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Electro-Disinfection of Municipal Waste Water using Direct Current

A Thesis

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

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In
Engineering (Environmental Engineering)

Submitted by

Julio A. Acosta

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Thanks to my parents, who’s emotional and financial support allowed to me achieve this academic goal.

To my friends, either close or far away always reminded me of my intellectual potential and capacity.

A special thank to Dr. Enrique La Motta, whose support as a professor and mentor made this paper and my graduate education possible. Thanks to him and his broad knowledge and excellent teaching techniques I discover my interest in this specific field of civil engineering.

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List of acronyms

EPA	Environmental Protection Agency
CWA	Clean Water Act
NPDES	National Pollutant Discharge Elimination System
DEQ	Department of Environmental Quality
LDEQ	Louisiana Department of Environmental Quality
BOD5	The Five-day Measure of the Biochemical Oxygen Demand
TSS	Total Suspended Solids
E. coli	Escherichia coli
TCR	Total Coliform Rule
CDC	Center for Conventional Disease Control and Prevention
PVC	Polyvinyl Chlorine
CERM	Center for Energy Resource Management
USEPA	United States Environmental Protection Agency
TRC	Total Residual Chlorine

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Abstract

Wastewater treatment has always been a problem to human settlers. Events such as the great stink of London during the summer of 1858 have pushed engineers into developing new technologies to deal with such wastes in effective and safe ways. Research projects like this aim to find ways of improving performance, economics, or environmental friendliness of treatment and disinfection methods.

This thesis deals with the effectiveness, mechanisms of action, by-products and side effects of using DC current to disinfect secondary effluent. It is proposed as an alternative to conventional methods, such as chlorination, which are not always environmentally friendly or feasible.

It was demonstrated that DC current efficiently kills pathogens. DC properly used achieves disinfection significantly higher than the minimum required by the EPA. Nevertheless disinfection by products such as residual chlorine should be taken into account when considering this technology as an alternative to conventional chlorination.

Keywords: Electrodisinfection, Waste Water, Direct Current, Electrochemical disinfection

Introduction

Disinfection is a crucial process in municipal wastewater. Conventional methods include chlorination using different compounds like chlorine gas, hipochlorous acid, sodium hypochlorite and chloramines, each one of them with a different dosage necessary to provide proper disinfection and achieve a maximum allowable coliform count of 200 CFU/ 100ml of water. These methods have proven to be effective over many decades and are an effective way to not only kill pathogens but to also to provide water with residual disinfection that protects from later contamination (Kraft, 2008). This residual protecting quality however might also be a concern due to possible by products that could be carcinogenic and must be regulated. (USEPA, 1999)

Other methods like ozonation and ultraviolet light also prove to be effective but they lack the residual protecting effect of chlorination. These are limited to disinfect the water in that moment and then discharge it.

Electrodisinfection is a viable yet not commonly used method to get rid of pathogens. The lack of popularity for this method is due to the fact that the materials necessary to make electrodes that would not easily corrode had not been commercially available until the early 2000’s. Hence not much research has been done on these topics and not many products of this type are available in the market. (Kraft 2008)

Dimensionally stable anodes (DSA) such as titanium coated with low reactive oxides like Iridium seem to be the most efficient material available now days to use as electrodes for reactors of this nature.

The main, but not only mechanism for disinfection is the generation of Chlorine, Fortunately Chlorate and Perchlorate are not formed at DSA® type and Pt electrodes given that this electrodes' material low over potential. (Kraft,1999).

In order to determine the importance of chlorine in the disinfection using this method, concentrations of the total residual chlorine will be compared to those of conventional chlorination methods.

Literature Review

For any given activity involving water and wastewater treatment, application of discharge regulations must be implemented. NPDES must be attained which are federally enforced by the EPA and each state's DEQ must enforce its own PDES which in the case of Louisiana would be the LPDES that must be at least as strict as NPDES.

The Clean Water Act sets goals for a body of water based on its intended use. Criteria are set to keep the water body safe for such uses. The quality standards for this consist of the following elements: (USEPA, 2012)

1. Use designated to the water body
2. Quantifiable pollutant maximum allowances and narrative requirements
3. A policy to maintain such quality.
4. Implementation and enforcing policies for example low flows, variances and mixing zones

The clean Water act (CWA) regulates the discharge of pollutants into surface water bodies and its quality for the United States. Originally named Federal Water Pollution Control Act, was significantly expanded upon in 1972 with several amendments and renamed "Clean Water act"

Under CWA regulations for both industrial and municipal water were established. No point source discharge can be released into any navigable water unless a permit is obtained. (USEPA, 2012)

Point sources are discrete conveyances such as pipes or man-made ditches. Individual residences not connected to a municipal system, use a septic system, or discharging into surface waters do not need an NPDES permit. Minicupal discharges do require an NPDES permit as administrated by states. (USEPA, 2012)

Permits can be general or individual; General permits are easier and faster to obtain but have more stringent regulations. Most of the time if chlorination is used for disinfection an Individual permit must be obtained. Individual permits require a deeper analysis of the water in which water is going to be discharged and the effluent characteristics (LADEQ, 2007)

Title 33, Part IX of EPA determines the procedures, frequency of tests, and documentation required to maintain a permit. The National Pollutant Discharge Elimination System Permit regulates wastewater treatment in Louisiana under the permit number LA0038091. This defines limits to which municipal wastewater has to be treated before discharging into the Mississippi River (Pulido, 2005).

For the permit to be granted to each wastewater treatment plant to discharge its effluent into any body of water, such effluent must comply with all NPDES criteria, such as BOD,TSS, fecal coliform, pH, solids and foam, and total residual Chlorine. Table 1 below shows some of these criteria applicable to the Mississippi River.

Table 1- NPDES Permit Summary (Cagle, 2012)

Parameter	Weekly	Monthly
BOD_5	45 mg/l	30 mg/l
TSS	45 mg/l	30 mg/l
Fecal Coliform	400 MPN/100 ml	200 MPN/100 ml
Escherichia Coli	235 cfu/100 ml (one dose)	200 MPN/100 ml (30 day rolling)
pH	Between 6 and 9	Between 6 and 9
Total Residual Chlorine	0.05 mg/l	0.05 mg/l
Other requirements	No floating solids or visible foam	No floating solids or visible foam

- **BOD_5:** The five-day measure of the biochemical oxygen demand.
- **Total Suspended Solids (TSS):** The amount of solid material suspended in water, commonly expressed as a concentration in terms of mg/L.
- **pH:** Measure of acidity of an aqueous solution.
- **Fecal Coliform:** A gram negative, non-spore-forming, rod-shaped bacteria found in the intestinal tract of warm-blooded animals.

Bacteria Quantification:

Waterborne and water-related diseases are among the most serious threats for human health. The most common waterborne disease is diarrhea which causes excessive dehydration, caused by manly by bacteria such as: Shigella dysenteriae, Escherichia Coli, Salmonella typhi and Campylobacter, viruses and parasites: Entamoeba histolytica or protozoa: Giardia lamblia and Cryptosporidium, worms and rotifers usually spread by the presence of feces.

Shigella Dysenteriae: Any rod-shaped bacteria that make up the genus *Shigella*, normal inhabitants of the human intestinal tract and can cause dysentery, or shigellosis. Shigellae are gram-negative, non-spore-forming, stationary bacteria.

S. Dysenteriae, spread by contamination of water and food sources, causes the most severe dysentery due to its potent toxin, but other species may also cause dysentery.

Salmonella Typhi: Pathogen usually located in the lymphatic tissues of the small intestine, spleen, liver and bloodstream of infected humans. This pathogen is common in developing countries. Infection of S. Typhi is a major cause of of typhoid and enteric fever. "(Pollack, 2003)

"Campylobacter: Campylobacteriosis is an infectious disease caused by bacteria of the genus Campylobacter. Campylobacter occasionally spreads to the bloodstream and causes a serious life-threatening infection. Campylobacter organisms are spiral-shaped bacteria that can cause disease in humans and animals". (CDC, 2013)

"Entamoeba Histolytica: Is an anaerobic parasitic protozoan that infects the digestive tract of predominantly primates including humans.

"Giardia Lamblia: Also known as Giardia intestinalis or Giardia duodenalis, is a parasite often found in food or water that has been contaminated with feces. This parasite has an outer shell that protects it from chlorine disinfection and allows it to survive for long periods of time outside the body"". (CDC, 2011)

Cryptosporidium: Similar structure and effects as Giardia Lamblia

Escherichia Coli (E. Coli): Is one of several types of bacteria that normally inhabit the intestine of humans and animals. Some strains of *E. coli* are capable of causing disease under certain conditions when the immune system is compromised or disease may result from an environmental exposure. This bacterium has been used as a biological indicator since 1890. (Shanson, 1999)

For the purpose of this research E.coli was used as an indicator of the degree of pathogen contamination and disinfection in the samples. The method is later described in detail and it is the following:

“Method 1603 describes a membrane filter (MF) procedure for the detection and enumeration of Escherichia coli bacteria in ambient waters and disinfected wastewaters. This method is a single-step modification of EPA Method 1103.1 (mTEC). Unlike the mTEC media method, it does not require the transfer of the membrane filter to another substrate. The modified medium contains a chromogen (5-bromo-6-chloro-3-indolyl-Beta-D-glucuronide), which is catabolized to glucuronic acid and a red- or magenta-colored compound by E. coli that produces the enzyme Beta-D-glucuronidase. The apparatus and equipment, and sampling, filtration, and verification procedures for the modified mTEC method are identical to those of the original mTEC method. E. coli is a common inhabitant of the intestinal tract of warm-blooded animals, and its presence in water samples is an indication of fecal pollution and the possible presence of enteric pathogens. The E. coli test is recommended as a measure of ambient recreational fresh water quality. Epidemiological studies have led to the development of criteria, which can be used to promulgate recreational water standards based on established relationships between health effects and water quality. The significance of finding E. coli in recreational fresh water samples is the direct relationship between the density of E. coli and the risk of gastrointestinal illness associated with swimming in the water. “(EPA, 2009)

Conventional wastewater treatment:

Conventional waste water treatment plants consist of a set of defined units. First a pretreatment is applied which consists of a bar rack or some other type of screening to remove all coarse solid material.

