinate's origin at the front right corner.

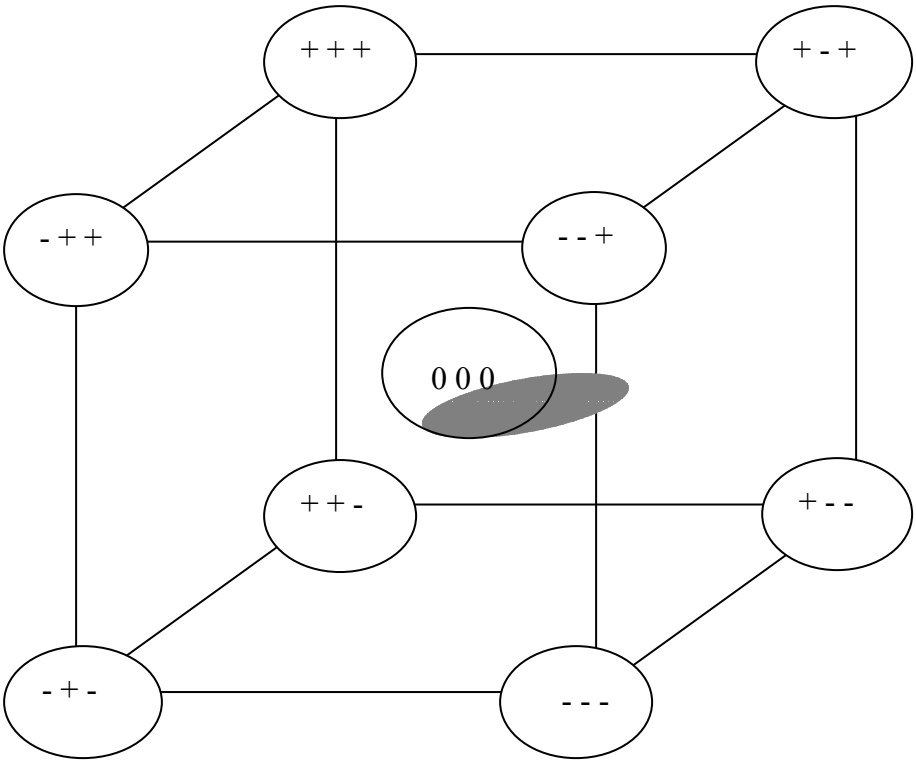

Figure 1. Portrayal of Factorial Design Test Space

Calculating the averages of the variables at the high tested level against their average values at the low tested level is compared against the process response. The magnitude of these changes is compared to the standard deviations to calculate if it is significant affect from the variable. This is done for each variable and response at each point. Each variable factor has 4 points at its high test level and 4 points at its low test level.

1.6.2 Analysis of Variances

Statistical analyses calculate the probability that a change measured in process response happened due to a change in operation variables or simply from natural variability of the process. The probability analysis compares the variation (standard deviation) and normal performance (average) at the two sets of operating conditions and establishes a probability with a given confidence level that the difference between them is a result of the difference in operating

parameters or just simply the variation (noise) inherent in the process. The probability is based on an analysis of the variances (ANOVA) between the standard deviation for all of the tests and the standard deviation for the subsets of tests at the high and low levels.

An F-test is employed to compute this statistical comparison. Mathematically stated, the F-test is:

$$\frac{\left[\dfrac{Model\ Sum\ of\ Squares}{Model\ \deg rees\ of\ freedom}\right]}{\left[\dfrac{\mathrm{Re}sidual\ Sum\ of\ Squares}{\mathrm{Re}sidual\ \deg rees\ of\ freedom.}\right]} = \frac{Model\ Mean\ Square}{\mathrm{Re}sidual\ Mean\ Square} = F\ statistic$$

Model Sum of Squares (S.S.) = $\Sigma\boldsymbol{\sigma}^2\times$degrees of freedom using averages for each group (high level and low level points). Each term from the model can also be evaluated in the same fashion against the residual S.S. with its single average.

Residual S.S. (or Total S.S.) = $\boldsymbol{\sigma}^2\times$degrees of freedom using a single (grand) average from all of the data.

2. LITERATURE REVIEW

2.1 Electrocoagulation

2.1.1 Aluminum reactions

A principal reaction in most electrochemical cells is the reaction of the electrode material itself. The strong oxidation or reduction condition at the surface causes most electrodes to react. In this thesis, the aluminum anode is dissolved according to the reaction:

$$Al_{(s)} \rightarrow Al^{3+} + 3e^- \quad E^o = +1.66V$$

The positive potential indicates the reaction is spontaneous in the direction written (aluminum being dissolved). According to Faraday's Law, the weight of metal dissolved at the anode (or deposited at the cathode) is a function of the current, time and number of exchanged electrons.

$$\text{Weight} = \frac{W_{eq} \times I \times time}{\Im}$$

$$W_{eq} = \text{Equivalent weight} = \frac{AtomicWeight}{Valence}$$

Valence representing the number of electrons required for the reaction; I = current in amperes, and $\Im$ = Faraday's constant. For aluminum, W_{eq} would be 26.98/3 = 8.99 g/equivalent.

Figure 2 is the stability diagram for aluminum in aqueous solutions from Pourbaix's Atlas [Pourbaix, 1974]. In Figure 1 the lines labeled a and b form the stability regions for water, representing the equations $2H_2O \rightarrow O_2\uparrow + 4H^+ + 4e^-$ ($E^o = -1.23V$) and $2H^+ + 2e^- \rightarrow H_2\uparrow$ ($E^0 = 0V$). The stable form of aluminum in water shown is aluminum oxide $Al_2O_3 \cdot 3H_2O$ (hydrargillite) which can be rewritten as $2Al(OH)_3$.

Aluminum metal is very reactive in air or water and quickly forms a stable (passive) oxide coating. After entering the aqueous phase, the Al^{3+} ions undergo hydrolysis, forming $Al(OH)_{3(S)}$ precipitate, and a variety of dissolved complex ions. Aluminum ions are very reactive due to their small (0.5×10^{-10}m) size and high charge density (+3) and become correlated with 6 water molecules in solution. The aluminum forms polynuclear complexes removing ions from solution and bridging particles by charge neutralization to aid settling of fine solids and precipitates. The charge of the hydrous oxide depends on the pH of the liquid resulting in amphoteric behavior [Stumm and Morgan, 1996].

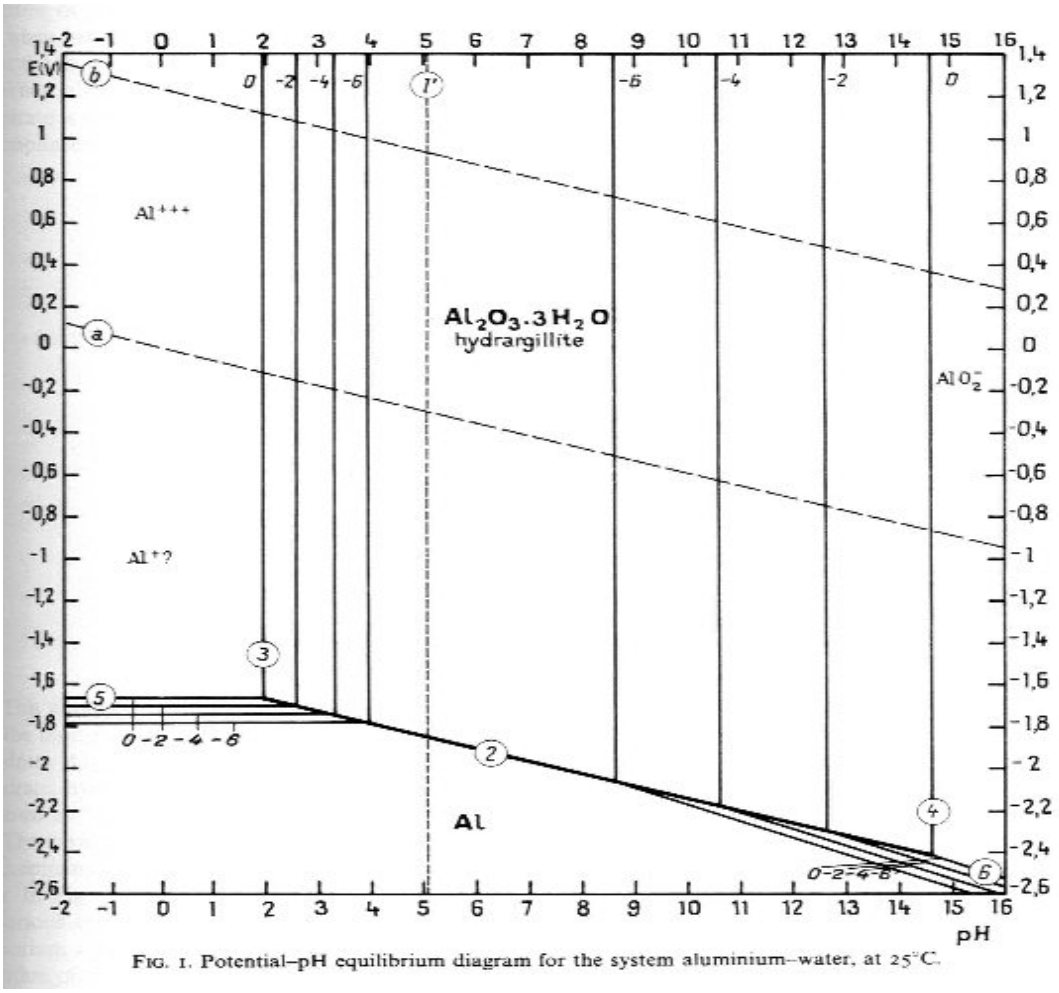

FIG. 1. Potential–pH equilibrium diagram for the system aluminium–water, at 25°C.

Figure 2. Aluminum electrochemical stability diagram [Pourbaix, 1974]

The complete chemistry of hydrolysis reactions and products is not well understood. Reported complex species formulae, charge, and thermodynamic data for hydrolysis vary with the investigators. The most recent report cited by Metcalf and Eddy from a number of investigators identifies aluminum hydrolysis products $AlOH^{2+}$, $Al(OH)_2^{+}$, $Al(OH)_4^{-}$, and $Al(OH)_3$. These data along with the reactions are presented in Table 3 and depicted graphically in Figure 3 [Metcalf and Eddy, 2003]. Similar data taken from Stumm and O'Melia [Stumm and O'Melia, 1968] in Figure 4, and Baes and Mesmer [Baes and Mesmer, 1976] in Figure 5 show the great variations observed in solubilities and pH for minimum observed solubilities. The Metcalf and Eddy data discount the effects of $Al(OH)_2^{+}$ because it doesn't intersect the solid phase region, but the dissolved $Al(OH)_2^{+}$ affects the total Al^{+3} in the system. The cross-hatched region appears in the Metcalf and Eddy solid phase region, but this region would be shifted from the $Al(OH)^{2+}$ border to the Al^{3+} border as considered by the other authors. Other proposed species identified by these investigations include: $Al_2(OH)_2^{4+}$, $Al_4(OH)_8^{4+}$, $Al_2(OH)_5^{4+}$, $Al_6(OH)_{15}^{3+}$, $Al_7(OH)_{17}^{4+}$, $Al_8(OH)_{20}^{4+}$, $Al_{13}(OH)_{34}^{5+}$,$Al_3(OH)_4^{5+}$, and $Al_{13}O_4(OH)_{24}^{7+}$. [Pourbaix, 1974][Metcalf and Eddy, 2003][Sillen L. and Martell, 1964]

Table 3. Aluminum Hydrolysis Products and Equilibrium Constants

Reaction	Log K
$Al^{3+} + 3(OH)^- \rightarrow Al(OH)_{3(S)}$	+31.2 (inverse of solubility product constant)
$Al_3^+ + H_2O \rightarrow Al(OH)^{2+} + H^+$	-5
$Al_3^+ + 2H_2O \rightarrow Al(OH)_2^+ + 2H^+$	+1.5
$Al(OH)_{3(S)} + H_2O \rightarrow Al(OH)_4^- + H^+$	-12.2

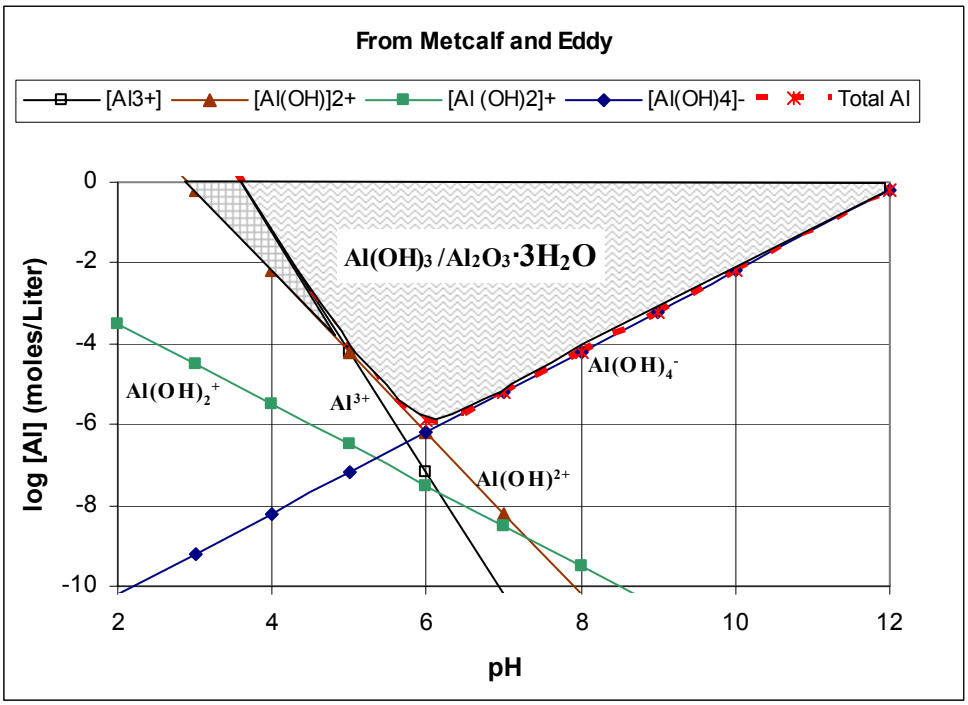

Figure 3. Aluminum Hydrolysis Solubility Diagram [Metcalf and Eddy, 2003]

Analyzing aluminum coatings reveals layers form onto aluminum in 3 stages of aluminum hydrated oxides below 60°C [Hart, 1957]. The initial layer of amorphous aluminum hydroxide gel crystallizes gradually into AlOOH, and finally crystallizes into bayerite ($Al_2O_3 \cdot 3H_2O$). The precipitates formed by aluminum hydrolysis in water gradually age by crystallizing through a series of hydroxyl compounds. The hydroxide gel crystallizes to orthorhombic böhmite, then to monoclinic bayerite, and then to monoclinic hydrargillite. The solubilities of these compounds vary as summarized in Table 4. The increasingly negative values of the log $\{[Al^{3+}][OH^-]^3\}$ values indicates that the solubilities of the progressive crystallization products decrease with time and increasing molecular stability. Their study also reports that hydrolyzing solutions tend to supersaturate allowing hydrolysis to proceed further than the expected value of the reaction constant K with higher dissolved species concentrations reported than thermodynamic data supports [Pourbaix, 1974]. Pourbaix also notes great discrepancies in reported solubilities and doubt on what the actual aluminum compounds were obtained and measured. {Note: The 1955 dated Pourbaix data agrees most closely with the most recent data selected by Metcalf and Eddy on approximating the minimum solubility observed for the solids of the various sources referenced at $10^{-5.8}$ moles/L.}

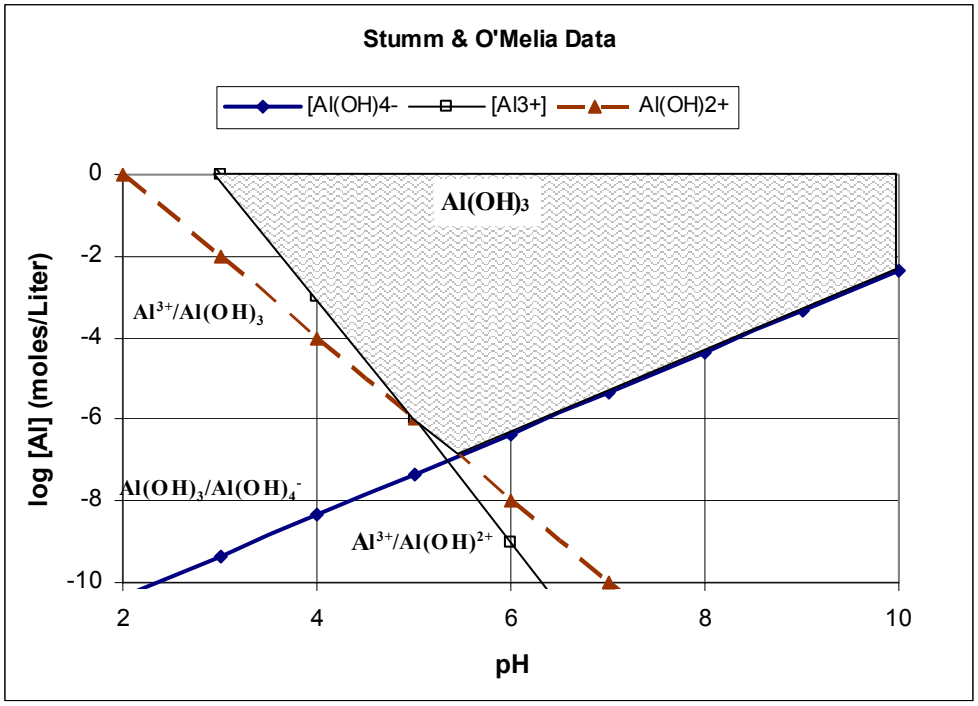

Figure 4. Aluminum Hydrolysis Solubility Diagram [Stumm and O'Melia, 1968]

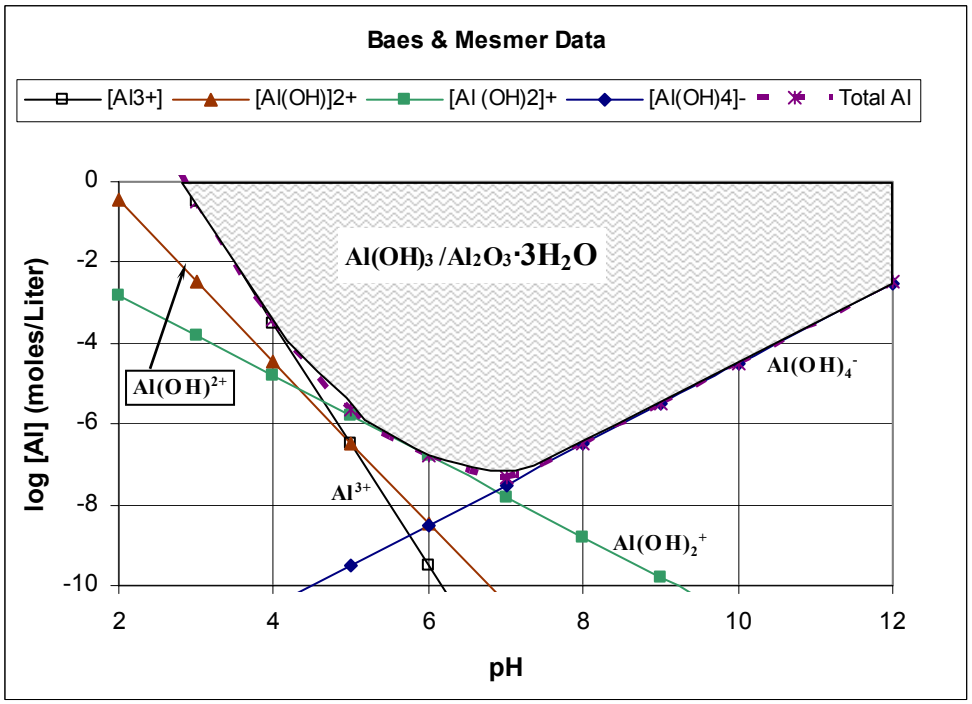

Figure 5. Aluminum Hydrolysis Solubility Diagram [Baes and Mesmer, 1976]

The aluminum precipitates and dissolved complexes adhere to oppositely charged surfaces (usually anionic [negative] charges), but also affect cationic charged materials as well. Neutralizing these charged surfaces stops their repelling forces allowing them to coalesce and settle. The charged aluminum species can further neutralize other dissolved charged species or suspended solids, removing surface charges which repel and cause colloids to remain suspended. The fine-sized neutral aluminum hydroxide precipitate further promotes coagulation and settling by providing solid surfaces to attach to and coalesce, building the particle size and increasing the settling rate of the solids.

Table 4. Solubilities of Aluminum Oxides [Deltombe et al. 1955]

Mineral	Formula	$Log\{[Al^{+3}][OH^-]^3\}$	Log solubility in pure H_2O
Hydrargillite	$Al_2O_3 \cdot 3H_2O$	-36.3	-7.8
Bayerite	$Al_2O_3 \cdot 3H_2O$	-35.5	-7.2
Böhmite	$Al_2O_3 \cdot H_2O$	-34.0	-6.2
Amorphous hydroxide	$Al(OH)_3$	-32.3	-5.3

Additionally, there can be many intermediate chemical reactions between products, reactants and contaminants in the bulk solution changing the composition and net energy balance. Because of the large number of potential reactions and unknown components in the uncontrolled samples treated in this study, determining the exact energy efficiency of the electrodes is beyond the scope of this study. The investigation focused on the applicability and disinfection efficiency achieved using aluminum pipes as expendable electrodes for disinfecting secondary sewage treatment effluent as a substitute for chlorine.

2.1.2 Previous Electrocoagulation Investigations

Coagulation by addition of alum ($Al_2(SO_4)_3 \cdot 14H_2O$) and ferric chloride ($FeCl_3$) is frequently used in potable water production to remove objectionable color, suspended solids and haze from colloidal suspensions. Electrocoagulation is electrochemical production of destabilization agents to cause charge neutralization for pollutant removal. Direct generation of the chemical species by electrolytic dissolution of these metal species to reduce costs followed in England back in the 1880s [Vik et al., 1984]. In addition to forming dissolved and precipitated chemical species, the applied electrical potential also can neutralize and precipitate dissolved ions, adsorb onto and flocculate suspended solids, and achieve varying levels of bacteria destruction [Mills, 2000]. Use of aluminum as the chosen metal varies with the availability, efficiency precipitating the process pollutant, pH, or aluminum's advantages being lower weight and not imparting color to the effluent. [Do and Chen, 1994][Donini et al., 1994]

Direct application of aluminum electrocoagulation and disinfection has successfully removed clay and paint suspensions, oil emulsions, and dissolved metals and phosphate from waste streams [Barkley et al., 1993]. Coagulated solids and precipitated metals were removed by settling after contact with aluminum pellets in the space between electrodes in an electrocoagulation cell. Salt was added to increase conductivity of the samples.

