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LOWER RIO GRANDE VALLEY TAP WATER HARDNESS REMOVAL
USING CONDUCTIVE CONCRETE CATHODE IN
ELECTROCHEMICAL PRECIPITATION

A Thesis
by
MIRZA ADDAITO BILLAH

Submitted to the Graduate College of
The University of Texas Rio Grande Valley
In partial fulfillment of the requirements for the degree of
MASTER OF SCIENCE

May 2021

Major Subject: Civil Engineering

LOWER RIO GRANDE VALLEY TAP WATER HARDNESS REMOVAL
USING CONDUCTIVE CONCRETE CATHODE IN
ELECTROCHEMICAL PRECIPITATION

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May 2021

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ABSTRACT

Billah, Mirza Addaito, Lower Rio Grande Valley Tap Water Hardness Removal Using Conductive Concrete Cathode in Electrochemical Precipitation, Master of Science in Civil Engineering (MS), May 2021, 56 pp, 24 figures , 11 tables, 99 references

Electrochemical water softening has obtained significant attention in recent years due to being environmentally friendly. One of this method's significant problems is the requirement of high cathodic surface area for enough efficiency. As most electrochemical cells use costly metal cathodes with meager service life and low structural integrity, practical large-scale implementation is scarce. In this research, graphite-concrete cathodes were proposed to replace conventional metal cathodes in the electrochemical water softening process. Graphite concrete has been used for heating pavement and electrical conductive roads. In this research concrete mixed with graphite powder by volume was successfully used as a cathode to treat LRGV(Lower Rio Grande valley) tap water. In this Electrochemical precipitation process, the total removal efficiency was achieved up to 53% at 35.5 Volt with a retention time of 60 minutes and an inter-electrode distance of .5cm. The 10% graphite-concrete cathodes were responsible for this performance the decent efficiency suggested that graphite concrete cathodes are viable alternatives to metal ones in hardness removal from tap water. Also, optimal performance parameters such as inter electrode distance, material and retention times were discovered. In this research it was concluded that with perfect conditions, conductive concrete can be used as

structural material. Furthermore, it can provide efficient and effective water hardness treatment for the tapwater. Further research is needed on flowing water and heavy metal removal to know the true potential of this electrochemical precipitation method.

DEDICATION

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 mother Nazifa Khatun and Father Mirza Moatasim Billah. It all started from their struggle and sacrifices. All credit goes to them that I enjoy living every single day of my life.

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I would acknoweldge my supervisor Dr.Jong min Kim. He was not only an excellent academic supervisor, but also turned me into the person I am today. Thanks to him I was able to conduct this research with confidence and pride. Also I would like to mention Dr.Philip park who had been meticulously guiding us through thick and thin.

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CHAPTER I

INTRODUCTION

Despite having a high-demand, a continuous supply of high-quality water for human consumption is very limited in the modern world. The rapid growth of the human population and unprecedented industrialization is continuously increasing the demand for suitable water (Gude, 2015; Jury and Vaux, 2005). This phenomenon led to a heightened interest in developing cost-effective, safe, and environmentally friendly water treatment technologies that can serve both domestic and industrial needs for water.

Soluble solids in water are the significant impurities to be removed in the water treatment processes. Among them, water hardness has been an essential quality criterion (Saurina et al., 2002). Water hardness is a condition mainly found in groundwater, a crucial water usage source in the modern world. In the united states alone, 44% of the population that includes 99% of the rural population, depends on groundwater as a primary source of drinking water (Kenny et al., 2009). In the water cycle, precipitated water enters the ground and recharges the water table underneath. During the process, water encounters limestone and dolomite deposits resulting in the dissolution of calcium and magnesium, two major hardness cations. Other minor water hardness contributors like strontium, aluminum, barium, iron, manganese, and zinc enter the water in the same process. (Entezari and Tahmasbi, 2009; Kabay et al., 2002; Sengupta, 2013; Soltanieh and Mousavi, 1999; Yildiz et al., 2003).