Water leaves pretreatment and passes into a settling unit. Settleable solids are removed on a primary clarifier by plain settling. After primary settling, water enters the biological treatment. Biological treatment can take a variety of forms:

- Activated sludge process
- Extended aeration process
- Anaerobic then aerobic digestion
- Trickling filters
- Other biofilm mechanisms
- A combination on several methods

After biological flocculation occurs in the selected biological process by means of polymers excreted by the aerobic bacteria water enters a secondary clarifier in which further settling and solids removal take place. This process ideally takes care of most of the NPDES requirements for water disposal like BOD, TSS, and COD under the maximum allowable limits. (Metcalf and Eddy, 2013)

It is here where disinfection is needed to finally make water safe for disposal into natural streams by pathogen removal. E coli which is the indicator to be used in this research would need to be reduced to 200 CFU/ 100ml

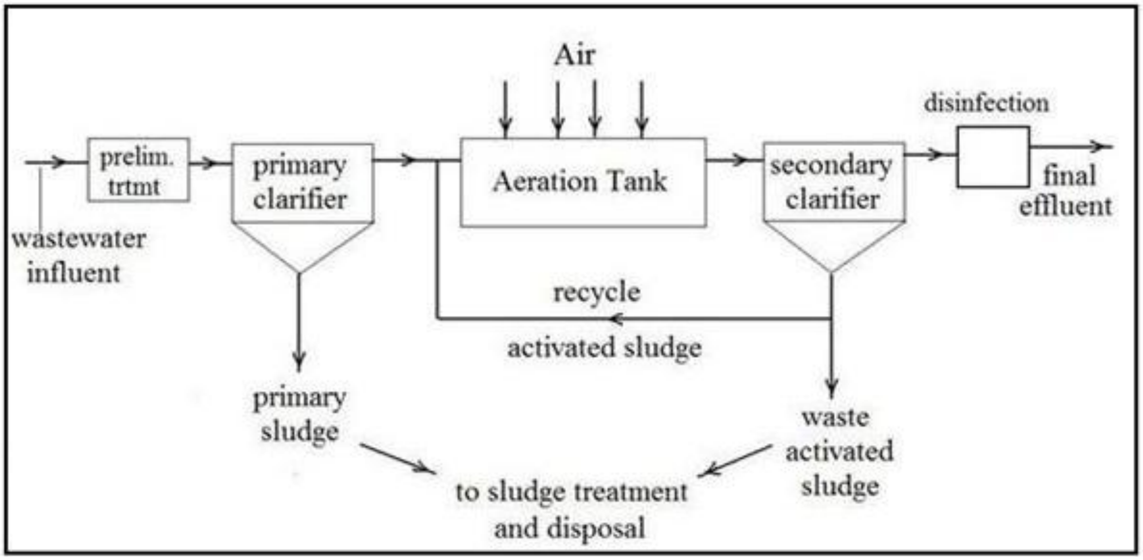

Figure 1-Wastewater Treatment Process flow diagram (Metcalf, 2013)

Once chlorine is added to the wastewater, two reactions take place: hydrolysis and ionization.

Hydrolysis occurs as follows

$$Cl2 + H2O \rightarrow HOCl + H+ + Cl-$$

Chlorine gas is mixed with water to form hypochlorus acid (HOCl).

Then hypochlorus acid is ionized to form a hypochlorite ion (OCl-):

$$HOCl \rightarrow OCl- + H$$

The relative proportion between HOCl and OCl- is something to be aware of because the killing of HOCl is up to 80 times more efficient than OCl- (Metcalf, 2013).

Chlorination mechanism of action is as follows:

- Direct oxidation of cell wall allowing cellular constituents to flow outside of the cell
- Modifies cell wall permeability
- Alteration of cell protoplasm
- Inhibition of enzyme activity
- Damage to the cell DNA and RNA (Metcalf and Eddy, 2013)

The effectiveness of chlorination is not in doubt but it has several drawbacks:

- Highly toxic, has to be transported, prone to accidents
- Poses health risks to treatment plan operators and public if accidental contact occurs.
- Stringent requirements for containment and neutralization , Uniform Fire Code.
- Reacts with organic constituents to produce odorous compounds and byproducts.
- Residual chlorine is toxic to aquatic life
- Long term effects of chloro-organic compounds are unknown. (Metcalf and Eddy,2013)

Dechlorination is the process of removing total and free residual chlorine residuals from water in order to reduce its toxicity due to chlorination and before being into natural water bodies. NPDES permits requires that the amount of residual chlorine in the discharged water to be “non-detectable”, therefore, dechlorination must be applied. Currently there are a few chemicals commonly use to address this problem such as: Sodium bisulfate, sulfur dioxide, sodium metabisulfite and activated carbon.

Sulfur dioxide is the commonly used option when dealing with residual chlorine from wastewater due to the high cost for activated carbon. This type of dechlorination dissolves the sulfur dioxide to form ionic sulfur in the S (IV) state, such as SO3-2. This causes a reduction of residual chlorine within minutes. However, too much sulfite addition can be detrimental to the environment if not controlled, a decrease in pH, lowered dissolved oxygen and health and safety hazards are among the most common threats (Cagle, 2012)

Research performed at the University of New Orleans showed that under similar circumstances but using alternating current free chlorine was generated, yet chlorine at these concentrations was not capable of achieving high disinfections. “Chlorine in concentrations of 0.152mg/L and 0.342mg/L, free and total respectively were generated yet chlorine alone was not enough to generate sufficient disinfection.” (Chavez 2014)

Electrodisinfection:

Electrolysis is the process through wich electrical energy is added to a salution in order to induce a chemical reaction that wouldn't take place otherwise. Regarding disinfectants chlorine is an important one generated while during electrolysis therefore the chemical equations between chlorine and hipochlorous acid and hipochloric acide are the same as with conventional chlorination.

The only difference is that chlorine is not mechanically added but chemically produced by the anode while supplying the electrodes for the reaction summarizing the whole process:

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

$$Cl_2 + H_2O \rightarrow HClO + HCl$$

$$HClO \leftrightarrows ClO^- + H^+$$

$$HClO \rightarrow O + Cl^- + H^+$$

$$ClO^- \rightarrow O + Cl^-$$

At the same time the electrolysis with these highly stable electrodes will generate other oxidative species like ozone and hydrogen peroxide that will aid in the disinfection.(Kraft 2008)

$$3H2O \rightarrow O3 + 6e– + 6H+$$

$$O2 + 2H2O + 2e– \rightarrow H2O2 + 2OH–$$

Most of the disinfectants might be synthesized at the anode; hydrogen peroxide may also be produced at the cathode if its synthesis is achieved. This process has been used by (Dhar *et al*. 1981) and (Drogui *et al*.2001) for water disinfection.

One important product of certain types of electrochemical reactors is Hydroxyl radicals (ROS). Some technologies have been developed specifically to avoid hydrogen production and focus on Hydroxyl radicals under the following equation. (Kraft, 2008)

$$O2 + 2H2O + 4e^{-} \rightarrow 4OH^{-}$$

However this hasn't been tested for this specific type of reactor, since testing for highly oxidative species like hydroxyl radicals is very hard to achieve due to their almost immediate reaction with surrounding compounds.

The generation of chlorine and other oxidative species depends highly on the material used for the electrodes. Figure 2 shows different chlorine production efficiencies.

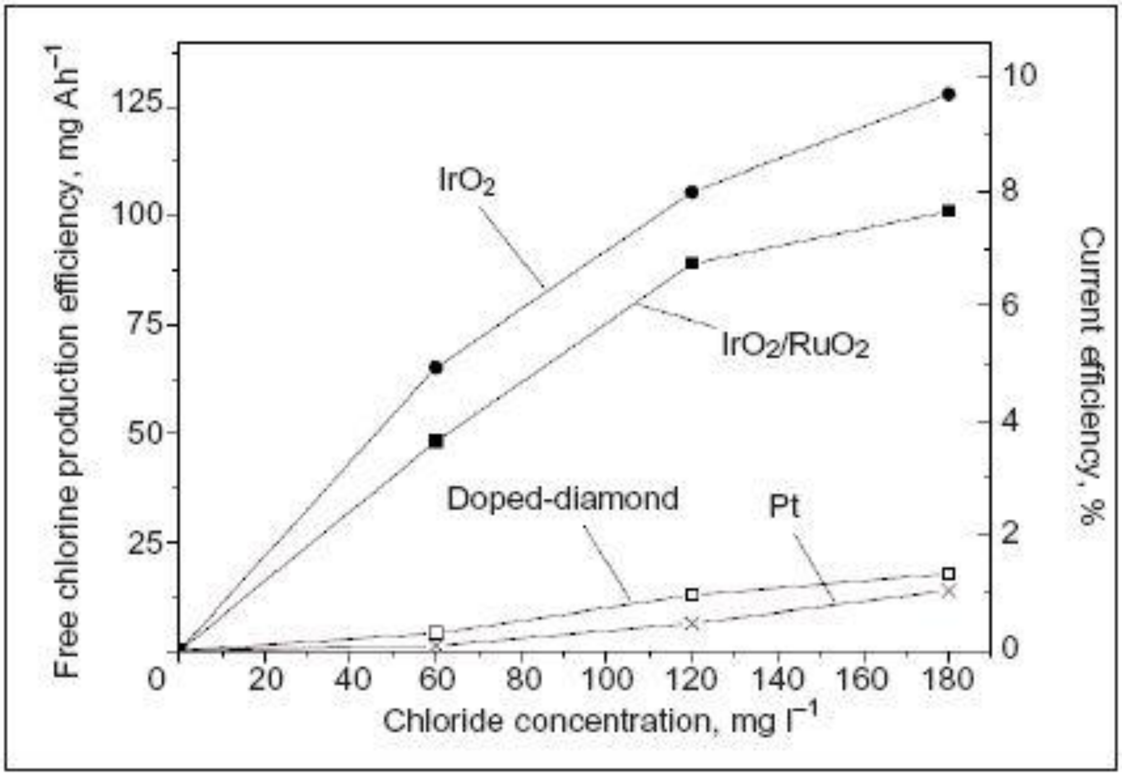

Figure 2- Electrode material and efficiencies for Chlorine generation (Kraft, 2008)

As shown in Figure 2 by (Kraft, 2008), the electrodes here tested give the highest efficiency of the non reactive electrodes.

Several authors have testified for the efficiency of this method when using highly stable electrodes.

Kraft concludes "Electrochemical water disinfection has many advantages compared with conventional disinfection technologies. It has proven its reliability in several practical applications, mainly for the disinfection of drinking water, swimming pool water and industrial cooling water. Electrochemical water disinfection has also been used or tested for the reduction of bacterial contamination in dental water supplies, and for the disinfection of contact lenses and ion exchange resins etc. However, only a few electrochemical water disinfection products are currently available on the market. This is due to the relative unfamiliarity of the technology, and to fierce market competition with other technologies. Eventually, the cost and performance advantages of electrochemical technology should lead to its wider use." (Kraft, 2008)

Research performed at the University of New Orleans focused on Ballast water disinfection done by Katherin McCraven states "Electricity breaking oxygen compounds create which can be attributed to a high annihilation rate of bacteria and D. Magna in ballast water. Generated Cl2 from treatment of brackish water by electricity is the predominant factor in destroying bacteria in brackish waters. Fresh water (low salinity) chlorides that have been transformed into Cl2 are within EPA's scope of tolerance for discharge into aqueous waters. This research demonstrates Cl2 along with ROS can be utilized for disinfection of bacteria in a cost effective manner."(McCraven, 2009)

Nakajuma et al, tried a similar process and tested it for different bacteria and concentrations, these are some of the conclusions:

"Electrolysis exerted a complete bactericidal effect on a relatively small number of B. subtilis cells, but not on a relatively large number of bacterial cells.

