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Tap Water Disinfection in the Electrochemical Precipitation Process by Using Novel Conductive Concrete

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TAP WATER DISINFECTION IN THE ELECTROCHEMICAL PRECIPITATION PROCESS BY USING NOVEL CONDUCTIVE CONCRETE

A Thesis

by

MD SHORIFUL ISLAM

Submitted in partial fulfillment of the

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TAP WATER DISINFECTION IN THE ELECTROCHEMICALPRECIPITATION PROCESS BY USING NOVEL CONDUCTIVE CONCRETE

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August 2022

ABSTRACT

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Electrochemical precipitation (EP) is tested to learn about its impact on disinfectants in tap water. The EP process involves applying electricity through electrodes submerged in water to precipitate dissolved metals like water hardness out without chemical additives. In the study, tap water was tested to learn about EP's impact on disinfectants. Tap water contains not only hardness but also residual disinfectants. It is very essential for the safety of drinking water against the potential ingress of pathogens before the water reaches the end-users. USEPA mandates that the free chlorine level in drinking water should be 0.2mg/L to 4 mg/L. Since pH near the electrodes changes during the EP process, the chemical composition of chlorine species may change as well. In conventional EP, sacrificial metallic cathodes are widely used. Instead, a newly developed conductive concrete block is used in this study. Conductive concrete blocks are made of concrete and conductive graphite flakes. Total and free chlorine concentrations in tap water during the EP process will be collected per different chloride concentrations, current density, treatment time, and pH. Preliminary research findings showed that an increase in the chloride concentration, time, and current density resulted in more free chlorine disinfectant concentrations in water. This study will introduce a novel hardness removal method, EP with conductive concrete, and provide preliminary proof of its added value as a disinfecting technology.

DEDICATION

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Thanks to the almighty. My Journey at UTRGV for the Master’s studies would not have been possible without the love and support of my family and friends. I dedicate this work to my brother Md Ashraful Islam. It all started from his struggle and inspiration. All credit goes to that person whom I enjoyed every single moment with him

.

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TABLE OF CONTENTS

Page

ABSTRACT....iii
DEDICATION....iv
ACKNOWLEDGEMENT....v
TABLE OF CONTENTS.... vi
LIST OF TABLES....vii
LIST OF FIGURES.... viii
CHAPTER I. INTRODUCTION....1
CHAPTER II. LITERATURE REVIEW....6
  Disinfection mechanism....6
  Physical Disinfection Methods....7
  Chemical Disinfection Methods....8
  Electrochemical disinfection....9
  Electro-Disinfection Mechanism....10
  Free Chlorine....11
  Factor affecting the production of free chlorine 13
  Electrochemical Precipitation 17
CHAPTER III. OBJECTIVES....23
CHAPTER IV. METHODOLOGY....24
  Conductive Concrete....24
CHAPTER V. RESULT AND DISCUSSION....36
  Resistivity....36
CHAPTER VI. CONCLUSION....54
REFERENCES 55
APPENDIX....60
BIOGRAPHICAL SKETCH….62

LIST OF TABLES

Page

Table 1: Free chlorine production in different electrodes (Alexander et al.,2008) ……………14

Table 2: The effect of chloride concentration on the active chlorine production rate per A using iridium oxide and platinum-coated titanium expanded metal electrodes (Kraft et al.,2008)..15

Table 3: Effect of current density on free chlorine production (Saha et al.,2020)……………..16

Table 4: Effect of pH on free chlorine production (Saha et al.,2020)…………………………16

Table 5: Effect of retention time on free chlorine production (Saha et al.,2020)………………17

Table 6: Summery of previous Research on disinfection...21

Table 7: Summery of previous research on Free chlorine production.......................................22

Table 8: The specification of graphite flake used in the experiment is shown in the table 25

Table 9: Mix Design of Graphite conductive concrete cathode used in this experiment...........26

Table 10: Compressive strength test of 10%,7.5% and 5% graphite concrete cylinder...............53

Table 11: Summery of total experimental data...61

LIST OF FIGURES

Page

Figure 1: Electrochemical Precipitation……………………………………...………………..18

Figure 2: a) Coarse Aggregate b) Fine aggregate ....……………...………………..24

Figure 3: a) Natural graphite flake b) Cement ....………………...……………25

Figure 4: a) Applying nickel paste on the conductive concrete cathode b) Applying copper tape over the nickel paste in this experiment...27

Figure 5: Schematic diagram of Electrochemical Precipitation ………………...………………..28

Figure 6: Top view of experimental setup…………………………………...………………..29

Figure 7: Compressive strength specimen…………………………………....……………..31

Figure 8: (a) Conducting compressive strength test on FORNEY F250. (b) specimen setup for the compressive strength test 32

FIgure 9: Failure of specimens 10T1, 10T2, and 10T3 under lateral loading 33

Figure 10: Specimens for the impedance measurement..34

Figure 11: Basic input parameter before impedance measurement...35

Figure 12: Nyquist diagram for 10 percent graphite...36

Figure 13: Bode plot diagram for 10 percent graphite...37

Figure 14: Phase angle diagram for 10 percent graphite 37

Figure 15: Nyquist diagram for 7.5 percent graphite...38

Figure 16: Bode plot diagram for 7.5 percent graphite...39

Figure 17: Phase angle diagram for 7.5 percent graphite...40

Figure 18: Nyquist diagram for 5 percent graphite...41

Figure 19: Phase angle diagram for 5 percent graphite..42

Figure 20: Effect of time on free chlorine production 43

Figure 21: Effect of time on total chlorine production...44

Figure 22: Effect of pH on free chlorine production 45

Figure 23: Effect of pH on total chlorine production...46

Figure 24: Effect of pH on total chlorine production...47

Figure 25: Effect of current density on total chlorine production 48

Figure 26: Effect of chloride concentration on total chlorine production....................................49

Figure 27: Effect of chloride concentration on total chlorine production....................................50

Figure 28: Compressive strength for 5%,7.5%, and 10% graphite content 52

CHAPTER I

INTRODUCTION

Water is one the most vital natural resources for human beings' existence and good quality of life in the world. But the clean water sources are getting depleted due to population growth, excess use, climate change, etc. WHO and UNICEF have adopted Millennium Development Goals (MDG) program for water supply and sanitation to ensure that half the population has access to safe drinking water (WHO, 2014). Although MGD fulfills the drinking water target, it has been estimated that more than 748 million people still lack access to drinking water sources. A huge number of the population are stipulated to depend on contaminated water sources which are microbiologically unsafe and have a higher risk of water-borne diseases including typhoid, hepatitis A and E, polio, and cholera . From both medical and economic point of view, Water quality remains one of the most serious problems in most countries and the most challenging issues of 21 century is water shortage and deficiency **(**Sadiq R et.al 2007 & Li Q et.al 2008) According to the WHO report, Around 1.1 billion people worldwide lack access to clean drinking water.**(**Li Q et.al 2008). A reliable and safe water distribution network is necessary to supply safe drinking water to the consumer in quantity and quality. The quality of drinking water depends on water sources and treatment. The sources of fresh drinking water are at risk due to environmental pollution. There are lots of mechanisms and pathways water gets contaminated and polluted.

(Islam et.al 2015). Any kind of contaminants intrusion either chemical or biological directly affect public health. The use of organic and inorganic products in Industrial and agricultural activities promotes the growth and transmission of different micro-organisms in drinking water (Abderrahmane et.al 2008). The drinking water contaminants create serious health risks to the consumer by causing(detected) water-borne outbreaks of (undetected) endemic levels (Berry et al. 2015). In developing countries, 5 out of 400 children under age 5 die per hour due to waterborne diarrheal diseases (WHO 1996), and approximately 80 percent of all sicknesses and diseases among people are caused by unsafe drinking water. The requirement of safe drinking water must be free from pathogenic microbes and detrimental substances that can transmit diseases and illnesses to the consumer such as typhoid fever, dysentery, cholera, and gastroenteritis, and received in terms of color taste odor and turbidity, etc (Levinson W et.al 2008 &Haider et.al 2006). Current progress develops a disinfection process to prevent this phenomenon. (Abderrahmane et.al 2008). The water treatment process consists of several steps like storage, sedimentation, coagulation and flocculation, and rapid filtration, and disinfection to reduce different physical and chemical contaminants present in water. Disinfection is a common and essential process for any kind of drinking water treatment technology. Because pathogens, bacteria, and other micro-organisms which may present in the water, can cause infectious and water-borne diseases to the consumer, are killed in this process (Li, H et.al 2011). The main objectives of the disinfection process are to kill all microorganisms or bacteria contain in water before distribution and provide enough residual disinfectant for future ingress of bacteria in the water distribution system (Haider et.al 2006).

There are several disinfection processes have already been developed in the world. Three common disinfection processes are widely used, and these are chlorination ozonation and ultraviolet radiation (Haider et.al 2015). It's proved that ozone is a better disinfectant than chlorine and chloramine for *Cryptosporidium parvum* as well as *Bacillus subtilis* oocysts inactivation. Cho et al showed that ozone has damaged the greatest level of the surface of bacteria cells than free chlorine and its oxidizing ability to react with organics of the cell wall before penetration into cell plasma (Li, H et.al 2011). Ozone oxidizes a minor amount of ammonia during disinfection, is desirable but its rapid reaction with organic and inorganic substances further reduces the time of persistence in water (Singer, P.C., & W.B. Zilli et.al 1975). Ozone is not suitable for developing countries due to the high cost of application and the need operational and maintenance infrastructure and it does not provide any kind of residual disinfectant in the distribution system. (Haider et.al 2006). Ultraviolet radiation has many applications ranging from drinking water purification at home to the entire township as well as industrial areas, but it does not provide residual disinfectant in the water distribution system. On the other hand, chlorination is used for water disinfection for more than 100 years due to its low capital and operating cost and produces free residual chlorine which acts as a secondary disinfectant and is stable in the water distribution system until it reaches the end consumer **(**Sadiq et.al 2007). According to the drinking water and health (National Academy and Science 1977), chlorination is the most standard method for disinfection compared to the other techniques. Free residual chlorine helps to prevent biofilm growth in the water and maintain taste and odor and finally reduces disinfection bioproduct (Sadiq R et al 2007, Haider et.al 2015). The world health organization (WHO) and Environmental

Protection Agency (EPA) recommends a minimum of 0.2mg/l free chlorine in the water distribution network. Because a low dosage of free chlorine is unable to kill all microorganisms in the water. It is important to maintain a minimum level of free chlorine concentration in water, but over-chlorinated water increases the growth of disinfectant Bio product (DBP). Besides, corrosion can be occurred due to elevated chlorine as well as higher temperature and lower ph. as a result of chlorine demand increase. (Frateur I et.al., 1999). USEPA recommends that the maximum free chlorine level in drinking water should be 0.2mg/L to 4 mg/L. But the formation of chlorination bioproduct tetra halo Methene (THM) and other hydrocarbons which are responsible for un-favorable taste and cariogenic disease, has created a major concern and promoted reexamining the available disinfection method and finding alternative procedure. Nowadays Electrochemical disinfection has gotten more attention and is considered a promising method due to providing both primary and residual disinfection, in addition, it is environment friendly, inexpensive, and easy to operate (Cossali et al., 2016). Bergman et al (2014) found that inline electrolysis is comparable to the chemical chlorination process in case of THM formation and conforming that ED is a more advantageous method due to producing onsite residual disinfectant that eliminates the handling, storage, and transport of hazardous chemical. There are also some disadvantages of the ED process, are the formation of a calcareous deposit on the cathode surface. Polarity reversal is used to eliminate the deposit from the cathode surface. But this polarity reversal leads to shortening the lifetime of the cathode and reduces the production of active chlorine production (A. Kraft, Blaschke, et al., 1999). EP is another electrochemical process and similar mechanism principle. EP needs a high cathodic area for sufficient precipitation which leads to a limited scope of innovative usage, also cleansing the cathode is a hazardous responsibility (Hasson et al., 2010)& (Hasson et al., 2011). The main challenge of these processes is to prevent or reduce electrical resistance due to precipitated scales on cathodes This may be mitigated by reversing circuits occasionally to electrochemically repulse precipitates and remove them. Scaled cathodes can be replaced with new ones as well.

Recently, conductive concrete has become a promising technology in the civil engineering industry. In the transportation and infrastructure field, electrically conductive concrete has been providing a great service and established a reliable method to remove ice from the surface. (Abdualla et al., 2018; Ceylan et al., 2014). To make these concrete conductive, electrically conductive materials like carbon fiber, and steel shavings were mixed with a normal concrete mixture. In this research, graphite powder has been used to make the conductive concrete. This is a novel technique where conductive concrete has been used as a cathode in the EP system.

CHAPTER II

LITERATURE REVIEW

In this chapter theoretical background of the disinfection mechanism and the latest progress in the field of electrochemical water, disinfection have been discussed.

Disinfection mechanism

In the world, a number of different disinfection techniques have already been created. A few of these techniques use chemical disinfectants such hypochlorite, chlorine dioxide, hydrogen peroxide, hydrochloric acid, ozone, and others in addition to physical treatment (heating and radiation).. Many researchers have studied the mechanism of the traditional disinfection method **(**Ding et al., 2016). According to Juneja & sofos et al 2002, The primary factor in the physical disinfection process is temperature. In this method, bacteria are subjected to the inhospitable temperature condition and temperature is the primary factor in bacteria inactivation temperature inhibits the intracellular proteins and enzymes activity and destroys the cellular membranes and nucleic acids. Setlow et al., 2002 made an experiment on the disinfection mechanism of strong acid and alkali treatment on *Bacillus subtillis* pores and reported that *Bacillus subtillis* inactivation was due to the intracellular enzymes' inactivation and permeability barrier disruption. On the other hand, chemical disinfection mechanism action is different from each other. Their distinctions are influenced by antimicrobial components as well as the disinfectant concentration that has been used, which can affect how the chemical agent or physical process behaves. There are three general mechanisms of action that might cause creatures to perish. These include oxidation, structural and function disturbance, cross-linking, coagulating, and clumping..

In cross-linking, coagulating, clumping disinfection mechanism, disinfectants clump cell protein. As a result, cell protein loses its function, and more specifically the cell membrane loses its structure and finally collapses and gets killed. Alcohol follows this mechanism and kills the organism. Oxidizing is another mechanism of action in which pathogens are killed by disinfectants. In this mechanism, disinfectants exabit fast bactericidal action and target the key metabolic enzymes, and finally destroy the organism structure. **(**Ashkin, S. P. 2022, January 31).