The cost of aluminum electrocoagulation systems varies with the power consumption, so salt (NaCl) is typically added to increase conductivity. The freshly produced aluminum polyhydroxide species are more reactive resulting in reduced aluminum consumption in comparison to alum use, and less bound water in the settled sludge reduces waste volume [Donini et al., 1994]. Donini also found that the amount of aluminum dissolved was over 100% of the expected value from the equation

$$Al_{(s)} \rightarrow Al^{3+} + 3e^- \quad E^o = +1.66V$$

This is due to the reaction of both the cathode and anode dissolving in addition to the reaction of the anode because aluminum favors reacting in the presence of any oxygen and especially an oxidizing atmosphere and when the surface passive layer can be acted on to further reaction. Actual aluminum dissolution reported was 165-215%.

Applying the aluminum electrolytic cell for disinfection of fecal coliforms found success when coupled with addition of 2-4 mg/L silver ions (Ag) or UV light [Robinson, 1999]. But investigations applying only the aluminum electrodisinfection are limited.

2.2 Water Disinfection

Electrochemical disinfection has been reported as effective in killing many pathogenic microorganisms, but the exact mechanisms and their comparative importance are still not completely established. The effectivity varies with the electrode construction, geometry of the reactor, concentration and makeup of the pollutants and microorganisms, and the type of electric current applied. The combination of electrical field, magnetic field, and *in situ* chemical disinfectant and electrochemical radical formation can produce effective destruction of pathogens. A partial list of known, identified, and proposed reactions of electrochemical disinfecting species which can or may be involved with pathogen destruction previously published is offered below.

Three sources of reactions involved in water disinfection and byproduct reactions during the electrolysis of water are cited in this thesis:

- Measured values for reactions from databases
- Reactions identified or detected without measured yields or thermodynamic data.
- Postulated reactions reported by investigators

2.2.1 Chemical Disinfection Reactions

It is established that electrolysis of water produces oxygen, hydrogen, hydrogen peroxide and ozone (and chlorine if chloride ions are present), and applied in the commercial production of these chemicals. The reactions, their standard reduction potentials (versus the standard hydrogen electrode in the cited reaction direction) follow: (Except where noted, the reduction potentials cited are from Handbook of Chemistry and Physics, CRC Press, 1975):

Anode Reactions

D1 $2H_2O \rightarrow O_2 + 4H^+ + 4e^-$ $E^o = -1.23V$

D2 $2H_2O \rightarrow H_2O_2 + 2H^+ + 2e^-$ $E^o = -1.776V$

D3 $2Cl^- \rightarrow Cl_2 + 2e^-$ $E^o = -1.36V$

D4 $Cl^- + 2OH^- \rightarrow ClO^- + H_2O + 2e^-$ $E^o = -0.90V$

D5 $Cl^- + H_2O \rightarrow HClO + H^+ + 2e^-$ $E^o = -1.49V$

Cathode Reaction

D6 $2H_2O + 2e^- \rightarrow H_2 + OH^-$ $E^o = -0.83V$

D7 $O_2 + 2\ H_2O + 4e^- \rightarrow 4\ OH^-$ $E^o = +0.401V$

The energy consumed in producing these oxidants is reflected in the magnitude and the negative sign of the reduction potential, indicating these reactions consume energy. The energy is released during disinfection when these reactions (except D6 which produces the reductant hydrogen gas) occur in the reverse direction, reducing the oxidants, destroying microorganisms, and oxidizing other molecules and atoms present in the water. The magnitude of the potential also can be used as a rough measure of the probability and relative amounts of each oxidant formed based on the required energy, with probability decreasing with the magnitude of the negative voltage. The concentration of the reactants also strongly affects the likelihood of the reaction. Reactions requiring more potential (more negative voltage) are less likely to acquire the higher voltage in the competing reactions and have the all the required reactants present at the site.

Secondary reactions also take place with these electrolysis products to form other chemical disinfecting species. These reactions (written as anodic reactions) have a lower probability of occurring because their reactants must first form, and then react again with water or another species and the electrode.

D8 $O_2 + H_2O \rightarrow O_3 + 2H^+ + 2e^-$ $E^o = -2.07V$

D9 $O_2 + 2OH^- \rightarrow O_3 + H_2O + 2e^-$ $E^o = -1.24V$

D10 $O_2 + 2H^+ + 2e^- \rightarrow H_2O_2$ $E^o = +0.68V$

D11 $O_2 + H_2O + 2e^- \rightarrow H_2O_2 + 2OH^-$ $E^o = -0.146V$

D12 $HOCl + e^- + H^+ \rightarrow \frac{1}{2}\ Cl_2 + H_2O$ $E^o = +1.63V$

D13 $Cl_2 + H_2O \rightarrow HOCl + Cl^- + H^+$

D14 $HOCl \rightarrow H^+ + OCl^-$

D15 $3O_2 \rightarrow 2O_3$

Evaluating the required potential for these reactions, D10 could occur spontaneously (depending on the oxygen gas concentration available to provide reactants) and D11 has a low required potential and would be more likely to be formed of the above oxidants. Equation D7 does not appear in the investigations noted in this thesis, but notably appears commonly in corrosion analysis [Fontana and Greene, 1967].

2.2.2 Free Radical Reactions

Finally, activated chemical species that have high reduction potentials are very short-lived due to their reactivity, and hence more difficult to detect, characterize or measure. The activated species may be charged (+ or -), or may simply be a free radical species with no charge and only a highly reactive specie. The mechanism of the formation and reaction of these highly reactive

species in disinfection is not completely understood and is still being studied. The radical species with measured reduction potentials reported include:

D16 $H_2O \rightarrow O\cdot_{(g)} + 2H^{+} + + 2e^{-+}$ $E^o = -2.42V$
D17 $H_2O_2 \rightarrow HO_2\cdot + H^{+} + e^{-}$ $E^o = -1.5V$
D18 $3OH^{-} \rightarrow HO_2^{-} + H_2O + 2e^{-}$ $E^o = -0.87V$ [-0.88V Tatapudi & Fenton, 1994]
D19 $OH^{-} \rightarrow OH\cdot + e^{-}$ $E^o = -2.8V$ [Hoigné J. and Bader, 1978]
D20 $O_2 \rightarrow O\cdot + O\cdot$
D21 $O_3 + H_2O + 2e^{-} \rightarrow O_2 + 2\,OH^{-}$ $E^0 = +1.24V$ [EPA 815/R/99/014]

2.2.3 Previous Electrochemical Investigations

Porta and Kulhanek applied current densities of 0.5-2 A/dm^2 (5-20mA/cm^2) to depollute water with $6*10^6$ germs per liter. Their documents reported that using alternating current (AC) as the power source produced no germ destruction and proposed that direct destruction of microorganisms from the applied potential may occur. Additionally, their patent documents postulated 3 reactions involving hydroperoxide ion (HO_2^-) as potentially responsible for the germ destruction: [Porta and Kulhanek, 1986]

D22 $O_2 + H_2O + 2e^{-} \rightarrow HO_2^{-} + OH^{-}$ $E^o = -0.076V$ [-0.065V Rajeshwar &Ibanez]
D23 $HO_2^{-} \rightarrow OH^{-} + 1/2O_2$
D24 $HO_2^{-} + 2e^{-} + H_2O \rightarrow 3OH^{-}$

Patermarakis & Fountoukidis found a disinfection factor proportional to the square of the current applied to the cell and treatment time, yielding the Chick-Watson format equation:

$$\frac{N}{N_O} = kt = ki^2 t = 8.4 \times 10^{-3}\, mA^{-2} cm^4 \times i^2 \times t$$

for current densities, i, from 2.5 to 5 mA/cm^2 and time, t, in minutes. They hypothesized that the reactions taking place (in addition to the 3 hydroperoxide ion reactions postulated in the Porta Patent above) were [Patermarakis and Fountoukidis, 1990]:

D25 $2OH^{-} \rightarrow H_2O + [O] + 2e^{-}$
D26 $O_3 + H_2O \rightarrow HO_3^{+} + OH^{-}$
D27 $HO_3^{+} + OH^{-} \rightarrow 2HO_2\cdot$ ($HO_2\cdot$ perhydroxyl radical)
D28 $O_3 + HO_2\cdot \rightarrow HO\cdot + 2O_2$ (Hydroxyl radical, $HO\cdot$)
D29 $3H_2O + \rightarrow O_3 + 6H^{+} + 6e^{-}$ $E^o = -1.51V$

and the chlorine based disinfection reactions:

D3 $2Cl^{-} \rightarrow Cl_2 + 2e^{-}$
D13 $Cl_2 + H_2O \rightarrow HOCl + Cl^{-} + H^{+}$
D14 $HOCl \rightarrow H^{+} + OCl^{-}$

A later paper by Li et al. provides a good summary of proposed electrochemical mechanisms responsible [Li et al., 2002]. Their paper reports the disinfection relation equation for saline wastewater:

$$\log \frac{N}{N_O} = kt = kI_d t = -0.01 \times (I_d t)^{1.87}$$

This relation shows the current density, I_d, and time in seconds, t, affecting the disinfection rate. 3-Log reduction in coliforms was recorded with 5 mA/cm^2 current densities in 9 seconds from the saline secondary wastewater treatment effluent. A 1-Log reduction using up to 30 mA/cm^2 current density was achieved with the fresh water secondary wastewater effluent for 15 seconds. A rare-earth coated electrode system was employed in this work.

The importance of chloride and the electrochemical formation of chlorine are not agreed upon by researchers. The work cited previously by Li [Li et al, 2002] discounts the effects of byproduct chlorine produced *in sit*u or direct destruction of germ cells by the electric field because of the low levels of chlorine measured and the ineffectivity on non-saline samples at the same voltage effective with saline samples. Applying direct chlorination to disinfect the samples tested at the levels produced by the electrochemical treatment did not achieve the same level of bacteria inactivation. A subsequent paper by Li found no disinfection with sulfate or nitrate salt addition but did note higher levels of disinfection if chloride salt was added in electrodisinfection tests, though he estimated only 10% of the inactivation was due to chlorine synthesis for disinfection [Li et al. 2004].

3. EXPERIMENTAL PHASE

3.1 Experimental Materials and Procedures

3.1.1 Feed Samples

Secondary wastewater treatment effluent was obtained courtesy of the Sewerage and Water Treatment Board of New Orleans from their New Orleans East Bank sewage treatment plant. The plant, located in Eastern New Orleans, consists of standard screening, degritting, and pure oxygen-activated sludge treatment followed by secondary clarification and chlorination. The samples were removed from the overflow trough of the secondary clarifier prior to contact in the chlorination trench. Typical analyses of the secondary settling overflow stream are presented in the Table 5.

Table 5. Permit Discharge Limits for Sample Wastewater from New Orleans Sewage Treatment Plant and Typical Values

Parameter	Permit Limit	Influent Range	Effluent Range
Fecal Coliforms, per 100 mL	200	2.1 - 7.1 ($\times 10^6$)	0.1 - 25.8 ($\times 10^5$)
Biological Oxygen Demand (BOD_5), mg/L	30	46.4 - 108 [1]	13.2 - 58.4 [1]
Total Suspended Solids (TSS), mg/L	30	7 - 26	363 - 6,543
Residual Chlorine, mg/L	0.5	0.025 – 0.05	0.19 – 1.03
pH	6 - 9	6.9 – 7.4	7.6 – 9.1

The coliforms could survive only limited time (6 hours is the time limit in the USGS field Manual) so a new sample was retrieved each day tests were conducted. A Proven Pony pump was used to remove the sample from the overflow trough from and discharge the sample to a plastic 5-gallon bucket. The pump had a 6-vane flexible rubber impeller mounted on an eccentric axis with the inlet opening located on the impinged side of the eccentric located axis. As the impeller rotated to the wider portion of the bowl, the rubber would unbend and fling the water to the discharge port of the bowl supplying the suction pressure to prime and force the water up the line (up to 10-feet of water head pressure). The sample was brought to the Center for Energy Resource Management (CERM) laboratory building on the University of New Orleans campus to conduct the tests and analyses.

3.1.2 Electrodisinfection Cell (EC)

The reactor was made up of 2 lengths of schedule 40 aluminum pipes mounted concentrically on a PVC closet flange base. Each pipe had a 2.5-cm × 4 cm tab fabricated from 3-inch i.d. pipe cutoff welded near the top. These pieces were threaded (3/8-inch × 18 thread) for attachment to the power supply using stainless steel bolts of the same size and thread spacing.

The aluminum pipes are connected to an Aldonex Model P-120 rectifier power supply that transforms the utility supplied alternating current (AC) into direct current (DC). The 1 KVA rectifier provided a controllable current (0-40A) with a variable output voltage (0-20V). The test amperage (and the resultant voltage) was adjusted by turning a knob that was attached to a rheostat. Voltage was not controlled and varied as required by the system power consumption to maintain the set current. The power supply had internal volt and amp meters, but amperage was

[1] Chemical Oxygen Demand (COD) values cited because BOD_5 was not analyzed

measured using an Amprobe ACDC-400 multimeter to permit better resolution of current (±0.01 amp). The multimeter was clamped around the wire attached to the outer pipe, which was the cathode at the beginning of the test (before polarity reversal).

On each cell, the pipe connected to the negative rectifier terminal acts as the anode and the pipe connected to the positive terminal acts as the cathode. The unit had ATC Model 422-100-F10X automatic polarity reversal timers installed. Polarity reversal allows both electrodes to alternate service as cathode and anode, distributing electrode consumption evenly between the pipes. Polarity reversal also inhibits polarization fields in the cell that interfere with current flow to the reaction and material diffusion within the sample.

Reactor assembly

The reactor was made up of 2 lengths of seamless schedule 40 aluminum pipes mounted concentrically on a PVC closet flange base. The inner pipe measured 61-cm in length with a 6.03-cm outside diameter (o.d.) pipe. The inner pipe was separated from the 54-cm long. The outer pipe measured 54-cm in length with a 7.74-cm inside diameter (i.d.). The bottom of the inner pipe was closed with a rubber cap to prevent the sample from seeping into the pipe. A 0.86-cm wide annular ring separated the pipes and formed the reactor sample compartment. The closet flange consisted of a 3.9-cm deep cylindrical cup, 8.9-cm in diameter, in the center of a 17.9-cm diameter base. The annular spacing in the assembled reactor measured 1.72 cm.

The outer pipe did not insert completely to the bottom of the cup. Because solution below the outer pipe would not be situated between the reactor electrodes, sample in this location would not be treated at the same electrolytic conditions. This void volume from the cup wall to the plug capping the inner pipe and below the outer pipe had to be filled to assure a uniformly treated effluent sample. Aquarium silicone was chosen to seal and join the components because it was guaranteed no chemicals would leach from the silicone into the fresh or salt water.

The pipes were cleaned by using a methanol wash to remove paints, machining oils and other organic residues, followed by a soap and water scrub to dislodge dusts and other foreign solids. The inner pipe cap was wrapped with several layers of Teflon™ tape to prevent silicone from adhering to allow the inner pipe to be removed for cleaning between tests. Four spacers were cut from a flexible (rubber) 3-inch x 2-inch reducer coupling so they were the proper width and curvature to fit snugly between the inner pipe bottom plug and the wall of the closet flange. Two additional spacers were cut from the 2-inch diameter end to insert between the pipes at the top of the outer pipe to maintain the annular spacing during operation.

The four rubber spacers centered the rubber plug for the bottom of the 2-inch inside diameter (inner) pipe in the closet flange cup. Silicone was injected into the annular region between the inner pipe bottom cap and the base cup wall to the height above the base where the bottom of the 3-inch pipe stopped when inserted (2.3 cm). The spacers were removed one at a time as the annular region was filled with silicone. The Teflon-wrapped cap was rotated at 10-minute intervals for the first 2 hours to keep it from attaching to the silicone, while retaining the silicone annulus formed around the center of the cup.

After the silicone had cured (48 hours), the cap was removed and the Teflon tape was brushed from the silicone and cap. The inner pipe bottom cap was cut to the same height as the silicone annulus in the cup of the closet flange and glued to the bottom of the inner pipe. Another layer of cement was applied to the top of the annular cement base and to the bottom of the 3-inch pipe before inserting it into the closet flange cup. Silicone was injected around the circumferential seam where the 3-inch pipe exited the top of the closet flange cup. The pipe was aligned to a vertical position using a level placed on the top of the 3-inch pipe. Figure 6 depicts the reactor assembly.

Effluent analyses required a total of about 850 mL of sample. After placing 850 mL of water in the reactor, the length of the pipe walls from the bottom edge to the surface were measured. The electrode surface area (wetted pipe wall) corresponding to the 42.3 cm height with an 850 mL sample volume was:

Electrode Area $= \pi \times \text{height} \times (\text{diameter}_{\text{inner}} + \text{diameter}_{\text{outer}}) = \pi \times \text{height} \times (6.025+7.74) =$ **$\underline{1830\ \text{cm}^2}$**

The annular volume $= \text{height} \times (\text{diameter}^2_{\text{outer}} - \text{diameter}^2_{\text{inner}})/4 = \pi \times \text{height}\ (6.025^2 + 7.74^2)/4$

For 850 mL the calculated height required was 45.85 cm. At 42.3 cm length, the annular volume calculates to 784 cm^3. This means there was some settling of the silicone annular ring leaving a 66 mL sample volume not uniformly between the electrodes.

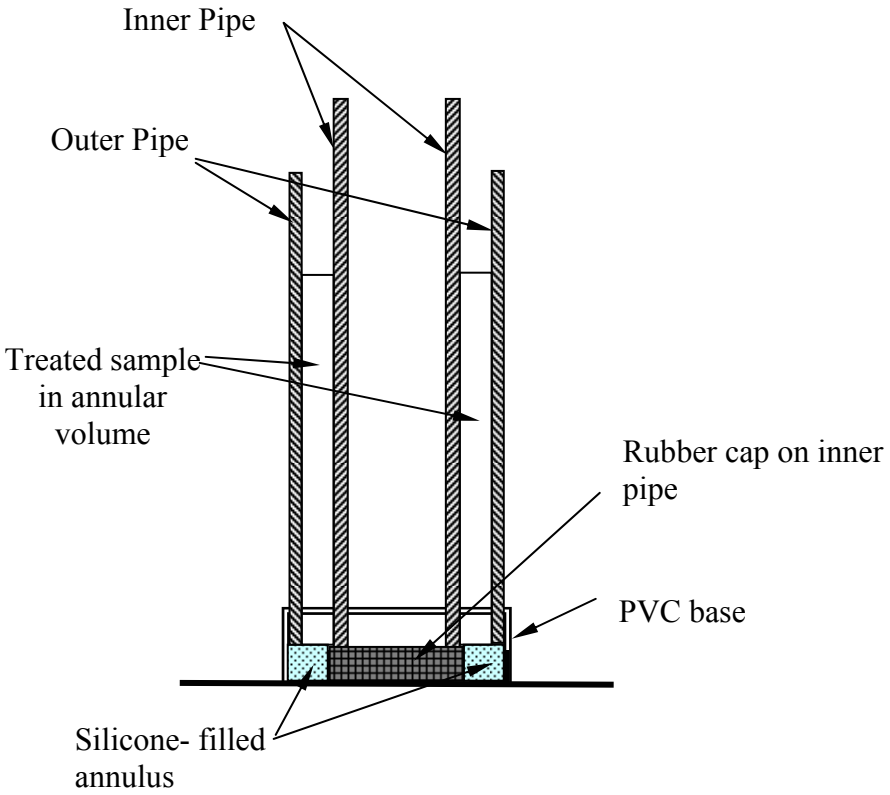

Figure 6. Electrodisinfection reactor schematic diagram.

3.1.3 Sterilization

All sample bottles, dilution water, and transfer glassware were sterilized using a Ritter M9 Ultraclave according to manufacturer recommendations and sterilization and cleaning procedures for dealing with coliform determinations recommended by the U.S. National Water Quality Laboratory. (US Geological Survey Field Manual, Ch. 7, Equipment and Equipment Sterilization Procedures TWRI 7/17/97). Lab ware was washed with soap, rinsed 3 times with tap water, followed by 3 deionized water rinses before autoclaving at 132°C and 27 pounds per square inch (psi) steam pressure for 30 minutes. Deionized water used for dilutions was sterilized at 121°C and 15 psi pressure for 30 minutes for 500 mL.

3.1.4 Dechlorination

Sodium thiosulfate ($Na_2S_2O_3$) was added to dechlorinate the effluent samples used for coliform analysis. Sodium thiosulfate reduces chlorine in solution to prevent destruction of bacteria from residual levels produced by electrochemical anodic oxidation of chloride in the reaction cell. One source recommended a 25% excess addition:

$Na_2S_2O_3 + Cl_2 + H_2O \rightarrow Na_2S0_4 + S + 2HCl$
158 g/mole $Na_2S_2O_3$ + 25% excess
70.9 g/mole $Cl_2$158 × 1.25 / 70.9 = 2.79

Standard Methods for Water Analyses [Greenberg et al., 1999] recommends a ratio of 2.85 : 1 for neutralization of halogens. A sample of effluent acquired using 1.5 mA/cm^2 current density analyzed 0.4 mg Cl_2/L. At the maximum current density of 5.5 mA/cm^2 the theoretical sodium thiosulfate required would be:

0.4 mg /L × 5.5/1.5× 1L/1000 mL × 200 mL × 2.85 = 4.18 mg $Na_2S_2O_3$ required. This would be sufficient for a residual chlorine concentration of 1.47 mg/L = 0.4 mg/L × 5.5/1.5.