Methodology

Sample collection:

Samples of partially treated waste water for this research were collected mostly from the overflow effluent of one of the secondary clarifiers at Jefferson Parish Marrero wastewater treatment plant, located at 6250 Lapalco Blvd. in Marrero, LA. The samples were collected before it went into the pipe that takes it to the chlorination stage. This was done in order to have a real life sample of what would be expected to be treated with the reactor being tested. Water was pumped into either a 20-L or 40-L container depending on the amount of tests to be run each day. Water was taken directly to the lab under temperature controlled conditions to prevent any harm to the bacteria due to temperature. The secondary clarifier is shown on the picture below:

Figure 3- Secondary clarifier at Marrero WWTP

For quantifying bacterial density in all of the samples, the EPA Method 1603: Escherichia coli (E. coli) in Water by Membrane Filtration Using Modified membrane-Thermotolerant Escherichia coli Agar (Modified mTEC) was used, which is a reliable method that allows to visually count the amount of colony forming units in the water sampled.

This method is a single- step modification of the mEPA method 1103.1, given that the membrane does not need the extra step of being transferred into another substrate. The agar contains 5-bromo-6-chloro-3-indolyl- Beta-D-glucuronide, which is catabolized into glucoronic acid by the Beta-D-glucuronidase enzyme present in E.Coli, this is what gives the red-magenta color that allows the visual enumeration of CFU. (USEPA, 2009)

Petri dishes Preparation:

The preparation of the agar starts by carefully measuring 11.4g of mTec Modified Agar in a precision balance. The agar was carefully mixed with 250ml of deionized water in an Erlenmeyer flask that was previously sterilized for 20 minutes in the autoclave at 121°C, placed on a string hot plate and mixed for 15 minutes or until full and homogenous mix was achieved. Immediately the mix was placed in the Autoclave for 20 minutes on the liquids configuration. After a cool down time of around one hour (until gauge pressure and temperature dropped under 0Pa and 100◦C respectively).The now sterilized agar was poured into the 9 × 50 mm Petri dishes at a depth of around 4-5mm adding to a total volume of approximately 5ml ± 1ml.

Bacterial counting:

Since this is a membrane method uses a filtration unit with flask and porous media. A sterile membrane filter paper of 47mm in diameter and pore size of 0.45um was used to filter the water samples. The dilutions ratios were made depending on the expected amount of bacterial CFU to make it a significant number to count but not too numerous to count either. Initial dilutions were of 2-μl of sample water per filtration, along with 200 ml of dionized water to dilute in, and later rinse the funnel above the filtration unit with approximately 20 ml of dionized water to make sure all residual bacteria went to the filter paper. The importance was given to the amount of sample since we expected this 2-μl to have about 100 to 200 CFU. These 100 to 200 CFU are equivalent to the expected 10^7 amount of bacterial CFU per 100 mL of undiluted water. Later tests showed that water from the secondary clarifier, which was the source, was sometimes cleaner than this, which caused inconsistencies on test results and different dilutions had to be made. When high disinfection was achieved it was necessary to significantly increase the amount of sample water being filtered. At the best disinfection result 10ml of wastewater per filtration were used in order for the result to show more accurately if the colony concentration was the equivalent of 200 or less CFU/ 100 mL of sample (20 CFU or less in each dish).

The filtration unit, the funnel, the tweezers and all surfaces that came into contact with the sample water were properly disinfected in between each filtration and sample using a 10% ethanol solution and thoroughly rinsed with dionized water to remove all alcohol traces and preventing it from altering the results.

After filtering the water, the paper filtered was placed into one of the previously prepared Petri dishes. The dishes were then incubated at 35°C for 2±0.5 hours to repair any bacteria that might have been stressed during the sample preparation process. Finally the Petri dishes are transferred to Whirl- Pak® bags, sealed and then rested inside a water bath at 44.5°C ± 0.2°C for 22 ± 2 hours. After this time passed the CFU that would be red/ magenta colored were visually counted. Three tests per sample were performed and the number of bacteria is the average of the three Petri dishes count.

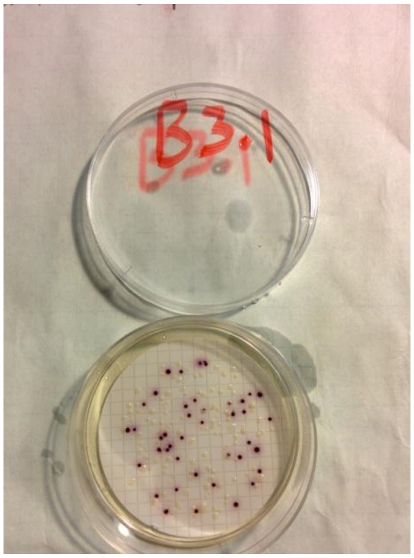

Figure 4- Petri dish used to count CFUs

Chloride Measurement:

To determine the initial amount of chlorides present in the wastewater to be disinfected The Hach Chloride Low Range Test Kit, Model 8-P was used. This is a silver nitrate titration method approved by the EPA to determine chloride concentrations in two ranges; 5-100 mg/l and 20-400 mg/l of Chlorides as Cl^- . Throughout all the research concentrations were always higher than 100mg/l, Therefore the higher range had to be used. The precision of this method is ± 10mg/l (half of the smallest step increment of 20mg/l). Chloride was measure on the raw sample and once after passing through the reactor.

Free Chlorine Measurement:

For free chlorine determination, EPA's approved DPD Method1 equivalent Method 10231 using Hach TNT867 and Hach DR-5000 photospectrometer were used. After collecting a significant amount of sample in a beaker that was properly cleaned to avoid any contamination the vial was filled almost to the neck as instructed by Hach's method. The zero vial was placed in the DR 5000 apparatus for zeroing. The sample vial was inserted and the DR 500 gave a reading. This reading was higher than the actual value given that the sample was highly turbid. The method recommended using a blank vial to correct for turbidity. After using the blank vial prepared with the same sample of water the corrected value of Chlorine as mg/l of CL_2 was recorded. The range of the readings was 0.05 to 2.00 mg/L Cl_2

Total Chlorine:

Total Chlorine was measured using the same TNT867 but with the DPD Method equivalent Method 10232 (Total Chlorine). The procedure is the same but after measuring free chlorine a drop of potassium dichromate was added and a measurement was taken three minutes later. The blank correction was performed as well. . The range of the readings was 0.05 to 2.00 mg/L $Cl_{2.}$

Free and total chlorine were measured for the raw sample and after every run in the reactor. In some cases further measurements were taken after resting the sample for minutes or even hours in a stirring Erlenmeyer flask that worked as a CFSTR.

Experimental Procedure

During the experiments some practical problems arose. It is necessary to have these problems and the solutions used to deal with them present when analyzing the results. These problems are a possible source for human error. It is necessary to keep them in mind when proposing ways to improve the results and accuracy of the results.

The first problem was how to keep a constant flow through each run. This issue had two sources. One the valves used did not handle well low flows. This first source of inconsistency was taken care of by the use of needle valves that are more precise. But the biggest cause of inconsistencies in flow was the static level.

The static level of any container used to hold the waste water was going to vary with time, therefore the flow provided by its water column would change accordingly. A solution that was tested against this problem was the use of Marriotte bottles. These bottles, according to many sources are a reliable way to keep a constant flow regardless of water level. "Once "bubbling" begins, the injection rate will be constant."(Moore, 1990) These bottles provide a relatively constant head partially solving the rate problem. It was not a hundred percent effective, at least not with the ones that were built in the lab but it did provide a relatively constant flow. To understand how these bottles work Figure 6 shows a diagram of a typical Marriotte bottle. Figure 5 shows the Marriotte bottle assembled at the lab.

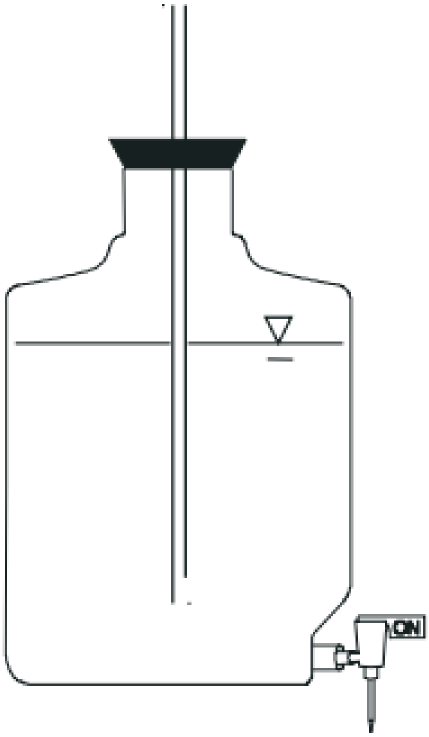

Figure 6- Typical Marriotte bottle (Moore, 1990)

Figure 5- Marriotte bottle used

The bottles had one disadvantage, while providing a relatively constant flow rate, it was hard to make sure complete mixing was achieved inside the closed bottle. A test was performed with clear water over a magnetic stirrer and it was observed that the magnetic bar would not spin properly inside the bottle that was being used. Complete mix is something that is was needed to assure consistency in results and bacterial distribution throughout all tests in each day and to have a reliable base line to which to compare against after disinfection.

In order to achieve complete mixing a large tank with a mechanical mixer was used. In order to try to control and stabilize the flow the micropump Model GC-M35-PVS.E with a

BALDOR Adjustable speed drive were used. The pump was helpless at maintaining the flow constant for it was affected by the static head of the reservoir. The pump was removed and everything was done using just the static head. Consistency in flow was never fully achieved therefore the needle valves were adjusted every 3 minutes to make sure the flow remained around the desired one for each of the required detention times..

Figure 7-CFSTR used during tests

As a power source a BK Precision High Current DC regulated Power 1791 supply was utilized. This apparatus allowed for change in current and observe the voltage needed or apply the voltage and observe the current given. This was very helpful as later these two data were used to calculate resistance and other electric characteristics of each array.