Physical Disinfection Methods

There are several disinfection processes are available in the world. They are classified into two types,physical and chemical disinfection methods. Heating was the earliest physical disinfection method that was used for medical instruments and special purpose equipment disinfection. But the thermal stability of the processed sample and their larger size were the limitations of that process. Hard gamma radiation was another physical disinfection technique, but it had more limitation. Currently, several physical disinfection methods are available for water and air: filtration, ozonation, and ultraviolet radiation, photocatalysis. Filtration disinfection is widely used for air disinfection. In this process, microorganism in the air is reduced to an acceptable level. Disinfection by ultra-violated radiation has long been traditionally used and most effective physical disinfection method. Ultra-violate radiation is used in this method. The efficiency of microorganism inactivation by UV radiation is dependent on the energy of the UV lamp. The advantages of UV disinfection are environment friendly, low operating cost, and zero chemicals used. But the main drawback of this method is high energy consumption. Ozone is one of the most effective physical disinfection methods for drinking water treatment. But it is not widely used in the world due to operation and maintenance problems in the first-generation system, and some effluent requires high ozone dosage for treatment. (Robson, C.M. et al., 1991)

Chemical Disinfection Methods

There are lots of chemical disinfectants available in the world which are used in the disinfection process. Three chemical disinfectants most used as primary disinfectants are chlorine, chlorine dioxide, and ozone (WHO 2004). Chlorine is the most widely used disinfectant in the world. Several types of free chlorine like HOCl, CLO-, and Cl2(aq) are widely used as a potential disinfectant to protect water from future contamination for potable, industrial, and agricultural water due to their low cost and high inactivity. When chlorine molecule enters the micro-organism cell. It dysfunction the enzymes group. As a result, micro-organism cells are inactivated. (Saha et al., 2017). But there are several health-related issues associated with safety problems that arise during the gaseous and liquid chlorine transportation and storage. Moreover, different types of organic matter are present in the water reacts with chlorine and creates disinfection bio-product tetrathlon methene.

Electrochemical disinfection

The process of eliminating germs through the use of appropriate electrodes and electrical current in water is known as electrochemical water disinfection. Electricity causes the creation of water-borne disinfection species at the phase boundary between the electrode and the water.. (Alexander Kraft et al., 2008). In this process, without any kind of chemical or oxidizing and bacterial substances are not added, they are naturally produced from the substance which present in the water by electrolysis. The oxidizing substance are short lived and free radical species are O*, OH* and more stable substance HOCl , Clo^{-}, H_2O_2, $O_3$and others.(A. Kraft, Blaschke, et al., 1999). A range of different kind of oxidant produced in this disinfection process. For example, chloride content electrolyte produced active chlorine species like Cl2 HOCl and OCl- which are very fundamental oxidant for killing pathogenic bacteria present in the water. (Ghernaout et al., 2019) From the nineteen centuries, electrolysis was used to clean or disinfect water by using Baghdad battery. But from the end of nineteen century many attempts have been taken for electrochemical disinfection and none of them have been successful and not for long lasting until now. Different term has been used according to their water treatment types and water produced by this technique. For example, electrolytic disinfection, anodic oxidation and functional water and electronically activated water among others. Electrochemical disinfection has recently reached technical maturity for three reasons. The first of these is the creation of adequate and ideal electrode materials. 2) The functional interaction between the chloride content of the water, the electrode material, the current density, and the production of hypochlorous acid and hypochlorite ions during disinfection. 3) Development of water chemistry and electrochemistry research projects carried out by amateurs (Alexander Kraft, et al., 2008)

Electro-Disinfection Mechanism

The mortality of electrochemical exposure has been attributed to a number of different processes. These include electrochemical oxidation of essential cellular components, irreversible permeabilization of the cell membrane by the applied electric field, and oxidative stress and cell death caused by electrochemically generated oxidant. Chemical oxidants, such as hydrogen peroxide and ozone as well as free chlorine and chlorine dioxide when chloride ions are present in the solution, are created when electricity is applied to aqueous suspensions of microorganisms. (Ghernaout & Ghernaout, 2010) According to recent studies, these oxidants account for the majority, but not all, of the lethality of the applied current. Additionally, research shows that antibacterial drugs and electric current collaborate to inactivate microbes. It also proved that 100 mg/L of chloride is adequate to produce free chlorine and effectively disinfect water..(Drees et al., 2003). In electrochemical water disinfection process, two electrodes (one anode and one cathode) are inserted into the water and a DC voltage is applied between the electrodes for water electrolysis. At the anode the main product is oxygen at equation 1

$$2\ \mathrm{H_2o} \rightarrow \mathrm{o_2} + 4\mathrm{H^+} + 4\mathrm{e^-} - \tag{1}$$

At the cathode, hydrogen is produced at equation 2

$$2\ \mathrm{H_2o} + 2\ \mathrm{e^-} \rightarrow \mathrm{H_2} + 4\mathrm{OH^-} \tag{2}$$

Water near the cathode becomes alkaline due to the formation of hydrogen which is generally unwanted, and it must be separated from the water stream.(Alexander Kraft, et al., 2008). Because it can be dissolved or outgas which can accelerates pipe corrosion and other effects. (Bergmann & Koparal, 2005). Recently a new electrochemical disinfection method developed which completely avoid hydrogen production. In this process, the atmospheric oxygen is reduced to hydroxyl ions at a gas diffusion cathode (Equation (iii)

$$o_2 + 2\,H_2o + \ 4e^- \rightarrow 4OH^-$$

The cathodic reaction replaces the hydrogen producing reaction. The porous graphite-polytetrafluorethylene (PTFE) layer with a metal mesh as current collector are used in gas diffusion electrodes gas diffusion electrodes. The electrodes are backed by an oxygen permeable PTFE layer to prevent water leakage.

Free Chlorine

Free chlorine is the sum of chlorine, hypochlorous acid and hypochlorite and oxidize organic compound that can result in the formation of disinfectant bio-product (DBP) including trihalomethanes and halo acetic acid. This kind of DBP is undesirable in drinking water due to possible cardiogenic and mutagenic properties. But to inactivate microorganism in the water, it is essential to chlorinate water sufficiently. The world health organization recommends the free chlorine concentration should not be exceeded 5ppm in drinking water to prevent toxic from chlorination (Wilson et al., 2018). Hypochlorous acid and hypochlorite ions are the main disinfecting agent for water disinfection process like anodic oxidation, chemical disinfection, electrochemical disinfection and produced from the chloride ion in the processed water. The chloride content in the naturally drive water (tap water) varies 10 up to 250 mg/l. (A. Kraft, Stadelmann, et al., 1999). The disinfectant HOCl and OCl- are produced at anode during ED process. First of all, chlorine is electronically produced from the dissolve chloride ion present in the water. (Equation 3)

$$Cl^- \to Cl_2 + 2e^- \tag{3}$$

Chlorine hydrolyses in water and create HOCl and OCl- (Equation 4)

$$Cl_2 + H_2o \to \text{HOCl} + \text{HCl} \tag{4}$$

HOCl and OCl- are a pH dependent anion. (Equation 5)

$$HOCl \longrightarrow \text{OCl- } + H_2 \tag{5}$$

In the nomenclature of water disinfection hypochlorous acid and hypochlorite concentration are termed as active chlorine or free chlorine. According to the following equation, disinfecting effect of free chlorine depends on the atomic oxygen release.(Alexander Kraft et al., 2008)

$$\text{HOCl ® O}+Cl^- + H^+$$

$$ClO^-\text{®O}+Cl^-$$

When chlorine is added in water, it will not produce any kind of free chlorine. Firstly, it will react with reducing agent and ammonia present in the water. As a result, chlorine is destroyed, and no residual chlorine is produced. Chlorine will react with ammonia and produced inorganic chloramine. The production of monochloramine and di-chloramine depends on the pH of water and ration of chlorine and ammonia. When additional chlorine is added, the ratio of chlorine and ammonia changes as a result with that monochloramine converted into dichloramine. When all the ammonia is reacted with the chlorine free chlorine begins to develop.(Drinking et al., 1980).

Factor affecting the production of free chlorine

There are several factors that affect the production of free chlorine in electrochemical disinfection system. These factors are described below.

Electrode materials

Many different electrodes had been studied for electrochemical disinfection process. The electrodes material current efficiency is very crucial for free chlorine production at low chloride concentrated water to be treated. There have been great differences found between different electrodes in the free chlorine production at low chloride concentrated water. Alexander Kraft, et al., 2008 showed that the variation of free chlorine production efficiency on chloride concentration for two dimensionally stable anode (DSA) type electrodes (active coatings of $IrO2$ or $IrO2/RuO2$), platinum and boron doped diamond electrodes. From the figure, In the synthesis of free chlorine, DSA type electrode material performs better than Pt and diamond electrodes. Due to their high overvoltage for both oxygen and chlorine evolution, diamond electrodes also have the potential to oxidize hypochlorite into the disinfection bioproducts chlorate and perchlorate. Very little amounts of this DBP are permitted in drinking water.. Carbon and platinum

were used as anode in electrochemical water disinfection, but their efficiency of free chlorine production is very low. Electrode lifetime is another important factor for selecting electrode material. During electrolysis, water containing calcium and magnesium ion deposited on the cathode surface. It is called scaling. Polarity reversal is used to remove the scaling from the cathode. But frequent use of this technique, the lifetime of the cathode is reduced (Alexander et al.,2008).

Table 1: Free chlorine production in different electrodes (Alexander et al.,2008)

IrO_2	IrO_2/RuO_2	Doped diamond	Platinum
62.5	50	5	0
75	60	8	3
100	80	10	5
125	100	15	10

Chloride Concentration

According to Kraft et al., 1999 active chlorine production depends on chloride concentration in the water up to 1500 mg/L. He also shown that active chlorine production rate on IrO2 is always higher than platinum at same chloride concentration.

Table 2: The effect of chloride concentration on the active chlorine production rate per A using iridium oxide and platinum coated titanium expanded metal electrodes (Kraft et al.,2008)

Chloride Concentration (mg/L)	IrO_2(Active Free Chlorine)	Pt (Active Free Chlorine)
20	0.018	0.002
120	0.025	0.002
300	0.05	0.003
500	0.07	0.02
1000	0.10	0.04

Free chlorine production rate increased with the increase of chloride concertation for both electrodes. Chloride ion which is present in water reacts at Ru sites to become absorbed and dissolved in water. Then it reacts with the hydroxyls ion and produce hypochlorous acid and hypochlorite ion.

Current Density

According to the saha et al., 2020, current density is also another factor that affect free chlorine production. In his electro chlorination research, chloride concentration is positively corelated with current density and free chlorine and total chlorine production increase to certain number under the increase of current density.

Table 3: Effect of current density on free chlorine production (Saha et al.,2020)

Current Density (mA/cm^2)	Free chlorine (mg/L)
0.5	0.3
1	0.6
1.5	0.65
2	0.7
2	0.7

pH

Saha et al., 2020 showed in his electro-chlorination experiment that free chlorine and total chlorine formation improved in slight alkaline condition. The maximum active chlorine 0.75mg/L was produced at pH 8.5.

Table 4: Effect of pH on free chlorine production (Saha et al.,2020)

Free Chlorine (mg/L)	pH
0.5	7
0.55	8
0.75	9
0.75	10

Retention Time

Free and total chlorine increases proportionally with the increase the electrolysis time. Saha et al., 2020 performed a 50-minute electrolysis experiment by using graphite electrodes and he analyzed the sample every 10 minute. He found that maximum free chlorine concentration was 0.75 mg/L in 30 minutes. Kerwick et al.,2005 reported that 0.75 mg/L was observed in 60 min by using titanium electrodes.

Table 5: Effect of retention time on free chlorine production (Saha et al.,2020)

Time (Min)	Free Chlorine (mg/L)
10	0.30
30	0.6
60	0.7

Electrochemical Precipitation

Standard EP cell consists of two electrodes, a DC power source and treatment water. Electrodes are submerged in water and connected to the DC power source. During the experiment, alkaline environment is created near the cathode and scale deposition on the cathode surface. A typical EP cell is shown in the figure below.

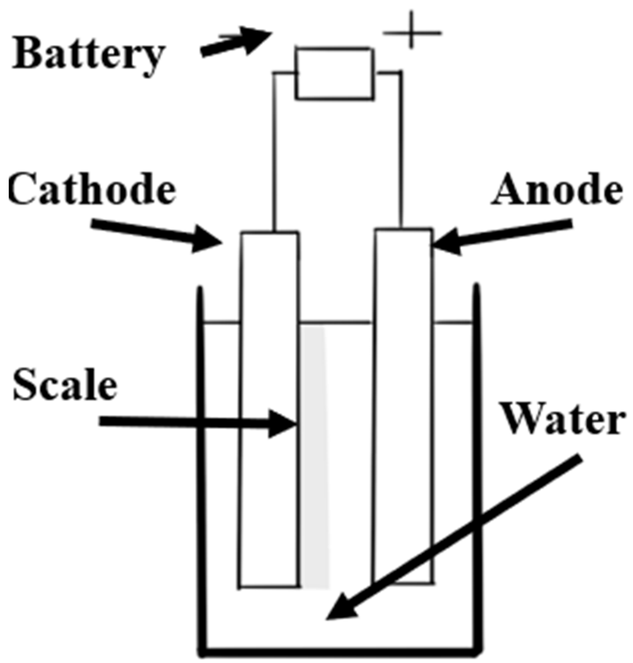


Figure 1: Electrochemical Precipitation

There is no separation medium between cathode and anode. For promoting precipitation near the cathode surface as a thin layer, high pH condition is maintained. As a result, dissolve oxygen is greatly reduced near the cathode. Following reaction occurs near the cathode

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \quad (1)$$

$$2H_2O + 2e^- \rightarrow H_2\uparrow + 2OH^- \quad (2)$$

Hydroxyl ion starts to destabilize the calco-carbonic equilibrium solution (Legrand et al.,1981). The high alkaline environment covert HCO_3^- into carbonate ions by the following reaction

$$HCO^-_3 + OH^- \rightarrow CO_3^{2-} + H_2O \tag{3}$$

Carbonate ions reacts with Ca^{2+} and create $CaCo_3$ which is precipitated and Mg^{2+} ions react with OH^- and form $Mg(OH)_2$ by the following reaction