To permit a 1 mL addition to supply sufficient $Na_2S_2O_3$:
4.2 mg/mL × 1000 mL stock solution × 1g/1000mg ×= 4.18 g/L

To assure sufficient neutralization capacity in case chlorine production was greater than a linear increase and to compensate for moisture or activity loss from the unanalyzed reagent used, a 1% $Na_2S_2O_3$ solution was used. This solution would be sufficient for up to 17.5 mg/L Cl_2.
(1% = 10g/L = 10 mg/mL) 10 mg / (2.85 × 0.2 L) = 17.5 mg/L

The solution was prepared by adding 5 g of the solid to 500 mL of sterilized water in a sterilized dark-colored glass bottle to minimize degradation from light.

The Petrifilm instructions advised that thiosulfate solutions could affect results. To check effects from using the dechlorination solution on coliform destruction measurements, two sets of duplicate plates were inoculated from a feed sample at three different dilutions with and without addition of the sodium thiosulfate addition. Count results from this test are summarized in Table 6. Samples diluted at 10:1 were all too numerous to count (TNTC).

Table 6 Comparison of total coliform counts with and without sodium thiosulfate addition.

	Coliforms (mL^{-1})	Dilution Factor	Sample Averages and Standard Deviations				
			Diluted Coliforms		Sample Coliforms		Sample Range
no	65	500		std. dev.		std. dev.	colonies/mL
$Na_2S_2O_3$	66	500	65.5	±0.5	32,750	±250	32,500-33,000
with	64	500					
$Na_2S_2O_3$	75	500	69.5	±5.5	34,750	±2,750	32,000-37,500
no	151	100					
$Na_2S_2O_3$	174	100	162.5	±11.5	16,250	±1,150	15,100-17,400
with	174	100					
$Na_2S_2O_3$	145	100	159.5	±14.5	15,950	±1,450	14,500-17,400

Sodium thiosulfate addition did not affect the coliforms counted, but may have increased the standard deviation of the samples. The 1 mL volume of the solution added was not factored into the calculation, making the resulting ranges even closer. Typical variations of coliform counts were greater than the observed variation from sodium thiosulfate addition.

3.1.5 Chemical Analyses

The COD is the measure of the oxygen required by the organic (carbon-, nitrogen-, phosphorus- and sulfur-based) materials and metals in the sample to stabilize them in their oxide forms. COD measurements used a Hach DR/2010 direct reading spectrophotometer and were conducted with triplicate specimens. Except for Test 1, supernatant samples were used for COD analyses to distinguish the COD of dissolved species.

Aluminum (Al^{+3}), Chlorine (Cl_2^0) and Chloride (Cl^{-1}) were measured using a Hach Model DR/2010 Direct Reading Spectrophotometer and the specified protocols from the Hach Method Manual. Aluminum analyses used Method 8012 (Aluminon method), chlorine was analyzed by Hach Method 8167 (equivalent to Standard Method 4500-Cl G for drinking water), and chloride was measured by Method 8113 (Mercuric Thiocyanate Method). Appendix 1 contains the detailed procedures along with the required dilutions and accuracy for these analyses.

Conductivity of the samples and effluents was measured using an Orion model 420 pH meter was used to measure pH (±0.01 pH unit) and redox potential (± 1 mV) with a model 900011 platinum combination electrode. Sample conductivity was measured (±1 mg/L) with a Jenway model 4150 conductivity meter. Dissolved oxygen was measured (±0.01 mg/L) using an Oakton DO 100 meter with internal temperature correction. Total dissolved solids in the samples were calculated based on the conductivity value. Spot checks found the actual dissolved solids at 80-90% of the calculated value. Total dissolved samples used approximately 25 grams of liquor weighed before and after drying at 105°C to ±0.1 milligrams (mg).

To convert redox reading using the platinum electrode to potentials compared to the standard hydrogen electrode, the values had to be converted to compensate for the Platinum electrochemical reactions. According to the manufacturer, the conversion for our 900011 electrode follows:

$E_{SHE} = E^0 + Correction$
$E_{SHE} =$ Observed Electrode Reading (in mV) – (Temperature in °C) + 224mV

3.1.6 Total Suspended Solids (TSS)
The TSS test was performed to quantify the amount of solid matter suspended in the samples. The samples were filtered through a Hach No. 30 qualitative filter paper, with a pore size of 0.45 micron, using a 1/4–horsepower GAST vacuum pump (model 0523-V4) attached to a 2-liter filter flask with a Millipore® glass filter holder assembly with a stainless steel mesh filter support. All filtration used Hach No. 30 qualitative filter paper, with a pore size of 0.45 micron pore size. Filtered samples were dried at 105°C and cooled in a dessicator for 1 hour before weighing on an Ohaus GA 200 electronic scale with 0.1 mg sensitivity. Feed samples used 50-100 mL samples, but effluent samples used 25-50 mL due to the poor filtration characteristics of the alum gel for the high CD and long time tests. Filtered samples were washed with deionized water 3 times to remove residual dissolved solids.

3.1.7 Total Coliform
Coliforms were counted from samples diluted inoculated and incubated on 3M Petrifilm™ count plates. The Petrifilm plates consisted of 2 thin sheets measuring about 4-inches by 4-inches. The bottom sheet was marked in a grid and had a 3-inch diameter circle of water-soluble gelling agar which contained nutrient, a red indicator dye for the coliforms, and a blue/violet indicator (Violet Red Bile) for fecal coliforms. The top sheet functioned as a cover, joined to the bottom sheet at one end. Samples were inoculated with a 1 mL of the diluted sample using a 1.0 mL TD (total delivery) pipette, covered to spread the sample, and incubated 48 hours at 35°C before counting. Each test effluent and feed sample was analyzed in triplicate when conducting coliform counts to assure accuracy and as a precaution against accidental contamination of a plate. Fecal coliforms could be distinguished by their blue color in contrast to the red colored common coliforms. Total coliforms were the sum of the blue fecal (*E. Coli*) and red common coliforms.

Feed samples were diluted 1000:1 (except the first three tests at 500:1) and effluent samples diluted to supply an appropriate level (30-80 colonies per plate) for accurate counting. Effluent dilution samples varied from 200 to 500 to 1 depending on current and time employed in the test and guessing the coliform destruction result. Some counts were run at multiple dilutions due to variations in the feed and uncertainty on process coliform destruction efficiency. Appendix Table A1 summarizes the dilution requirements and resultant variation produced on accuracy. Table 7 summarizes the analyses, equipment and precision obtained in this study.

3.2 Test Procedures
3.2.1 Electrodisinfection Tests
After mixing the sample by pouring between buckets 2-3 times and stirring, 850 mL of sample was measured by graduate cylinder and transferred to the reactor annulus ring. Initial temperature was measured in the electrocoagulation cell before and after the test. During the test, the current had to be adjusted to maintain the set point, usually requiring an increase in the rectifier output with a corresponding increase in the voltage required to maintain the amperage setting. Each polarization change took about 10-15 seconds to restabilize the settings and repeat the cycle of adjustments. Each test was repeated using the same conditions 3 times to assure accuracy and account for process variation. Average values for the 3 test responses are reported in this thesis unless otherwise stated.

Table 7. Test parameters measured in electrodisinfection tests

Parameter	Method and Precision
Current	Amprobe ACDC 400 ±0.01 amp
Time	Electronic timer ± 1 sec.
Dissolved chloride (Cl^-)	Hach 2010 DR. ±0.1 mg/L
Chemical oxygen demand (COD)	Hach 2010 DR. ±0.1 mg/L
Dissolved aluminum (Al^{+3})	Hach 2010 DR. ±0.01 mg/L
Total chlorine	Hach 2010 DR. ±0.01 mg/L
Dissolved oxygen	Oakton DO-100 ± 0.01 mg/L
Total suspended solids (TSS)	Gravimetric ±0.1 mg
Total dissolved solids (TDS)	Jenway 4150 ±10 mg/L
Conductivity	Jenway 4150 ± 1 µS/cm
Volume (>100 mL)	Graduate Cylinder ± 5mL
Volume (<100 mL)	Graduate Cylinder ± 0.5mL
Coliforms	Petrifilm ± 1 colony
Temperature	Mercury thermometer, ±0.5°C
Redox (Reduction/oxidation) potential	Orion ±0.01 amp, 900011 combination electrode
pH	Orion ±0.01 pH unit, 900011 combination electrode

Test effluent was transferred to sample jars with screw caps and sealed. The coliform count sample jar had previously been inoculated with 1 mL of 1% sodium thiosulfate solution to remove residual chlorine and stop further coliform destruction (Appendix T). Free chlorine content was measured immediately. Conductivity, TDS, dissolved oxygen (DO), pH, temperature, Chloride (Cl^-), total chlorine (Cl_2), total aluminum, and COD measurements were by Maria E. Pulido. Triplicate readings were taken for each parameter measured, and the average values are reported in this Thesis unless otherwise stated. Appendix Table A1 summarizes the dilution requirements and resultant variation produced on accuracy.

3.2.2 Settling Tests

250 mL of effluent sample from each test was placed in a 250-mL graduate cylinder, inverted slowly 3 times to mix, and allowed to settle into a clear supernatant and concentrate the solids into a settled sludge. The supernatant/settling solids interface volume was recorded with the settling time. The volume calibration of the graduate cylinder was translated into mL/cm to calculate settling velocity at each or the data points. When settling stopped the sample supernatant was decanted and the sludge was dried at 105°C, and then cooled in a dessicator before weighing.

The settling data obtained was converted from interface height versus time to settling velocity versus time and then plotted settling velocity as a function of the settled solids concentration for each data point to determine rise rate for sizing requirements for thickening of the effluents as reported by Wilhelm and Naide [Wilhelm and Naide, 1979]. Solids concentration was calculated from the total solids in the test and the volume settled below the supernatant interface.

3.2.3 Response Analyses

Unless otherwise stated, reported analyses are the averages of triplicate analyses, and test responses are the averages of the test runs using averages of the triplicate analyses. These averaged values were analyzed using a statistical software program named DesignEase® 6.0.10 by StatEase, Inc. to measure the effects and interactions of the variables. The averaged triplicate from all 3 test replicates are the values reported in the Discussion and Conclusions sections.

4. DISCUSSION OF RESULTS

A 2-level full factorial test series examined the electrodisinfection variables of current density, detention time, and chloride concentration. 2 replicate tests were conducted at midrange of the variable range to check experimental variation. The experimental test design space is illustrated in Figure 7. Each set of test conditions were conducted in triplicate to compensate for expected variability in performance and analyses. In addition, 2 other tests were repeated to check for abnormality and reproducibility: test 1 (conducted with the unused aluminum electrodes) and test 2 (due to difficulty in solids settling data) were repeated in test 10 and 12. Analyses were conducted in triplicate to reduce variations. Experiment variables and their set points, and test responses and their measured values are summarized in Table 8. A detailed summary of the measured test parameters and responses are tabulated in the appendix.

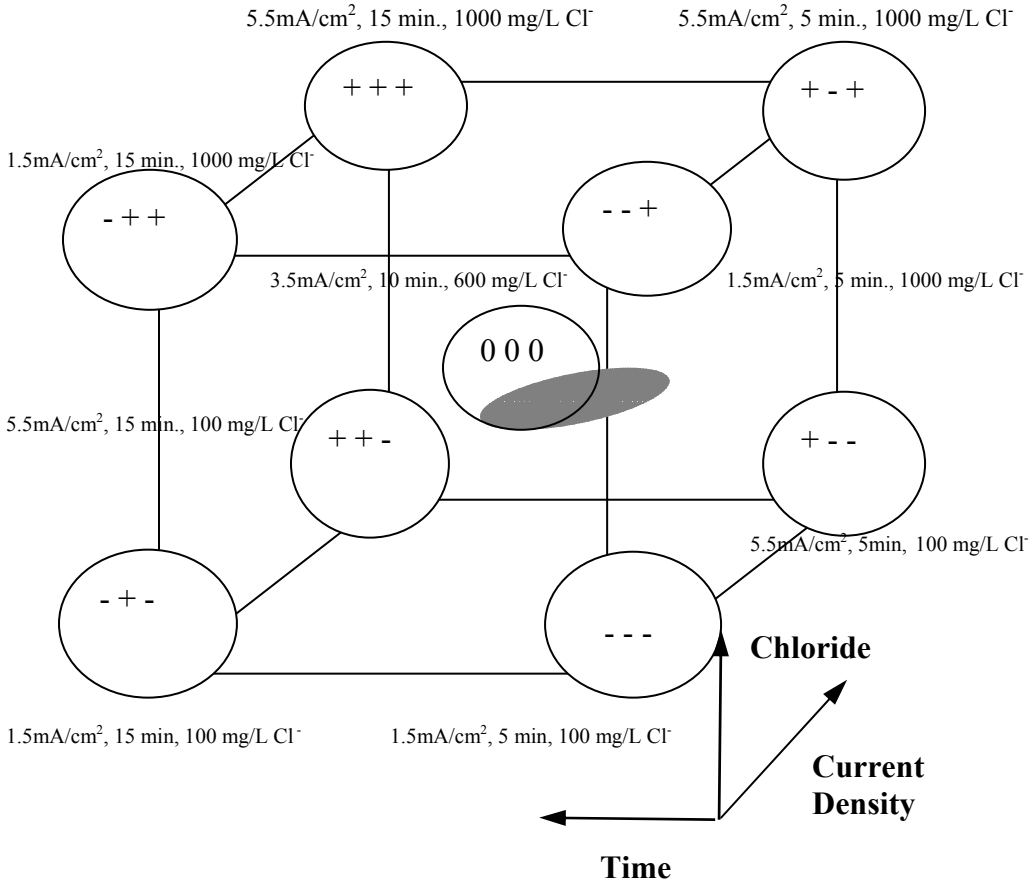

Figure 7. Factorial Experiments Design Space Illustrating Variables and Values

Table 8. Test Variables and Response Summary

Test	Time	Current Density	Chloride Conc.	Coliform Removal	Energy Input	Effluent TSS	% COD Removal
Units	minutes	mA/cm^2	mg/L	$Log[1-N/N_0]$	$kW\text{-}h/m^3$	mg/L	mg/L
1	5	5.5	139	0.61	13.81	1676	3.5[2]
2	15	5.5	137	2.18	41.42	6543	61.4
3	10	3.5	583	0.97	5.02	2919	60.4
4	5	1.5	136	0.31	1.07	363	67.4
5	15	1.5	129	0.62	3.63	1110	78.5
6	5	5.5	868	0.90	3.75	2287	62.2
7	5	1.5	1005	0.69	0.54	484	59.3
8	15	1.5	1010	0.68	1.61	1409	65.6
9	15	5.5	1075	2.14	10.93	5915	65.9
10 (1r)	5	5.5	123	0.85	13.81	1974	49.5
11 (3r)	10	3.5	580	0.76	4.77	2531	75.4
12 (2r)	15	5.5	162	1.79	38.46	5723	76.4

4.1 Variables Tested

Current density was tested at 1.5 and 5.5 milliamps per square centimeter (mA/cm^2), with times of 5 and 15 minutes, using as-received chloride (varying from 129 to162 milligrams per liter chloride (mg/L Cl^-) and with chloride added to about 1000 mg/L Cl^- using reagent grade sodium chloride. Duplicate tests performed at the experimental design space center point used 3.5 mA/cm^2 for 10 minutes at 580 mg/Cl^- conditions.

4.2 Responses Measured

Total coliform, COD, TSS, pH, Cl_2, aluminum concentrations, and redox potential measurements of the feed and effluent samples monitor the process effects on the sample. Applied amperage and corresponding voltage and time were used to evaluate process responses based on energy input to the system. Settling rates of the solids were also recorded in relation to process influences.

4.3 Voltage Variations

Typical variation in cell potential during the course of the tests required a rapid adjustment to the set point amperage by adjusting the rheostat to the target current. A slow increase in voltage (and power output) was then required until the scheduled polarization reversal (about every 2 minutes). After polarization reversal, the amperage would reach set point again after 10-20 seconds, and in some tests exceed the set point using the same dial setting, operating with a slight increase in current efficiency for the power level supplied. The trend would reverse after

[2] Test 1 measured COD from the effluent slurry instead of supernatant only as recorded in the other tests.

30-50 total seconds into the cycle and again require small adjustments increasing the power to maintain the set point current.

After each polarization reversal at 1.5 and 3.5 mA/cm^2 current densities, the amount of power adjustments required to maintain set point decreased. At 5.5 mA/cm^2 this conditioning was not observed, and the behavior became more erratic with time in the low chloride tests. The tests using chloride addition moderated this effect, but still exhibited a more erratic behavior than the low current densities.

The increased voltage (and resultant power consumption) could probably be attributed to the buildup of a resistant corrosion film on the anode. On polarization reversal, the reaction reverses briefly for partially dissolved material or aluminum ions still in contact with the cathode. Overall the process quickly returned to the buildup and loss of oxidized material. The high current density tests were more unstable in operation performance, again likely due to the greater corrosion levels and rate of oxide material buildup on the anode.

4.4 Coliform Destruction

Analysis of coliform destruction by the statistical software identified 2 variables strongly affecting the response: current density (CD) and time. This agrees well with disinfection theory and previous investigations. Additionally, an interaction of CD and time added to the coliform destruction effect. Figure 8 illustrates how these variables were selected. Plotting the responses of each experimental datapoint on log-probability paper produced graph CDFX. Nonlinearity indicates a point which does not have an equal probability of effect on the response for normal data. Points A, B and AB correspond to the CD, time, and CD-time interaction data points. Analysis of variance (ANOVA) using this model gives an F-statistic of 40, with a probability of less than 0.0001 that this effect could occur randomly and the relation is insignificant.

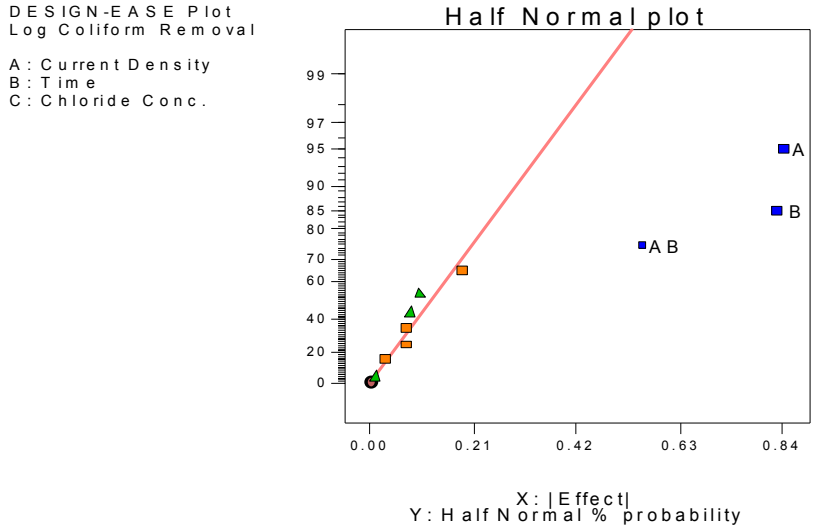

Figure 8. Log Coliform Destruction Effects Selection Plot

The model equation calculated by the program is:

Log Coliform Removal =
Log (1-[N/N_0]); for N= effluent coliforms/mL, N_0 = feed coliforms/mL
= 0.524 - 0.066 ×Current Density - 0.026 ×Time + 0.0275 ×[Current Density ×Time]

This equation can be simplified to = 0.0275×[Current Density ×Time] with only minor errors added underpredicting at the low end and overpredicting at the top end of the range tested.

Figure 9 illustrates the nonlinearity in response caused by the CD-time interaction. Coliform destruction plotted as a function of time for the 2 current density levels show a steeper response slope at the high CD level. The midpoints appear near the middle of the plot.

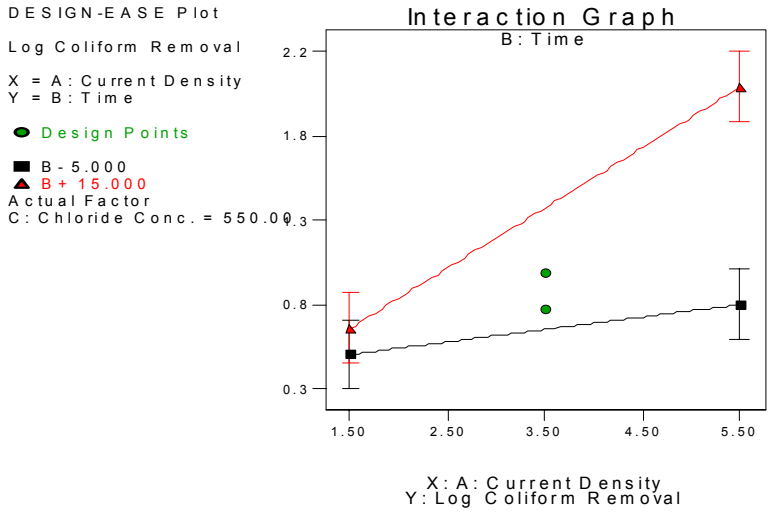

Figure 9. Interaction Plot of Current Density and Time on Coliform Destruction

4.4.1 Total and Fecal Coliform Destruction

Coliform content from the treatment plant samples used as feed varied greatly depending on recent rainfall (it took 3 days for dilution from infiltration to dissipate), temperature, and proximity to Mardi Gras. Total coliforms varied from 1.7 to 7.1 (×10^4) colonies per mL without rain dilutions, peaking the 2nd week after Mardi Gras. Feed samples were diluted 1000:1 (except the first three tests at 500:1) to supply an appropriate level (30-80 colonies per plate) for accurate counting. Because coliform destruction was not known until after inoculation and counting, some variation in accuracy is present. Over dilution of the inoculated samples reduced accuracy, but under dilution resulted in overloaded plates with colony impingement, reduced size, nutrient shortage and color change from the indicator resulting in a sample TNTC.

Total coliforms and fecal coliforms were counted in all samples. Fecal (*E.coli*) coliforms would become blue colored and generally numbered about 1/10th of the total coliforms. Destruction of fecal and total coliforms occurred in the same ratio, so only total coliforms are reported here because the greater quantity and accuracy.

Figure 10 presents the graphic relation between coliform destruction (1-(N/N_0)) as a function of the energy input to the reactor and chloride content. Due to the increased conductivity with higher chloride (dissolved solids) content, energy consumption was notably lower than with the low chloride system although the test current densities and time varied over the same lengths for both sets of data. Applying a log function to the data sets yielded fairly good correlation coefficients (0.98 and 0.95 for the high and low chloride series, respectively), with an expected higher scatter for the low chloride. The model also under-predicts coliform removal at high CD and time as expected according to the statistical evaluation because the model does not account for a time-CD interaction.