Water hardness can be classified into soft (0-75 mg/L as CaCO_3), moderate (75-150 mg/L as CaCO_3), hard (150-300 mg/L as CaCO_3), and very hard water (above 300 mg/L as CaCO_3) (Kalash et al., 2015b). It is known that people tend to pleasantly consume water with the hardness not exceeding 300 mg/l as CaCO_3 (Organization, 2010). American Water Works Association recommended keeping the water hardness between 80-100 mg/l as CaCO_3 (Kalash et al., 2015b). Hardness in untreated groundwater is much higher than those ranges recommended by WHO (2010) and Kalash *et al.* (2015).

High hardness in the groundwater causes severe problems in both residential and industrial applications. Hard water forms scales on metal surfaces and clogs water supply lines gradually. Scaling issues accelerate in the presence of a heating element, which can reduce heat transfer efficiency significantly in boilers and cooling towers (Ghizellaoui et al., 2005; Lima et al., 2004; Park et al., 2007; Saurina et al., 2002). The alarming fact is that more than 85% of boilers in the United States were reported to be supplied with hard water supplies (Brastad and He, 2013). A large percentage of United States population is prone to hard water (Fig 1). Areas like Lower Rio Grande valley (LRGV) has been supplied with tap water with high hardness of 298.3 mg/L as CaCO_3 (Perez and Freese, 1997) since hardness removal is not mandated by the US environmental law. Most of the high-quality manufacturing processes require process water with very low hardness (Viero et al., 2002). If not adequately removed, high hardness in water leads to expensive repairs on broken water supply lines, boilers and colossal energy loss. Resolving this issue is a significant concern for many leading industries.

Numerous methods have been introduced to reduce water hardness. Popular ones include chemical precipitation and ion exchange, which require extensive chemical use (Bergman, 1995; Fu et al., 2009; Gabrielli et al., 2006; Yeon et al., 2004). More advanced methods that do not

necessitate chemical addition are reverse osmosis, electrodialysis, nano-filtration, crystallization, distillation, and evaporation (Bequet et al., 2000; Low et al., 2008; Saurina et al., 2002; Tabatabai et al., 1995) However, these technologies are expensive and require substantial energy input. Some of those advanced technologies need additional treatment afterward, causing them to be undesirable. Also, the advanced technologies' concentrated byproducts can be a serious threat to nature (Malakootian et al., 2010; Tabatabai et al., 1995; Tlili et al., 2003a; Zeng et al., 2007; Zhi and Zhang, 2016).