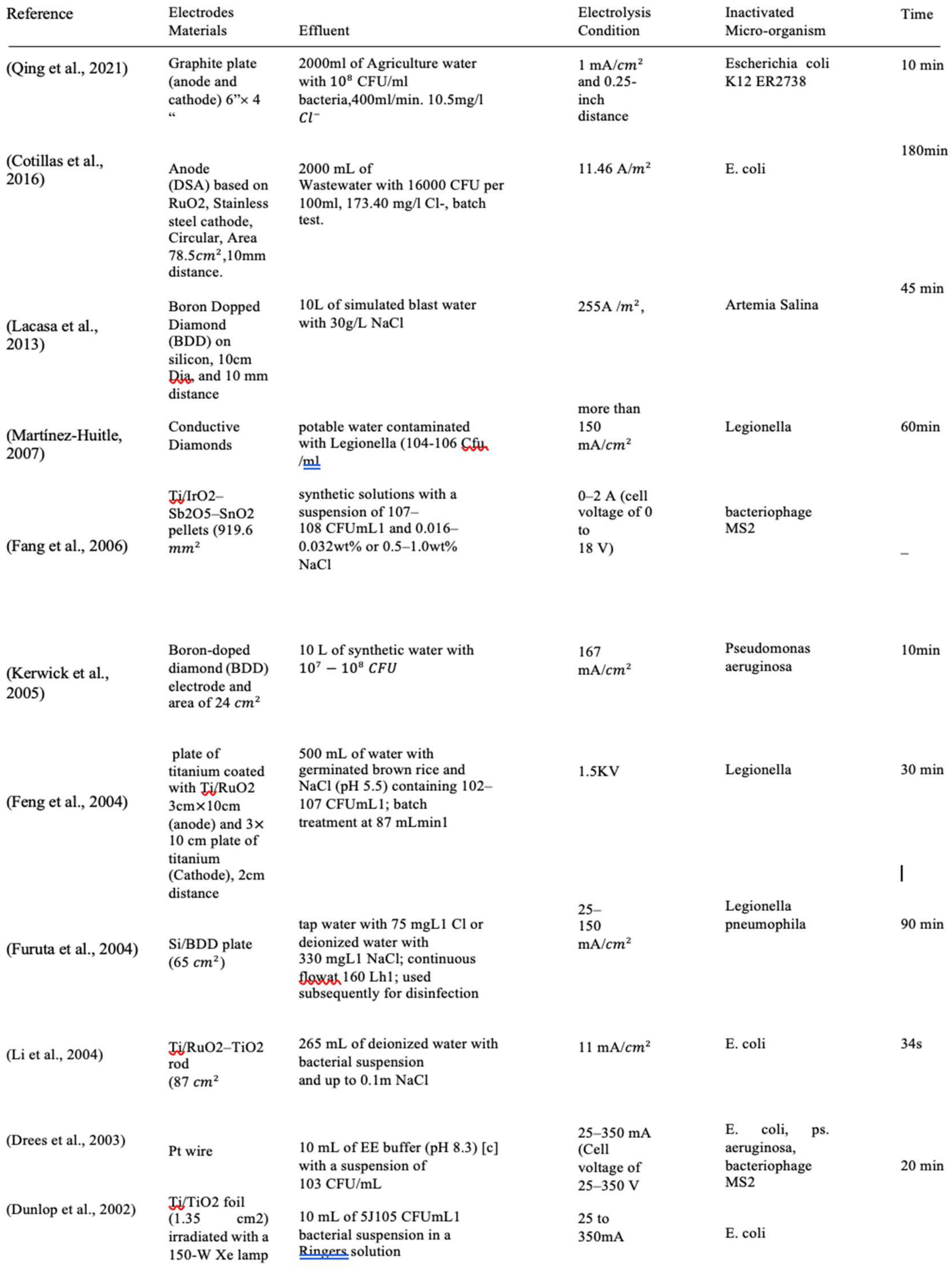
$$Ca^{2+} + OH^- + HCO_3^- \rightarrow CaCO_3 \downarrow + H_2O \tag{4}$$

$$Mg^{2+} + 2OH^- \rightarrow Mg(OH)_2 \downarrow \tag{5}$$

Recent progress in hardness removal by electrochemical process includes 1) Tap water softening 2) Cooling water treatment 3) Descaling cathode surface 4) Cell design and process enhancement

Previous methods were expansive, large energy requirement, heavy chemical use etc. EP is an alternative approach of hardness removal from water. Most of EP process were conducted by using metallic cathode which are expansive and require large cathodic area. Conductive concrete can be replacement of metallic cathode. Previous researcher Mirza Aidditto Billah conducted research on tap water hardness removal in EP process by using conductive concrete as cathode. During the EP process, pH increased near the cathode which help to chloride ion to free chlorine formation. Free chlorine is very important for maintaining water safety against potential intrusion of pathogen and bacteria in the water supply system. In this research EP effect on chlorine species and factors affecting the production of free chlorine will be discussed.

Table 6: Summery of previous Research on disinfection



Reference	Electrodes Materials	Effluent	Electrolysis Condition	Inactivated Micro-organism	Time
(Qing et al., 2021)	Graphite plate (anode and cathode) 6”× 4 “	2000ml of Agriculture water with 10^8 CFU/ml bacteria,400ml/min. 10.5mg/l Cl^-	1 mA/cm^2 and 0.25-inch distance	Escherichia coli K12 ER2738	10 min
(Cotillas et al., 2016)	Anode (DSA) based on RuO2, Stainless steel cathode, Circular, Area 78.5cm^2,10mm distance.	2000 mL of Wastewater with 16000 CFU per 100ml, 173.40 mg/l Cl-, batch test.	11.46 A/m^2	E. coli	180min
(Lacasa et al., 2013)	Boron Dopped Diamond (BDD) on silicon, 10cm Dia, and 10 mm distance	10L of simulated blast water with 30g/L NaCl	255A /m^2,	Artemia Salina	45 min
(Martínez-Huitle, 2007)	Conductive Diamonds	potable water contaminated with Legionella (104-106 Cfu /ml	more than 150 mA/cm^2	Legionella	60min
(Fang et al., 2006)	Ti/IrO2–Sb2O5–SnO2 pellets (919.6 mm^2	synthetic solutions with a suspension of 107–108 CFUmL1 and 0.016–0.032wt% or 0.5–1.0wt% NaCl	0–2 A (cell voltage of 0 to 18 V)	bacteriophage MS2	–
(Kerwick et al., 2005)	Boron-doped diamond (BDD) electrode and area of 24 cm^2	10 L of synthetic water with $10^7 - 10^8$ CFU	167 mA/cm^2	Pseudomonas aeruginosa	10min
(Feng et al., 2004)	plate of titanium coated with Ti/RuO2 3cm×10cm (anode) and 3× 10 cm plate of titanium (Cathode), 2cm distance	500 mL of water with germinated brown rice and NaCl (pH 5.5) containing 102–107 CFUmL1; batch treatment at 87 mLmin1	1.5KV	Legionella	30 min
(Furuta et al., 2004)	Si/BDD plate (65 cm^2)	tap water with 75 mgL1 Cl or deionized water with 330 mgL1 NaCl; continuous flowat 160 Lh1; used subsequently for disinfection	25–150 mA/cm^2	Legionella pneumophila	90 min
(Li et al., 2004)	Ti/RuO2–TiO2 rod (87 cm^2	265 mL of deionized water with bacterial suspension and up to 0.1m NaCl	11 mA/cm^2	E. coli	34s
(Drees et al., 2003)	Pt wire	10 mL of EE buffer (pH 8.3) [c] with a suspension of 103 CFU/mL	25–350 mA (Cell voltage of 25–350 V	E. coli, ps. aeruginosa, bacteriophage MS2	20 min
(Dunlop et al., 2002)	Ti/TiO2 foil (1.35 cm2) irradiated with a 150-W Xe lamp	10 mL of 5J105 CFUmL1 bacterial suspension in a Ringers solution	25 to 350mA	E. coli	

Table 7: Summery of previous research on Free chlorine production

Reference:	Electrode	Effluent	Electrolysis condition	Free Chlorine	Time
(Qing et al., 2021)	Graphite plate (anode and cathode) 6”□ 4	2000ml of Agriculture water with ,400ml/min. 10.5mg/l Cl-, Flow rate 400ml/min	0-3 mA/$cm^2$and 0.25-inch distance	0.06 mg/l	20 min
(Saha & Gupta, 2017)	graphite anodes (32.97 cm2) and stainless-steel cathodes (24.78 cm^2)	150 ml water with 8.5 mg/l Cl-	1.5 mA/$cm^2$and 0.5 cm	0.8 mg/l	30 min
(De Moura et al., 2014)	Ti/Ru0.3Ti0.7O2 as anode (1 cm2 exposed area) and pt wire as Cathode.	500 ml of water with 1.2 g/l NaCl	25, 50 and 75 mA / cm^2	145 mg /l	40 min
(Zaviska et al., 2012)	Ti/IrO2 (anode) and stainless steel (cathode)	400 ml of water 0.11M of H_3O^+ and 0.8 M of NaCl	1.6 A	38.5 mg /l	35 min
(Oh et al., 2010)	A grid shaped Pt/Ti electrode (3 cm × 3 cm)	300 ml of synthetic and natural seawater with 20000 mg/l Cl- (200mg/l)	110 mA/cm^2, 0.1 mm electrode distance	200 mg/l	5 min
(Khelifa et al., 2004)	ruthenium-coated titanium anode (S, = 24 cm”) and titanium cathode, (18.04 cm^2)	250 ml of water with reagent grade 2M NaCl and 0.1% potassium bicarbonate	35 A/dm-3, electrode distance 0.5 cm	65.67g/l	120 min
(Nakajima et al., 2004)	platinum-iridium electrodes (8*34 mm each, distance = 3 mm apart)	10 ml glass taste tube with tap water	110-0.6A, Electrode distance 3 mm	30 ppm	30 min
(Kraft et al., 1999)	titanium expanded metal coated with either iridium oxide or Platinum	1530 ml deionized water with concentrated NaCl solution	15 mA/ cm^2 and 5 mm electrode distances	0,15 mmol Dm-3.	---

CHAPTER III

OBJECTIVES

This study aims to develop a lab scale EP cell using graphite conductive concrete.

- Investigating the compatibility of graphite concrete electrodes as cathodes in an EP cell. The goal was to reduce the resistivity as low as possible by using an efficient amount of graphite
- Setting up the lab scale setup, which included finding the optimal power source, setting up equipment to produce the maximum output, and ensuring safety procedures.
- Testing the best operational factors for this setup, such as pH, current density, retention time, chloride concentration, and cathode material.
- Determine the free and total chlorine formation by varying pH, current density, retention time, chloride concentration, and cathode material

CHAPTER IV

METHODOLOGY

Conductive Concrete

The mold size was 76.3 mm×17.77 mm× 20.33mm. Coarse aggregate was #8 no sieve passing, and maximum and minimum sizes were 9.5 mm and 2.38 mm respectively. Fine aggregate was #8 sieve passing and #100 sieve retaining. Water cement ratio and air content of concrete were used at 0.45 and 4.5%. Natural flake graphite was used to make concrete conductive. The physical properties of graphite are shown in the fig

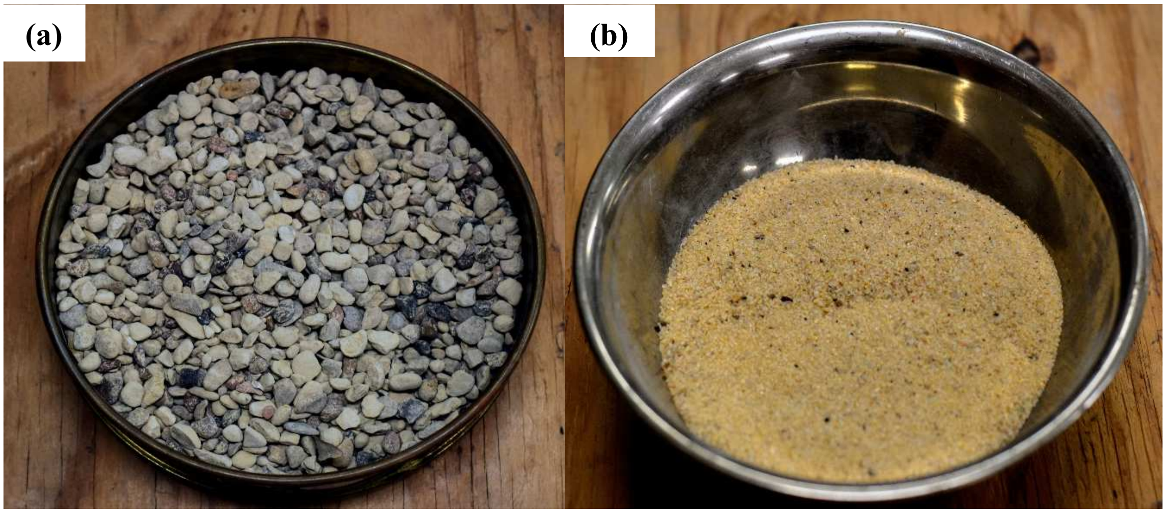


Figure 2: a) Coarse Aggregate b) Fine aggregate

Table 8: The specification of graphite flake used in the experiment is shown in the table.

Percent of Carbon	Typical Size (μm)	Specific Gravity	Surface Area (m^2/g)	Typical Resistivity (Ω-cm)	Note
95.46%	50-60	2.6	5	0.03-0.05	Flake

Graphite was added with the cement, coarse and fine aggregate homogenously with the volume of the specimen. Enough water was added to the mixture to make the concrete paste. Three specimens were cast containing 10% graphite powder. The mix design and casting were followed by ASTM standards.