Coliform destruction = 1-(N/N_0) =0.086 ×Ln(Energy) + 0.423 with chloride addition
= 0.078 ×Ln(Energy) + 0.329 without chloride addition.

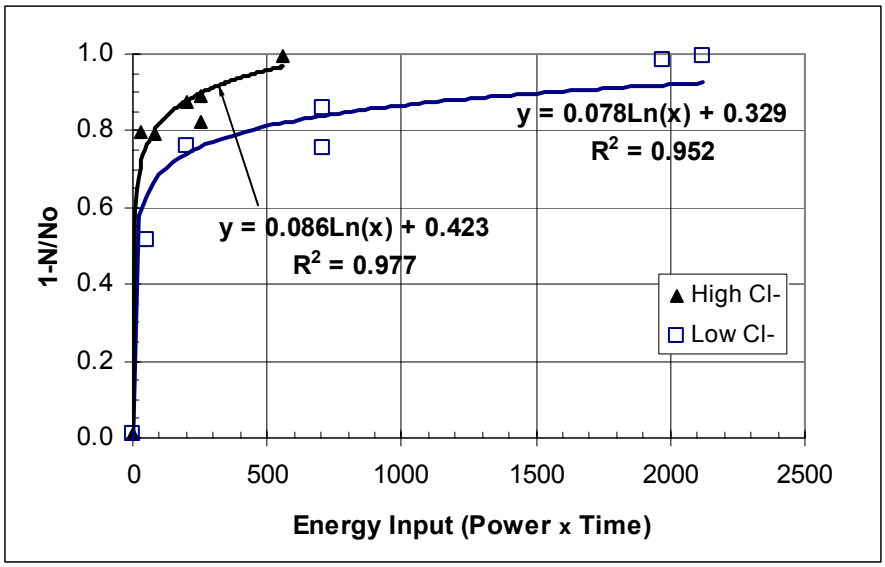

Figure 10. Coliform Destruction Plotted as Functions of Energy Input and Chloride Level

4.5 Solids Production

Precipitated aluminum from the electrodissolution of the electrodes to neutralize and aid settling dispersed solids is desirable in many water and wastewater treatment applications. In this application the level of energy input to the system to reduce the coliform population produced excessive levels of solids.

The net half-cell reactions expected for solids production expected based on thermodynamic favorability are presented below. The cathode reaction equation is expected for neutral solutions with oxygen available according to corrosion engineering [Fontana and Greene].

Cathode: $3O_2 + 6\ H_2O + 12e^- \rightarrow 12\ OH^-$	$3 \times 0.401V = +1.20$ V
Anode: $4Al \rightarrow 4Al^{3+} + 12e^-$	$4 \times 1.66V = +6.64$ V
$4Al + 6\ H_2O + 3\ O_2 \rightarrow 4\ Al^{+3} + 12\ OH^-$	$E^0 = 7.84$ V = +1.96V/mole Al

followed by: $4\ Al^{+3} + 12\ OH \rightarrow 4\ Al(OH)_3\downarrow$

Statistical evaluation of the data identified the variables time and current density as responsible for solids production. Solids production will parallel coliform destruction and vary with the time and current density (and energy input). Figure 11 portrays the effects selection plot from the StatEase software.

Again, if all of the test points were equally involved or not involved in the response, they would fall in to a normal (bell-shaped) distribution based on allowed measures of deviation from the average when divided among all of the members of the population, and form a line on the probability plot. Current density and time strongly affected the solids production. The ANOVA for the model generates an F-statistic of 250, extremely definitive that the model is a result of the variable effects. The probability of the effect happening due to random chance is less than 0.00001. The model equation calculated by the software is:

Ln (Suspended Solids, mg/L) = 4.89+ 0.39 × Current Density + 0.11 × Time

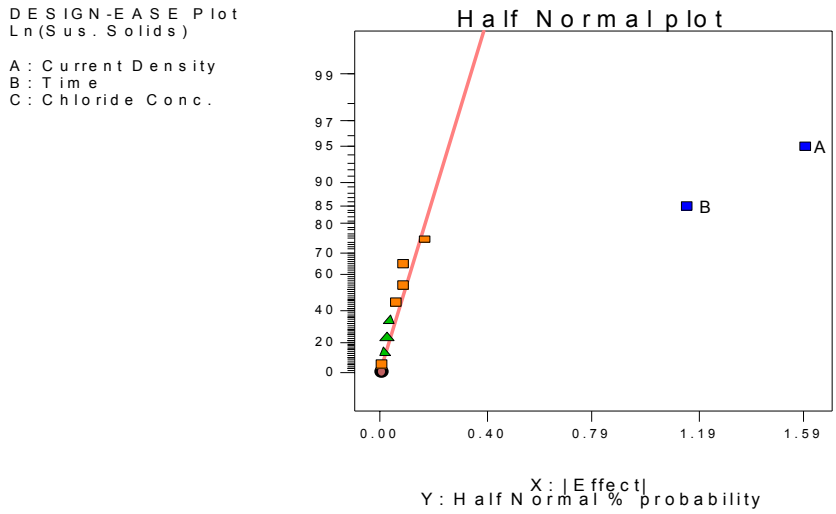

Figure 11 Solids Production Effects Selection Plot from Statistical Analysis

Analyzing solids production based on energy input (current×voltage×time per unit volume) allows solids production to be predicted. Conductivity again strongly affects cell resistance (voltage required at a given current density) requiring the data to be broken down by chloride concentration level. Solids production is plotted as functions of chloride concentration and energy input (in watt-minutes) in Figure 12. Linear functions can describe the suspended solids production as follows:

TSS (mg/L) = 10.742 × Power (watts) × Time (minutes); with high chloride
= 2.97 × Power (watts) × Time (minutes); with low chloride

Energy requirements to dissolve a given weight of aluminum decreased by afactor or 3.6 with increased chloride and conductivity in the reactor.

When the overall operating time and current is considered, discounting the difference due to cell voltage and resistance, there is no difference in effluent suspended solids per amp-second for the high chloride or low chloride tests. Figure 13 displays the plot of suspended solids as a function of charge consumption (current×time) at high and low chloride concentrations. No significant difference between current applied and electrode dissolution can be noted in this study.

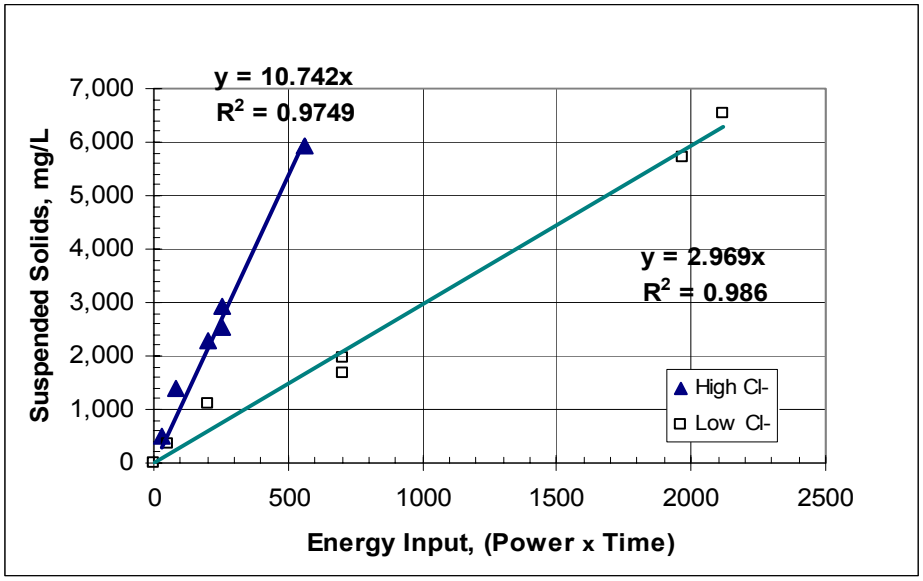

Figure 12. Solids Production as Functions of Chloride Concentration and Energy Input

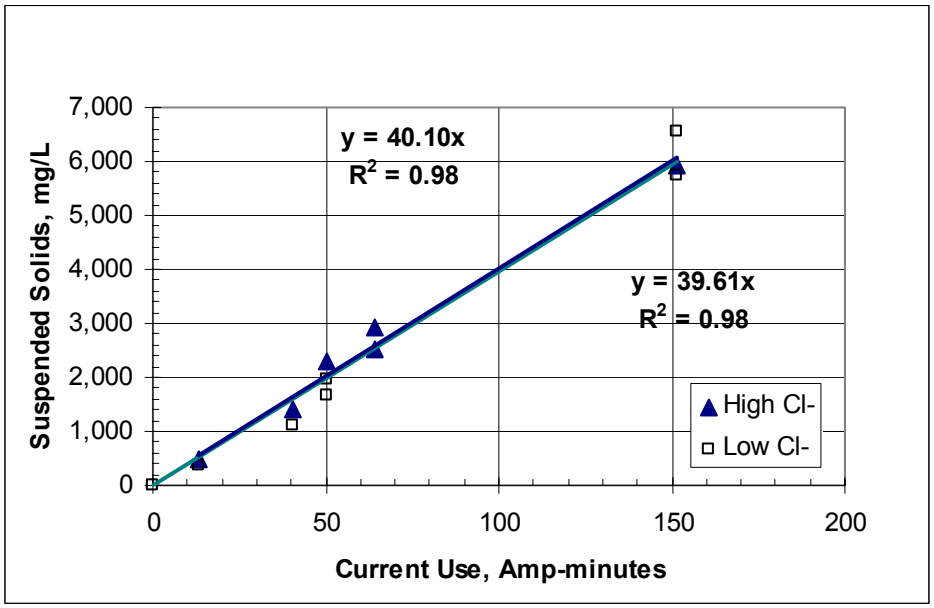

Figure 13. Suspended Solids as Functions of Chloride Level and Current Consumption

4.6 Solids Settling

Test effluents settled into a clear supernatant coagulating the amber color and solids from the feed into a white voluminous sludge from the aluminum anode. No solids were measured in the supernatants and no color was observed. Settling rates for the final effluent solids ranged widely, depending on the solids concentration produced by the varying current density and detention time, from 0.1 meter/day (m/d) at 15 minutes DT and 5.5 mA/cm^2 CD to 50 m/d or more at 5 minutes DT and 1.5 mA/cm^2. Figure 14 portrays the settling rate data from tests using high current densities, sorted according to test chloride level.

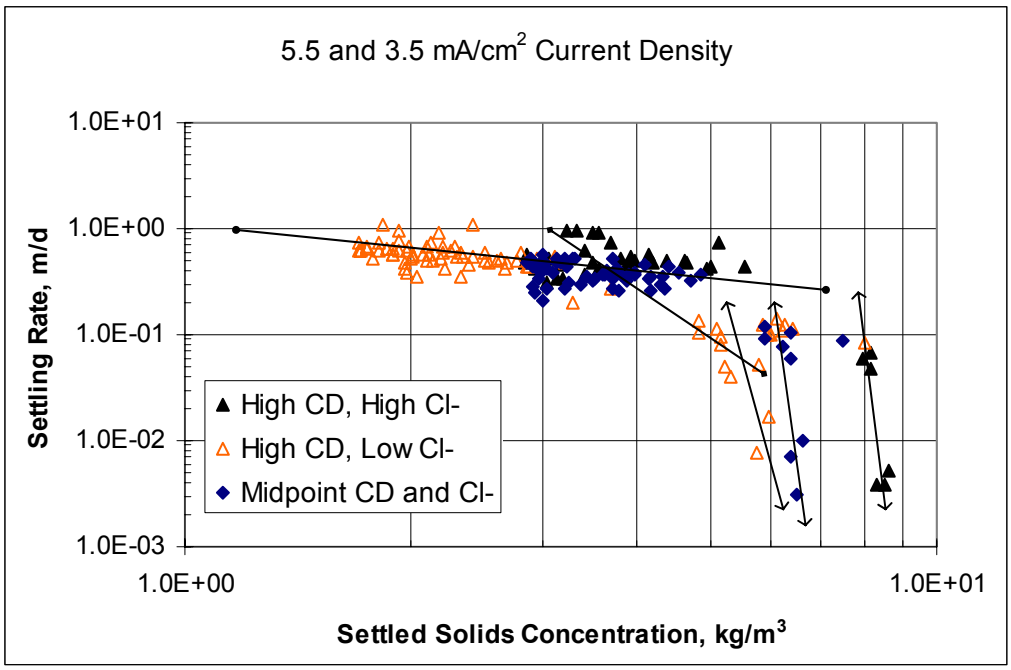

Figure 14. Solids Settling Rates as a Function of Settled Solids Concentrations for 3.5 and 5.5 mA./cm^2 Current Density Tests

Settling solids generally progress through 3 zones of settling:
1) Free Settling
2) Hindered Settling
3) Compaction

The initial free settling occurs with unencumbered solids slipping through the liquid at a constant velocity. Hindered settling is when the solids are in proximity to other solids particles and space is becoming limited for liquid to escape. This hindered settling region exhibits a continually reducing settling velocity and is called the knee of the curve connecting the 1st and 3rd settling regimes. In compaction the particles are in contact with one another and gradually fill in the interstices but liquid removal, surface charges, and trapped liquid slow the settling rate to a near stop. These 3 settling regions can be observed in Figure 14 marked by the added construction lines. In the compaction zone the settling data exhibits different compaction velocities depending on the chloride content, with the solids compacting to greater solids concentrations with higher chloride content in the system.

Effluent solids settled slowly in cases of high current density due to the high level of solids produced. Evolution of dissolved gas and coalescing gas bubbles trapped in the gel/sol mixture also interfered with settling and floated the solids in the tests at 15 minutes DT and 5.5 mA/cm^2 CD, halting settling completely after some time.

Figure 15 depicts the settling rate data from the tests conducted at low current density. Initial settling rate data is flawed because it was difficult to discern an interface on the rapidly settling particles. Variation in particle size added to the variability of the settling rates for the particles. Settling rates for later data points reflect this error because they depend on accurate readings from the previous point to calculate settling rates and solids concentrations. The actual settling rates for the free-settling zone will be near the average of the hyperbolic curve. Using 1.5 mA/cm^2 the compaction zone settling rates depended more upon the time length of the test, with the 15 minute tests generating higher final settled solids concentrations. This could be due to the greater amount of solids weight contributing to compaction of the solids. The scale of these settling tests was too small to estimate process solids concentration reliably.

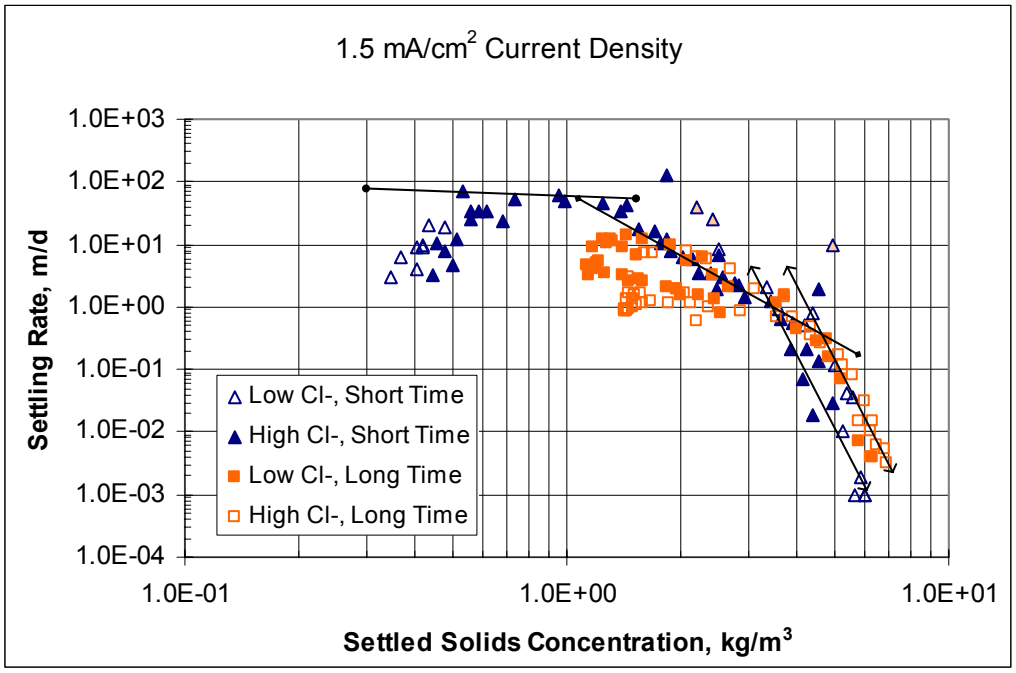

Figure 15. Solids Settling Rates as a Function of Settled Solids Concentrations for 1.5 mA./cm^2 Current Density Tests

Insufficient sample was obtained from most of the tests for positive identification of the exact aluminum species. The solids also altered with time from the high current density and long detention time tests. This was more pronounced for the 5.5mA/cm^2, 15 minute test. Solids began floating in the settling test after 2 hours. They were stirred to dislodge the gas bubbles and resettled, but re-floated overnight. The color of the precipitate gradually faded from a dark gray metallic color to a dull white color over the 3 days attempts to settle the repeatedly floating solids. Gas production had ceased after 1 day, but the gelled precipitates and dissolved species continued to form gas bubbles causing flotation. This also happened when the test was repeated

at the end of the series (test 12). Gradual oxidation of the fine-sized solids in solution from contact with the charged solution, gases trapped inside the gelled precipitate, and morphology changes as solids crystallization likely occurred. This behavior is based on observations of researchers and operators with aluminum coagulation as recorded in the Literature Review section.

The gelled solids filtered poorly, requiring small samples (25-50 mL) for solids analysis from the tests with high effluent solids content. Solids analyses of the bulk settled solids had the weight corrected by subtracting residual dissolved solids dried along with the sample to calculate solids contents. A large variation in weight was probably observed due to the adsorption of water by the samples after removal from the dessicator and possibly crystallization morphology changes as discussed by Pourbaix. Sample weight varied with time in the dessicator, order of samples measured (number of times the dessicator was opened before weighing the sample) and even from day to day, probably due to atmospheric or building temperature and humidity. A 20% variation in weight was observed from a solids sample from test 12, 11% from tests 9 and 10, and only 3% variation from test 7.

The possible alteration of the aluminum hydroxide into hydrated oxide forms, which would also explain some of the variations observed in weights, include

Amorphous aluminum hydroxide	$Al(OH)_3$	= 34.6% Al
Böhmite	$Al_2O_3 \cdot H_2O = AlO(OH)$	= 45.0% Al
Hydrargillite, Bayerite, Nordstrandite	$Al_2O_3 \cdot 3H_2O$	= 34.6% Al
Corundum	Al_2O_3	= 52.9% Al

X-ray diffraction analysis of well aged and dried solids identified only $Al(OH)_3$ from a bulk sample removed from a pilot scale cell composed of the same pipes that treated a more concentrated simulated landfill leachate. The identified solids crystal pattern closely followed that of bayerite ($Al(OH)_3$) with possible inclusion of some nordstrandite ($Al(OH)_3$) crystalline material. The peak scale matches for the bayerite and nordstrandite diffraction patterns were 0.980 and 0.706, respectively. Appendix 4 contains the X-ray diffraction report provided by Paul Schilling of the University of New Orleans Mechanical Engineering Department.

4.7 Electrode Consumption

Effluent suspended solids increased for a given set of test conditions for each consecutive run of the three replicates. This behavior was consistent throughout the test program. In long DT or low CD tests the effect was not as large, but all tests showed a marked increase in solids production in the second replicate and slightly higher or equivalent in the third. This behavior can be expected as the electrode becomes conditioned to its service, finally forming the oxide coating proportionate to the service conditions and stabilizing solids production (corrosion rate). Suspended solids from the third replicate would represent the effluent solids expected from the test conditions.

Expected aluminum consumption according to the Nernst equation would be:

$$\frac{W_{eq} \times I \times time}{\Im} = \frac{\frac{27 g / mole}{3e^-} \times Amps \times \text{sec}onds}{96{,}500\ Coulombs / mole}$$

Aluminum solids measured by weight and aluminum analysis were 115-320% of theoretical aluminum production. Previous investigations report 165-215% of theoretical due to the dissolution of the cathode and anode due to aluminum corrosion in oxidized water. Aluminum assays may be in error on the high side, and solids content is susceptible to 5-22% variation depending on test conditions. No conclusions can be drawn regarding aluminum consumption other than that it was well above the theoretical consumption rate. Figure 16 plots solids aluminum content as a function of energy input. A rough correlation can be drawn for the low chloride tests, but the high level series can not. An outlier point may occur with a 54% Al concentration that may change the trend into a steep drop in aluminum concentration with energy input as would be expected due to oxidation. It is also possible, though not likely, that it was oxidized to the point of crystallization to corundum. No conclusion can be drawn on aluminum concentration of the solids based on the data.

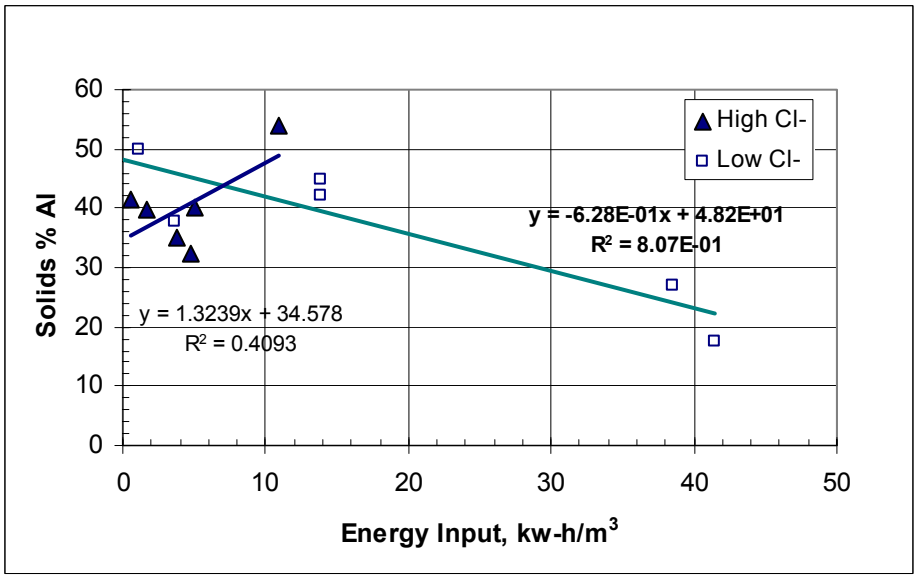

Figure 16. Suspended Solids Aluminum Content as Functions of Energy Input and Chloride Level

4.8 pH

The effluent sample pH increase ranged from 1.3 units (from 6.9 to 8.2 at 2.5 A/cm^2 with a 5 minute detention time) to 2.9 units (from 7.1 to 9.0 using 5.5 A/cm^2 for 15 minutes) in the process. Increased pH results from the cathodic reaction: $O_2 + 2\ H_2O + 4e^- \rightarrow 4\ OH^-$ Figure 17 displays the correlation observed between ph and energy input in this study.