Reactor:

In order to have flexible properties in the reactor an ECOLOTRON reactor that allowed for change in volume of the reactor and number of electrodes in a number of different combinations was used. The reactor consisted of plastic plates with squares holes in the center of 10.2cmx10.2cm. The plates have rubber seals that would align with each other to prevent leakage and the space of the aligned holes would make up our reactor volume. The plates were held together by an ENERPAC P39 hydraulic jack. One of the used reactor configurations is shown of Figure 8

Figure 8-Example of Reactor configuration

All configurations except for those highlighted in blue (SS1,SS2) were performed using highly stable electrodes made of Titanium coated with Iridium Oxide. Like the one on Figure 9

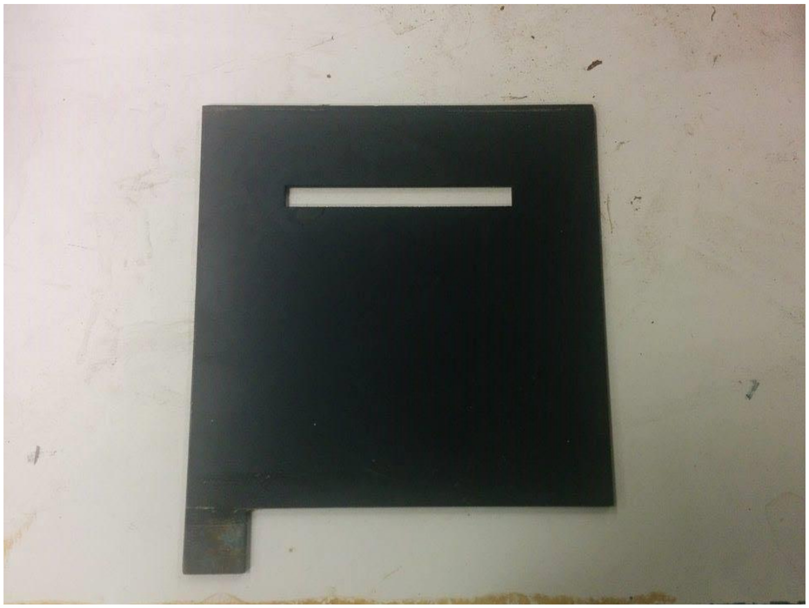

Figure 9- Titanium coated with Iridium Oxide Electrode

Tests Runs

All runs labeled as 1A-7A, and 1B-7B are different configurations using titanium electrodes, covered with iridium oxide given their corrosion resistance. Runs SS1 and SS2 were performed with stainless steel electrodes in order to compare the performances and corrosion of this two different types of electrodes after the ideal conditions for disinfection and residual chlorine using the titanium electrodes where determined.

Titanium/IrO2 electrodes (Runs 1A, 1B)

The first set of tests was run parallel in two reactors of around one liter volume and with a flow rate of 100ml/l. This was intended to test the disinfection of DC using one anode and one cathode for configuration 1A, and two anodes and two cathodes for configuration 1B. Both configurations were operated with a detention time of 10 min and with a current intensity of 0.6 A. In this case the usual 2-microliter sample to be filtered for the initial and the disinfected samples was used. The conditions for 1A were later on repeated to verify and to obtain more accurate results with a different dilution. Chlorides, free chlorine and total chlorine were recorded prior to disinfection and right after disinfection as explained in the methodology.

Titanium/IrO2 electrodes (Runs 2A, 2B)

This set of tests was run parallel in two reactor configurations. Tests 2A and 2B where similar to runs 1A, and 1B, but this time with a lower current (0.4 A). The purpose of this runs was to find the lower threshold at which high disinfection efficiency was still reached, while minimizing energy costs. The reactor volumes around were kept at 1-L and the number of anodes (1 for A, 2 for B) and cathodes (1 for A, 2 for B). Chlorides, free chlorine and total chlorine were measured prior to disinfection and right after sampling the disinfected water. This time the sample was allowed to rest in a CFSTR and both free chlorine and total chlorine were measured every 10 minutes up to 40 minutes in order to determine how fast chlorine would dissipate. 2-microliter samples were collected.

Still keeping the same reactor volumes and electrodes configuration the current was reduced to 0.2A and the chlorides were measured at the beginning, while free and total chlorine were measured up to 30 minutes to observe its dissipation. Since after 30 minutes the residual chlorine change was negligible, the 40 - minute reading was not performed. 2-microliter samples were collected.

Titanium/IrO2 electrodes (4A)

The same electrode configuration of one anode one cathode was maintained and the current was raised back to 0.4A in order to verify the results from 2A but this time using a different dilution (10 ml of disinfected water), since much lower bacterial counts were expected than the initial. This would also allow verifying if water ended up with less than 200 CFU/100ml which is the maximum allowable by USEPA regulations. As will be explained in more detail this was not the case, the sample was high in CFU making them too numerous to count (TNC) therefore the exact disinfection was not quantified and the water is classified as too high in pathogens. It is worth noticing that for some reason the reactor presented lower resistance hence less voltage was needed than for the same configuration on run 2A. Chlorines were tested up to 20 minutes after passing through the reactor. The volume, flow and detention time were kept the same as before, around a liter, 10 minutes, and 100ml/min respectively.

Titanium/IrO2 electrodes (5A)

This run was very similar to 4A in this case the purpose was to verify and obtain more precision on previously obtained results. The volume, flow and detention time were kept the same as before, around a liter, 10 minutes, and 100ml/min respectively. This run returned to the configuration used in run 1A one anode and cathode, same volume and 0.6A current. As with test 4A we realized disinfection was not as high as it was initially thought. Chlorines were measured up to 30 minutes after passing through the reactor. Results did not show a significant disinfection, this will be explained thoroughly in results and analysis. 10 ml were sampled.

Titanium/IrO2 electrodes (4B)

This run was done to test the theory that adding non connected anodes and cathodes increases the resistance of the reactor and the electrolysis inside of it, theoretically increasing disinfection. The first cathodes and the last anodes were connected, leaving two plates in the middle without being connected. The current intensity was set at 0.4 A. Indeed, the voltage was higher so the resistance was higher. As usual, chlorides and chlorine were measured before disinfection and right after disinfection and every ten minutes until 30 minutes passed to record the dissipation of chlorine. Given the mentioned higher resistance and voltage a high disinfection was expected. Hence 10ml of disinfected water were collected for the CFU count. Chlorides were tested before and right after disinfection while chlorine (free and total) were monitored up to 20 minutes after leaving the reactor. The volume, flow and detention time were kept the same as before, around a liter, 10 minutes, and 100ml/min respectively.

Titanium/IrO2 electrodes (5B)

As with 4B the purpose of this run was to test the disinfection with a previously used current, 6A, but using two not connected plates. As with 4B resistance was higher to the point it was not possible to reach 0.6 A with the maximum voltage of 65 V that the power supply was able to provide. Therefore, the test was performed at 0.5A. Chlorides were tested prior to disinfection and right after disinfection. Free and total chlorine were monitored every ten minutes up to 30 minutes after leaving the reactor. Around one liter of reactor volume was used and 100ml/min to keep detention time at 10min. 10 ml were sampled when testing for bacterial CFU.

Titanium/IrO2 electrodes (6A)

This run had had as its purpose to determine if current alone was the determining factor in the disinfection efficiency and the amount of residual chlorine. Determining the effect of plate distance was the main goal. The current of 05.Amps from test 5B was kept as well as the two real and two not connected electrodes. A variable analyzed here was reducing the total volume of the reactor to around 0.7L and the flow rate to 70ml/min in order to account for changes due to plate distance and volume but not for detention time, which was kept at 10 minutes. The shorter distance between plats reduced the resistance, making easier to achieve 0.5Amps. Chlorides as usual were measured prior and post disinfection. Chlorine right after passing the reactor was really high. Hence it was decided to monitor it for the next three hours and see if such an amount eventually dissipates and to what extent. 10 ml were filtered for bacterial CFU count since high disinfection was expected.

Titanium/IrO2 electrodes (7A)

Just as with 6A with 6A two connected and two not connected plates were used, a current of 0.5Amp was kept. Two connected and two not connected electrodes were kept since previous tests showed greater disinfection using this configuration. The detention time was reduced to around five minutes by increasing the flow. If a high disinfection was to be achieved then 10 minutes would be redundant and 5 minutes would be a more economical option to keep for future design parameters. Only immediate chlorines and chlorides were measured besides initial values. As usual 10 ml were filtered given the expected disinfection.

Titanium/IrO2 electrodes (6B)

The purpose of this run was to compare disinfection and chlorines production at a larger volume of approximately 1.7L while keeping the most commonly used detention time of 10 minutes by also increasing the flow to 172ml/min. The same 2 real electrodes and two not charged electrodes configuration was kept as well as 10ml for filtration.

Chlorides, Free and total Chlorine was monitored prior and immediately after disinfection. Since resistance increased the current was set at 0.3Amps since the power source couldn't provide more than 65V.

Titanium/IrO2 electrodes (7B)

The same conditions as in 6B of two real and two non connected electrodes was used, as well as a volume of around 1.7-L but the detention time was reduced to around five minutes by increasing flow to around 334ml/min, a comparison of this would give us a more economical value to use as a design parameter later on. Chlorides, free and total chlorine were monitored right after and prior to disinfection. 10ml sample for filtration in the bacterial count was kept given that disinfection was expected to be high.

Stainless Steel electrodes, (SS1)

The best results from the disinfection using titanium covered with iridium oxide electrodes was used as a guide to pick the ideal configuration for stainless steel runs. In this case the following configuration was used; 2 connected and 2 not connected electrodes, 1.5L of reactor volume, 10 minutes detention time, 0.4A current. Water was tested prior and immediately after for chlorides and chlorines.

Stainless Steel electrodes, (SS2)

The same configuration as with SS1 regarding flow rate, detention time, test sampling and volume was used. The only change made to SS2 compared to SS1 was the current that was brought down to 0.3Amps in order to try to reduce the amount of ferric oxide precipitate that was being generated and will be discussed later on.

Results

Configurations:

Before being able to compare the results of each run a table with each configuration summary and its name will be presented. More details about the configurations and why they were selected was explained in the previous section (Test Runs)

Table 2- Reactor Configurations

Run	Voltage (V)	Current (Amp)	Anodes	Cathodes	Volume (ml)	Flow rate (ml/min)	Detention Time (min)
1A	59.7	0.6	1	1	1036.1	100	10.36
2A	47	0.4	1	1	1036.1	100	10.36
3A	28.5	0.2	1	1	1036.1	100	10.36
5A	36	0.4	1	1	1036.1	100	10.36
4A	54	0.6	1	1	1036.1	100	10.36
6A	40	0.5	1(+1 fake)	1(+1 fake)	698.904	70	9.98
7A	40	0.5	1(+1 fake)	1(+1 fake)	698.904	140	4.99
1B	9.5	0.6	2	2	1073.4	100	10.73
2B	7.9	0.4	2	2	1073.4	100	10.73
3B	6.4	0.2	2	2	1073.4	100	10.73
4B	51	0.4	1(+1 fake)	1(+1 fake)	1073.4	100	10.73
5B	65.1	0.5	1(+1 fake)	1(+1 fake)	1073.4	100	10.73
6B	65	0.3	1(+1 fake)	1(+1 fake)	1728.9	172	10.05
7B	65	0.3	1(+1 fake)	1(+1 fake)	1728.9	344	5.026
SS1	64.7	0.4	1(+1 fake)	1(+1 fake)	1509.084	150	10.0
SS2	52	0.3	1(+1 fake)	1(+1 fake)	1509.084	150	10.06

Configurations highlighted with yellow are those with desired or higher disinfection and levels of residual Free Chlorine lower than 0.4mg/L.