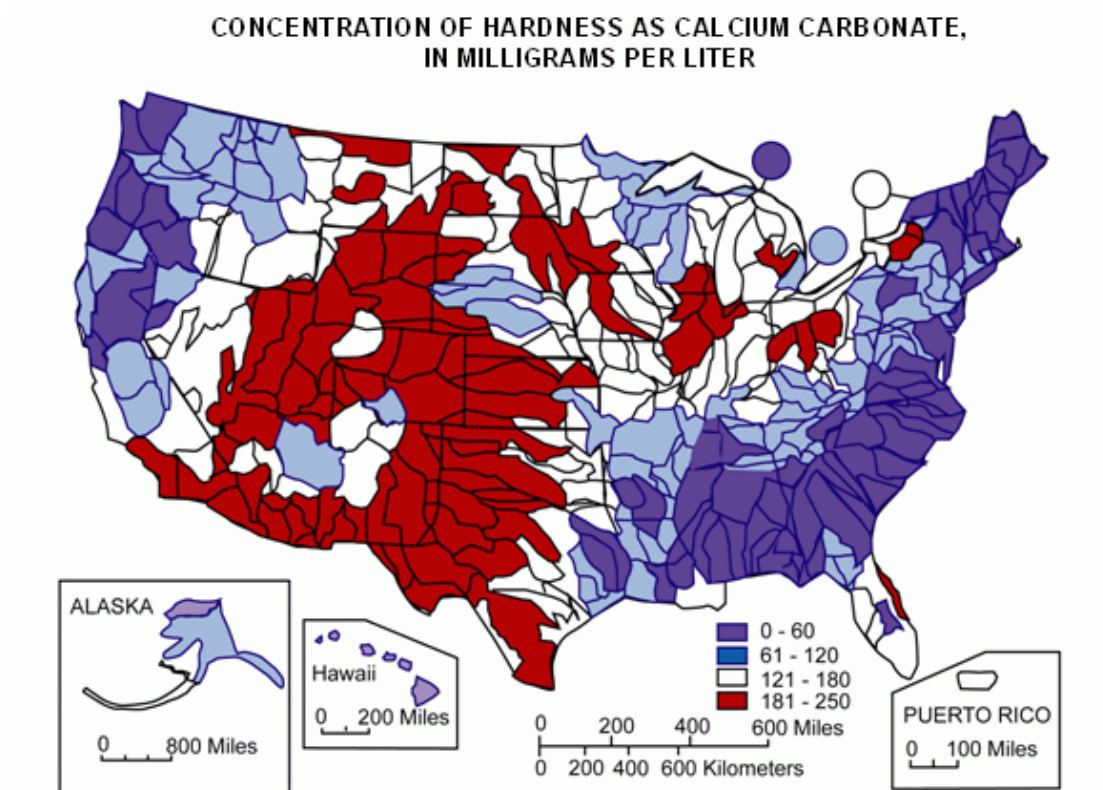


Figure 1:Hardness Concentration map of United States(USGS)

One technology that has the potential to overcome such shortcomings of existing hardness removal technologies is electrochemical water treatment (Gabrielli et al., 2006; Low et al., 2008; Malakootian et al., 2010; Tlili et al., 2003a, 2003b). In the last decades, electrochemical

technologies have advanced drastically as environmentally friendly treatments (Anglada et al., 2009; Cañizares et al., 2009; Comninellis and Chen, 2010; Emamjomeh and Sivakumar, 2009; Martínez-Huitle et al., 2015; Martínez-Huitle and Ferro, 2006; Rajeshwar and Ibanez, 1997; Sirés et al., 2014, 2014; Subba Rao and Venkatarangaiah, 2014; Wu et al., 2014). Electro flotation (Casqueira et al., 2006; da Cruz et al., 2016; Emamjomeh and Sivakumar, 2009; Hosny, 1996; Janssen et al., 1984; Maksimov and Ostsemin, 2015; Sarkar et al., 2011), electrocoagulation (Ahmadzadeh et al., 2017; Barrera-Díaz et al., 2014; Fajardo et al., 2017, 2014; Gönder et al., 2017; Hernández et al., 2017, 2017; Sahu et al., 2017; Vasudevan et al., 2013, 2011, 2009), and electrochemical precipitation (EP) are some of many electrochemical treatments that have produced promising study results in removing heavy metals, dyes, organic and inorganic compounds.

Sustainable hardness removal from the water was reported from previous electrochemical treatment studies (Gabrielli et al., 2006; Hasson et al., 2008; Lédion and Leroy, 1994; Sanjuán et al., 2019a). Among them, the EP process is newly developed, and the number of research works focusing on EP is on the rise in recent years. The EP process has shown potential in hardness removal without chemical additives and with minimum sludge generation. However, EP has been reported to require large cathodic areas for sufficient precipitation (Hasson et al., 2011, 2010). For industrial scale implementation, cathode with high surface area is required for sufficient performance. Metal cathodes are exposed to rust and deteriorate very fast. Frequent change of cathodes is required very frequently due to mentioned problems. This not only makes the process hard to implement but also makes it economically not feasible.

Conductive concrete is a promising technology in the civil engineering industry. Electrically conductive concrete (ECON) has been providing excellent service to transportation infrastructure in recent years. It is an established and structurally reliable method to remove ice and snow from the surface. (Abdualla et al., 2018; Ceylan et al., 2014; Pan et al., 2015; Sadati et al., 2018; Sassani et al., 2018). These conductive concretes are made by adding electrically conductive materials in normal concrete mixtures. Carbon fibers, steel shavings, graphene powder are added as conductive materials for conductive concrete. (Arabzadeh et al., 2019; Notani et al., 2019; Sassani et al., 2017). In this research graphite powder has been used to make the concrete conductive. This is a novel method where conductive concrete has been used as an cathode in an electrochemical precipitation system.

CHAPTER II

LITERATURE REVIEW

In this chapter theoretical background of electrochemical precipitation and latest progress in the field of electrochemical water hardness treatment has been discussed.