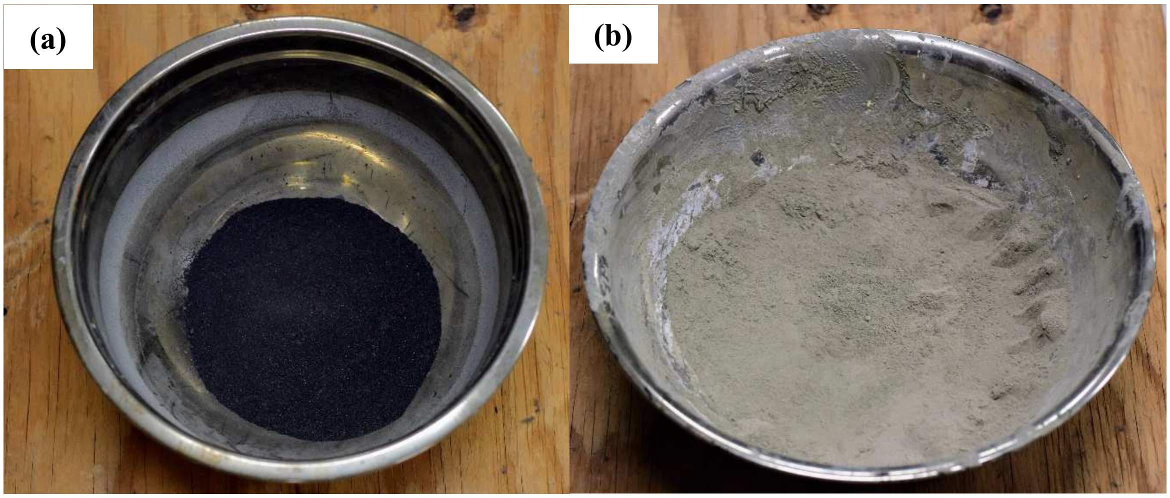


Figure 3: a) Natural graphite flake b) Cement

Table 9: Mix Design of Graphite conductive concrete cathode used in this experiment

Materials	10% graphite concrete Mix design (gm)
Graphite	31.14
Cement	56
Fine Aggregate	128
Coarse Aggregate	106
Water	25

A rubber rectangular mold was cleaned with grease so that the final specimen could be removed easily. A wooden stick was put horizontally between the rubber mold to make it strong. Cement paste was poured into the mold and light temping was done to remove the void space. On the other hand, temping should not be too hard so that aggregate can be segregated. The cast specimens were kept under the 25-degree centigrade room temperature for 24 hours. After setting the concrete, specimens were kept in the water for 14 days.

Preparation of specimen

Cured specimens were checked for cracks and submerged again into the water minimum of 24 hours for creating a saturated condition. Because it was needed to create a similar condition to a concrete pipe or reservoir. Then saturated conductive concrete specimen was coated with PELCO conductive nickel paste. This nickel paste was from Tedpella USA and contain pure nickel flakes (8-14 µm). It has also fast drying properties. This nickel paste creates a thin, conductive, and flexible layer on the conductive concrete specimen. Nickel paste conductivity is 10% greater than silver paste and 10% better than graphite. The copper foil was wrapped up over the nickel paste and an electrical connection was made over the copper foil tape. It created ample space for electrical connections.

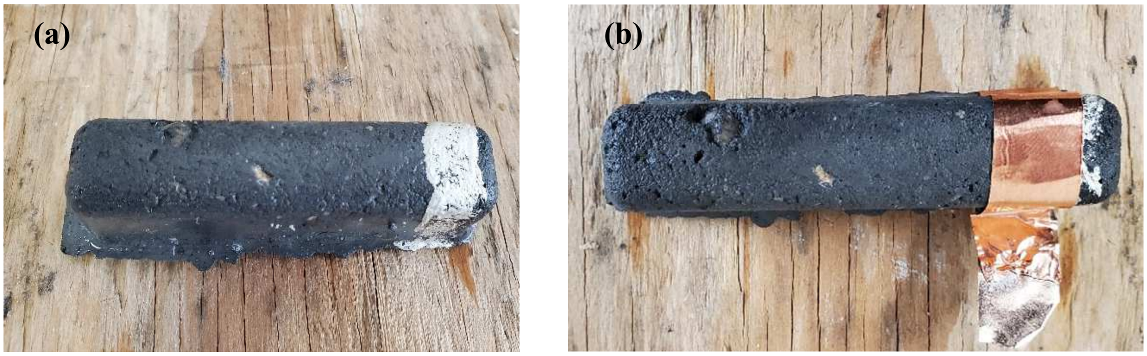


Figure 4: a) Applying nickel paste on conductive concrete cathode b) Appling copper tape over the nickel pastes in this experiment

Treatment Water

Tap water was used in the experiment and it was collected from the Edinburg Environmental engineering lab. The water sample was collected in a 1000 ml beaker after running the tap for 5 min. The initial hardness and ph. of sample water were 378 mg/L and 7.18. The initial free chlorine and total chlorine of tap water were 0.18 mg/L and 2.82 mg/L. The temperature of the sample water was 23 degrees centigrade.

Experimental Setup

The experimental setup was started by connecting the anode and cathode with a DC power source. The power source consists of a maximum voltage of 31 V and a maximum current of 5 A. Voltage and current can be regulated according to the requirement. Anode and cathode were connected with one end of two clamps and the other ends tied with two stands to make specimens vertical side by side. Dimensionally stable anode (DSA) was used as anode and Conductive concrete was used as cathode. Anode and cathode were kept parallelly and the distance was 0.5 cm.

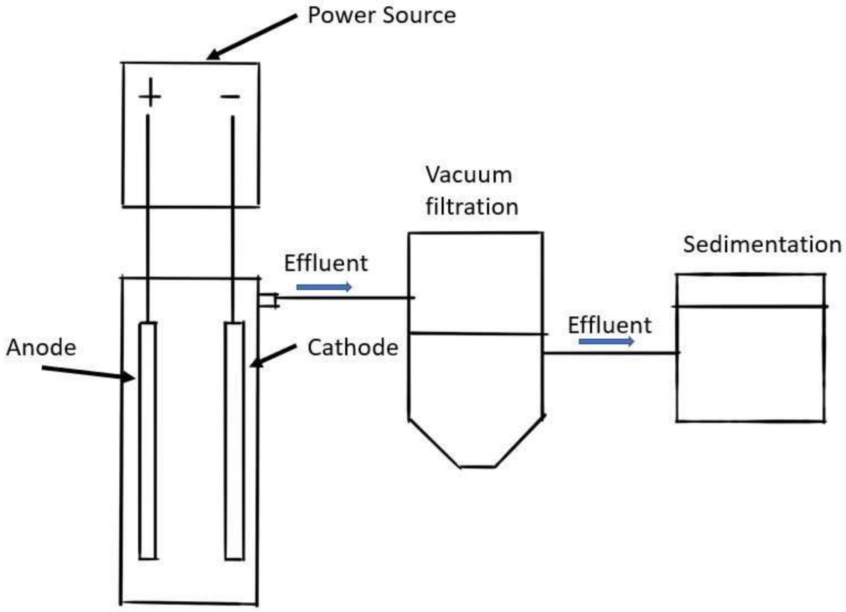


Figure 5: Schematic diagram of Electrochemical Precipitation

The dimension of the anode was 2inch * 6 inches. One end of the 18-gauge wire was attached with a crocodile clamp to connect with the electrodes and the other end was connected to the power supply. Finally, both electrodes were put into the 1L beaker. Sample tap water was put into the beaker and electrodes were submerged into the water as much as possible. Because maximum area will give better efficiency. There was no medium between the anodic and cathodic environment.

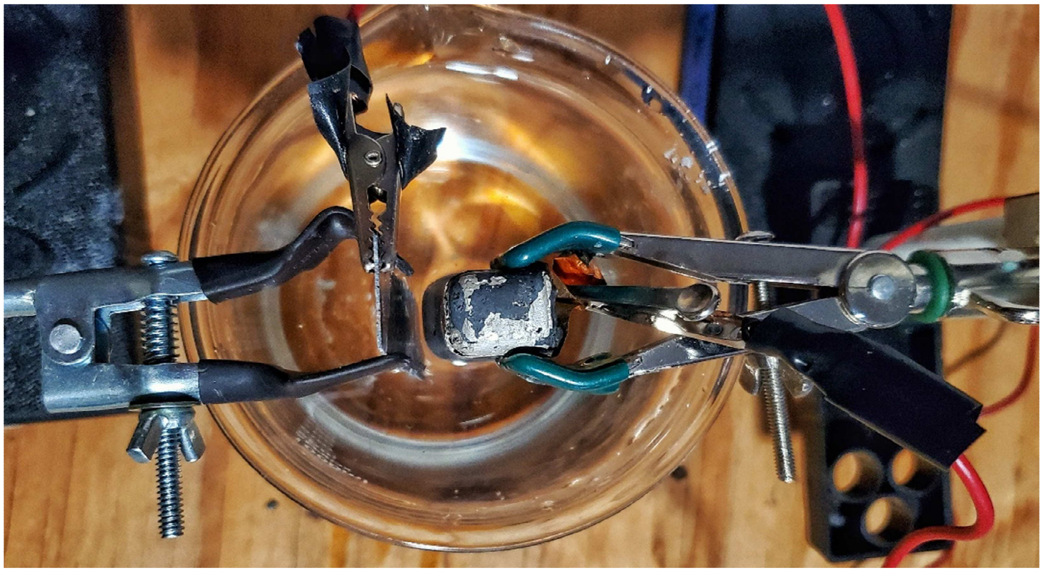


Figure 6: Top view of the experimental setup

In this experiment, the effect of chloride concentration, current density, pH, electrode distances, treatment time on free and total chlorine production, and hardness removal had been investigated. The experiment was continued for 10 min, 30 min, and 60 min. Constant 35 V was applied to each experiment. Inter electrode distances were 0.5 cm, 1 cm, and 1.5 cm. The concrete was used as a cathode with 10 % graphite content of the total volume.

Before conducting each experiment, initial pH, hardness, free chlorine, and total chlorine of sample water were measured. The experiment was started by pressing the power supply power on. Output current was recorded every 5 minutes during the experiment. After specified treatment time, treated water precipitation was removed by filtering through the 1 µm glass fiber filter connected to vacuum filter (WELCH 2534B-01A, Louisiana, USA). Then treated water pH, free and total chlorine, final hardness was measured. HACH DR 3900 was used to measure the free and total chlorine and a DPD powder pillow was used as a reagent.

For measuring the compressive strength of conductive concrete, three sets of cylindrical specimens with 5%, 7.5%, and 10% graphite content were made. Each set had three specimens. The diameter and height of the cylindrical mold were 10cm and 20 cm respectively. Graphite content concrete was placed into the mold and compaction was done to remove the void space between the concrete. The whole casting procedure was followed and maintained according to ASTM standards. After 24-hour casting, specimens were removed and put into the water for 28 days of curing. Specimens were named 10 T1, 10 T2, 10 T3 for 10% and 7.5T1, 7.5T2, 7.5T2 for 7.5% and 5T1, 5T2, 5T3 for 5 % graphite content.

Figure 7: Compressive strength specimen

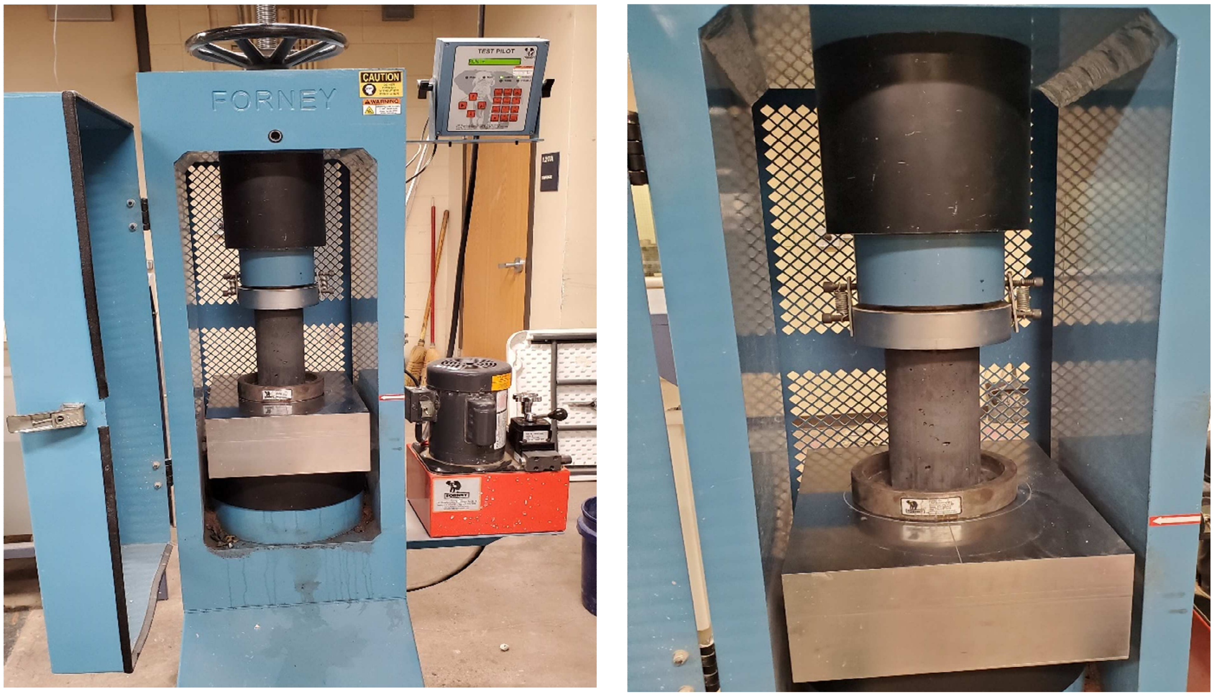


Figure 8: (a)Conducting compressive strength test on FORNEY F250. (b) specimen setup for the compressive strength test.