Configurations highlighted with red are those with residual Free Chlorine higher than 0.4mg/L

More details will be shown in later tables

Disinfection:

Not all configurations had a significant disinfection and a lot of them had such good results that it is hard to quantify how close to 100% was achieved.

In order to calculate the approximate number of CFU in a given sample the following formula was used for every sample and initial values:

$$\frac{colonies}{100\ ml} = \frac{ACS * 0.1(L)}{VS(L)}$$

Where:

ACS= Average colony count in the sample

VS= Volume Sampled in liters

In order to determine the percentage disinfection the following expression was computed for each run:

$$\%Disinfection = \frac{Sample\ count\left(\frac{colonies}{100ml}\right) - Initial\ count\left(\frac{colonies}{100ml}\right)}{Initial\ count\left(\frac{colonies}{100ml}\right)} * 100$$

The CFU counts in Table 3 will have some mathematical operators to show how it was calculated.

~ : Since 100 ml were not physically sampled due to practical reasons (the filter paper would clog) the value is a calculated based on a 1:10 dilution therefore an approximate one.

Table 3- Volume sampling per run and disinfection %

Run	Volume Sampled for Initial count (L)	Initial CFU /100ml	Volume Sampled for disinfected colony count (L)	Average Colony count	Effluent CFU /100ml	Disinfection %
1A	2.00E-06	2.50E+06	2.00E-06	0	0	~100
2A	2.00E-06	1.63E+06	2.00E-06	0	0	~100
3A	2.00E-06	1.63E+06	2.00E-06	0.33	~1.65E+04	~99.0
4A*	0.01	1.75E+07	2.00E-06	TNC	very high	~0
5A*	0.01	1.75E+07	2.00E-06	TNC	very high	~0
6A*	0.01	1.75E+07	2.00E-06	0	0	~100
7A*	0.01	1.75E+07	2.00E-06	0	0	~100
1B	2.00E-06	2.50E+06	2.00E-06	0	0	~100
2B	2.00E-06	1.63E+06	2.00E-06	34.7	~1.73E+06	~0
3B	2.00E-06	1.63E+06	2.00E-06	30	~1.50E+06	~8.2
4B*	0.01	1.75E+06	0.01	159.3	~1.59E+03	~99.9
5B*	0.01	1.75E+06	0.01	2.7	~26.7	~100
6B*	0.01	1.75E+06	0.01	0	0	~100
7B*	0.01	1.75E+06	0.01	0	0	~100
SS1	1.00E-05	1.86E+06	0.01	TNC	very high	~0
SS2	1.00E-05	1.86E+06	0.01	140	~1400	~99.92

The runs marked with an asterisk * are those which originally had an initial value of zero that didn't allow for a proper comparison. This was due to the plant working better than usual. To account for this, a sample on a different day was taken from the same spot and initial values were taken at different dilutions; 2μL, 20μL, and 100μL. Confirming our hypothesis the usual 2μL sample was giving zero CFU, the 100μL was almost impossible to count, almost uniform in color, but the 20μL gave an average of 350 CFU. This value of 350 CFU translated into CFU/100ml was used to compare against for the tests with original CFU counts of zero. Later tests on stainless steel were performed with a 10μL sample to prevent this issue from happening again.

Yellow highlighted are the ones with sufficient disinfection and lower than 0.4mg/L residual free chlorine. Red highlighted are runs those with higher than 0.4mg/L of residual free chlorine. These parameters are going to be highlighted throughout the thesis since they are the ones that are intended to be optimized.

Chlorine:

Waste water Characteristics:

Average Chlorides: 150 mg/L (fresh non saline) (food and Agricultural organization of the United Nations)

Average Free Chlorine: 0

Average Total Chlorine: 0.07 mg/L

As it can seen from this average values of not disinfected water the reactors are dealing with fresh water. Not a single sample had any detectable free chlorine. Some cases did show some total chlorine. The focus of the calculations below is going to be free chlorine since it is the chlorine that actively disinfects and is believed to be the main agent for disinfection in these reactors. Final values of total residual chlorine (TRC) will be used to compare with regulations.

Free Chlorine is a parameter that needs to be minimized for environmental and cost reasons. As extra information total chlorine, which also includes compounds like chloramines, were also measured but the emphasis was given to free residual chlorine. Results from all runs regarding free chlorine in order of highest to lowest are shown in Table 4

Table 4- Free Chlorine for each run

Run	Free Chlorine as mg/L
6A	1.34
1A	0.562
7A	0.389
5B	0.351
4B	0.268
5A	0.209
1B	0.098
6B	0.061
2SS	0.057
1SS	0
2A	0
2B	0
3A	0
3B	0
4A	0
7B	0

As with previous tables the red highlights are the ones with high free chlorine as it can be clearly seen in Table X, and the yellow ones are the ones with the desired disinfection and low chlorine.

Table 5- Free and Total Chlorine in successful runs

TEST	Cl2 free(mg/L)	TRC (mg/L)
7B	0	0.08
6B	0.061	0.142
5B	0.351	0.837
7A	0.389	0.807

This table shows the total residual chlorine of the successful runs to later compare with regulations.

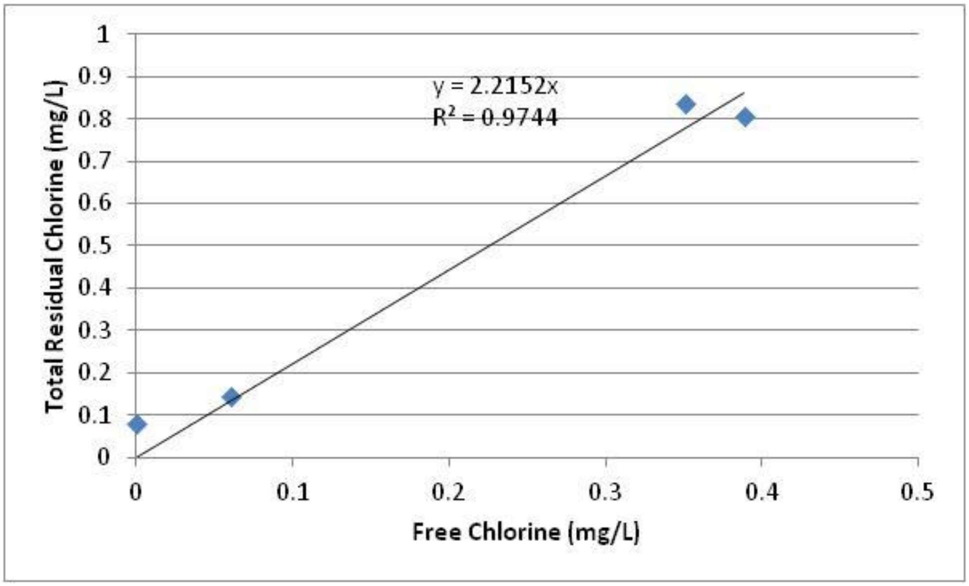

Figure 10-relationship between Free and Total Chlorine

As it can be seen, TRC tends to be twice as much as free chlorine in each of the successful runs obtained

Electrical Characteristics:

Since all of the research was performed using a DC power source all the formulas for the electrical properties are those for DC current.

$$R(ohms) = \frac{V(Volts)}{I(Amps)}$$

$$Power(watts) = I * V$$

$$Energy\left(\frac{KW * h}{L}\right) = \frac{Power(watts) * time\ applied(\frac{h}{L})}{1000\frac{W}{Kw}}$$

The time applied for each litter had to be calculated for each case as well in order to be able to use the equation above to calculate the energy per litter. The time for each liter case is as follows:

$$time\ applied(\frac{h}{L}) = \frac{1000(\frac{ml}{L})}{Q\left(\frac{ml}{min}\right) * 60\frac{min}{h}}$$

Current density was calculated in order to see if there was a tendency between it and other properties, residual chlorine or disinfection percentage. The basic definition of current density is the current in the reactor per unit area of cross section.

$$CDx = \frac{Current\ (Amps)}{Cross\ sectional\ Area}$$

The cross sectional area is considered to be the area through which the electric field is crossing, the area of the plates in contact with water and facing each other. For all plates the area was 0.01040m^2.

Since the unexpected phenomenon of adding not connected plates seemed to change the electrical properties a second type of current density was calculated in order to analyze if any tendency with chlorine, resistance, resistivity or residual chlorine was a function of the total exposed area of the electrodes to the electric field as shown in the expression below.

$$CDt = \frac{Current\ (Amps)}{N * Cross\ sectional\ Area}$$

Where:

CDx= Current density per cross sectional area

CDt= Current density per exposed electrode area.

N= Number of electrode faces exposed to the electric field.

The charge applied was calculated per liter to make easier comparisons later against chlorine concentration.

$$Ch\left(\frac{coulombs}{L}\right) = Current(A) * time\ applied\left(\frac{h}{L}\right) * (3600\frac{s}{h})$$

For reasons that require a deeper electrochemical analysis electrical properties of the configurations with just two or four regular electrodes were different from those of the configurations with two connected electrodes and two not connected which for purpose of this analysis will be called fake electrodes.

Theoretically speaking the amount of chlorine generated should be directly proportional to the current and time the water is exposed to it given the following

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

$Cl_2 + H_2O \rightarrow HClO + HCl$

$HClO \leftrightarrows ClO^{-} + H^{+}$

$HClO \rightarrow O + Cl^{-} + H^{+}$

$ClO^{-} \rightarrow O + Cl^{-}$

Since reactions that take place between the generation of chlorine and the time measurements of free or active chlorine (of hipochlorous acid and hypochlorite ion), a correlation between the charge in Coulombs and free chlorine was approximated for each group of configurations. Results are shown in Table X

For no fake electrodes (All titanium covered with Iridium Oxide)

Table 6- Charge applied and free chlorine with no fake electrodes

TEST	Coulombs/L	Cl2 free(mg/L)
3B	120	0
2B	240	0
3A	120	0
4A	240	0
2A	240	0
1B	360	0.098
5A	360	0.209
1A	360	0.562

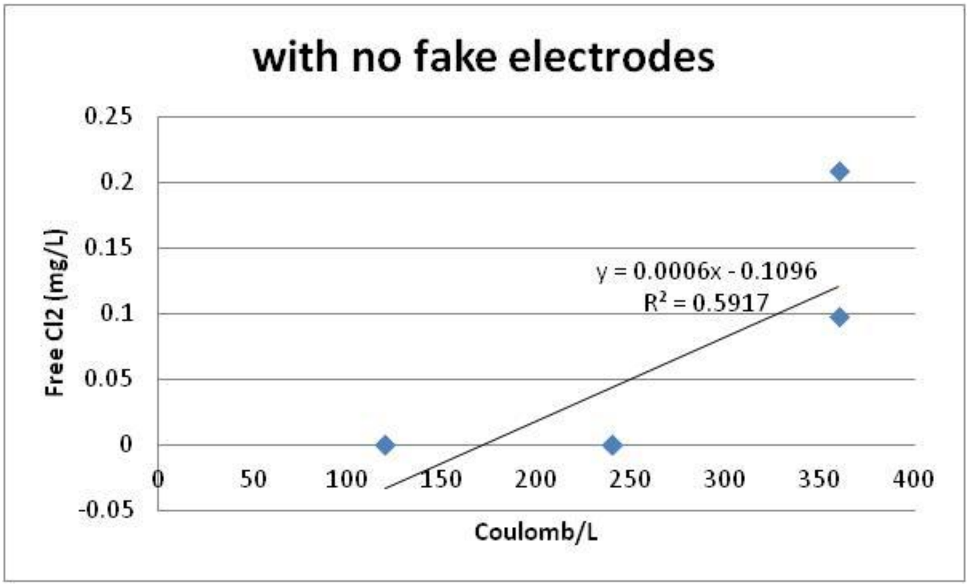

Figure 11-Relationship between applied charge and free Chlorine with no fake electrodes

The correlation is not strong but a tendency can be seen. 1A was considered an outlier since in had the same 360 Coulombs/L as 5A with more than twice the chlorine. Also from a thermodynamic point of view the amount of electrolysis is proportional to the energy input, since at standard conditions it is necessary to input the equivalent to the Gibbs free energy to make a non spontaneous reaction like the oxidation of chlorides into free chlorine to occur. Table X and Figure X show the correlation between energy per liter and free chlorine detected.