A standard electrochemical precipitation cell consists of a DC power source, pair or multi electrodes setup and treatment water. In a conventional EP setup (Fig. 1), both electrodes are submerged in water. Function of the cathode is to generate alkalinity and be the scale deposition

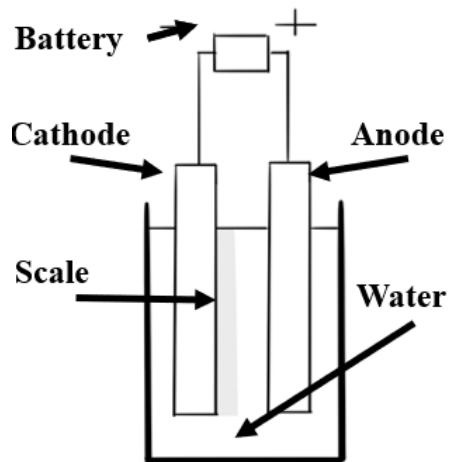


Figure 2:Electrochemical precipitation

surface. No medium is needed for the separation of anodic and cathodic environments. The high pH condition is necessary to promote precipitation near the cathodic surface as a thin layer. As a

result, the precipitation reaction occurs exclusively in the thin water film adjacent to the cathodic surface (Tlili et al., 2003b, 2003a).

During water electrolysis, dissolved oxygen is reduced on the cathode in a large potential range. A basic environment near the cathodic surface is created by the following cathodic reactions (Gabrielli et al., 2006; Kalash et al., 2015b):

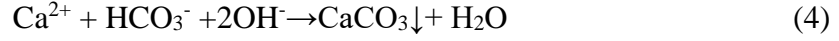


The reaction rate is not limited by mass transport, and the current intensity can be very high (Gabrielli et al., 2006).

The hydroxyl ion generates either by (1) or (2) and starts to destabilize the Calco-carbonic equilibrium of the solution (Legrand et al., 1981). The high alkaline environment converts HCO_3^- ions into carbonate ions by the following reaction:



In the next step, carbonate ions react with calcium ions to start the nucleation and create CaCO_3 crystals by the following reaction:



The rate of hydroxyl generation is directly related to Faraday's law. Hydroxyl generation is proportional to i ampere, and the following equation describes it. (Eq. 6)

$$\frac{i}{F} \eta = R_{OH} \quad (6)$$

Where F is Faraday constant (96,485 coulombs/mole), and η is the current efficiency of the ratio of moles CaCO_3 precipitated to a specific hydroxyl ion mole.

The passing current causes these specific hydroxyl ions. The current density of the EP process controls both anodic and cathodic reactions. Faraday's law describes the connection between current density and the number of metallic electrodes dissolved. (Eq 7) (Comninellis and Chen, 2010)(Aguilar et al., 2005; Wang et al., 2002).

$$n = \frac{it}{zF} \quad (7)$$

Where n is the number of moles of metals dissolved, i is the current in amperes, t expresses the operation time of electrolysis in seconds, F is the Faraday constant, and z is the charge of the cation (Martínez-Huitle et al., 2018).

Recent progress in electrochemical hardness removal from water can be portioned into four parts.

Tap water softening: Tap water is less favorable than brackish water to remove hardness with the EP process since tap water is much less conductive ($\sim 0.5 \text{ mS cm}^{-1}$) than seawater ($\sim 50 \text{ mS cm}^{-1}$) (Clauwaert et al., 2020). However, some achievements have been documented by a few studies. (Kalash et al., 2015b) applied the EP process to remove hardness from municipal tap water containing hardness of 330mg/L as calcium carbonate and achieved about 85% hardness removal. Aluminum cathodes and graphite anode plates were used as electrodes in this experiment. (Agostinho et al., 2012) achieved 80% hardness removal from tap water with initial hardness of 355 mg/L as calcium carbonate in Campina Grande Paraiba State, Brazil. Steel and aluminum electrodes produced these results in 40 minutes of treatment.