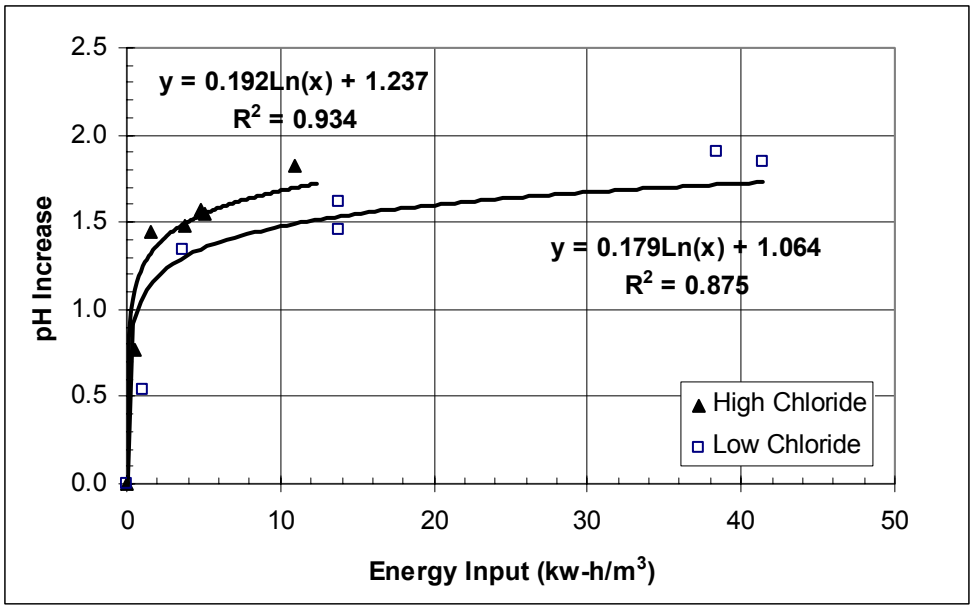

Figure 17. Effect of Energy Input on Effluent pH

4.9 COD Removal

COD removal was measured for the supernatant only after the first test. In the first test, COD of the total effluent suspension was analyzed. COD actually increased in the third replicate of the first test (72.2±3.1 mg/L) compared to the average feed content (62.0 ±4.9 mg/L). The average of the three runs averaged 58.4±12.1 mg/L for a 3.5 mg/L (5.7%) reduction in COD. Suspended solids production in the three replicates increased from 1613 to 1645 to 1739 in these first test runs. As TSS increased, the level of oxygen demand of the aluminum hydroxide and the gradual crystallization and aging it exhibits changing to a hydrolyzed oxide form increased, adding COD demand to the sample or interfering with further dissolved COD removal.

Figure 18 plots the COD reduction in mg/L for each test (except test 1) as a function of energy input at high and low chloride addition level. The correlation is poor, but illustrates the COD reduction behavior as a function of the variables used to remove COD and coliform (current and time). An element of oxygen demand could not be removed by simply increasing electrodisinfection level.

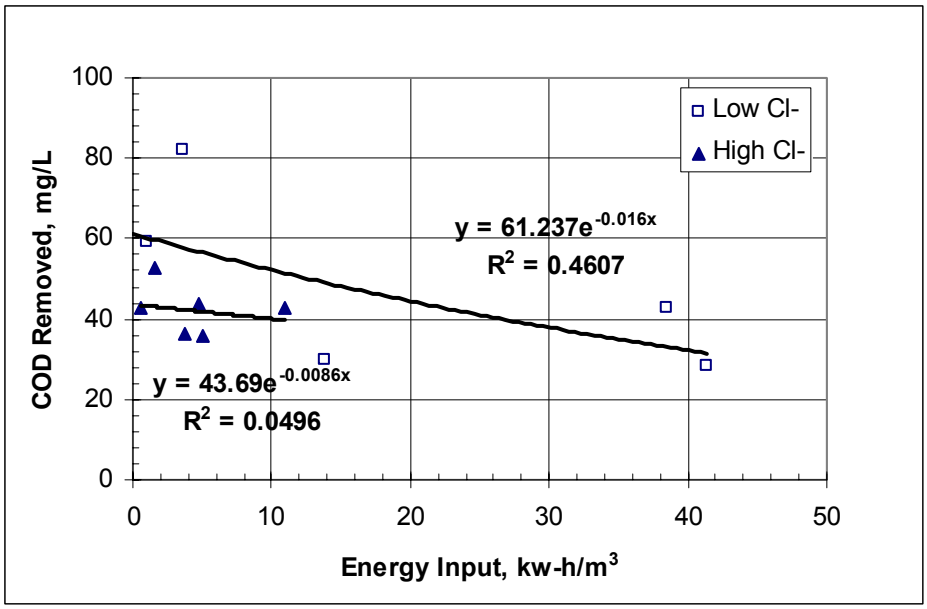

Figure 18. COD Removal as a Function of Energy Input

4.10 Chloride Addition

Chloride addition level was expected to affect operation on several levels, including synergistic effects:

1) Chloride concentration affects the levels of free chlorine and hypochlorite ions formed to cause disinfection;
2) Chloride (and the balancing positive sodium counterions) increases the conductivity of the water, increasing the current efficiency and charge transfer to the water;
3) Chloride will increase the level of aluminum dissolved from the electrodes by increasing the corrosivity of the water, and initiating the pitting corrosion cycle.

The maximum free chlorine measured and generated in this test work was 1.0 mg/L from test 9 with high CD, long time, and high chloride content. According to the CT (Concentration×Time) Tables used for disinfection, this would only account for a low level of disinfection. Most of the disinfection noted in this test work resulted from combinations of charged or free radical oxidation in combination with some chlorine-based destruction and possible direct electrical field or contact to deactivate coliform from the wastewater. Table 9 summarizes COD removal, chlorine data, and test conditions from this study.

Chloride content did not control the level of free COD removal, because tests 4 and 5 produced the highest percentage COD removal and COD concentration reductions in the tests, but contained no added chloride. Statistical analyses of the data did not reveal any of the variables significantly controlling chlorine production, including feed chloride concentration. No dependence between COD or coliform removal and chlorine production could be discerned from this study data. Chlorine production was a function of the energy (or current and time) input to

the system, with the chloride content affecting the energy due to conductivity changes. Figure 19 depicts the dependence of chlorine production on energy input to the reactor.

Table 9. COD and Chlorine Production Data Summary

Test ID	Current Density	Time	Feed Chloride	Effluent Cl_2	% COD Removal	COD Reduction	Coliform Removal
Units	mA/cm^2	min.	mg/L	mg/L	%	mg/L	$1-N/N_0$
1	5.5	5	139	0.389			0.755
2	5.5	15	137	0.942	61.4	28.4	0.993
3	3.5	10	583	0.833	60.4	35.9	0.892
4	1.5	5	136	0.343	67.4	59.3	0.514
5	1.5	15	129	0.573	78.5	82.3	0.758
6	5.5	5	868	0.470	62.2	36.3	0.875
7	1.5	5	1005	0.390	59.3	43.0	0.797
8	1.5	15	1010	0.193	65.6	52.5	0.790
9	5.5	15	1075	1.033	65.9	42.8	0.993
10	5.5	5	123	0.433	49.5	30.1	0.860
11	3.5	10	580	0.410	75.4	43.9	0.825
12	5.5	15	162	0.647	76.4	42.6	0.984

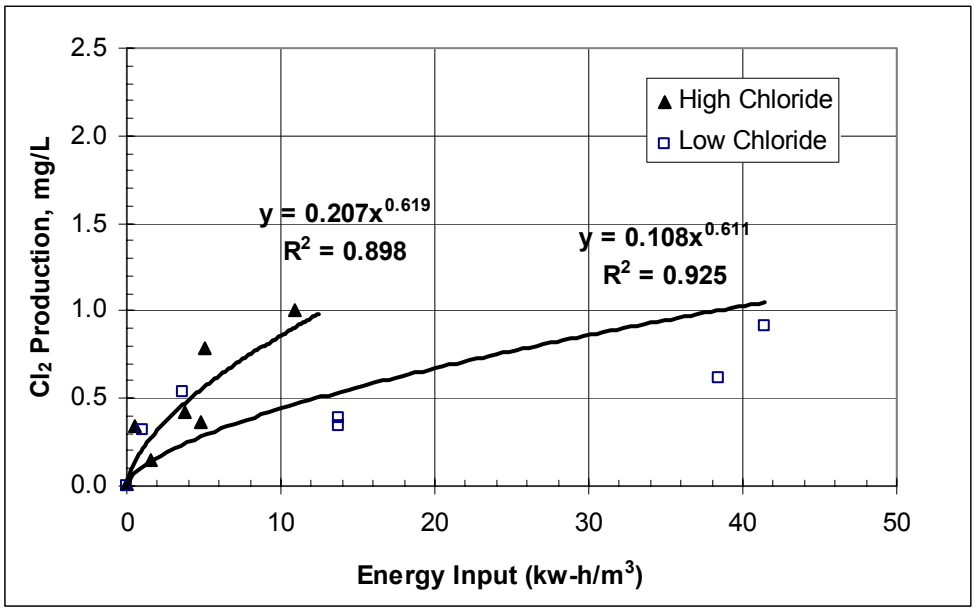

Figure 19. Chlorine Production versus the Energy Input

Although Figure 18 appears to show higher COD removal with the low chloride addition level, a regrouping of the data excluding energy addition shows chloride addition, chlorine production, and %COD removal appear unrelated in the regions examined in this work in Figure 20.

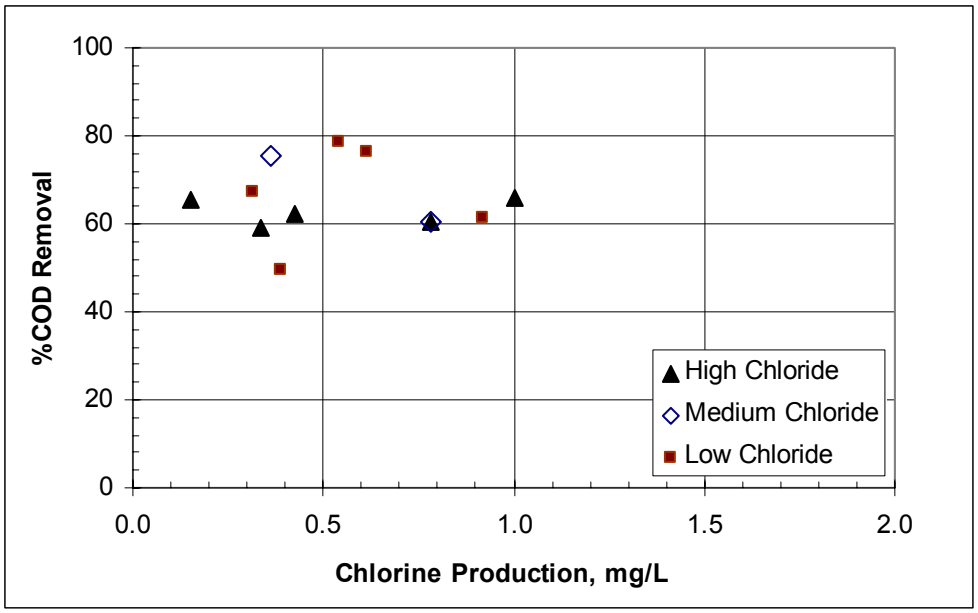

Figure 20. COD Removal as a Function of Chlorine Production and Chloride Addition Level

5. CONCLUSIONS AND RECOMMENDATIONS

Coliform removal in the aluminum electrodisinfection cell can exceed 2-log (99%) using 15 minutes time and 5.5 mA/cm^2 current density. Coliform destruction is dependent on current, time, and an interaction of current×time.

Time based dissolution of the aluminum did not notably increase by the addition of 0.5 to 1.0 g/L chloride. Increased current efficiency due to reduced voltage drops with higher conductivity water reduced energy consumption per weight of aluminum consumed by a factor of 3.6 with additions below 0.01% chloride.

Current efficiency increased and power consumption decreased by a factor of nearly 10. Even the addition of only 500 mg/L Cl^- (824 mg/L NaCl) for 0.08% addition greatly reduced power consumption.

Power and current requirements for disinfection of secondary wastewater treatment effluent generates excessive levels of aluminum dissolution and suspended solids for the level of clarification and problematic solids to be removed. Aluminum electrodes in an electrodisinfection unit are more appropriate in applications involving high levels of emulsions and fine solids dispersions which require the additional surface charge neutralization form the dissolved hydroxy-complexes and fresh aluminum solids.

Chlorine production and chloride addition did not significantly affect COD removal or coliform destruction. The mechanisms of these effects were associated with the reactor current density and detention time. Chloride concentration did not significantly affect free chlorine production.

Future work should examine stainless steel and titanium electrodes construction and determine electrodisinfection lethality on *giardia* and *cryptrosporidium*.

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APPENDICES

Appendix 1 Analytical Procedures
Table A1. Sample dilutions and resulting analytical precision

Appendix 2 USGS Sterilization Procedures

Appendix 3 Experimental Data
Test Sheets
Data Summary Table
Statistical Analyses Output

Appendix 4 X-ray Diffraction Report on Aluminum Precipitates

Appendix 1 Hach Analytical Procedures

ALUMINUM (0 to 0.80 mg/L) For water and wastewater
Method 8012 Aluminon Method*
* Adapted from Standard Methods for the Examination of Water and Wastewater.

1. Enter the stored program number for aluminum (Al). Press: 1 0 ENTER
The display will show: Dial nm to 522
Note: The Pour-Thru Cell can be used if rinsed well with deionized water between the blank and prepared sample.
2. Rotate the wavelength dial until the small display shows: 522 nm
When the correct wavelength is dialed in, the display will quickly show: **Zero Sample** then: **mg/L Al3+**
Note: Total aluminum determination needs a prior digestion; use any of the three procedures given in Digestion (Section II).
3. Fill a 50-mL graduated mixing cylinder to the 50-mL mark with sample.
Note: Rinse cylinder with 1:1 Hydrochloric Acid and deionized water before use to avoid errors due to contaminants absorbed on the glass.
Note: The sample temperature must be between 20-25 °C (68-77 °F) for accurate results.
4. Add the contents of one Ascorbic Acid Powder Pillow. Stopper. Invert several times to dissolve powder
5. Add the contents of one AluVer 3 Aluminum Reagent Powder Pillow. Stopper. Invert repeatedly for one minute to dissolve.
Note: A red-orange color develops if aluminum is present.
Note: Inconsistent results will be obtained if any powder is undissolved.
6. Pour 25 mL of mixture into a 25-mL sample cell (the prepared sample).
7. Add contents of one Bleaching 3 Reagent Powder Pillow to the remaining 25 mL in the mixing Graduated cylinder. Stopper. Vigorously shake for 30 seconds.
Note: This solution should turn a light to medium orange upon bleaching. It will not become colorless.
8. Pour the remaining 25 mL of mixture in the cylinder into a second 25-mL sample cell (the blank).
9. Press: SHIFT TIMER
A 15-minute reaction period will begin. When the timer beeps, the display will show: mg/L Al3+
10. Within five minutes after the timer beeps, place the blank into the cell holder. Close the light shield.
11. Press: ZERO The display will show: **Zeroing**. . . . then: **0.00 mg/L Al3+**
12. Immediately place the prepared sample into the cell holder. Close the light shield.
13. Press: READ The display will show: **Reading**. . . . then the result in mg/L aluminum will be displayed.
Note: Clean the graduated cylinder and sample cells with soap and brush immediately following the test.
Note: For most accurate results, analyze a reagent blank (deionized water) and subtract the amount determined on each lot of reagents from the sample reading.

Precision : ±0.016 mg/L Al^{3+}.

CHLORIDE (0 to 20.0 mg/L Cl-) For water and wastewater
Method 8113 Mercuric Thiocyanate Method*
* Adapted from Zall, et. al., Analytical Chemistry, 28 (11) 1665 (1956).

1. Enter the stored program number for Chloride (Cl-). Press: 7 0 ENTER
The display will show: **Dial nm to 455**
Note: The Pour-Thru cell can be used with this procedure. Collect the waste solution for proper disposal.
2. Rotate the wavelength dial until the small display shows: 455 nm
When the correct wavelength is dialed in the display will quickly show: **Zero Sample** then: **mg/L Cl-**
3. Fill a sample cell with 25 mL of sample. Note: Filter turbid samples.
4. Fill another cell with 25 mL of deionized water (the blank).
5. Pipet 2.0 mL of Mercuric Thiocyanate Solution into each cell. Swirl to mix.
6. Pipet 1.0 mL of Ferric Ion Solution into each sample cell. Swirl to mix.
Note: An orange color will develop if chloride is present.
7. Press: SHIFT TIMER A two-minute reaction period will begin.
8. When the timer beeps, the display will show: **mg/L Cl-**
Place the blank into the cell holder. Close the light shield.
9. Press: ZERO The display will show: **Zeroing**. . . then: 0**.0 mg/L Cl-**
10. Place the prepared sample into the cell holder. Close the light shield.
11. Press: READ The display will show: **Reading**. . . then the result in mg/L chloride (Cl-) will be displayed.
Note: The prepared sample and blank contain mercury and must be disposed of according to current Federal, State, and local hazardous waste regulations.

Precision: ±0.3 mg/L Cl^-.

CHLORINE, TOTAL (0 to 2.00 mg/L) For water, wastewater and seawater
Method 8167, DPD Method* (Powder Pillows or AccuVac Ampuls)
USEPA accepted for reporting water and wastewater** Using Powder Pillows
* Adapted from Standard Methods for the Examination of Water and Wastewater.
** Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water.

1. Enter the stored program number for free and total chlorine (Cl2) powder pillows. Press: 8 0 ENTER
The display will show: **Dial nm to 530**
Note: The Pour-Thru Cell can be used with 25-mL reagents only.
2. Rotate the wavelength dial until the small display shows: 530 nm
When the correct wavelength is dialed in the display will quickly show: **Zero Sample** then: **mg/L Cl2**
3. Insert the 10-mL Cell Riser into the sample compartment.
4. Fill a 10-mL sample cell with 10 mL of sample.
Note: Samples must be analyzed immediately and cannot be preserved for later analysis.

5. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Stopper the sample cell and shake for 20 seconds. Remove the stopper.
Note: Shaking dissipates bubbles which may form.
Note: DPD is a Salt of N,N-Diethyl-p-Phenylenediamine, and may contain sodium phosphate dibasic and carboxylate salts.
6. Press: SHIFT TIMER
A three-minute reaction period will begin.
Note: A pink color will develop if chlorine is present.
7. When the timer beeps, the display will show: mg/L Cl2
Fill another sample cell (the blank) with 10 mL of sample. Place it into the cell holder. Close the light shield.
8. Press: ZERO
The display will show: **Zeroing**. . . then: **0.00 mg/L Cl2**
9. Within three minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield.
10. Press: READ The display will show: **Reading**. . . then the result in mg/L chlorine (Cl2) will be displayed.
Note: It the sample temporarily turns yellow after sample addition, or shows **OVER-RANGE**, dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the appropriate dilution factor; see Sample Dilution Techniques (Section I).

Precision: ±0.012 mg/L Cl_2.

CHEMICAL OXYGEN DEMAND For water, wastewater and seawater
Method 8000 Reactor Digestion Method*; USEPA approved for reporting wastewater analysis** Digestion
* Jirka, A.M.; Carter, M.J. Analytical Chemistry, 1975, 47(8). 1397.
** Federal Register, April 21, 1980, 45(78), 26811-26812.
1. Homogenize 100 mL of sample for 30 seconds in a blender.
Note: Mix the sample before homogenizing it. To improve accuracy and reproducibility, pour the homogenized sample into a 250-mL beaker and gently stir with a magnetic stir plate. For samples containing large amounts of solids, increase the homogenization time.
Note: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and clothing for adequate user protection. If contact occurs, flush the affected area with running water. Follow instructions carefully.
2. Turn on the COD Reactor. Preheat to it 150 °C. Place the plastic shield in front of the reactor.
Note: Ensure safety devices are in place to protect analyst from splattering should reagent leaking occur.
3. Remove the cap of a COD Digestion Reagent Vial for the appropriate range:

Note: The reagent mixture is light-sensitive. Keep unused vials in the opaque shipping container, in a refrigerator if possible. The light striking the vials during the test will not affect results.
4. Hold the vial at a 45-degree angle. Pipet 2.00 mL of sample into the vial.
For greater accuracy a minimum of three replicates should be analyzed and the results averaged.
Note: Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Do not run tests with vials which have been spilled. If spills occur, wash with running water. Use Low Range vial type for 0-150 mg/L range.
5. Replace the vial cap tightly. Rinse the outside of the COD vial with deionized water and wipe the vial clean with a paper towel.
6. Hold the vial by the cap and over a sink. Invert gently several times to mix the contents. Place the vial in the preheated COD Reactor.
Note: The vial will become very hot during mixing.
7. Prepare a blank by repeating Steps 3 to 6, substituting 2.00 mL deionized water for the sample.
Note: Be sure the pipet is clean.
Note: One blank must be run with each set of samples. Run samples and blanks with the same lot of vials.
8. Heat the vials for 2 hours.
Note: Many samples are digested completely in less than two hours. If desired, measure the concentration (while still hot) at 15 minute intervals until the reading remains unchanged. Cool the vials to room temperature for final measurement.
9. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120 °C or less.
10. Invert each vial several times while still warm. Place the vials into a rack. Wait until the vials have cooled to room temperature.
Note: If a pure green color appears in the reacted sample, measure the COD and, if necessary, repeat the test with a diluted sample.
11. Use one of the following analytical techniques to measure the COD:
• Colorimetric method, 0-150 mg/L COD
• Colorimetric method, 0-1,500 mg/L COD
• Colorimetric method, 0-15,000 mg/L COD

Colorimetric Determination, 0 to 150 mg/L COD
1. Enter the stored program number for chemical oxygen demand (COD), low range. Press: 4 3 0 ENTER The display will show: **Dial nm to 420**
2. Rotate the wavelength dial until the small display shows: 420 nm
When the correct wavelength is dialed in, the display will quickly show: **Zero Sample** then: **mg/L COD LR**
Note: Approach the wavelength setting from the higher to lower values.
3. Place the COD Vial Adapter into the cell holder with the marker to the right.
4. Clean the outside of the blank with a towel.
Note: Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.
5. Place the blank into the adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.
Note: The blank is stable when stored in the dark; see Blanks for Colorimetric Determination following these procedures.