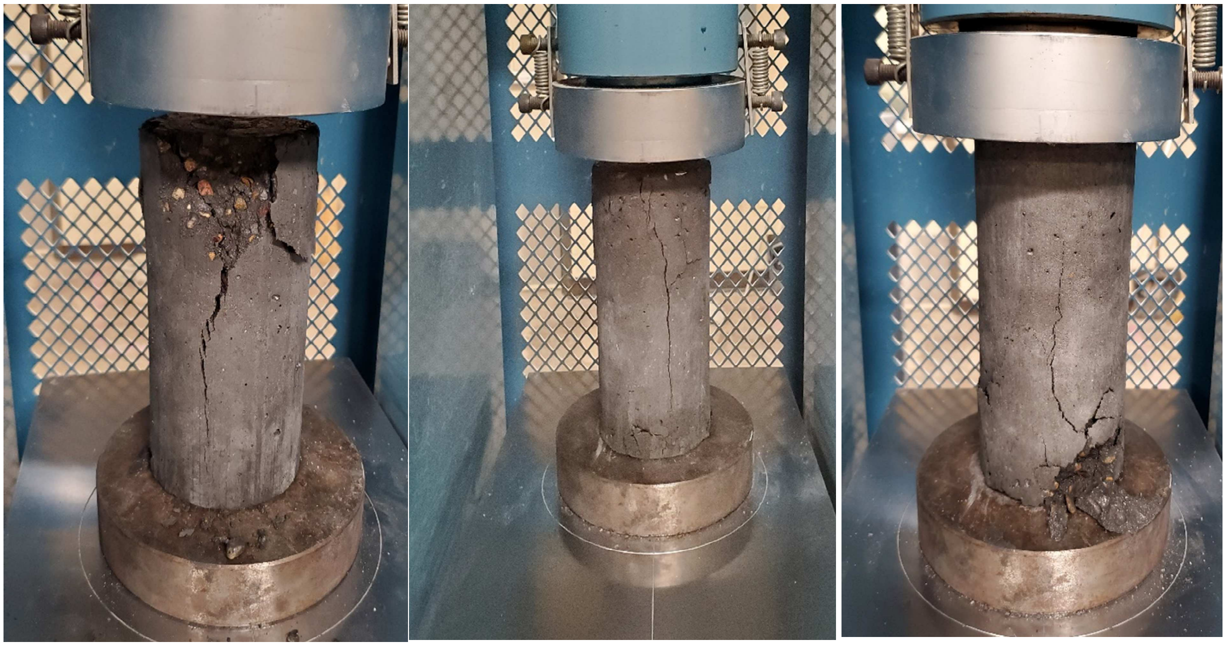
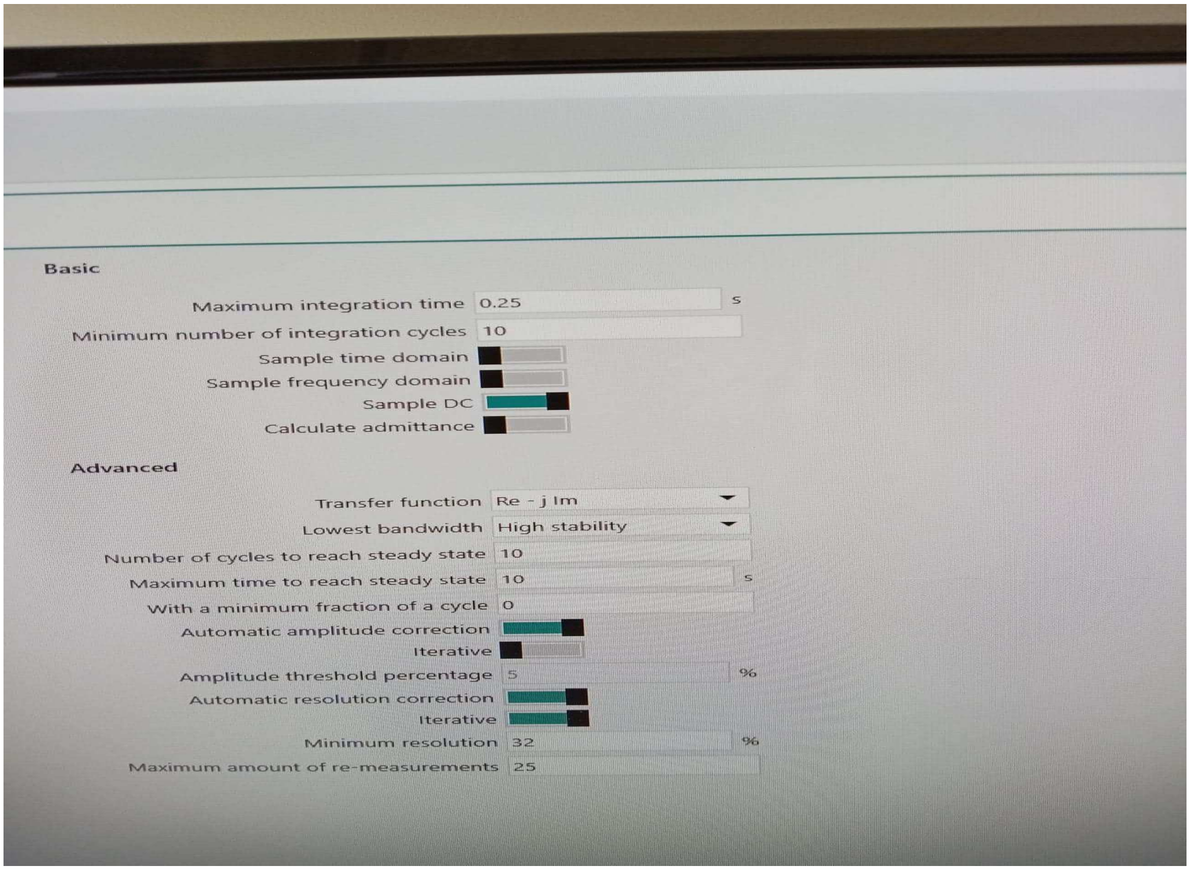


Figure 9: Failure of specimens 10T1, 10T2, and 10T3 under lateral loading.

For determining the resistivity of the conductive concrete, three sets of 5%,7.5%, and 10% graphite content of cubical specimens were made according to the same concrete mix design for compressive strength. The size of the specimen was 2 inches × 2 inches × 2 inches. After 28 days of curing, specimens were attached with copper tape by nickel paste and covered with polyethylene paper to keep the moisture constant. Then impedance is measured by impedance spectrometer.

Figure 10: Specimens for impedance measurement.

Before the measurement, several parameters are inputted. The maximum integration time was 0.25, the maximum number of integration cycles was 10, the amplitude threshold percentage was 5%, the number of cycles to reach a steady state was 10, the maximum resolution was 32 and the maximum amount of measurement was 25. Bode plot, Nyquist diagram, and resistance reading were measured by using an impedance spectrometer.



Basic

Maximum integration time 0.25 s
Minimum number of integration cycles 10
Sample time domain ☐
Sample frequency domain ☐
Sample DC ☑
Calculate admittance ☐

Advanced

Transfer function Re - j Im
Lowest bandwidth High stability
Number of cycles to reach steady state 10
Maximum time to reach steady state 10 s
With a minimum fraction of a cycle 0
Automatic amplitude correction ☑
Iterative ☐
Amplitude threshold percentage 5 %
Automatic resolution correction ☑
Iterative ☑
Minimum resolution 32 %
Maximum amount of re-measurements 25

Figure 11: Basic input parameter before impedance measurement.

CHAPTER V

RESULT AND DISCUSSION

Resistivity

To measure the electrical properties of specimens, Impedance Spetroscopy (IS) was used through out the experiments. Four types of information-real and imaginary impedance, impedance and phage angle-have been collected through IS using Alternate Current (AC) to visualize three graphs -Nyquist Diagram, Bode Plot Impedance and Bode Plot Phage Angle. One Hz frequcency of 1MHz frequency have been used for this study. Nyquist diagram has been drawn plotting real impedance in X-axis and imaginary impedance in Y-axis while bode plot impedance has been drawn plotting frequency in horizontal and impedance in vertical and bode plot phase angle has been drawn plotting frequency in horizontal and phase angle in vertical.

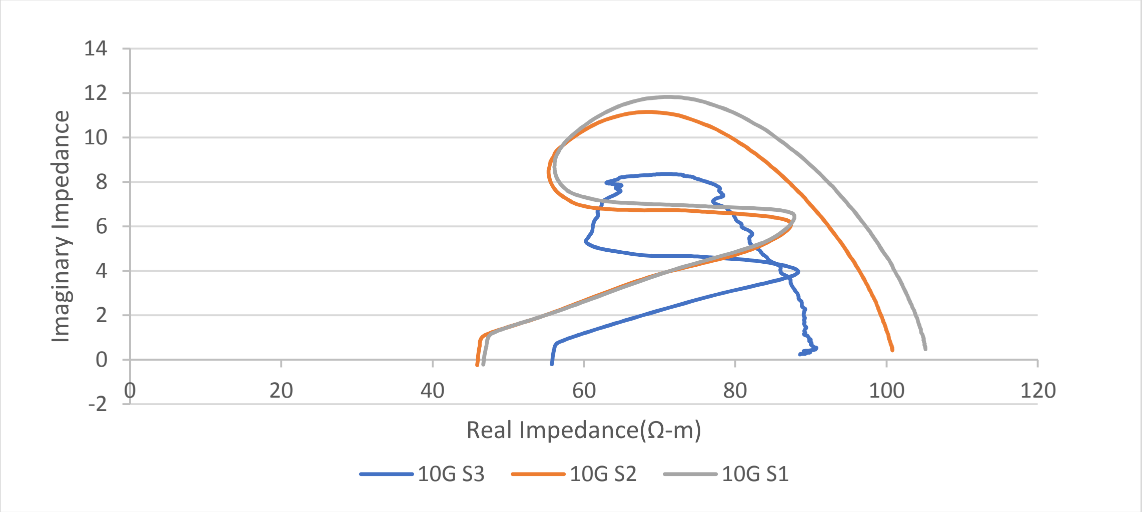


Figure 12: Nyquist diagram for 10 percent graphite

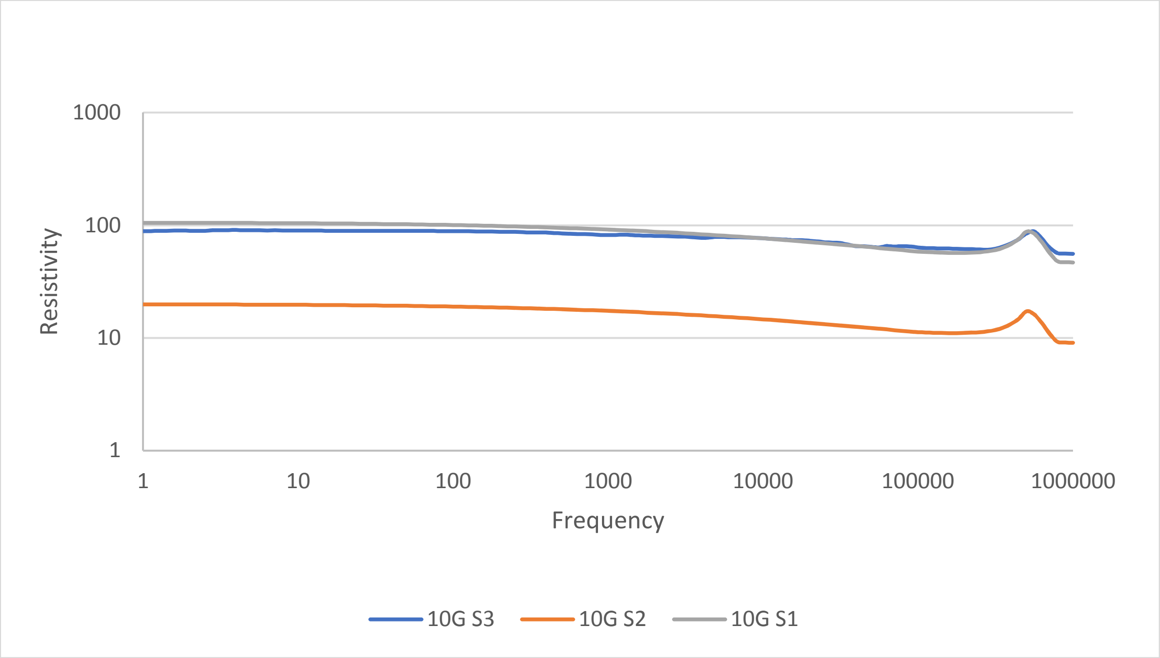


Figure 13: Bode plot diagram for 10 percent graphite

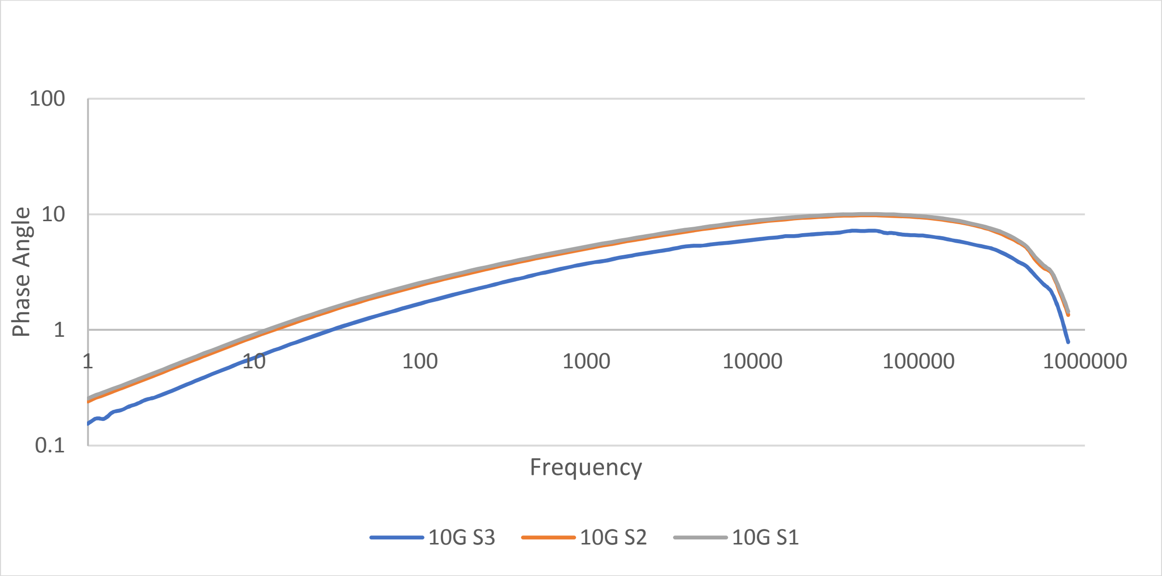


Figure 14: Phase angle diagram for 10 percent graphite

There is no significant difference in Impedance for 10 percent of graphite and 7.5 percent graphite. In 10 percent of graphite, impedance is slightly lower then 100 Ω.cm (Figure 13) and in 7.5 percent of graphite impedance is slightly above of 100 Ω.cm (Figure 16) (White for 5 percnet, it is not here). However, both of the specimens are conductive enough to pass the electivity. In higher frequency, the impedance decrease slowly, it express that the materials have some capacitance effects but it is negligible.