Cooling water treatment: Cooling water is a medium that is used to reject excess process heat from the industrial application (Song et al., 2018). An evaporative cooling water system consists of a cooling tower and piping around it (Becker et al., 2009). Evaporation of cooling water causes hardness ions scaling in the circulation system. This phenomenon makes water softening for cooling water very important, especially for industrial purposes. In industries mixing cooling water with softened water to keep the total hardness less than 400 mg/L as CaCO_3 is common (Moran, 2018). (Yu et al., 2019) used a multistage electrochemical precipitation reactor to increase total hardness removal efficiency up to 21.6% in hard cooling water. (Luan et al., 2019) successfully reduced the test solution's total hardness from a Chinese manufacturing company in Shan Dong from 350 mg/L as CaCO_3 to below 100 mg/L as CaCO_3 by using a multi-mesh system. (Yu et al., 2018a) tested a lab-scale batch hardness removal unit to remove total hardness

up to 22.8%. In a separate study, (Yu et al., 2018b) achieved final total hardness removal efficiency of 12.2 to 15.2 %. (Jin et al., 2019) achieved total hardness removal efficiency of 16.4-21.4% from cooling water using the EP process with titanium DSA (Dimensionally Stable Anode) for both anode and cathode. The EP process with high efficient multi-layer mesh coupled cathode was also tested to treat cooling water by (Li et al., 2020). The study results suggested that internal and external layers synergically enhanced the performance, which implies a step towards exceeding the cathode area requirement of the EP system.

Descaling cathode surface: One of the significant challenges of the EP system is to prevent scaling on the cathode surface. In Table 1, a comparison between different types of methods for descaling is introduced. There has been a significant growth in research to find a better descaling technology for the EP process. A novel method proposed by (Yu et al., 2018a) was pulsating current during the EP process. The research showed that increased current density created higher gas pressure on the crystals attached to the cathode. As a result, higher descaling was observed (Table 2). The relationship between the current density and turbidity is described. Also, the study claimed that a repetitive descaling performance without decay was possible. Yu *et al.* (2018b) proposed air scoured washing for descaling the cathode. This method has been mainly known for filter backwashing (Liu and Liu, 2016; Park et al., 2016). It was observed that the airflow rate per unit cathodic area was the main factor behind scale detachment performance. The study provided a promising outlook of air scoured washing as a promising method for descaling. (Jin et al., 2019) used polarity reversal to descale the cathode. This has been a proven descaling method in various technologies like electrode ionization and electrodialysis (Lee et al., 2006; Valero and Arbós, 2010; Yeon et al., 2007). When the polarity of the electrodes is reversed, poles of

electricity change and initiate descaling. In an experimental result, it was observed that the increased current density during polarity reversal resulted in a higher descaling rate during polarity reversal.

Table 1: Comparison of different descaling techniques (Yu et al., 2018b)

Descaling Technique	Advantages	Drawbacks
Mechanical Scraping	Easily operational	Low softening efficiency and high energy consumption
Polarity Reversal	Simple configuration	Costly and environmentally harmful
Acid Washing	High detachment efficiency	Severely reduced electrode lifetime
Ultrasonic	Satisfactory descaling efficiency and environmental friendliness	Complicated configuration, difficulty in maintenance
Air-Scoured Washing	Simple configuration, easy operation	Require a separate air supply system

Cell design and process enhancement: Recently developed novel softening methods involve process enhancements and improved cell designs. Most of these approaches achieved better hardness removal than the predecessors by engaging one of the most prevailing problems of EP, lack of cathodic surface area. (Luan et al., 2019) increased the active cathodic surface area using a multi mesh system instead of single cells. (Yu et al., 2018a) tested a multistage EP reactor with eight cells. The multistage system showed better water softening performance than the conventional approaches at comparatively less energy consumption. However, a greater ohmic drop was observed from the same multilayer system when normal unsalted water was treated.

(Yu et al.,2018b) used a combination of electrochemical cell compartments of cathode and anode with ion-exchange membranes between them. The method removed 73-78% of calcium and 40-44% of magnesium.

Table 2:Effects of current density on the turbidity of water (Yu et al., 2018a)

Current Density, A/m²	Turbidity, NTU	Voltage, V
100	29.4	6.3
200	155.7	9.3
300	561.7	12.4
400	986.2	16.8
500	1270.1	19.8
600	1305.6	23.1

Factors affecting electrochemical precipitation: Several parameters are known to significantly influence the performance of the EP process to remove hardness from water. These parameters not only affect the hardness removal rate, but also the sustainability of the system itself. Major factors are discussed below.

Applied voltage and current density: The hardness removal rate directly relates to the current density (Yu et al., 2019). The increased current density and voltage make bubbles in treated water-dense and smaller. It increases the active surface area of the bubbles, and greater flotation

efficiency can be achieved. This all leads to an increased OH^- production (Table 3) (Clauwaert et al., 2020) and greater hardness removal from the water.