6. Press: ZERO The display will show: **Zeroing**. . . . then: **0. mg/L COD LR**
7. Clean the outside of the sample vial with a towel.
8. Place the sample vial into the adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.
9. Press: READ The display will show: **Reading**. . . . then the result in mg/L COD will be displayed.
Note: For most accurate results with samples near 150 mg/L COD, repeat the analysis with a diluted sample.

Precision: ±2.7 mg/L

Table A1. Sample dilutions and resulting analytical precision

Test	**coliforms**	**Cl^-**	**Cl_2^0**	**COD**	**Supern't Al^{3+}**	**Total Al^{3+}**
1	500	10 / 3	0	0 / 2.7	0 / 0.013	2000 / 26
2	500	10 / 3	2	0 / 2.7	0 / 0.013	2000 / 26
3	500	50 / 15	2	0 / 2.7	10 / 0.13	2000 / 26
4	500	10 / 3	2	0 / 2.7	10 / 0.13	1000 / 13
5	250	10 / 3	2	0 / 2.7	10 / 0.13	1000 / 13
6	500	100 / 30	2	0 / 2.7	10 / 0.13	1000 / 13
7	500	100 / 30	2	0 / 2.7	10 / 0.13	1000 / 13
8	500	100 / 30	2	0 / 2.7	10 / 0.13	1000 / 13
9	200	100 / 30	2	0 / 2.7	100 / 1.3	4000 / 52
10	500	10 / 3	2	0 / 2.7	1000 / 13	2000 / 26
11	500	50 / 15	2	0 / 2.7	10 / 0.13	2000 / 26
12	200	10 / 3	2	0 / 2.7	100 / 1.3	2000 / 26

APPENDIX 2

Excerpts from Sterilization Guide from USGS TWRI

EQUIPMENT AND EQUIPMENT 7.1.1

STERILIZATION PROCEDURES

Fecal Indicator Bacteria U.S. Geological Survey TWRI 7/17/97

Equipment for collection and analysis of bacterial samples must be clean and sterile (table 7.1–3). Wrap equipment in kraft paper, autoclavable bags, or aluminum foil. Sterilize and store the equipment in a clean area. Resterilize equipment if foil, bag, or kraft paper is torn.

Add sodium thiosulfate ($Na_2S_2O_3$) to sample bottles before sterilization if the water to be collected contains residual chlorine or other halogens added for disinfection. Residual chlorine can be found in samples collected from sources such as treated potablewater taps, in effluents, and surface-water samples collected from the mixing zones of wastewater-treatment plants. A 10-percent solution of $Na_2S_2O_3$ is prepared in the following manner. In a volumetric flask, dissolve 100 g $Na_2S_2O_3$ into 500 mL of deionized or distilled water; stir until dissolved, and fill flask to 1,000 mL (Bordner and Winter, 1978, p. 6; American Public Health Association and others, 1992, p. 9–18). Add 0.1 mL of 10-percent $Na_2S_2O_3$ solution for every 100 mL of sample. Keep $Na_2S_2O_3$ refrigerated and in a dark bottle; after 6 months prepare a fresh solution.

Add ethylenediaminetetraacetic acid (EDTA) to sample bottles when water to be collected contains trace elements such as copper, nickel, and zinc at concentrations greater than 10 mg/L (Britton and Greeson, 1989, p. 5–6; Bordner and Winter, 1978, p. 6; American Public Health Association and others, 1992, p. 9–18). A 15-percent solution of EDTA is prepared by dissolving 372 mg in 1,000 mL of distilled or deionized water. Before sterilization, add 0.3 mL of the EDTA solution per 100 mL of sample to sample bottles. EDTA can be combined with the $Na_2S_2O_3$ solution in the sample bottle before sterilization.

Autoclaving is the preferred method for sterilizing equipment.

Sterilize the filtration apparatus between sites or for each sample collected at the same site at different times. Autoclaving is the preferred method of sterilization. Use only autoclaves that have temperature, pressure, and liquid- and dry-utensil-cycle controls. Steam sterilizers and vertical autoclaves are not recommended because the temperature cannot be held constant.

Table 7.1–3. Equipment cleaning and sterilizing procedures
[$Na_2S_2O_3$, sodium thiosulfate; >, greater than value shown; µg/L, micrograms per liter; EDTA, ethylenediaminetetraacetic acid; °C, degrees Celsius; mg/L, milligrams per liter]

Equipment	**Cleaning and sterilizing procedures**
All equipment	Wash equipment thoroughly with a dilute nonphosphate, laboratory-grade detergent. Rinse three times with hot tap water. Rinse again three to five times with deionized or glass-distilled water.
Glass, polypropylene, or Teflon™ bottles	If sample will contain residual chlorine or other halogens, add $Na_2S_2O_3$. If sample will contain >10 µg/L trace elements, add EDTA. Autoclave at 121°C for 15 minutes, or bake glass jars at 170°C for 2 hours.
Stainless-steel field units	Flame sterilize with methanol (Millipore Hydrosol™ units only), or autoclave, or bake at 170°C for 2 hours.
Portable submersible pumps and pump tubing	Autoclavable equipment (preferred): Autoclave at 121°C for 15 minutes. Non-autoclavable equipment: Submerge sampling system in a 200-mg/L laundry bleach solution and circulate solution through pump and tubing for 30 minutes; follow with thorough rinsing, inside and out, with sample water pumped from the well. DO NOT USE THIS METHOD TO DISINFECT EQUIPMENT USED TO COLLECT SAMPLES FOR SUBSEQUENT DETERMINATIONS OF TRACE ELEMENTS AND ORGANIC SUBSTANCES.

Take care to ensure that materials to be autoclaved, such as tubing and containers, are thermally stable. Polymers (such as polycarbonate, polypropylene, polyallomer, and polymethylpentene) and Teflons™ and Tefzel™ (such as perfluoroalkyoxy-polymers or PFA™, ethylenetetrafluoro-ethylene or ETFE™, fluorinated ethylene propylene or FEP™, and polytetrafluoroethylene polymers or PTFE™) can be autoclaved. Each has different thermal characteristics and tolerances to repeated autoclaving.

APPENDIX 3

Experiment Test Sheets (1-12)
Data Summary Table
DesignEase Program Statistical Analysis Output

RUN # 1 Date: January 24, 2005

OPERATION PARAMETERS:

Contact Time, minutes	5
Sample Volume, mL	850
Current Density (mA/cm^2)	5.5
Cl^- Concentration (mg/L)	138.6

Power Consumption	
Volts :	14
Amps :	10.06
kWh/m^3 :	13.81

Solids Production

0.001668 **tonne/m^3**

2 hr delay between run A and B to get new fuse.

FEED	Cndctvy. (mS)	TDS (mg/l)	pH	ORP (V)	Temp (^{o}C)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C)	Evap'n Loss (%)
Influent 1	1012	667	6.97	0.197	16.7	8.33	142.4	19.0	23.79
Influent 2	966	638	6.92	0.198	17.7	8.35	134.7	18.7	
Influent avg.	989	653	6.95	0.1975	17.2	8.34	138.6	19.5	**ΔT,°C 7.2**
EFFLUENT									
Effluent A	811	524	8.50	0.104	20.6	5.29	176.1	26.7	
Effluent B	801	528	8.66	0.096	21.6	5.56	181.5	26	
Effluent C	741	483	8.53	0.100	22.6	4.47	187.8	26	
Effluent Avg	**784**	**512**	**8.56**	**0.100**	**21.6**	**5.11**	**181.8**		

LAB ANALYSES **Coliform dilution =** 500

FEED	Coliforms (mL^{-1})	Total Cl^{+1} (mg/L)	COD (mg/L)	TSS (mg/L)	Al^{3+} (mg/L)	**Sample Averages and Std. Deviations** **Coliforms**	**COD**	**TSS**	**Al^{3+}**
						INFLUENT			
Influent A	36	0.05	66.8	8	0.14				
Influent B	46	0.05	62.0		0.08	41.3	62.0	8.0	0.080
Influent C	42	0.04	57.1		0.02	5.0	4.85		0.085
SAMPLE A									
						EFFLUENT #1			
Effluent 1	11	0.36	46.3	1613	611.5				
Effluent 2	10	0.48	54.4		621.6	11.7	49.7	1613.0	614.9
Effluent 3	14	0.34	48.4		611.5	2.1	4.19		5.79
SAMPLE B									
						EFFLUENT #2			
Effluent 1	6	0.38	55.4	1645	852.1				
Effluent 2	11	0.46	54.4		711.8	9.3	53.4	1645.0	782.0
Effluent 3	11	0.40	50.4		782.0	2.9	2.67		70.18
SAMPLE C									
						EFFLUENT #3			
Effluent 1	9	0.40	69.5	1769	651.6				
Effluent 2	10	0.33	71.5		621.6	9.3	72.2	1769.0	638.3
Effluent 3	9	0.34	75.6		641.6	0.6	3.08		15.31

Test Averages	**Influent** Average	Std. Dev.	**Effluent** Average	Std. Dev.	**Change Δ**	**Removal (%)**	**Log Removal**	Feed stdv.
Coliforms (L^{-1})	41.3	5.0	10.11	1.3	-31.2	75.5		12.2%
Total Chlorine (mg/L)	0.045	0.005	0.389	0.053	0.34	-763.9		
COD (mg/L)	62.0	4.9	58.4	12.1	-3.5	5.7		
TSS (mg/L)	8.0		1676	82.4	1668	(20,846)		
Al^{3+} (mg/L)	0.1	0.1	678.4	90.5	678.3	(847,848)		
Conductivity (µS)	989	32.5	784	37.9	-205	20.7		
TDS (mg/L	653	14.5	512	24.9	-141	21.6		
pH	6.95	0.02	8.56	0.09	1.6			
ORP (mV)(*vs NHE)	0.0	0.00	0.0	0.00	0.0			
Temperature (°C)	17.2	0.50	21.60	1.00	4.4			Eff. Stdv.
Dissolved Oxygen (mg/L)	8.34		5.11		-3.2			
Actual Coliform Counts: ↑(per 100 mL)	2.07E+06	2.52E+05	5.06E+05	6.74E+04	-1.56E+06		0.61	13.3%

RUN # 2 Date: January 26, 2005

OPERATION PARAMETERS:

Contact Time, minutes	15
Sample Volume, mL	850
Current Density (mA/cm^2)	5.5
Cl- Concentration (mg/L)	136.9

Power Consumption	
Volts :	14.0
Amps :	10.06
kWh/m^3 :	41.42

Solids Production Rate

0.006539 **tonne/m^3**

Sludge floated into a gel, then floated again after degassing once.

SAMPLE 1	Conduct. (mS)	TDS (mg/l)	pH	ORP (V)	Temp (°C)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C)	Evapr'n Loss (%)
Influent	970	642	6.89	0.194	20.2	7.69	131.3	20.5	22.33
Influent	970	639	6.97	0.190	20.1	7.06	142.4		
Influent	970	641	6.93	0.19	20.15	7.38	136.9		**ΔT,°C**
EFFLUENT									**21**
Effluent A	657	429	8.85	0.066	31.1	1.52	162.7	41.5	
Effluent B	706	452	8.66	0.079	30.9	1.52	190.5	47	
Effluent C	677	432	8.83	0.069	31.9	1.02	175.4	43	
Effluent Avg	**680**	**438**	**8.78**	**0.071**	**31.3**	**1.35**	**176**		

LAB ANALYSES **Coliform dilution =** 500

	Coliforms (mL-1)	Total Cl^{+1} (mg/L)	COD (mg/L)	TSS (mg/L)	Al^{3+} (mg/L)	**Sample Averages and Std. Deviations** **Coliforms**	**COD**	**TSS**	**Al^{3+}**
FEED									
Influent A	34	0.03	44.7	4	0		**INFLUENT**		
Influent B	35	0.03	46.35		0.06	33.7	46.4	4.0	0.06
Influent C	32	0.02	48.0		0.11	1.5	1.65		0.08
SAMPLE A			supernatant						
Effluent 1	0	1.00	26.2	5881	1167.3		**EFFLUENT #1**		
Effluent 2	0	1.22	34.3		1207.5	0.0	29.2	5881.0	1220.9
Effluent 3	0	1.04	27.2		1288.0	0.0	4.39		61.48
SAMPLE B									
Effluent 1	0	0.66	11.1	6750	1046.5		**EFFLUENT #2**		
Effluent 2	0	0.56	10.1		1066.6	0.0	9.1	6750.0	1039.8
Effluent 3	0	0.66	6.0		1006.3	0.0	2.67		30.74
SAMPLE C									
Effluent 1	0	1.20	13.1	6999	1207.5		**EFFLUENT #3**		
Effluent 2	2	0.96	19.1		1187.4	0.7	15.4	6999.0	1174.0
Effluent 3	0	1.16	14.1		1127.0	1.2	3.24		41.89

Test Averages	**Influent**		**Effluent**		**Change**	**Removal**	**Log**	
	Average	Std. Dev.	Average	Std. Dev.	Δ	**(%)**	**Removal**	Feed stdv.
Coliforms (L-1)	33.7	1.5	0.22	0.4	-33.4	99.3		4.5%
Total Chlorine (mg/L)	0.025	0.005	0.942	0.253	0.92	-3669.4		
COD (mg/L)	46.4	1.7	17.9	10.3	-28.4	61.4		
TSS (mg/L)	4.0	0.0	6543	587.0	6539	(163,483)		
Al3+ (mg/L)	0.1	0.1	1144.9	94.0	1144.8	-2.E+06		
Conductivity (µS)	970	0.00	680	24.6	-290	29.9		
TDS (mg/L	641	1.5	438	12.5	-203	31.7		
pH	6.93	0.04	8.78	0.10	1.9			
ORP (mV)(*vs NHE)	409.2	10.15	251.5	3.97	-157.7			
Temperature (°C)	20.2	0.05	31.30	0.53	11.2			Eff. Stdv.
Dissolved Oxygen (mg/L)	7.375		1.35		-6.0			
Actual Coliform Counts: ↑(per 100 mL)	1.68E+06	7.64E+04	1.11E+04	1.92E+04	-1.67E+06		2.18	173.2%

RUN # 3 Date: January 31, 2005

OPERATION PARAMETERS:

Contact Time, minutes	10		**Power Consumption**		**Solids Production Rate**
Sample Volume, mL	850		Volts :	4	
Current Density (mA/cm^2)	3.5		Amps :	6.4	**0.002898** **tonne/m^3**
Cl- Concentration (mg/L)	583	*596.5*	kWh/m3 :	5.02	

SAMPLE 1	Conduct. (mS)	TDS (mg/l)	pH	ORP (V)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C)	Evap'n Loss (%)
Influent	2.46	1620	7.40	0.166	18.8	7.64	596.5	20.5	-10.62
Influent	2.43	1604	7.48	0.161	19.6	7.50	569.8	20.8	
Influent	2.45	1612	7.44	0.16	19.20	7.57	583.15		**ΔT,°C**
SAMPLE 3									**10.7**
Effluent A	2.19	1434	8.90	0.078	21.9	2.75	506.3	24.1	
Effluent B	2.17	1421	9.00	0.071	22.3	1.59	594.0	24.3	
Effluent C	2.17	1417	9.07	0.067	22.6	2.43	481.2	25	
Effluent Avg	**2**	**1424**	**8.99**	**0.072**	**22.3**	**2.26**	**527**		

LAB ANALYSES **Coliform dilution =** 500

FEED	Coliforms (mL-1)	Total Cl+1 (mg/L)	COD (mg/L)	TSS (mg/L)	Al3+ (mg/L)	**Sample Averages and Std. Deviations** **Coliforms**	**COD**	**TSS**	**Al3+**
Influent A	105	0.05	60.0	21	0.02		**INFLUENT**		
Influent B	111	0.05	59.5		0.02	105.7	59.5	21.0	0.02
Influent C	101	0.05	59.0		0.02	5.0	0.50		0.00
SAMPLE A									
Effluent 1	6	1.00	26.0	2820	1122.8		**EFFLUENT #1**		
Effluent 2	6	0.90	22.0		supern't	5.7	24.7	2820.0	1122.8
Effluent 3	5	1.06	26.0		1.9	0.6	2.31		
SAMPLE 2									
Effluent 1	16	1.08	27.0	2876	1162.9		**EFFLUENT #2**		
Effluent 2	19	0.78	25.0		supern't	18.0	27.3	2876.0	1162.9
Effluent 3	19	0.84	30.0		2.0	1.7	2.52		
SAMPLE 3									
Effluent 1	8	0.70	26.0	3061	782		**EFFLUENT #3**		
Effluent 2	14	0.58	17.0		supern't	10.7	18.7	3061.0	782.0
Effluent 3	10	0.56	13.0		0.4	3.1	6.66		

Test Averages	**Influent**		**Effluent**		**Change**	**Removal**	**Log**	
	Average	Std. Dev.	Average	Std. Dev.	Δ	**(%)**	**Removal**	Feed stdv.
Coliforms (L-1)	105.7	5.0	11.44	6.2	-94.2	89.2		4.8%
Total Chlorine (mg/L)	0.050	0.000	0.833	0.195	0.78	-1566.7		
COD (mg/L)	59.5	0.5	23.6	4.4	-35.9	60.4		
TSS (mg/L)	21.0	0.0	2919	126.1	2898	(13,800)		
Al3+ (mg/L)	0.0	0.0	1022.6	209.3	1022.5	-5.E+06		
Conductivity (µS)	2	0.0	2	0.0	0	11.0		
TDS (mg/L	1612	8.0	1424	8.9	-188	11.7		
pH	7.44	0.04	8.99	0.09	1.6			
ORP (mV)(*vs NHE)	373.7	12.21	271.5	5.99	-102.2			
Temperature (ºC)	19.2	0.40	22.27	0.35	3.1			Eff. Stdv.
Dissolved Oxygen (mg/L)	7.57		2.26		-5.3			
Actual Coliform Counts: ↑(per 100 mL)	5.28E+06	########	5.72E+05	3.10E+05	-4.71E+06		0.97	54.2%

RUN # 4 Date: February 11, 2005

OPERATION PARAMETERS:

Contact Time, minutes	5
Sample Volume, mL	850
Current Density (mA/cm^2)	1.5
Cl- Concentration (mg/L)	136

Power Consumption

Volts :	4.0
Amps :	2.74
kWh/m^3 :	1.07

Solids Production Rate

0.000337 **tonne/m^3**

FEED	Conduct. (mS)	TDS (mg/l)	pH	ORP (mV)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C)	Evap. Loss (%)
Influent	1050	690	7.07	0.183	18.1	7.29	139	17.8	5.27
Influent	1040	686	7.00	0.187	18.2	7.24	143	18	
Influent avg.	1045	688	7.035	0.185	18.15	7.265	136	19	ΔT,°C 1.9
EFFLUENT									
Effluent A	952	627	7.61	0.152	19.6	6.52	141.3	20.5	
Effluent B	954	629	7.46	0.161	19.1	7.11	138.7	19.9	
Effluent C	950	627	7.64	0.150	19.1	4.43	150.7	20.2	
Effluent Avg	**952**	**628**	**7.57**	**0.154**	**19.3**	**6.02**	**144**		

LAB ANALYSES Effluent Coliform dilution = 500 Feed Coliform dilution = 1000

FEED	Coliforms (mL-1)	Total Cl^{+1} (mg/L)	COD (mg/L)	TSS (mg/L)	Al^{3+} (mg/L)	Sample Averages and Std. Deviations Coliforms	COD	TSS	Al^{3+}
Influent A	52	0.03	91.5	26	0.01		**INFLUENT**		
Influent B	53	0.03	88.0		0.02	53.0	88.0	26.0	0.02
Influent C	54	0.03	84.5		0.02	1.0			0.01
SAMPLE A									
Effluent 1	46	0.23	27.0	338	170.5		**EFFLUENT #1**		
Effluent 2	51	±0.01	±8.5		supern't	49.7	27.0	338.0	170.5
Effluent 3	52				4.0	3.2			
SAMPLE B									
Effluent 1	49	0.39	33.5	376	160.5		**EFFLUENT #2**		
Effluent 2	60	±0.01	±2.1		supern't	59.7	33.5	376.0	160.5
Effluent 3	70				5.7	10.5	#VALUE!		
SAMPLE C									
Effluent 1	41	0.41	25.5	374	170.5		**EFFLUENT #3**		
Effluent 2	44	±0.04	±12.0		supern't	45.3	25.5	374.0	170.5
Effluent 3	51				4.8	5.1			

Test Averages	Influent Average	Influent Std. Dev.	Effluent Average	Effluent Std. Dev.	Change Δ	Removal (%)	Log Removal	
Coliforms (L-1)	53.0	1.0	51.56	7.4	-1.4	51.4		1.887%
Total Chlorine (mg/L)	0.028	0.003	0.343	0.099	0.32	-1148.48		
COD (mg/L)	88.0	0.0	28.7	4.3	-59.3	67.4		
TSS (mg/L)	26.0	0.0	363	21.4	337	(1,295)		
Al^{3+} (mg/L)	0.0	0.0	167.2	5.8	167.1	-1.E+06		
Conductivity (µS)	1045	5.0	952	2.0	-93	8.9		
TDS (mg/L	688	2.0	628	1.2	-60	8.8		
pH	7.04	0.04	7.57	0.10	0.5			
ORP (mV)(*vs NHE)	390.7	1.72	357.8	5.89	-32.9			
Temperature (°C)	18.15	0.05	19.27	0.29	1.1			Eff. Stdv.
Dissolved Oxygen (mg/L)	7.265		6.02		-1.2			
Actual Coliform Counts: (per 100 mL)	5.30E+06	1.00E+05	2.58E+06	3.68E+05	-2.72E+06		0.31	14.26%

RUN # 5 Date: February 16, 2005

OPERATION PARAMETERS:

Contact Time, minutes	15
Sample Volume, mL	850
Current Density (mA/cm^2)	1.5
Cl- Concentration (mg/L)	128.9

Power Consumption

Volts :	5
Amps :	2.74
kWh/m3 :	3.63

Solids Production Rate

0.001092 **tonne/m^3**

FEED	Conductivity (mS)	TDS (mg/l)	pH	ORP (V)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C	Evapor'n Loss (%)
Influent i	988	652	6.99	0.184	21.3	6.52	120.1	21	1.87
Influent f	982	648	7.05	0.181	21.0	6.51	137.6	22.0	
Influent avg.	985	650	7.02	0.1826	21.15	6.515	128.9	22.2	ΔT,°C 2.6
EFFLUENT									
Effluent A	786	518	8.10	0.119	22.7	5.64	145.40	24.2	
Effluent B	752	495	8.46	0.099	22.7	6.17	128.10	24.3	
Effluent C	756	497	8.51	0.096	22.8	4.94	120.4	24.5	
Effluent Avg	**765**	**503**	**8.36**	**0.105**	**22.7**	**5.58**	**131**		

LAB ANALYSES Effluent Coliform dilution = 250 Feed Coliform dilution = 1000

	Coliforms (mL-1)	Total Cl+1 (mg/L)	COD (mg/L)	TSS (mg/L)	Al^{3+} (mg/L)	Sample Averages and Std. Deviations Coliforms	COD	TSS	Al^{3+}
FEED									
Influent A	66	0.03	91.5	18	0		**INFLUENT**		
Influent B	64	0.03	104.8		0.01	65.3	104.8	18.0	0.01
Influent C	66	0.04	118.0		0.02	1.2			0.01
SAMPLE A		Effluent supernatant COD							
Effluent 1	64	0.52	25.0	1093	371.9		**EFFLUENT #1**		
Effluent 2	74	±0.01	±2.8		supern't	74.0	27.0	1093.0	371.9
Effluent 3	84	0.50	29.0		4.5	10.0			
SAMPLE B									
Effluent 1	45	0.44	26.0	1067	472.4		**EFFLUENT #2**		
Effluent 2	64	±0.06	±2.1		supern't	55.0	27.5	1067.0	472.4
Effluent 3	56	0.52	29.0		5.7	9.5	#VALUE!		
SAMPLE C									
Effluent 1	56	0.76	12.0	1171	381.9		**EFFLUENT #3**		
Effluent 2	63	±0.07	±1.4		supern't	60.7	13.0	1171.0	381.9
Effluent 3	63	0.66	14.0		7.9	4.0			

Test Averages	**Influent** Average	Std. Dev.	**Effluent** Average	Std. Dev.	**Change** Δ	**Removal** (%)	**Log Removal**	Feed stdv.
Coliforms (L-1)	65.3	1.2	63.22	9.8	-2.1	51.6		1.767%
Total Chlorine (mg/L)	0.033	0.003	0.573	0.167	0.54	-1664.10		
COD (mg/L)	104.8	0.0	22.5	8.2	-82.3	78.5		
TSS (mg/L)	18.0	0.0	1110	54.1	1092	(6,069)		
Al^{3+} (mg/L)	0.0	0.0	408.7	55.4	408.7	-4.E+06		
Conductivity (µS)	985	3.0	765	18.6	-220	22.4		
TDS (mg/L	650	2.0	503	12.7	-147	22.6		
pH	7.02	0.03	8.36	0.22	1.3			
ORP (mV)(*vs NHE)	384.9	2.14	304.4	12.85	-80.5			
Temperature (ºC)	21.15	0.15	22.73	0.06	1.6			Eff. Stdv.
Dissolved Oxygen (mg/L)	6.515		5.58		-0.9			
Actual Coliform Counts: (per 100 mL)	6.53E+06	########	1.58E+06	2.44E+05	-4.95E+06		0.62	15.43%

RUN # 6 Date: February 21, 2005

OPERATION PARAMETERS:

Contact Time, minutes	5
Sample Volume, mL	850
Current Density (mA/cm^2)	5.5
Cl- Concentration (mg/L)	868

Power Consumption

Volts :	3.8
Amps :	10.06
kWh/m3 :	3.75

Solids Production Rate

0.002278 **tonne/m^3**

FEED	Conductivity (mS)	TDS (mg/l)	pH	ORP (V)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C)	Evapor'n Loss (%)
Influent A	3890	2570	7.11	0.177	22.0	6.07	880	23.1	-20.49
Influent C	3860	2550	7.43	0.159	22.1	6.34	855	23.6	
Influent avg.	3875	2560	7.27	0.16765	22.05	6.205	868	23.5	**ΔT,°C 3.0**
EFFLUENT									
Effluent A	3650	2400	8.71	0.084	23.8	3.36	720	25.9	
Effluent B	3620	2380	8.75	0.080	24.1	1.80	690	26.7	
Effluent C	3610	2370	8.78	0.079	24.0	3.06	750	26.5	
Effluent Avg	**3627**	**2383**	**8.75**	**0.081**	**24.0**	**2.74**	**720**		

LAB ANALYSES Effluent Coliform dilution = 500 Feed Coliform dilution = 1000

FEED	Coliforms (mL-1)	Total Cl^{+1} (mg/L)	COD (mg/L)	TSS (mg/L)	Al^{3+} (mg/L)	Sample Averages and Std. Deviations Coliforms	COD	TSS	Al^{3+}
Influent A	72	0.05	57.5	9	0		**INFLUENT**		
Influent B	71	0.05	58.3		0.00	71.3	58.3	9.0	0.00
Influent C	71	0.05	59.0		0	0.6	1.06		
SAMPLE A		Effluent supernatant COD							
Effluent 1	21	0.44	20.0	2187	804.0		**EFFLUENT #1**		
Effluent 2	10	0.44	21.0		supern't	16.3	20.5	2187.0	804.0
Effluent 3	18				17.1	5.7	0.71		
SAMPLE B									
Effluent 1	14	0.42	16.0	2319	783.9		**EFFLUENT #2**		
Effluent 2	15	0.46	22.0		supern't	16.3	19.0	2319.0	783.9
Effluent 3	20				15.1	3.2	4.24		
SAMPLE C									
Effluent 1	19	0.54	25.0	2354	814.1		**EFFLUENT #3**		
Effluent 2	21	0.52	28.0		supern't	21.0	26.5	2354.0	814.1
Effluent 3	23				15.1	2.0	2.12		

Test Averages	**Influent**		**Effluent**		**Change**	**Removal**	**Log**	
	Average	Std. Dev.	Average	Std. Dev.	Δ	**(%)**	**Removal**	Feed stdv.
Coliforms (L-1)	71.3	0.6	17.89	2.7	-53.4	74.9		0.8%
Total Chlorine (mg/L)	0.045	0.000	0.470	0.290	0.43	-944.44		
COD (mg/L)	58.3	1.1	22.0	4.0	-36.3	62.2		
TSS (mg/L)	9.0	0.0	2287	88.1	2278	(25,307)		
Al^{3+} (mg/L)	0.0	0.0	800.7	15.4	800.7			
Conductivity (µS)	3875	10.0	3627	20.8	-248	6.4		
TDS (mg/L	2560	6.7	2383	15.3	-177	6.9		
pH	7.27	0.11	8.75	0.04	1.5			
ORP (mV)(*vs NHE)	368.3	9.30	278.2	2.94	-90.0			
Temperature (ºC)	22.05	0.03	23.97	0.15	1.9			Eff. Stdv.
Dissolved Oxygen (mg/L)	6.205		2.74		-3.5			
Actual Coliform Counts: (per 100 mL)	7.13E+06	########	8.94E+05	1.35E+05	-6.24E+06		0.90	15.06%

RUN # 7 Date: March 1, 2005

Actual feed TDS=2297

Actual effluent TDS=2144

OPERATION PARAMETERS:

Contact Time, minutes	5
Sample Volume, mL	850
Current Density (mA/cm^2)	1.5
Cl- Concentration (mg/L)	1005.0

Power Consumption

Volts :	2
Amps :	2.74
kWh/m3 :	0.54

Solids Production Rate

0.00047 **tonne/m^3**

FEED	Conduct. (mS)	TDS (mg/l)	pH	ORP (V)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C	Evapor'n Loss (%)
Influent A	3930	2570	7.19	0.181	19.2	7.64	1015	20.8	4.29
Influent C	3830	2520	7.63	0.157	19.6	6.80	995	20.2	
Influent avg.	3880	2545	7.41	0.16895	19.4	7.22	1005.0	20.5	**ΔT,°C**
EFFLUENT		2545							**0.6**
Effluent A	3790	2480	8.02	0.131	20.6	6.43	1065	21.2	
Effluent B	3760	2470	8.25	0.117	21.0	6.43	1050	21.0	
Effluent C	3770	2480	8.27	0.116	21.0	6.74	1035	21.1	
Effluent Avg	**3773**	**2477**	**8.18**	**0.121**	**20.9**	**6.53**	**1050**		

LAB ANALYSES Effluent Coliform dilution = 500 Feed Coliform dilution = 1000

FEED	Coliforms (mL-1)	Total Cl+1 (mg/L)	COD (mg/L)	TSS (mg/L)	Al^{3+} (mg/L)	Sample Averages and Std. Deviations Coliforms	COD	TSS	Al^{3+}
Influent A	47	0.06	73.0	14	0.02		**INFLUENT**		
Influent B	49	0.053	72.5		0.02	47.3	72.5	14.0	0.02
Influent C	46	0.05	72.0		0.02	1.5	0.50		0.00
SAMPLE A		Effluent supernatant COD							
Effluent 1	12	0.32	30.0	444	190.5		**EFFLUENT #1**		
Effluent 2	27	0.32	32.0	440	supern't	18.7	31.0	442.0	190.5
Effluent 3	17				3.2	7.6	1.41		
SAMPLE B									
Effluent 1	20	0.30	26.0	496	190.5		**EFFLUENT #2**		
Effluent 2	22	0.32	30.0	496	supern't	22.0	28.0	496.0	190.5
Effluent 3	24				4.2	2.0	2.83		
SAMPLE C									
Effluent 1	16	0.54	28.0	512	200.5		**EFFLUENT #3**		
Effluent 2	16	0.54	31.0	516	supern't	17.0	29.5	514.0	200.5
Effluent 3	19				4.7	1.7	2.12		

Test Averages	**Influent**		**Effluent**		**Change**	**Removal**	**Log**	
	Average	Std. Dev.	Average	Std. Dev.	Δ	**(%)**	**Removal**	Feed stdv.
Coliforms (L-1)	47.3	1.5	19.22	2.5	-28.1	59.4		3.2%
Total Chlorine (mg/L)	0.053	0.003	0.390	0.116	0.34	-642.86		
COD (mg/L)	72.5	0.5	29.5	1.5	-43.0	59.3		
TSS (mg/L)	14.0	0.0	484	37.5	470	(3,357)		
Al3+ (mg/L)	0.0	0.0	193.8	5.8	193.8	(969,067)		
Conductivity (µS)	3880	33.3	3773	15.3	-107	2.7		
TDS (mg/L	2545	16.7	2477	5.8	-68	2.7		
pH	7.41	0.15	8.18	0.14	0.8			
ORP (mV)(*vs NHE)	372.5	12.15	324.2	8.33	-48.2			
Temperature (°C)	19.4	0.13	20.87	0.23	1.5			Eff. Stdv.
Dissolved Oxygen (mg/L)	7.22		6.53		-0.7			
Actual Coliform Counts: (per 100 mL)	4.73E+06	1.53E+05	9.61E+05	1.27E+05	########		0.69	13.24%

RUN # 8 Date: March 2, 2005

OPERATION PARAMETERS: **Actual effluent TDS=2214** **Actual feed TDS=2318**

Contact Time, minutes	15
Sample Volume, mL	850
Current Density (mA/cm^2)	1.5
Cl- Concentration (mg/L)	1010

Power Consumption

Volts :	2
Amps :	2.74
kWh/m3 :	1.61

Solids Production Rate

0.001389 **tonne/m^3**

FEED	Conduct. (mS)	TDS (mg/l)	pH	ORP (V)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C	Evapor'n Loss (%)
Influent A	3860	2550	7.27	0.177	19.0	6.12	1010	20.3	6.48
Influent C	3810	2520	7.35	0.171	19.3	6.61	1010	20.1	
Influent avg.	3835	2535	7.31	0.174	19.15	6.365	1010	20.1	**ΔT,°C**
EFFLUENT		2535							**1.9**
Effluent A	3660	2400	8.60	0.096	21.0	6.22	1095	22.0	
Effluent B	3610	2380	8.82	0.086	20.9	6.16	1055	22.2	
Effluent C	3620	2370	8.82	0.084	20.9	4.82	1090	22.0	
Effluent Avg	**3630**	**2383**	**8.75**	**0.089**	**20.9**	**5.73**	**1080**		

LAB ANALYSES Effluent Coliform dilution = 500 Feed Coliform dilution = 1000

	Coliforms (mL-1)	Total Cl+1 (mg/L)	COD (mg/L)	TSS (mg/L)	Al^{3+} (mg/L)	**Sample Averages and Std. Deviations** **Coliforms**	**COD**	**TSS**	**Al^{3+}**
FEED									
Influent A	51	0.06	78.5	20	0.01		**INFLUENT**		
Influent B	47	0.04	80.0		0.01	47.7	80.0	20.0	0.01
Influent C	45	0.03	81.5		0.01	3.1	1.50		0.00
SAMPLE A		Effluent supernatant COD							
Effluent 1	20	0.20	27.0	1402	541.1		**EFFLUENT #1**		
Effluent 2	20	0.20	29.0		supern't	25.0	28.0	1402.0	541.1
Effluent 3	35				7.5	8.7	1.41		
SAMPLE B									
Effluent 1	17	0.26	24.0	1422	611.5		**EFFLUENT #2**		
Effluent 2	14	0.26	30.0		supern't	16.3	27.0	1422.0	611.5
Effluent 3	18				9.0	2.1	4.24		
SAMPLE C									
Effluent 1	15	0.12	24.0	1402	501.3		**EFFLUENT #3**		
Effluent 2	18	0.12	31.0		supern't	18.7	27.5	1402.0	501.3
Effluent 3	23				11.8	4.0	4.95		

Test Averages	**Influent** Average	Std. Dev.	**Effluent** Average	Std. Dev.	**Change** Δ	**Removal** (%)	**Log Removal**	Feed stdv.
Coliforms (L-1)	47.7	3.1	20.0	4.5	-27.7	58.0		6.4%
Total Chlorine (mg/L)	0.043	0.013	0.193	0.063	0.15	-354.90		
COD (mg/L)	80.0	1.5	27.5	0.5	-52.5	65.6		
TSS (mg/L)	20.0	0.0	1409	11.5	1389	(6,943)		
Al^{3+} (mg/L)	0.0	0.0	551.3	55.8	551.3	-6.E+06		
Conductivity (µS)	3835	16.7	3630	26.5	-205	5.3		
TDS (mg/L	2535	10.0	2383	15.3	-152	6.0		
pH	7.31	0.03	8.75	0.13	1.4			
ORP (mV)(*vs NHE)	377.8	2.50	290.5	6.67	-87.3			
Temperature (ºC)	19.15	0.10	20.93	0.06	1.8			Eff. Stdv.
Dissolved Oxygen (mg/L)	6.365		5.73		-0.6			
Actual Coliform Counts: (per 100 mL)	4.77E+06	########	1.00E+06	2.24E+05	########		0.68	22.42%

RUN # 9 Date: March 7, 2005

OPERATION PARAMETERS:

Contact Time, minutes	15
Sample Volume, mL	850
Current Density (mA/cm^2)	5.5
Cl- Concentration (mg/L)	1075

Power Consumption

Volts :	3.7
Amps :	10.04
kWh/m3 :	10.93

Solids Production Rate

0.0059 **tonne/m^3**

FEED	Conduct. (mS)	TDS (mg/l)	pH	ORP (V)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C)	Evapor'n Loss (%)
Influent A	3990	2620	7.20	0.172	20.5	5.28	1065	22.4	0.00
Influent C	3890	2560	7.31	0.165	21.3	5.21	1085	23.0	
Influent avg.	3940	2590	7.26	0.1687	20.9	5.245	1075	23.8	**ΔT,°C**
EFFLUENT									**7.5**
Effluent A	3560	2340	8.93	0.069	23.8	3.18	1120	29.8	
Effluent B	3560	2320	9.10	0.059	24.8	2.75	1035	30.7	
Effluent C	3560	2340	9.20	0.054	24.5	2.32	1070	31.2	
Effluent Avg	**3560**	**2333**	**9.08**	**0.061**	**24.4**	**2.75**	**1075**		

LAB ANALYSES Effluent Coliform dilution = 200 Feed Coliform dilution = 1000

FEED	Coliforms (mL-1)	Total Cl+1 (mg/L)	COD (mg/L)	TSS (mg/L)	Al^{3+} (mg/L)	Sample Averages and Std. Deviations: Coliforms	COD	TSS	Al^{3+}
Influent A	51	0.03	64.0	15	0.01		**INFLUENT**		
Influent B	47	0.03	65.0		0.01	52.0	65.0	15.0	0.01
Influent C	58	0.03	66.0		0.01	5.6	1.00		0.00
SAMPLE A		Effluent supernatant COD							
Effluent 1	3	1.1	24.0	5802	2608.0		**EFFLUENT #1**		
Effluent 2	5	1.7	28.0		supern't	3.0	26.0	5802.0	2608.0
Effluent 3	1				5.0	2.0	2.83		
SAMPLE B									
Effluent 1	1	1.0	19.0	5964	3531.0		**EFFLUENT #2**		
Effluent 2	2	0.6	23.0		supern't	1.3	21.0	5964.0	3531.0
Effluent 3	1				10.0	0.6	2.83		
SAMPLE C									
Effluent 1	1	0.7	17.0	5980	3451.0		**EFFLUENT #3**		
Effluent 2	2	1.1	22.0		supern't	1.3	19.5	5980.0	3451.0
Effluent 3	1				10.0	0.6	3.54		

Test Averages	**Influent** Average	Std. Dev.	**Effluent** Average	Std. Dev.	**Change** Δ	**Removal** (%)	**Log Removal**	Feed stdv.
Coliforms (L-1)	52.0	5.6	1.89	1.0	-50.1	96.4		10.7%
Total Chlorine (mg/L)	0.030	0.000	1.033	0.388	1.00	-3344.44		
COD (mg/L)	65.0	1.0	22.2	3.4	-42.8	65.9		
TSS (mg/L)	15.0		5915	98.5	5900	(39,336)		
Al^{3+} (mg/L)	0.01	0.0	3196.7	511.4	3196.7	-3.E+07		
Conductivity (µS)	3940	33.3	3560	0.0	-380	9.6		
TDS (mg/L	2590	20.0	2333	11.5	-257	9.9		
pH	7.26	0.04	9.08	0.14	1.8			
ORP (mV)(*vs NHE)	369.6	3.95	254.1	8.41	-115.5			
Temperature (ºC)	20.9	0.27	24.37	0.51	3.5			Eff. Stdv.
Dissolved Oxygen (mg/L)	5.245		2.75		-2.5			
Actual Coliform Counts: (per 100 mL)	5.20E+06	#######	3.78E+04	1.92E+04	-5.16E+06		2.14	50.94%

RUN # 10 Date: March 10, 2005 (Repeat #1)

OPERATION PARAMETERS:

Contact Time, minutes	5
Sample Volume, mL	850
Current Density (mA/cm^2)	5.5
Cl- Concentration (mg/L)	123

Power Consumption

Volts :	14
Amps :	10.06
kWh/m3 :	13.81

Solids Production Rate

0.001962 **tonne/m^3**

FEED	Conductivity (mS)	TDS (mg/l)	pH	ORP (V)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C	Evapr'n Loss (%)
Influent A	1056	699	6.94	0.187	19.9	5.83	122	21.2	-0.82
Influent B	1051	692	7.09	0.179	19.9	5.56	124	22.4	
Influent avg.	1053.5	695.5	7.02	0.18	19.9	5.70	123	22.2	**ΔT,°C 8.1**
EFFLUENT									
Effluent A	763	502	8.33	0.105	23.2	5.64	122	29.9	
Effluent B	762	502	8.48	0.096	23.1	5.32	122	30.2	
Effluent C	741	488	8.62	0.089	23.3	6.21		30.0	
Effluent Avg	**755**	**497**	**8.48**	**0.097**	**23.2**	**5.72**	**122**		

LAB ANALYSES Effluent Coliform dilution = 500 Feed Coliform dilution = 1000

FEED	Coliforms (mL-1)	Total Cl+1 (mg/L)	COD (mg/L)	TSS (mg/L)	Al3+ (mg/L)	Sample Averages and Std. Deviations Coliforms	COD	TSS	Al^{3+}
Influent A	35	0.05	64.0	12	0.01		**INFLUENT**		
Influent B	36	0.04	60.8		0.01	37.7	60.8	11.5	0.01
Influent C	42	0.04	57.5		0.01	3.8	3.25		0.00
SAMPLE A									
Effluent 1	14	0.36	28.0	1849	930.0		**EFFLUENT #1**		
Effluent 2	9	0.44	26.0		supern't	13.7	27.0	1849.0	930
Effluent 3	18				15.0	4.5	1.41		
SAMPLE B									
Effluent 1	8	0.42	29.0	2040	829.0		**EFFLUENT #2**		
Effluent 2	11	0.36	19.0		supern't	10.3	24.0	2040.0	829
Effluent 3	12				20.0	2.1	7.07		
SAMPLE C									
Effluent 1	6	0.50	44.0	2032	869.4		**EFFLUENT #3**		
Effluent 2	6	0.52	38.0		supern't	7.7	41.0	2032.0	869
Effluent 3	11				25.0	2.9	4.24		

Test Averages	**Influent** Average	Std. Dev.	**Effluent** Average	Std. Dev.	**Change** Δ	**Removal** (%)	**Log Removal**	Feed stdv.
Coliforms (L-1)	37.7	3.8	10.6	3.0	-27.1	72.0		10.1%
Total Chlorine (mg/L)	0.043	0.003	0.433	0.271	0.39	-919.6		
COD (mg/L)	60.8	3.3	30.7	9.1	-30.1	49.5		
TSS (mg/L)	11.5	0.0	1974	108.0	1962	(17,062)		
Al^{3+} (mg/L)	0.0	0.0	876.1	50.8	876.1	-9.E+06		
Conductivity (µS)	1054	3.5	755	12.4	-298	28.3		
TDS (mg/L	696	3.5	497	8.1	-198	28.5		
pH	7.02	0.07	8.48	0.15	1.5			
ORP (mV)(*vs NHE)	385.2	4.66	290.5	8.22	-94.8			
Temperature (°C)	19.9	0.00	23.20	0.10	3.3			Eff. Stdv.
Dissolved Oxygen (mg/L)	5.695		5.72		0.0			
Actual Coliform Counts: (per 100 mL)	3.77E+06	########	5.28E+05	1.50E+05	-3.24E+06		0.85	28.48%