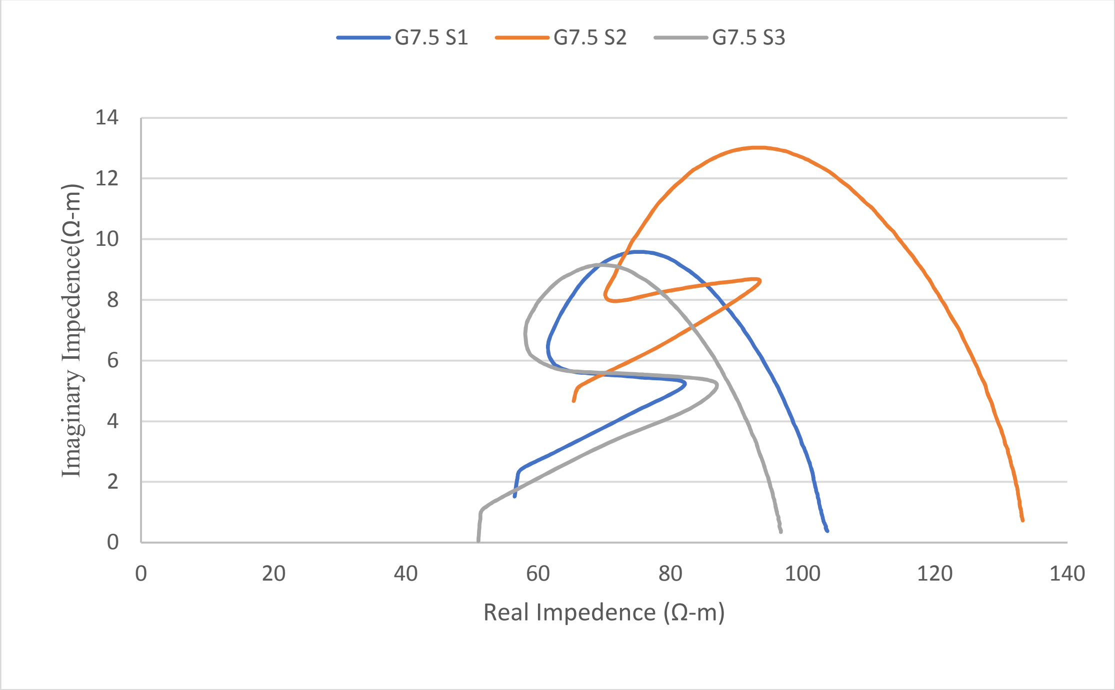


Figure 15: Nyquist diagram for 7.5 percent graphite

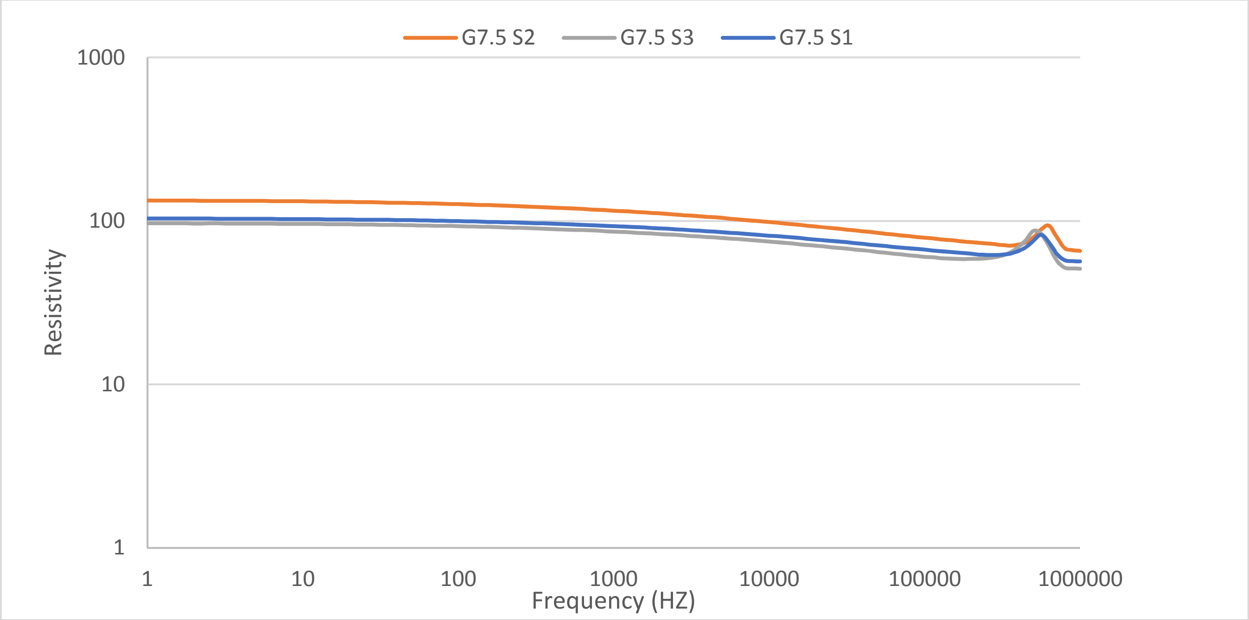


Figure 16: Bode plot diagram for 7.5 percent graphite

It is very difficult to describe the Nyquist diagrams for 10 and 7.5 percent of graphite as they are showing very fractuations (Figure 12 and 15) but graphs are semicircles for 5 percent of graphite (Figure 18). Two parallel metal plates are separated by insulator materials behaves like a capacitor and materials that hinder to pass electricity behave like resistor. Both types of process are available in cement concrete. This semicircle that come from bulk impedance expresses that, the capacitor and the resistor are connected in parallel way.

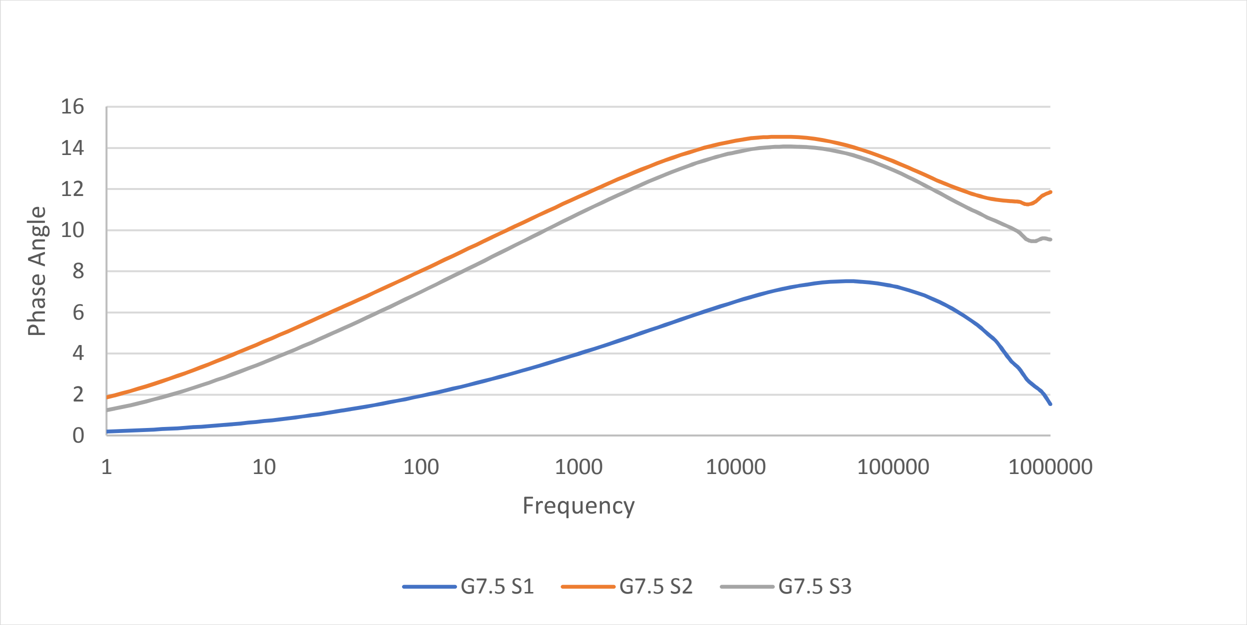


Figure 17: Phase angle diagram for 7.5 percent graphite

Phase angle for 10 percent and 7.5 percent of graphite start from almost 0 degree (Figure 14 and Figure 17), for 5 percent of graphite, graphs start from 5 degree (Figure 19). While frequency increase, the phase angle increase gradually. Pure resistor shows 0 degree phase angle, if capacitane behaviour raise phase angle increase. In low frequency, the specimens show no capacitance effects but in high frequency, they show very small capacitance effects

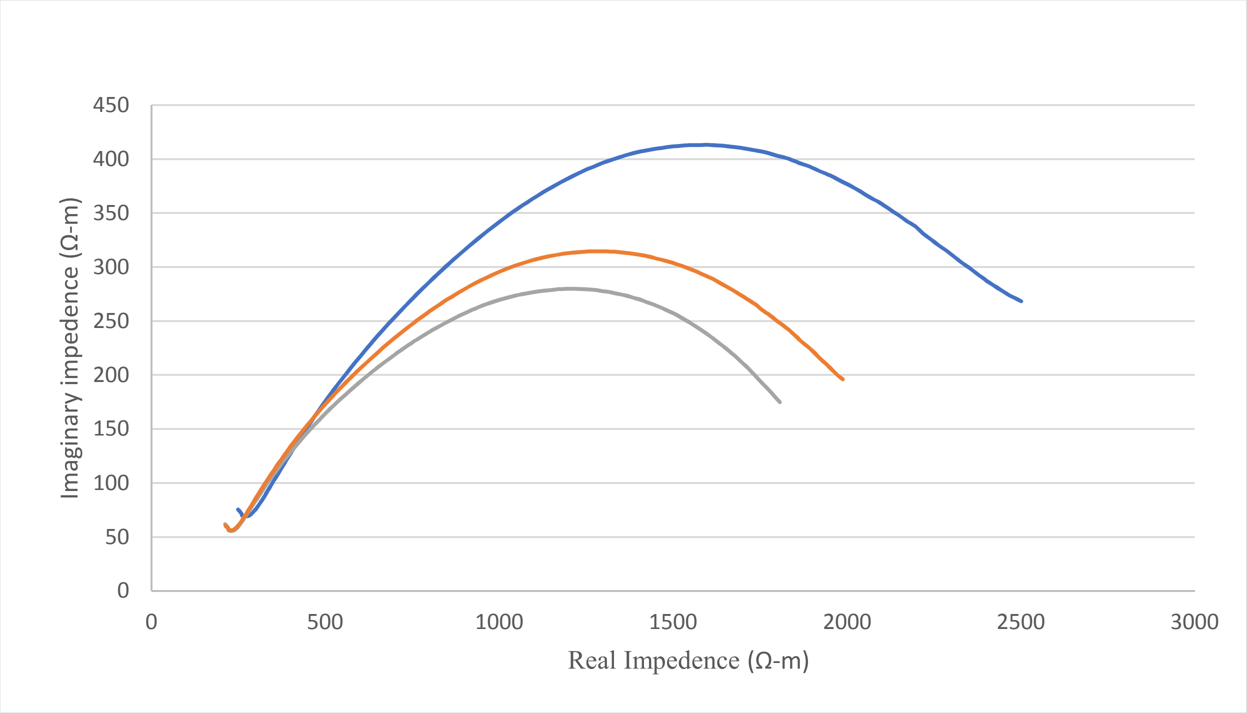


Figure 18: Nyquist diagram for 5 percent graphite

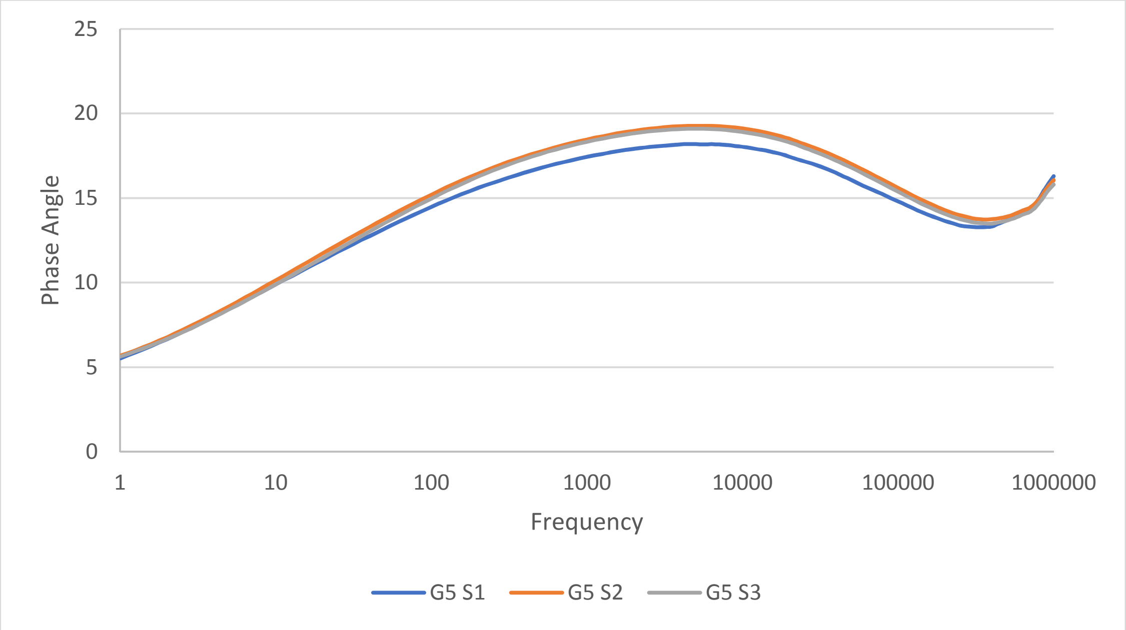


Figure 19: Phase angle diagram for 5 percent graphite

Retention Time

The performance of the innovative EP time is significantly influenced by retention time. In the experiment, a considerable increase in free chlorine was seen under operational settings of 0.5cm electrode gap, 31V power supply, and similar electrode configuration. The free chlorine formation increased from 0.09 mg/L to 0.34 mg/L after 30 minutes of treatment, and this rate increased with retention time. The free chlorine increased to 0.75 mg/L after 60 minutes of treatment in all setups. Other previous research discovered similar results. (saha et al.,2017, Ghernaout et al.,2010). The production of free chlorine rose as treatment time increased. Because the amount of OH- also increased with time which reacts with Cl- and creates free chlorine. On the other hand, total chlorine concentration decreased with an increase in retention time. Total chlorine in sample tap water was 1.53 mg/L and after 10 min of treatment, it increased to 1.36 mg/L. After 10 minutes, it deteriorated.