Table 3: Change of hydroxyl production with charge density (Clauwaert et al., 2020)

Charge Density (C L⁻¹ tap water)	Hydroxyl production (mmol OH⁻ L⁻¹ tap water)
175	181
524	543
1097	1137
2237	2318
3129	3243

Electrode Material

The electrode material is vital for all electrochemical processes. The electrode material must be (a) Physically and chemically highly stable, (b) Have high electrical conductivity, and (c) Low cost/life ratio (Chen, 2004)(Anglada et al., 2009) for optimum performance. Materials of the electrodes can not only change the efficiency of the hardness removal, but also the electrical efficiency. The importance of electrode material can be observed from Table 4. The initial hardness of the treatment water was 350 mg/l as CaCO_3 (Yu et al., 2018).

Table 4: Effect of electrode material on hardness removal (Yu et al., 2018b)

Calcium Removal (mg/L)	Titanium	Nickel	Normal Stainless Steel	Frosted Stainless steel	Mirror Stainless Steel
1st trial	46.06	54.14	55.75	53.65	61.15
2nd trial	48.48	51.71	54.94	53.33	62.22
3rdtrial	45.25	52.52	54.14	50.10	59.48

Inter electrode distance:

Distance between the electrodes is also a vital factor for the efficiency of the EP system. (Kalash et al., 2015b) studied the impact of inter-electrode distance on the EP system's hardness

Table 5:Current Density, Cell Voltage, Hardness removal efficiency .electrode materials and treatment water of previous researches.

Reference	Current Density (A/m ²)	Cell Voltage (V)	Hardness Removal Efficiency (%)	Reactor	Electrode Materials	Treatment water
(Yu <i>et al.</i> ,2019)	20-120		18.7-21.6	Continuous	DSA anode, Steel cathodes	Cooling water
(Clauwaert et al.,2020)	7-10		75 - 86 and 73 - 78	Contiguous-A Batch-B	Stainless steel mesh cathode, Titanium anode	Tap water
(Yu et al.,2018a)	100	5.3 - 7	17.8 - 22.8	Batch	DSA anode, Stainless steel cathode	Cooling water
(Zhi and Zhang 2016)	EP- 250 EC- 20			Batch	DSA anode, Al anode, Stainless cathode	Prepared solution
(Luan et al.,2019)	24.5 - 61.3			Batch	DSA anode & Stainless steel mesh cathode	Cooling water
(Jin et al.,2019)		5.2-7.2	16.4 - 21.4	Continuous	DSA anode & DSA cathode	Cooling water
(Zhi and Zhang.,2014)	5 - 150			Continuous	DSA titanium anode (EP), Al anode(EC), Stainless cathode	Prepared solution
(Wang et al.,2018)	50 - 250		72.6	Batch	Ti/IrO ₂ -RuO ₂ anode, Titanium cathode	Prepared Solution
(Li et al., 2020)	2 - 16			Batch	DSA anode, Stainless steel mesh cathode	Cooling water
(Yu et al.,2018b)	100		12.2 - 15.2	Batch	DSA Anode, Stainless Steel (Normal, Frosted, Mirror),	Recirculating Cooling water
(Kalash et al., 2015)	10 - 28.5		85	Batch	Graphite plate anode and aluminum plate cathode	Tap water
(Sharma and Chopra,2014)	0.16 - 1.68	5-40	59.7	Batch	Al electrode both as anode and cathode	municipal wastewater

Effect of retention time

The calcium and magnesium precipitation rate vary depending on the retention time. Electrolysis time has directly proportional to the hardness removal to a certain level when pH and the potential difference are constant (Yu et al., 2018b). observed that hydraulic retention time and electrolytic hardness removal were directly proportional for the first 4 minutes of the experiment (Fig. 3).