Table 7- Energy and free chlorine with no fake electrodes

TEST	Energy KW*h / L	Cl2 free(mg/L)
3B	0.000213333	0
2B	0.000526667	0
1B	0.00095	0
3A	0.00095	0.098
4A	0.0024	0
2A	0.003133333	0
5A	0.0054	0.209
1A	0.00597	0.562

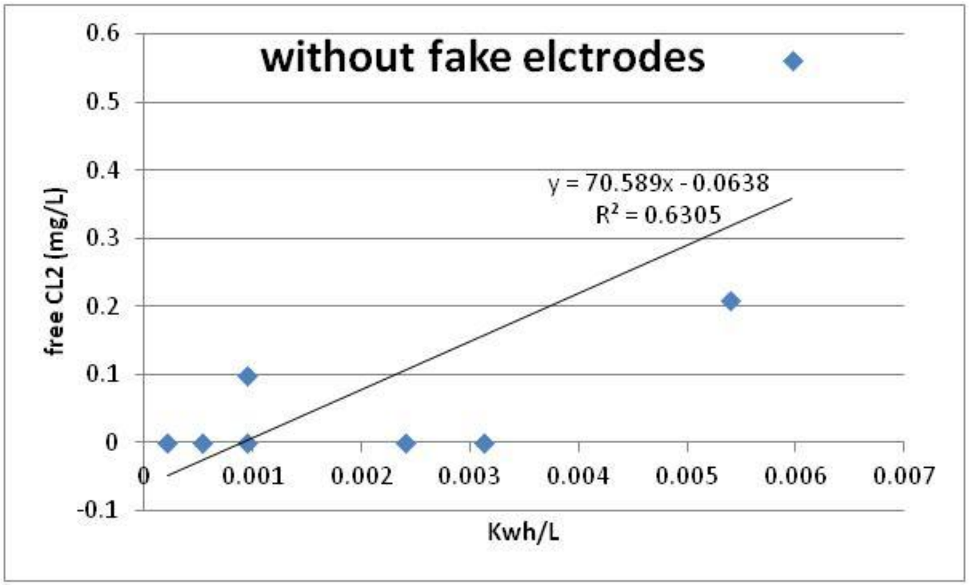

Figure 12- Relationship between free chlorine and energy without fake electrodes

4A and 1A might seem as outliers but not considering them in the correlation still doesn’t improve much the R^2 value

The same analysis was made for the titanium covered with iridium oxide plates in a 2 real two fake electrode configuration with much better results in correlations and as previously shown better disinfection. All the values of high disinfection with relatively low chlorine happened in this configurations.

These are the results for Charge per liter for the 2 real 2 fake titanium and iridium oxide configuration regarding charge per liter. 5B was considered an outlier and taking it out for the correlation gave us a higher R^2

Table 8- Charge applied and free chlorine with fake electrodes

TEST	Coulombs/L	Cl2 free(mg/L)
7B	52.3255814	0
6B	104.6511628	0.061
4B	240	0.268
5B	300	0.351
7A	214.2857143	0.389
6A	428.5714286	1.34

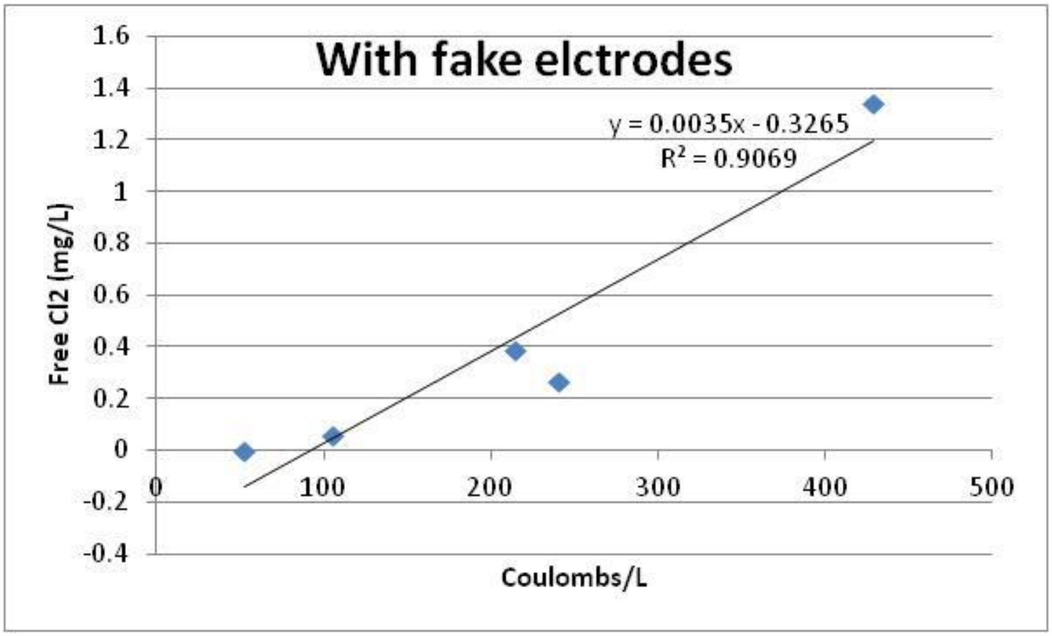

Figure 13- Relationship between free chlorine and charge applied with fake electrodes

5B was considered an outlier, since it has lower Chlorine than 7A and 6A even though it as higher energy. Eliminating 5B from the correlation elevated the R^2 from 0.806 to its current 0.9069 value. A tendency was obtained for the energy-chlorine correlation but less reliable than the Coulomb-chlorine one

Table 9- Energy and free chlorine with fake electrodes

TEST	Energy KW*h / L	Cl2 free(mg/L)
7B	0.000944767	0
6B	0.001889535	0.061
4B	0.0034	0.268
5B	0.005425	0.351
7A	0.002380952	0.389
6A	0.004761905	1.34

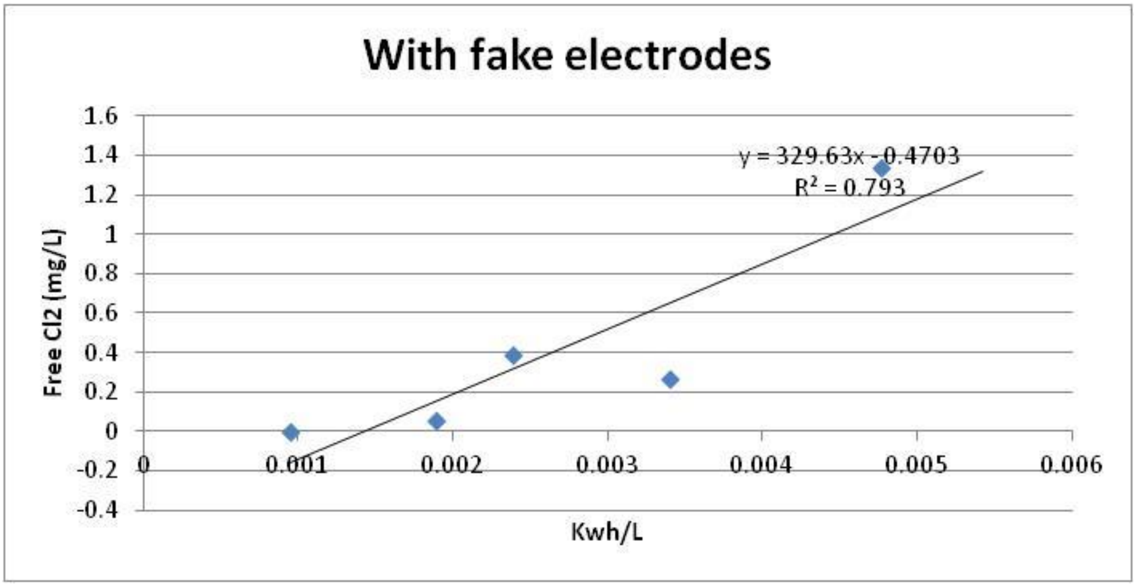

Figure 14- Relationship between free chlorine and energy with fake electrodes

Since results were clearly better and more consistent with the 2 connected and 2 fake electrodes the resistivity for this type of configuration was calculated in order to later be able to use this as a design parameter if a larger scale reactor was going to be designed.

Table 10- Resistnce and (L/A) with fake electrodes

TEST	Resistance (ohm)	L/A (1/m)
7B	216.6666667	11.83593095
6B	216.6666667	11.83593095
4B	127.5	7.348422862
5B	130.2	7.348422862
7A	80	4.784648902
6A	80	4.784648902

The slope of the curve would be the resistivity in ohms*m and the Y intercept would be set to zero.