RUN # 11 Date: March 15, 2005 (Repeat #3)

OPERATION PARAMETERS:

Contact Time, minutes	10
Sample Volume, mL	850
Current Density (mA/cm2)	3.5
Cl- Concentration (mg/L)	580

Power Consumption

Volts :	3.8
Amps :	6.4
kWh/m3 :	4.77

Solids Production Rate

0.002522 **tonne/m^3**

FEED	Conduct. (mS)	TDS (mg/l)	pH	ORP (V)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C)	Evapr'n Loss (%)
Influent A	2720	1797	7.19	0.174	20.0	5.83	580	21.9	-9.43
Influent B	2660	1767	7.39	0.162	20.5	5.90	580	22.0	
Influent avg.	2690	1782	7.29	0.168	20.3	5.87	580	22.1	**ΔT,°C**
EFFLUENT									**3.8**
Effluent A	2370	1561	8.77	0.081	22.6	5.36	555.0	25.6	
Effluent B	2380	1565	8.90	0.073	22.8	5.41	547.5	25.9	
Effluent C	2400	1577	8.92	0.072	22.7	4.94	487.5	26.0	
Effluent Avg	**2383**	**1568**	**8.86**	**0.075**	**22.7**	**5.24**	**530**		

LAB ANALYSES Effluent Coliform dilution = 500 Feed Coliform dilution = 1000

FEED	Coliforms (mL-1)	Total Cl+1 (mg/L)	COD (mg/L)	TSS (mg/L)	Al3+ (mg/L)	Sample Averages and Std. Deviations Coliforms	COD	TSS	Al^{3+}
Influent A	36	0.06	58.0	10	0.01		**INFLUENT**		
Influent B	43	0.05	58.3		0.01	40.7	58.3	9.5	0.01
Influent C	43	0.04	58.5		0.01	4.0	0.25		0.00
SAMPLE A									
Effluent 1	14	0.26	15.0	2400	881.0		**EFFLUENT #1**		
Effluent 2	11	0.42	17.0		supern't	15.7	16.0	2400.0	881.0
Effluent 3	22				<8	5.7	1.41		
SAMPLE B									
Effluent 1	13	0.30	12.0	2581	801.0		**EFFLUENT #2**		
Effluent 2	12	0.48	16.0		supern't	12.7	14.0	2581.0	801.0
Effluent 3	13				<8	0.6	2.83		
SAMPLE C									
Effluent 1	9	0.36	13.0	2613	741.0		**EFFLUENT #3**		
Effluent 2	17	0.64	13.0		supern't	14.3	13.0	2613.0	741.0
Effluent 3	17				<8	4.6	0.00		

Test Averages	**Influent** Average	Std. Dev.	**Effluent** Average	Std. Dev.	**Change** Δ	**Removal** (%)	**Log Removal**	Feed stdv.
Coliforms (L-1)	40.7	4.0	14.2	1.5	-26.4	65.0		9.9%
Total Chlorine (mg/L)	0.048	0.008	0.410	0.138	0.36	-763.2		
COD (mg/L)	58.3	0.3	14.3	1.5	-43.9	75.4		
TSS (mg/L)	9.5	0.0	2531	114.9	2522	(26,546)		
Al^{3+} (mg/L)	0.0	0.0	807.7	70.2	807.7	-8.E+06		
Conductivity (µS)	2690	42.4	2383	15.3	-307	11.4		
TDS (mg/L	1782	15.0	1568	8.3	-214	12.0		
pH	7.29	0.10	8.86	0.08	1.6			
ORP (mV)(*vs NHE)	369.5	6.05	273.5	5.01	-96.0			
Temperature (°C)	20.25	0.25	22.70	0.10	2.5			Eff. Stdv.
Dissolved Oxygen (mg/L)	5.865		5.24		-0.6			
Actual Coliform Counts: (per 100 mL)	4.07E+06	#######	7.11E+05	7.52E+04	-3.36E+06		0.76	10.57%

RUN # 12 Date: March 22, 2005 (Repeat #2)

OPERATION PARAMETERS:

Contact Time, minutes	15
Sample Volume, mL	850
Current Density (mA/cm^2)	5.5
Cl- Concentration (mg/L)	161.75

Power Consumption	
Volts :	13.0
Amps :	10.06
kWh/m3 :	38.46

Solids Production Rate

0.005716 **tonne/m^3**

FEED	Conductivity (mS)	TDS (mg/l)	pH	ORP (V)	Temp (oC)	D.O. (mg/L)	Chlorides (mg/L)	Temp. (°C)	Evapr'n Loss (%)
Influent A	1239	819	7.06	0.178	21.5	4.91	162	23.2	19.53
Influent B	1237	817	7.11	0.175	22.0	4.55	161.5	25.7	
Influent avg.	1238	818	7.09	0.177	21.8	4.73	161.75	25.8	**ΔT,°C 18.6**
EFFLUENT									
Effluent A	792	516	8.99	0.062	27.3	2.82	191.0	43.0	
Effluent B	823	538	8.96	0.063	27.2	2.17	212.5	43.8	
Effluent C	786	518	9.01	0.059	28.2	2.47	199.5	43.8	
Effluent Avg	**800**	**524**	**8.99**	**0.061**	**27.6**	**2.49**	**201**		

LAB ANALYSES Effluent Coliform dilution = 200 Feed Coliform dilution = 1000

FEED	Coliforms (mL-1)	Total Cl^{+1} (mg/L)	COD (mg/L)	TSS (mg/L)	Al^{3+} (mg/L)	**Sample Averages and Std. Deviations** **Coliforms**	**COD**	**TSS**	**Al^{3+}**
Influent A	36	0.04	54.3	7.0	0		**INFLUENT**		
Influent B	37	0.04	55.8		0.00	36.7	55.8	7.0	0.00
Influent C	37	0.04	57.3		0	0.6	1.50		0.00
SAMPLE A									
Effluent 1	6	0.92	14.0	5648	1628.0		**EFFLUENT #1**		
Effluent 2	4	0.68	15.0		supern't	5.7	14.5	5648.0	1628.0
Effluent 3	7				17.0	1.5	0.71		
SAMPLE B									
Effluent 1	1	0.52	11.0	5830	1547.0		**EFFLUENT #2**		
Effluent 2	1	0.28	13.0		supern't	1.0	12.0	5830.0	1547.0
Effluent 3	1				17.0	0.0	1.41		
SAMPLE C									
Effluent 1	1	0.72	11.0	5691	1467.0		**EFFLUENT #3**		
Effluent 2	4	0.76	15.0		supern't	2.3	13.0	5691.0	1467.0
Effluent 3	2				17.0	1.5	2.83		

Test Averages	**Influent** Average	Std. Dev.	**Effluent** Average	Std. Dev.	**Change** Δ	**Removal** (%)	**Log Removal**	Feed stdv.
Coliforms (L-1)	36.7	0.6	3.00	2.4	-33.7	91.8		1.6%
Total Chlorine (mg/L)	0.035	0.000	0.647	0.221	0.61	-1747.6		
COD (mg/L)	55.8	1.5	13.2	1.3	-42.6	76.4		
TSS (mg/L)	7.0	0.0	5723	95.1	5716	(81,657)		
Al^{3+} (mg/L)	0.0	0.0	1547.3	80.5	1547.3	#DIV/0!		
Conductivity (µS)	1238	1.4	800	19.9	-438	35.4		
TDS (mg/L	818	1.0	524	12.2	-294	35.9		
pH	7.09	0.03	8.99	0.03	1.9			
ORP (mV)(*vs NHE)	375.6	2.86	241.6	1.77	-134.0			
Temperature (°C)	21.75	0.25	27.57	0.55	5.8			Eff. Stdv.
Dissolved Oxygen (mg/L)	4.73		2.49		-2.2			
Actual Coliform Counts: (per 100 mL)	3.67E+06	########	6.00E+04	4.81E+04	-3.61E+06		1.79	80.12%
					Log removal = 2.22 without Effluent A			

DATA SUMMARY TABLE Part 1

Test #	Detention Time (min)	Current Density (mA/cm^2)	Total Current] (Amps)	Log Removal	Power Consumption ($kW\text{-}h/m^3$)	Voltage (V)	ΔTemp (°C)	Cl^{-1} (mg/L)	TSS Al conc. (wt.%)	Effluent TSS (mg/L)	Effluent Al Conc. (mg/L)	Solids Rate (t/m^3)
1	5	5.5	10.1	**0.61**	13.81	14	7.2	139	42.2	1676	678	0.00167
2	15	5.5	10.1	**2.18**	41.42	14	21.0	137	17.5	6543	1145	0.00654
3	10	3.5	6.4	**0.97**	5.02	4	10.7	583	40.1	2919	1023	0.00290
4	5	1.5	2.7	**0.31**	1.07	4	1.9	136	49.8	363	167	0.00034
5	15	1.5	2.7	**0.62**	3.63	5	2.6	129	37.6	1110	409	0.00109
6	5	5.5	10.1	**0.90**	3.75	4	3.0	868	35.2	2287	801	0.00228
7	5	1.5	2.7	**0.69**	0.54	2	0.6	1005	41.4	484	194	0.00047
8	15	1.5	2.7	**0.68**	1.61	2	1.9	1010	39.7	1409	551	0.00139
9	15	5.5	10.1	**2.14**	10.93	3.7	7.5	1075	**54.1**	5915	3197	0.00590
10 (1r)	5	5.5	10.1	**0.85**	13.81	14	8.1	123	44.8	1974	876	0.00196
11 (3r)	10	3.5	6.4	**0.76**	4.77	4	3.8	580	32.2	2531	808	0.00252
12 (2r)	15	5.5	10.1	**1.79**	38.46	13	18.6	162	27.1	5723	1547	0.00572

DATA SUMMARY TABLE continued, Part 2

		FEED		EFFLUENT								
Test #	Feed TSS	Feed Coliforms	Coliforms/ mL	Effluent Coliforms	Coliforms/ mL	Settling Rate	Clarifier Area	Solids Production	Solids Production	Effluent pH	pH Rise	D.O. Change
	(mg/L)					(m/min)	(m^2)	(tonne/day)	(m^3/day)			(mg/L)
1	8.0	41	20,667	10.1	5056	4.02E-04	1.31E+06	631	263	8.56	1.62	-3.2
2	4.0	34	16,833	0.2	111	**Floated**	#VALUE!	2,475	1031	8.78	1.85	-6.0
3	21.0	106	52,833	11.4	5722	2.65E-04	1.98E+06	1,097	457	8.99	1.55	-5.3
4	26.0	53	53,000	51.6	25778	7.40E-03	7.10E+04	127	53	7.57	0.54	-1.2
5	18.0	65	65,333	63.2	15806	4.82E-03	1.09E+05	413	172	8.36	1.34	-0.9
6	9.0	71	71,333	17.9	8944	3.75E-03	1.40E+05	862	359	8.75	1.48	-3.5
7	14.0	47	47,333	19.2	9611	1.94E-02	2.71E+04	178	74	8.18	0.77	-0.7
8	20.0	48	47,667	20.0	10000	1.87E-03	2.81E+05	526	219	8.75	1.44	-0.6
9	15.0	52	52,000	1.9	378	**Floated**	#VALUE!	2,233	931	9.08	1.82	-2.5
10 (1r)	11.5	38	37,667	10.6	5278	4.00E-04	1.31E+06	743	309	8.48	1.46	0.03
11 (3r)	9.5	41	40,667	14.2	7111	2.69E-04	1.95E+06	955	398	8.86	1.57	-0.6
12 (2r)	7.0	37	36,667	3.0	600	7.80E-05	6.74E+06	2,164	901	8.99	1.90	-2.2

DATA SUMMARY TABLE continued, Part 3

Test #	COD Reduction (mg/L)	Theor. Al dissolved (mg/L)	Operatiion Time	CD^2*t	1-N/No	I^2*t	% COD Removal	I*E*t	g per coulomb	Feed Cl_2 (mg/L)	Effluent Cl_2 (mg/L)	Δ Cl_2 (mg/L)	Al vs Theor.
1		332.5	15	151.25	0.755	561.1		707	45.1	0.045	0.389	0.344	2.04
2	28.4	997.4	45	453.75	0.993	561.1	61.4	2121.0	59.0	0.025	0.942	0.917	1.15
3	35.9	421.3	30	122.5	0.892	143.4	60.4	256.0	61.9	0.05	0.833	0.783	2.43
4	59.3	88.9	15	11.25	0.514	10.9	67.4	54.0	34.1	0.0275	0.343	0.316	1.88
5	82.3	266.6	45	33.75	0.758	10.9	78.5	202.5	36.9	0.0325	0.573	0.541	1.53
6	36.3	332.5	15	151.25	0.875	561.1	62.2	202.0	61.7	0.045	0.470	0.425	2.41
7	43.0	88.9	15	11.25	0.797	10.9	59.3	27.0	47.6	0.0525	0.390	0.338	2.18
8	52.5	266.6	45	33.75	0.790	10.9	65.6	81.0	46.9	0.0425	0.193	0.151	2.07
9	42.8	997.4	45	453.75	0.993	561.1	65.9	560.6	53.2	0.03	1.033	1.003	3.21
10 (1r)	30.1	332.5	15	151.25	0.860	561.1	49.5	707.0	53.1	0.0425	0.433	0.391	2.64
11 (3r)	43.9	421.3	30	122.5	0.825	143.4	75.4	256.0	53.9	0.0475	0.410	0.363	1.92
12 (2r)	42.6	997.4	45	453.75	0.984	561.1	76.4	1969.5	51.6	0.035	0.647	0.612	1.55

Statistical Analyses Output from DesignEase

Response: Log Coliform Removal
ANOVA for Selected Factorial Model Analysis of variance table [Partial sum of squares]

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	4.09	3	1.36	40.45	< 0.0001	significant
A	*1.68*	*1*	*1.68*	*49.79*	*0.0002*	
B	*1.18*	*1*	*1.18*	*34.85*	*0.0006*	
AB	*0.73*	*1*	*0.73*	*21.52*	*0.0024*	
Residual	0.24	7	0.034			
Cor Total	4.36	11				

The Model F-value of 40.45 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.

Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, AB are significant model terms.

Std. Dev.	0.18	R-Squared	0.9455
Mean	1.04	Adj R-Squared	0.9221
C.V.	17.63	Pred R-Squared	0.8395

Factor	Coefficient Estimate	DF	Standard Error	95% CI Low	95% CI High
Intercept	0.99	1	0.059	0.85	1.13
A-Current Density	0.42	1	0.059	0.28	0.56
B-Time	0.35	1	0.059	0.21	0.49
AB	0.28	1	0.059	0.13	0.42
Center Point	-0.13	1	0.14	-0.47	0.21

Final Equation in Terms of Actual Factors:

Log Coliform Removal = 0.52375 - (0.065833 * Current Density) - (0.026250 * Time)
+ (0.027500 * Current Density * Time)

Response: Sus. SolidsTransform: Natural logConstant: 0
ANOVA for Selected Factorial Model Analysis of variance table [Partial sum of squares]

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	8.70	2	4.35	250.85	< 0.0001	significant
A	*5.84*	*1*	*5.84*	*336.97*	*< 0.0001*	
B	*3.09*	*1*	*3.09*	*178.47*	*< 0.0001*	
Curvature	0.49	1	0.49	28.12	0.0007	significant
Residual	0.14	8	0.017			
Lack of Fit	*0.11*	*5*	*0.021*	*1.96*	*0.3077*	*not significant*
Pure Error	*0.033*	*3*	*0.011*			
Cor Total	9.33	11				

The Model F-value of 250.85 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.

Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The "Curvature F-value" of 28.12 implies there is significant curvature (as measured by difference between the average of the center points and the average of the factorial points) in the design space. There is only a 0.07% chance that a "Curvature F-value" this large could occur due to noise.

The "Lack of Fit F-value" of 1.96 implies the Lack of Fit is not significant relative to the pure error. There is a 30.77% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Std. Dev.	0.13	R-Squared	0.9843
Mean	7.59	Adj R-Squared	0.9804
C.V.	1.74	Pred R-Squared	0.9662

Factor	Coefficient Estimate	DF	Standard Error	95% CI Low	95% CI High
Intercept	7.36	1	0.042	7.27	7.46
A-Current Density	0.78	1	0.042	0.68	0.88
B-Time	0.56	1	0.042	0.46	0.65
Center Point	0.54	1	0.10	0.31	0.78

Final Equation in Terms of Actual Factors:

Ln(Sus. Solids) = 4.887 +0.39007 * Current Density +0.11126 * Time

APPENDIX 4

X-Ray Diffraction of Precipitate

Prepared by

Paul Schilling
Dileep Simhadri
Department of Mechanical Engineering
University of New Orleans
New Orleans, LA 70148
Phone: (504)280-6187 Fax: (504)280-5539

February 23, 2004

Prepared for

Mark Peterson
Enrique La Motta
Department of Civil and Environmental Engineering
University of New Orleans
New Orleans, LA 70148

Summary

X-ray diffraction (XRD) measurements were performed on material precipitated from a municipal solid waste leachate formed by dissolving aluminum in an electrocoagulation cell in which the dissolved aluminum pulls metal ions out of solution along with other species. In the XRD pattern, a large number of sharp peaks due to crystalline phases are observed, in addition to several broad features indicating the presence of an amorphous (glassy) material. A search-match routine was used to provide possible identifications of the crystalline phases. Phases identified include two aluminum hydroxides – Bayerite [$Al(OH)_3$] and Nordstrandite [$Al(OH)_3$]. A possible third phase is an iron-rich phase – Giniite [$Fe_5(PO_4)_4(OH)_3{\cdot}2H_2O$] – but this is based only on matching the most intense peak of Giniite. These matches are summarized in Table 1 and Figure 1. The matches are presented graphically in the 'Results' section and the JCPDS data for these three phases are presented in the appendix. The file Al_Al2O3_Fe.csv contains the data. This file can be read using Microsoft Excel.

Table 1. Identified Patterns List

Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
18-0050	59	Nordstrandite	0.706	$Al(OH)_3$
45-1436	33	Giniite, Fe-rich, syn	0.155	$Fe_5(PO_4)_4\ (OH\ )_3\ {\cdot}2\ H_2O$
74-1119	62	Bayerite	0.980	$Al(OH)_3$

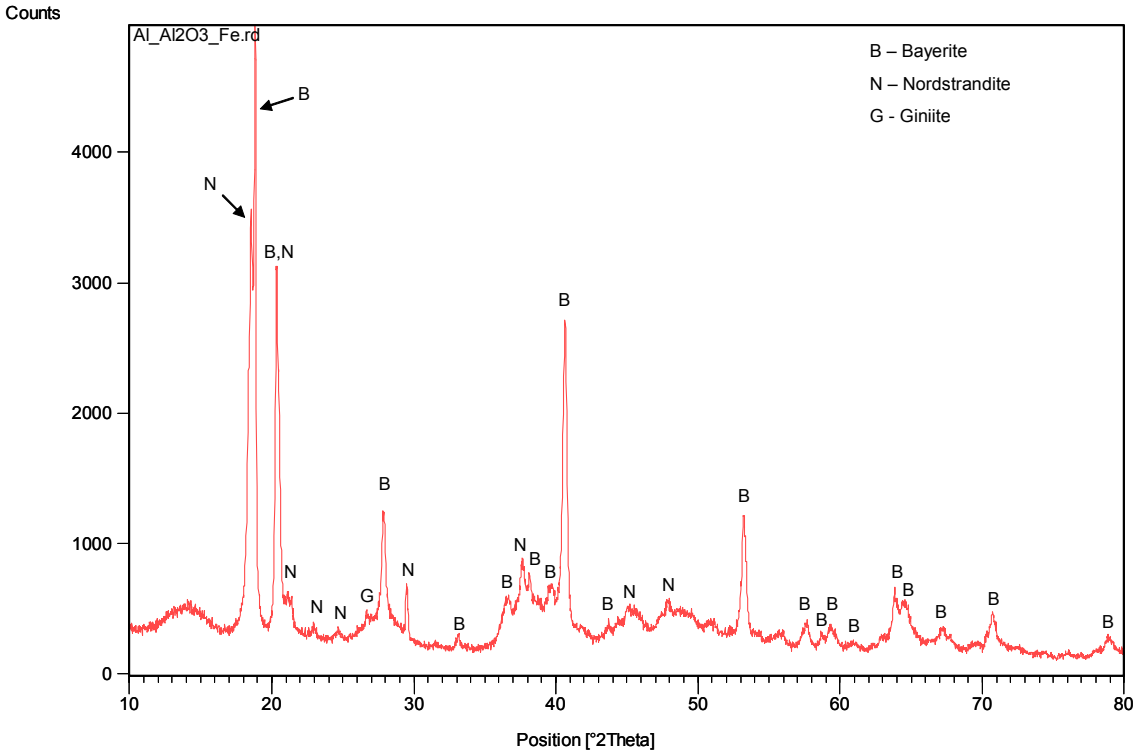

Figure 1. Plot of identified phases.

VITA

Mark Peterson was born in Anaconda, Montana near the westernmost headwaters of the Mississippi River. He received a B.S. degree in Metallurgical Engineering at Montana Tech in 1978. Following 3 years working for American Metals Climax in Colorado he returned for an M.S degree in 1985 working on arsenic removal from lead smelter waste. He worked for the US Bureau of Mines Salt Lake Research Center for 10 years publishing papers in column flotation and bioextraction of metals from acid mine drainage, until the abolition of the agency during the partisan political bickering called the "Contract for America." While working as a process engineer for EIMCO Process Equipment Company in Salt Lake City, he married Leslie Monahan. Following the buyout and reduction of EIMCO's U.S. facilities by the Canadian engineering company Groupe Laperrière and Verreault following NAFTA, he moved to New Orleans and pursued a degree in Environmental Engineering focusing on water treatment and management studies. Outsourced by American politics from the western highlands to the very edge of the continent, he continues to pursue improvements in water treatment and conservation.