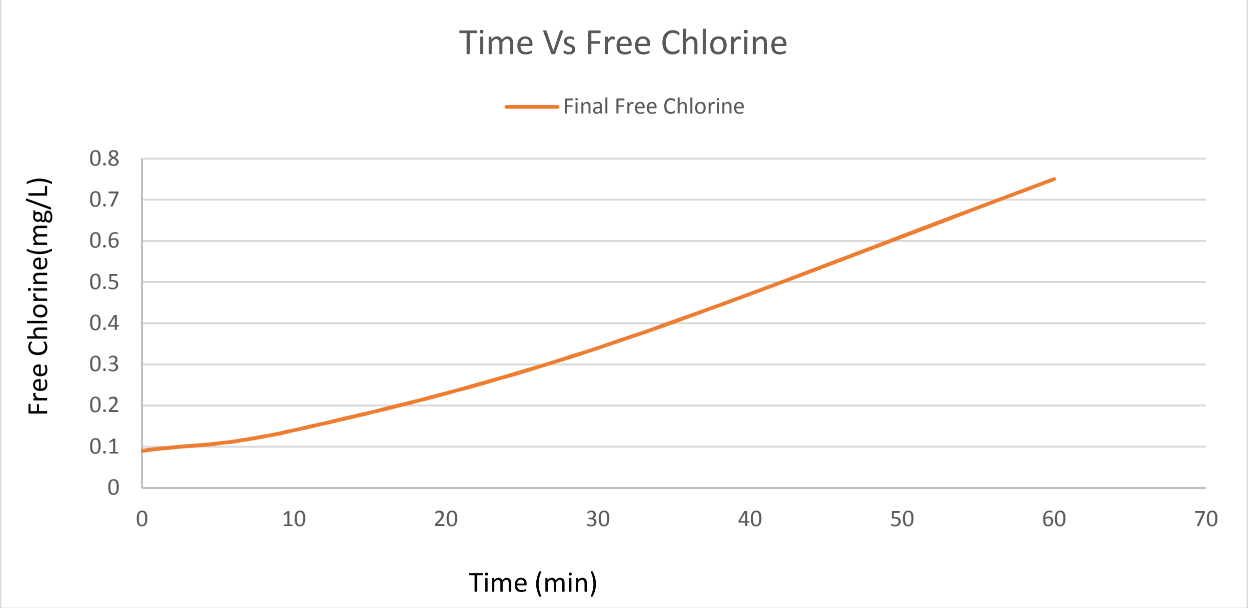


Figure 20: Effect of time on free chlorine production

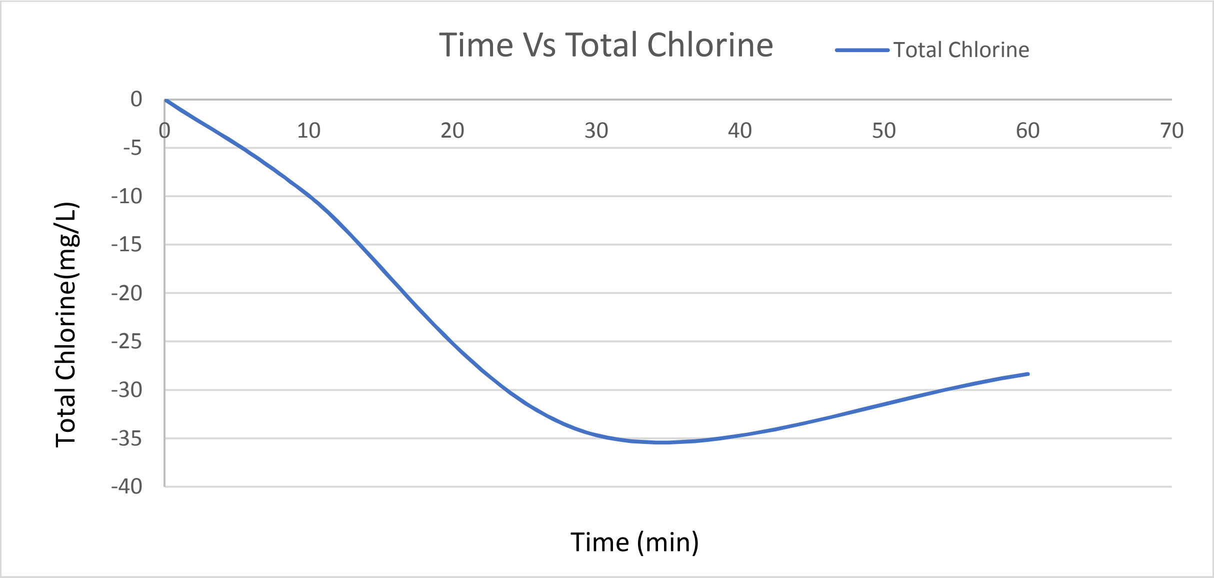


Figure 21: Effect of time on total chlorine production

pH

Previous studies found that pH affects the performance of EP. Malakootian et al. (2010) showed that pH has a significant impact on the performance of EP. He used EP process in hardness removal and found that initial pH (acidic or alkali?) gave better hardness removal performance. Saha et. (2017) conducted an electro chlorination experiment for about 50 min by varying pH ranging from neutral to alkaline conditions (7-10). He found that the formation of free and total chlorine slightly increased at pH 8.5. It was known that chloride oxidation accelerated in an alkaline environment (Patil et al.,2014). At pH 8.5, a current density of 2.5 mA/cm2 and an accessible chloride concentration of 8.5 mg/l, the maximum active chlorine concentration of 0.75 mg/l was found. (saha et al.,2017). From these studies, it has been found that after 60 min of treatment pH increased to 8.7 from 7.18 and free chlorine production was 0.78 mg/L which was very close to the previous studies. On the other hand, amount of total chlorine slightly reduces with the increase of pH, although the previous researcher found the increase of total chlorine with an increase in pH. It can be assumed that total chlorine reduction was due to the formation of DBP in the EP process.

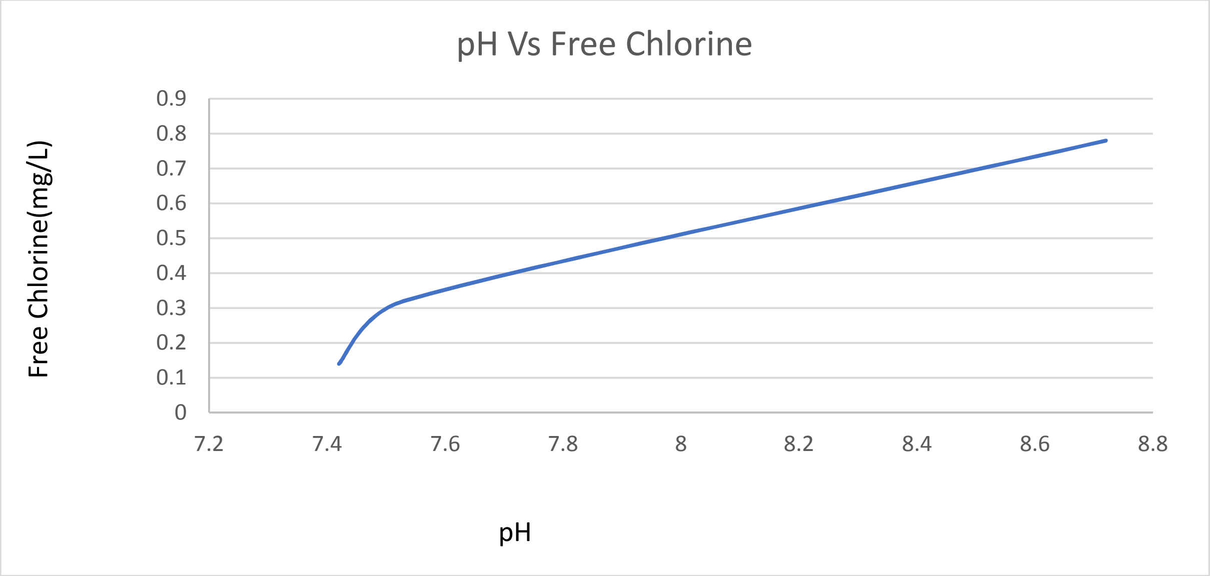


Figure 22: Effect of pH on free chlorine production

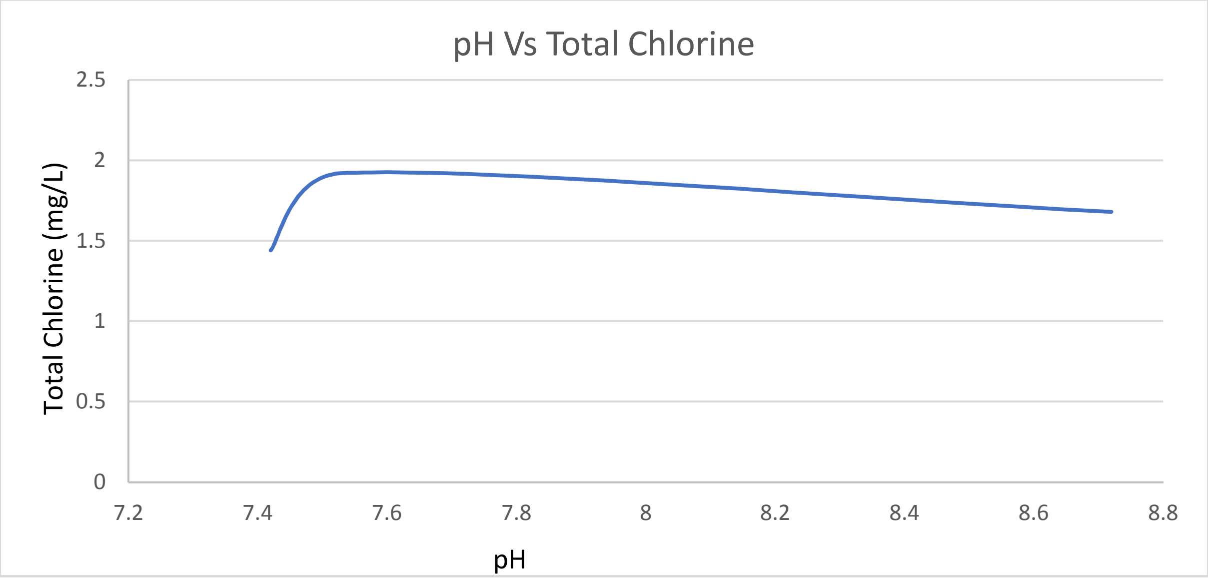


Figure 23: Effect of pH on total chlorine production

Current Density

Current density is a major factor in efficient electrochemical free and total chlorine production. In this EP process, the result of the production of free and total chlorine was studied with 10 % graphite conductive concrete cathode. During the experiment, the inter-electrode distance was 0.5 cm, the applied voltage was 31V and the initial free chlorine was 0.14 mg/L. It was seen that free chlorine increased with the current density. At the current density of 14.5 mA/cm2, free chlorine concentration was 0.14mg/L and it increased to to 0.78 mg/L at the elevated current density of 26.2 mA/cm2. The previous researcher showed the same result about free chlorine production. Saha et al. (2017) carried out an Electro-chlorination test for 60 minutes with various low current densities (0.1, 0.2, 0.3, 0.4, 0.5, 1, 1.5, 2, 2.5 mA/cm2), which evolved active chlorine species at a constant chloride concentration of 8.5 mg/l, which is usually measured in tap water. At 1.5 mA/l, the free chlorine and total chlorine reached a maximum of 0.7 and 0.75 mg/l, respectively.