Resistivity= 17.95 Ω*m

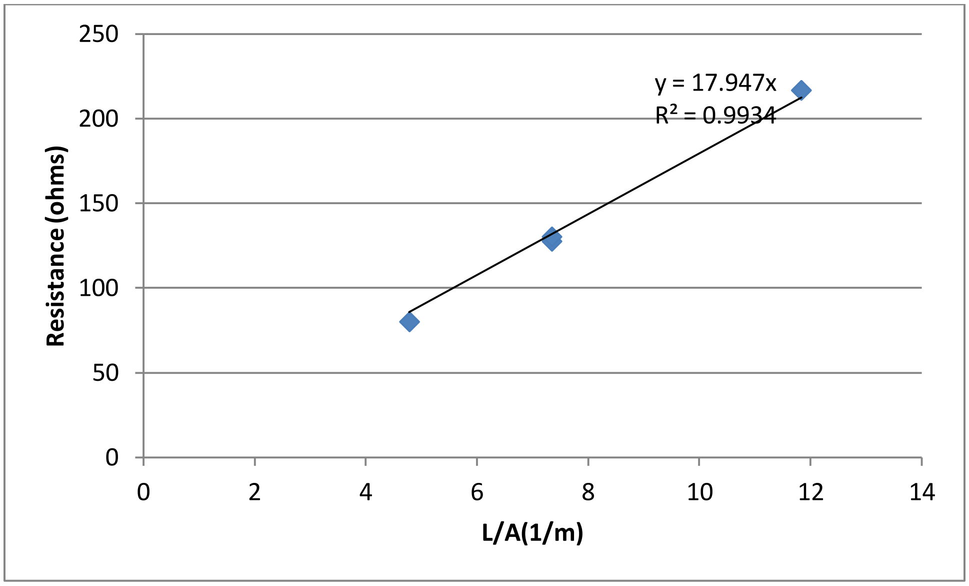

Figure 15- Resistivity with fake electrodes

Since Current density is considered an important parameter according to different authors, a correlation was approximated between current density and chlorine production. The highest chlorine for 6A of 1.34mg/L was considered an outlier and the relationship between current density and free residual chlorine for the 2 real and 2 fake configurations is shown in Figure 16

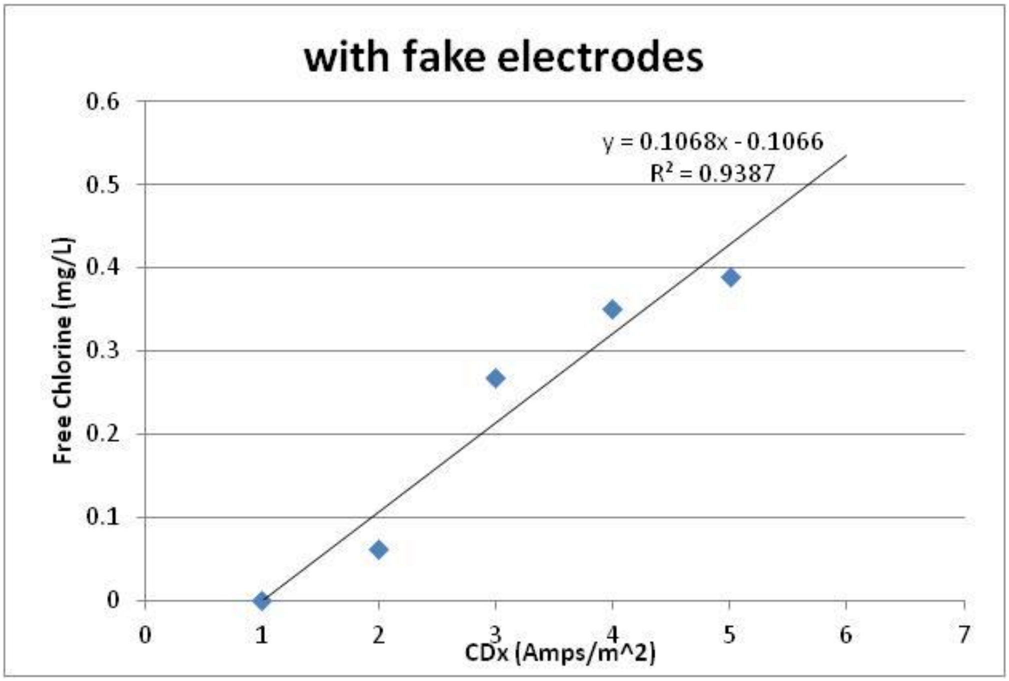

Figure 16- Relationship between free chlorine and cross sectional area

No tendency or explanation for the density using all exposed faces was found.

Economical Analysis:

Since each configuration had its own electrical behavior it was necessary to calculate the amount of energy used per liter to have a basic idea of the operational costs under similar configurations at a large scale; for this the energy per liter was multiplied by the average industrial cost of KWh of 7cts for 2014 and also shown the cost for a common flow of 4000 cubic meters per day plant, which would be a typical small-mid size waste water treatment plant.

Table 11- Energy and Cost for each run

TEST	Energy KW*h/ L	Energy KW*h for 4000 m^3	cost for 4000 m^3
3B	0.000213	853	59.7
2B	0.000527	2107	147.5
7B	0.000945	3779	264.5
1B	0.000950	3800	266.0
3A	0.000950	3800	266.0
2SS	0.001733	6933	485.3
6B	0.001890	7558	529.1
7A	0.002381	9524	666.7
4A	0.002400	9600	672.0
1SS	0.002876	11502	805.2
2A	0.003133	12533	877.3
4B	0.003400	13600	952.0
6A	0.004762	19048	1333.3
5A	0.005400	21600	1512.0
5B	0.005425	21700	1519.0
1A	0.005970	23880	1671.6

As can be seen the operational cost varies a lot depending on the configuration used. The minimum amount of energy that was used and still obtained the required disinfection gives an operational cost of $265. Later, this figure will be compared with energy consumption and operational costs of conventional chlorination plants.

Stainless Steel plates:

Table 12- Summary of results for stainless steel runs

Run	Cl-	Cl2 (mg/L)	TRC (mg/L)	Avg CFU/Petri	CFU/100ml	Disinfection %
SS1	160	approx 0	0.414	TNC	very high	Low
SS2	160	0.057	0.107	140	1400	99.92

This is a summary of the results that were obtained with the 2 real 2 fake stainless steel configuration.

An important note is that high quantities ferric oxide and hydroxide were present. It was theorized that eventually the production of this precipitates would cease but after 5 hours it did not stop. Figure 17 shows such precipitates.

Figure 17- Stainless steel electrodes with slimy precipitate

Figure 18- effluent water using stainless steel electrodes

This is the sample of water obtained after passing through the reactor with stainless steel electrodes.

Expecting some corrosion of the electrodes this were weighted clean before an after the test runs. Two electrodes became heavier and two lost significant mass.

Table 13- Change in mass for stainless steel electrodes

Plate	Mass (m)	Mass (g)	ΔMass (g)
SS7	1564	1566	2
SS1	1562.5	1538.5	-24
S2	1573	1576	3
S3	1593	1572	-21

No further calculations were done for this type of electrodes.

Chlorine Dissipation/Decay

For the highest value of chlorine obtained a dissipation plot was developed, in order to observe how much of this residual chlorine would eventually decay or dissipate.

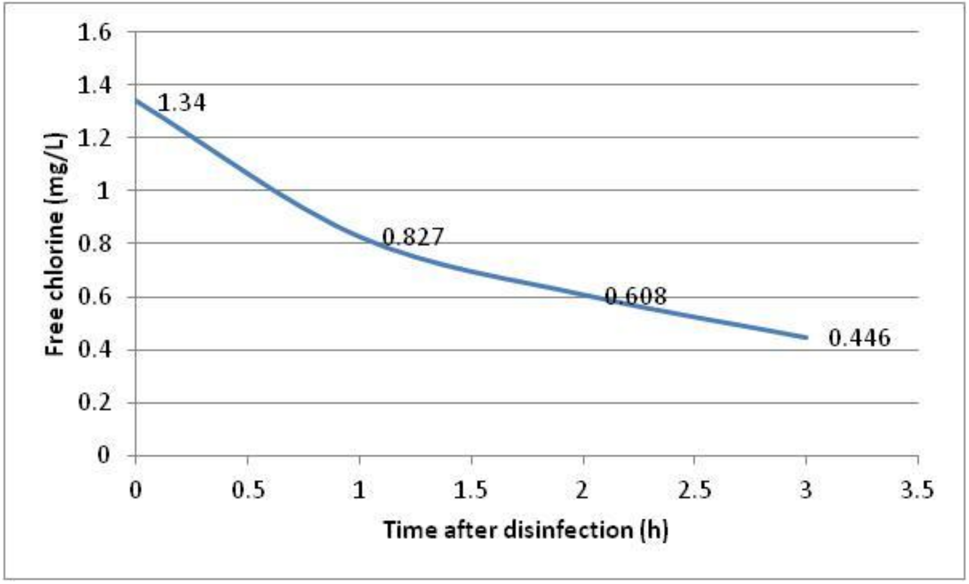

Figure 19- Chlorine dissipation

Total Chlorine remained greater than 2mg/L until the 3rd hour then it was finally measured at 1.39mg/L

Discussion

Disinfection:

It was not possible to obtain a detailed and gradual spectrum of disinfection percentages. Hence it is hard to correlate any of the independent variables with disinfection percentages. However, the results previously shown are very clear and convincing in demonstrating that DC current works efficiently in achieving high disinfection percentages, in most cases close to 100%. We cannot say it is a 100% given that the influent and effluent CFU/ 100ml were approximated using smaller samples. It can be said, however, that it definitively achieves the EPA standard of a maximum 200 CFU/100ml. Some of the 10 - ml samples didn't show a single colony in three of the Petri dishes

Figure 20 shows one of the mentioned samples of 10ml each with no CFU.

The mechanisms for disinfection are not 100% understood, but given that a measurable amount of free chlorine is generated it is reasonable to think it is at least in great part due to the oxidation of chlorides in water to hipochlorous acid and hypochlorite.

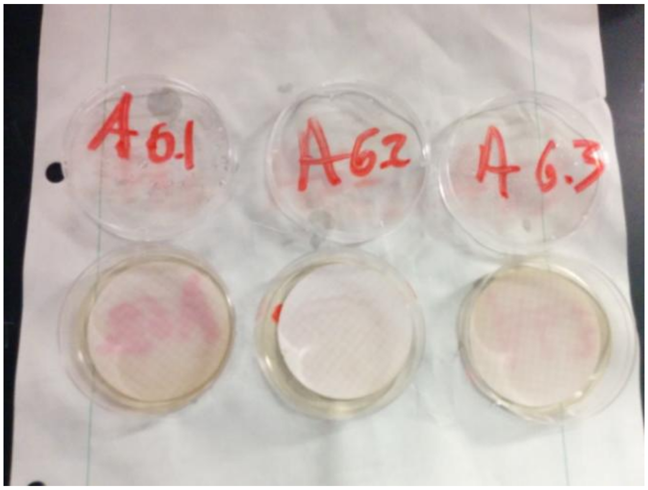

Figure 20- example of successfully disinfected effluent

Hydraulic properties:

The weak spots of all measurements and the biggest sources of possible errors were the hydraulic parameters. While the number of bacteria CFU was measured using high volume samples, and the electric properties were carefully measured and verified, flow rate, detention times, as well as real reactor volumes are not as precise. Due to the gas generating nature of the reactor it was virtually impossible to determine the effective volume of the reactor that was actively holding and treating water and what portion of the reactor was just holding gas generated at the electrodes. Given the high content of substances in wastewater, different products were generated during electrolysis, being hydrogen and oxygen the main two. This, however, only makes the findings here and later recommendations about detention time conservative for the actual volume of the reactor is probably smaller than the one here considered based on the plates' dimensions. This could be improved by using an open reactor or a system to recover the gases, one of which, hydrogen, is a useful gas and could potentially be used as a form of energy recovery. Also the design of the reactor could be modified to accumulate the gases in a specific place that would not be considered as part of the reactor volume and using air release valves removed once it reached certain pressure. The flow itself could have a high error for it was never constant. Given all the static head and pump issues explained in the experimental procedure the flow was readjusted every three minutes to something a bit higher than the desired since it will eventually drop below it. Lacking a flow meter the flow was measured by the use of a timer and a gradated cylinder and adjusted by turning the needle valve. This could be improved by the use of a pump that provides a constant flow regardless of the static head, hence controlling the flow entering the reactor instead of the flow leaving. This would be specially needed if the reactor would be modified to be open or if other gas removal and recovery methods were implemented to prevent over flooding it. For the difficulties mentioned with gas accumulation no tracer test was performed. If the test would have been run with clear water the amount of gas wouldn't be that of real conditions and any tracer added would react making it practically useless. Running the test without electricity would have been equally insignificant since no gas would have been generated making it totally different from real conditions.

Chlorine and disinfection:

Chlorine is most likely the main disinfecting agent generated during electrolysis but it doesn't seem to be the only one, and results might even suggest is not strictly necessary. Other disinfectants might be produced given the high stability of the electrodes, like hydrogen peroxide at the cathode. Or even oxidative species from oxygen like ozone and hydroxyl ions in low quantities in (Kraft, 2008) According to this three equations

$$3H_2O \rightarrow O_3 + 6e^- + 6H^+$$

$$O_2 + 2H_2O + 2e^- \rightarrow H_2O_2 + 2OH^-$$

$$O2 + 2H2O + 4e– \rightarrow 4OH–$$

The problem with calculating the amounts of dissolved oxidative species or any gas is that the pressure inside the reactor is unknown therefore affecting solubility and the role this gases play in disinfection. Hydroxyl radicals are really hard to detect given that they react almost immediately with surrounding compounds.

As mentioned these levels of chlorine alone are not sufficient to achieved such disinfection (Chavez ,2014) Therefore we infer other phenomena is occurring

Electrical properties and Chlorine generation:

There seems to be a relationship between the amount of chlorine and certain electrical characteristics of the reactor configurations. Focus is mostly given in this analysis to those results on the two real and two fake electrode configurations that are the ones which show clear tendencies in properties. At the same time these configurations obtained the satisfactory results in terms of low chlorine and high disinfection efficiency. The correlation between Charge, given in Coulomb per liter and the amount of free residual chlorine gave an R^2 of 0.9069 for the two real and two fake configurations. This is a strong correlation and should be taken into account for modeling and design. According to many authors, current density, as earlier defined and calculated, is crucial in the amount of electrolysis and chlorine generated. The data obtained for the successful runs clearly shows a correlation between detectable free chlorine and the current density with an R^2 of 0.9387. Since current density is directly proportional to the current intensity, and the cross sectional area, which is kept constant throughout all runs, plotting this two variables is just determining the relationship between chlorine and current without taking into account the contact time and the flow. Because of this, the coulomb-chlorine relationship is more significant given that chlorine and coulombs are both measured in a per liter basis making it more inclusive of hydraulic properties of the reactor. Nevertheless the effect of current density should be further researched to determine its effect on disinfection and chlorine production by electrolysis. Relationships between the energy input and chlorine were also observed, less significant but should be taken into account if design criteria were to be developed from this experimental data.

Resistivity is an intrinsic property of any material that describes how much does it oppose to the flow of electrons. When multiplied times the length and divided by the area it describes a specific element in a circuit its electrical resistance. Given that area and length of each configuration are available it was possible to plot resistance vs. L/A allowing for an approximation of the resistivity of 17.95 Ω*m for all the two real and two fake configurations. The R^2 of 0.9934 implies a very reliable value of the slope. More data points are needed to improve the accuracy of this correlation.

Temperature was monitored through the whole duration of the experiment. No significant temperature increases were observed. On average, water temperature increased from room temperature (23°C) to around 25°C or less. Hence all disinfection is due to electrolysis and its products and not to any heating effect as with pasteurization, which requires temperatures of minimum 62°C and detention times of around 30 minutes for proper disinfection to occur. (Metcalf and Eddy, 2014)

Since the disinfection efficiency of stainless steel electrodes was very low, and due to the fact there is a large generation of suspended solids, it was deemed unnecessary to further explore the usefulness of these electrodes.

Chlorine decay/dissipation

It was observed that chlorine tends to dissipate with time but not in a rapid manner. The plot clearly shows that after 3 hours chlorine still remained at high concentrations for run 6A. The rate of change also seemed to decrease with time.

Dechlorination requirements:

If calibrated properly reactors of this nature might not need much or no dechlorination at all depending on where they are discharging. Under the two most efficient successful run characteristics, dechlorination would not be needed to discharge in any natural marine or fresh stream of a dilution factor of 50:1 and above, maybe even lower than 50:1.

Table 14- Free and Total Chlorine for successful runs

TEST	Cl2 free(mg/L)	TRC (mg/L)
7B	0	0.08
6B	0.061	0.142
5B	0.351	0.837
7A	0.389	0.807

The limit for discharge is based on the Following Tables X and X for both fresh and marine water given by EPA’s NPDES

Table 15-TRC limitations (mg/L) for Discharges into fresh water based on the Dilution Factor

Dilution Factor	Avg month/ daily max	Dilution Factor	Avg month/ daily max	Dilution Factor	Avg month/ daily max
50:1	0.55/0.95	64:1	0.7/1	78:1	0.86/1
51:1	0.56/0.97	65:1	0.72/1	79:1	0.87/1
52:1	0.57/0.99	66:1	0.73/1	80:1	0.88/1
53:1	0.58/1	67:1	0.74/1	81:1	0.89/1
54:1	0.59/1	68:1	0.75/1	82:1	0.90/1
55:1	0.60/1	69:1	0.76/1	83:1	0.91/1
56:1	0.62/1	70:1	0.77/1	84:1	0.92/1
57:1	0.63/1	71:1	0.78/1	85:1	0.94/1
58:1	0.64/1	72:1	0.79/1	86:1	0.95/1
59:1	0.65/1	73:1	0.80/1	87:1	0.96/1
60:1	0.66/1	74:1	0.81/1	88:1	0.97/1
61:1	0.67/1	75:1	0.83/1	89:1	0.98/1
62:1	0.68/1	76:1	0.84/1	90:1	0.99/1
63:1	0.69/1	77:1	0.85/1	>=91:1	1.0/1.0

Table 16-TRC limitations (mg/L) for Discharges into marine water based on the Dilution Factor

Dilution Factor	Avg month/ daily max	Dilution Factor	Avg month/ daily max	Dilution Factor	Avg month/ daily max
50:1	0.38/0.65	67:1	0.50/0.87	84:1	0.63/1.0
51:1	0.38/0.66	68:1	0.51/0.88	85:1	0.64/1.0
52:1	0.39/0.67	69:1	0.52/0.90	86:1	0.65/1.0
53:1	0.40/0.69	70:1	0.53/0.91	87:1	0.65/1.0
54:1	0.41/0.70	71:1	0.53/092	88:1	0.66/1.0
55:1	0.41/0.72	72:1	0.54/0.94	89:1	0.67/1.0
56:1	0.42/0.73	73:1	0.55/0.95	90:1	0.68/1.0
57:1	0.43/0.74	74:1	0.56/0.96	91:1	0.68/1.0
58:1	0.43/0.75	75:1	0.56/0.98	92:1	0.69/1.0
59:1	0.44/0.77	76:1	0.57/0.99	93:1	0.70/1.0
60:1	0.45/0.78	77:1	0.58/1.0	94:1	0.71/1.0
61:1	0.46/0.79	78:1	0.59/1.0	95:1	0.71/1.0
62:1	0.47/0.81	79:1	0.59/1.0	96:1	0.72/1.0
63:1	0.47/0.82	80:1	0.6/1.0	97:1	0.73/1.0

Operational costs:

Operational costs would vary significantly depending on the configuration chosen. Another fact that would significantly affect the cost of this method would be the lifetime of the electrodes which has not been addressed in this paper. But purely operational non maintenance cost which translate into energy costs would be of $265 with the best configuration here found. No cost would come from chemical compounds since no chemical compounds are needed with this method.

Conclusions

Iridium oxide coated titanium is a very stable electrode material that can be used for electro disinfection through electrolysis. Using cheaper and less stable materials such as stainless steel is not a viable option due to the contamination of the water with ferric oxide and hydroxide.

Disinfection is achieved even in very low chloride water like the one used here (Avg Cl^- 150mg/L)

Given the fact that chlorine production was low dechlorination might not even be necessary or wouldn't be as costly as with conventional chlorination methods. This all depends on the regulatory agency, which for the USA is the EPA and each state's DEQ, and where is the water being discharged (Check Table C for Fresh and Marine water, EPA NPDES). Usual TRC is around 3-10mg/L before dechlorination lowers it to around 0.15mg/L. (EPA, Waste water technology Fact Sheet, Chlorine Disinfection, 1999)

It was not possible to determine a gradual change in disinfection based on the data here presented, nevertheless it was very clear which parameters had to be maximized in order to achieve the desired disinfection efficiency. Because of this if design criteria were to be developed it would have to be conservative. Given the disinfections of the selected runs are higher than required and very close to 100%, to the point it cannot even be measured. One can be confident of the following design criteria based on the minimum of each property tested that yielded successful runs:

- Use at least two not connected electrodes given that these configurations by far outperformed the “all connected “configurations. (more research on this is needed)
- Keep a minimum electrical input of 9.5E-5 KW*h/L of energy, special care on this for operational cost minimization.
- Keep a minim current of 0.3Amps
- Keep a minimum Current density (CDx) of 30 Amps/M^2
- Emphasis in keeping a minimum charge input (Ch) of 55 Coulombs/L
- Minimum detention time of 5 minutes

All of these criteria are based on all the minimum characteristics observed in the successful runs, all corresponding to run 7B which also yielded the lowest operation cost. More research should be done to determine which of the criteria is more defining and should be prioritized over the other. Separating in different chambers might be an option.

Operational Costs

Operational costs under the recommended criteria are as low as 265$ but depending on the design the electrodes would create a high maintenance and upfront cost.

This technology is perfectly applicable and can replace conventional chlorination, some of its advantages are:

- Low operational cost
- Almost no moving parts
- Requires low skilled labor to maintain
- No need to deal with handling and transport of chemicals like CL2 and or Sodium hypochlorite.

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Vita

Julio Acosta was born in Quito, Ecuador on January 1, 1990. In 2008 Graduated from high school specialized in physics and math. The same year he started a five year civil engineering program at ESPOL in Guayaquil, Ecuador. In 2011 He transferred to the University of New Orleans where he graduated the spring 2013.

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