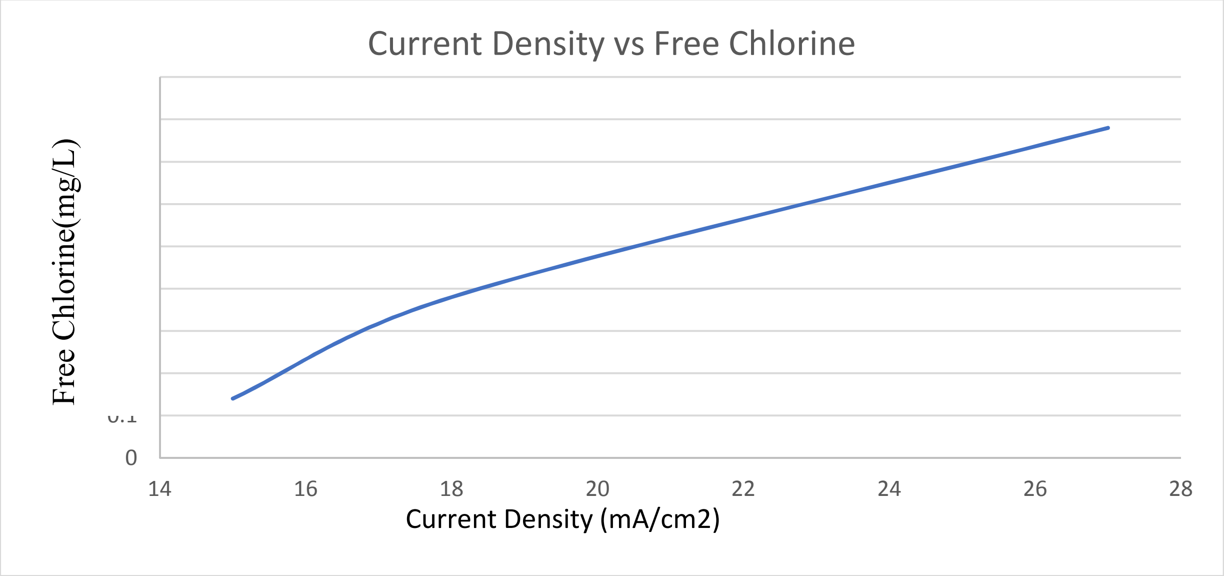


Figure 24: Effect of pH on total chlorine production

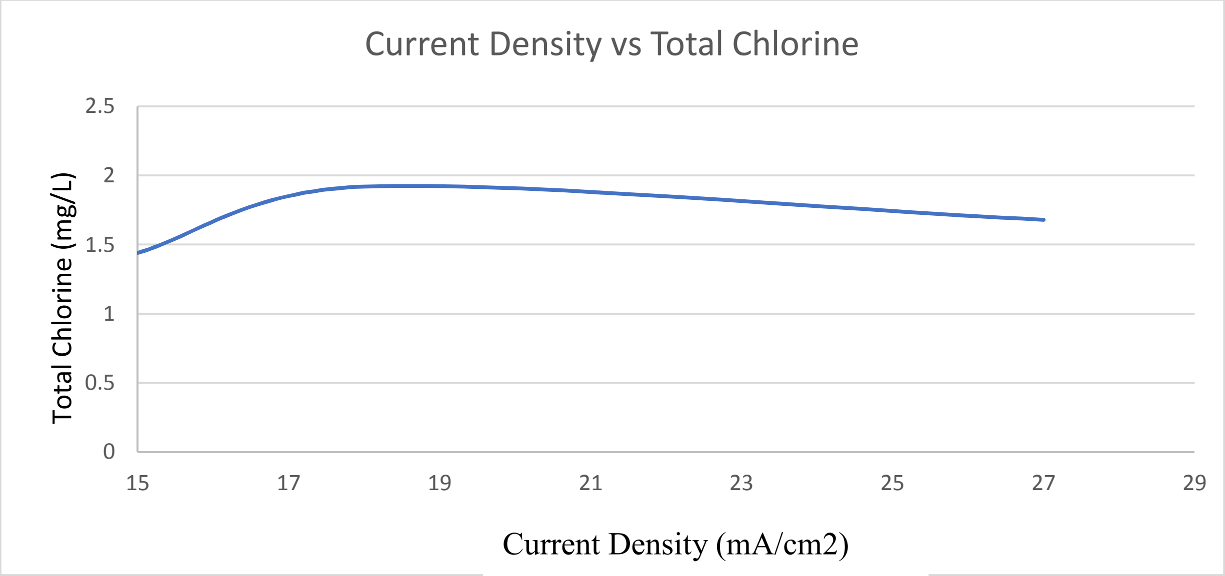


Figure 25: Effect of current density on total chlorine production

Chloride Concentration

The effect of chloride ions on the active chlorine production rate was investigated for the accessible number of chlorides in regular tap water at a concentration ranging from 38 mg/L to 140 mg/L at room temperature and current intensities ranging from 0 to 5A. The goal of this study was to see if hypochlorite could be made from chloride ions found in ordinary tap water. It was observed that at the chloride concentration of 38 mg/L, free chlorine production was 0.38 mg/l. After an increase in chloride concentration, free chlorine production increased, which is similar to the previous study. On the other hand, total chlorine concentration decreased with an increase in chloride concentration, which is different from the previous research. It may be due to precipitation of chloride ions and evaporation of combined chlorine.

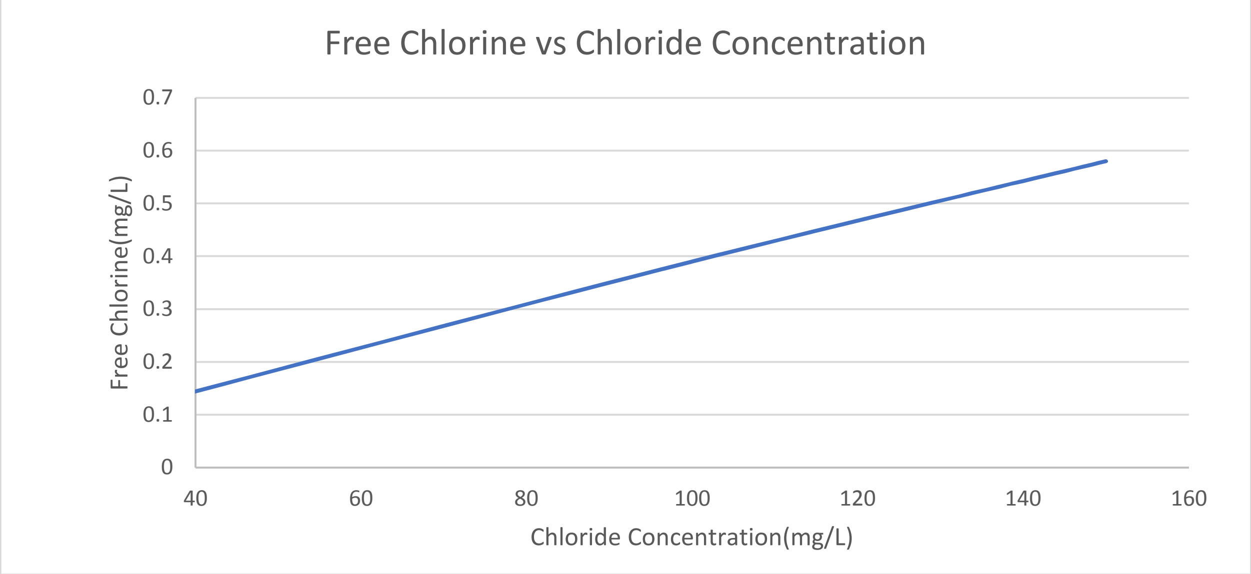


Figure 26: Effect of chloride concentration on total chlorine production

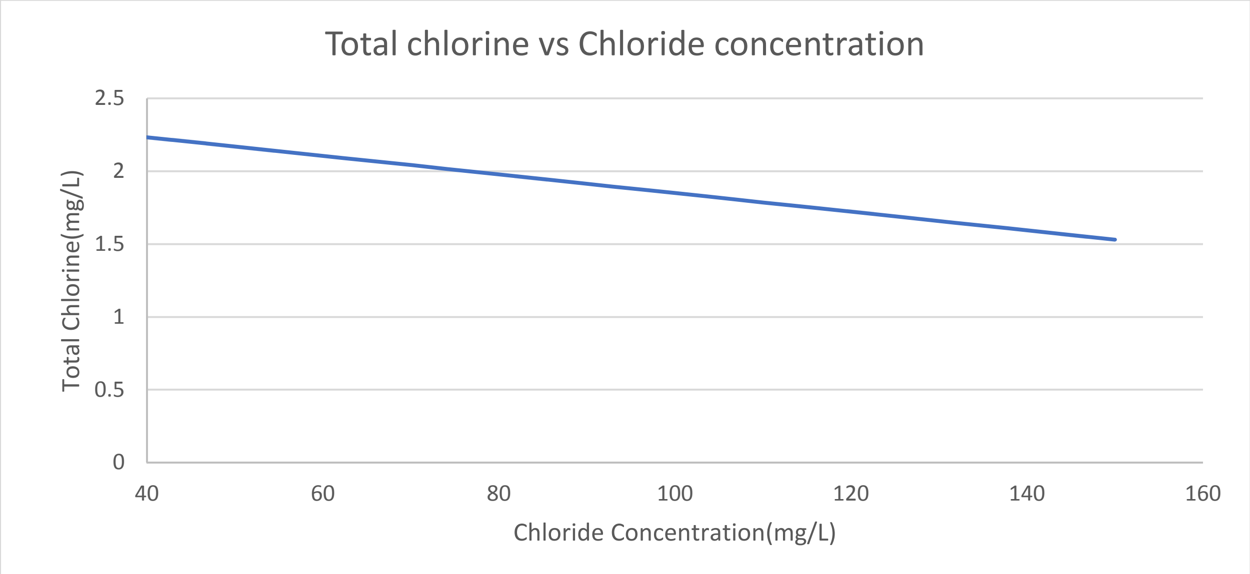


Figure 27: Effect of chloride concentration on total chlorine production

Cathode Material

Cathode material plays an important role in producing free chlorine. (saha et al.,2007). He showed that different electrode materials under the same condition produce different amounts of free chlorine. Energy consumption was found to be the cause of this phenomenon. More energy consumption resulted in more free chlorine production. Saha et al. (2007) also reported that platinum cadmium mesh with 580 mg/L electrolyte solution produces 1.6 mg/L free chlorine. In addition, iridium oxide with 50 mg/L electrolyte solution produces 1.6mg/L free chlorine. On the other hand, conductive concrete with 1000 mg/l electrolyte solution produce 0.85 mg/L free chlorine. It can be said that free chlorine production depends on cathode material.

Compressive Strength

Three cylindrical specimens for 5%, 7.5%, and 10% with a diameter of 10 cm and a length of 20cm were tested. Peak load bearing capability was measured while the lateral load was applied. The specimen's detailed result may be seen in the table8. The compressive strength decreased with the increase of graphite percentage because graphite reduces the cohesion between cement and aggregate. The compressive strength of standard concrete should be roughly 2500-4000 psi. There are two causes for the lack of compressive strength in these specimens. The first is the particle size. The coarse aggregate size has a significant impact on the concrete's performance. (Tasdemir et al., 1996; Mihashi et al., 1991). Concrete does not show linear fracture due to its heterogeneous nature. As a result, applying linear fracture mechanics to concrete becomes incredibly challenging. To overcome the low strength , particle size can be increased. Secoend is the reduction of the graphite content in the mixture. From the flowing figure , it was seen that low graphite content gives more strength. Please add a couple more lines about possible ways to overcome low strength.

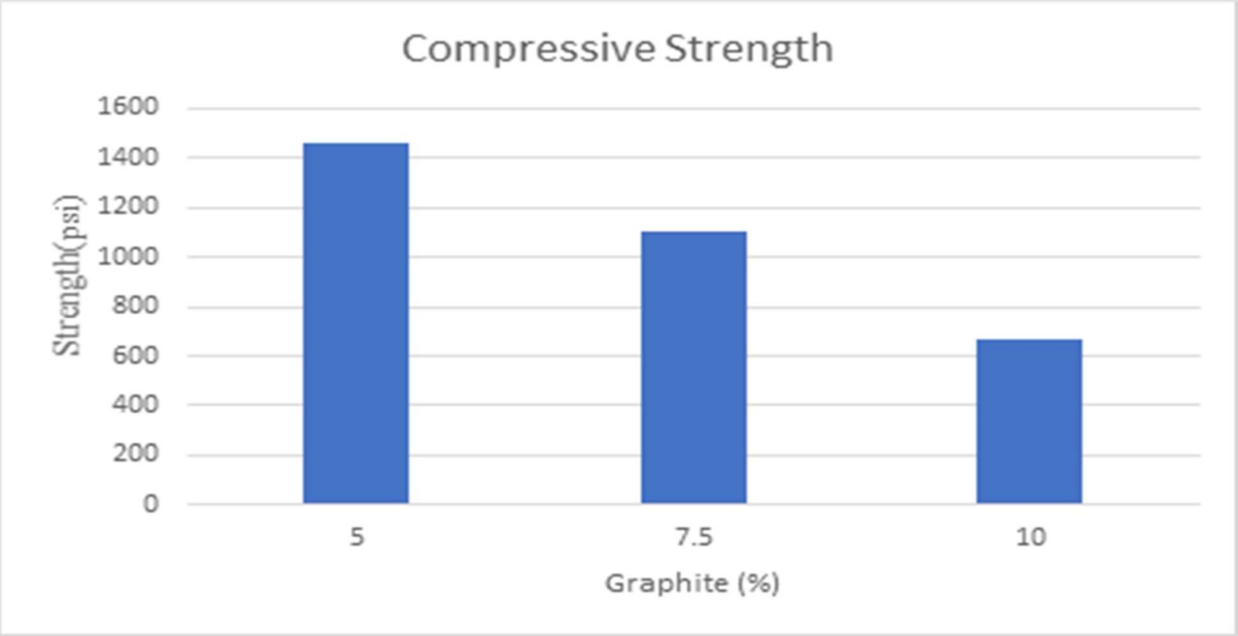


Figure 28: Compressive strength for 5%,7.5% and 10% graphite content

Table 10: Compressive strength test of 10%,7.5% and 5% graphite concrete cylinder

Specimen	Weight (lb)	Load	Area (Sq. Inch)	Compressive Strength (psi)
10 T1	7.55	11920	12.566	7600
10 T2	7.55	10855		10830
10 T3	7.60	9960		6590
7.5 T1	7.65	11085	12.566	11085
7.5 T2	7.60	16765		16765
7.5 T3	7.55	13790		13790
5 T1	7.65	18650	12.566	18650
5 T2	7.65	17830		17830
5 T3	7.60	18455		18455

CHAPTER VI

CONCLUSION

A series of experiments were performed to find the optimal operating condition for the novel EP system. The experiments were conducted in lab-scale batch operations. It was observed that operations were affected by retention time, chloride concentration, current density, pH andcathode material. The free chlorine production was 0.79 mg/L in 60 minutes. This is enough kill microbes present in water. According to EPA, 0.4mg/L free chlorine should be present in tap water for water safety against future contamination. The highest free chlorine production was achieved at the operational condition of 31V, .5cm of inter-electrode distance, 60 minutes of treatment, and 10% graphite concrete cathode. Thus, it was demonstrated that this process may be used as a pretreatment step not only for water hardness removal but also for free chlorine production. To incorporate conductive concrete into large-scale treatment programs, additional research on the material's structural stability is required. Additionally, it is crucial to conduct more research on the treatment facility's economic viability. Because in electrochemical treatment methods, effective and acceptable energy usage and operational costs are essential to the success of real-world applications. There is a need for larger-scale practical investigations and pilot-scale research

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APPENDIX

APPENDIX

Experimental Data

Table 11: Summery of total experimental data

Time(min)	Chloride content	Initial pH	Final pH	Current Density	Initial free chlorine	Initial Total Chlorine	Final Free Chlorine	Final Total Chlorine
10	39mg/L	7.18	7.57	0.202	0.08	1.42	0.14	1.36
10		7.18	7.27	0.212	0.09	1.58	0.14	1.49
10		7.12	7.42	0.209	0.08	1.54	0.11	1.48
30		7.21	7.59	0.246	0.19	2.94	0.3	1.86
30		7.23	7.43	0.257	0.17	2.95	0.34	2.08
30		7.2	7.57	0.263	0.16	2.82	0.38	1.82
60		7.2	8.76	0.383	0.09	2.12	0.75	1.62
60		7.18	8.72	0.376	0.1	2.15	0.78	1.54
60		7.22	8.69	0.379	0.8	2.19	0.73	1.61

BIOGRAPHICAL SKETCH

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