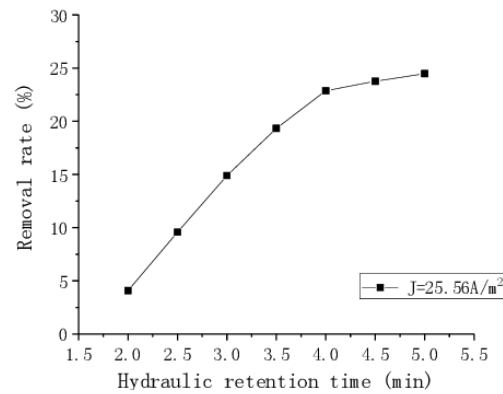


Figure 3:Effect of retention time(Yu et al., 2018b)

CHAPTER III

OBJECTIVES

This study aims to develop a lab scale electrochemical precipitation cell using graphite concrete electrodes.

- Finding the compatibility of graphite concrete electrode as cathode in an electrochemical precipitation cell. The target was to lower the resistivity as low as possible using efficient amount of graphite in the concrete.
- Setting up operational lab scale setup. This included finding the optimum power source, best setups for the maximum output and ensuring safety protocols.
- Determining the best operational factors for this setup. Effect of distance, voltage variation, retention time and cathode material to be tested thoroughly.
- Effect of electrochemical precipitation on the quality of the water after softening.
- Determining the maximum hardness removal capability of the experimental setup.

CHAPTER IV

EXPERIMENTS AND RESULTS

Conductive Concrete

Size of each mold was 76.2 mm×17.78 mm× 20.34mm. Due to the size of the mold maximum coarse aggregate size was 9.5mm and minimum of 2.38 mm (#8). Fine aggregate of #8 sieve passing and #100 sieve retaining was used (Fig 4). (Fig 5). The physical properties of the graphite are demonstrated in Table 6.

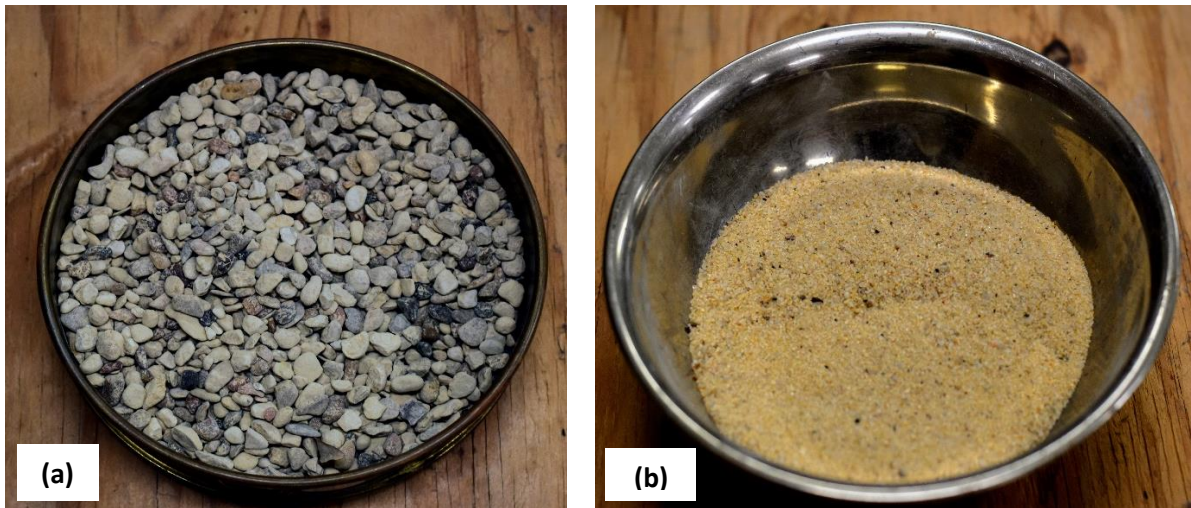


Figure 4: (a) Coarse aggregate used in the experiment. (b) Fine aggregate used in the experiment.

Table 6: Physical Properties of the natural flake graphite used in experiment

Percent Carbon	Typical Size (μm)	Specific Gravity	Surface area (m^2/g)	Typical Resistivity ($\Omega\text{-cm}$)	Note
95.46%	50-60	2.6	5	0.03-0.05	Flake

Graphite was added to the concrete respective to their volume. Three specimens were cast containing 5%, 7.5% and 10% graphite powder with respect to the total volume of the specimen. The specimens were cast following standard ASTM methodology. Specimens were mixed using Mixed Specimens were casted in rectangular silicon molds. (Table 7)

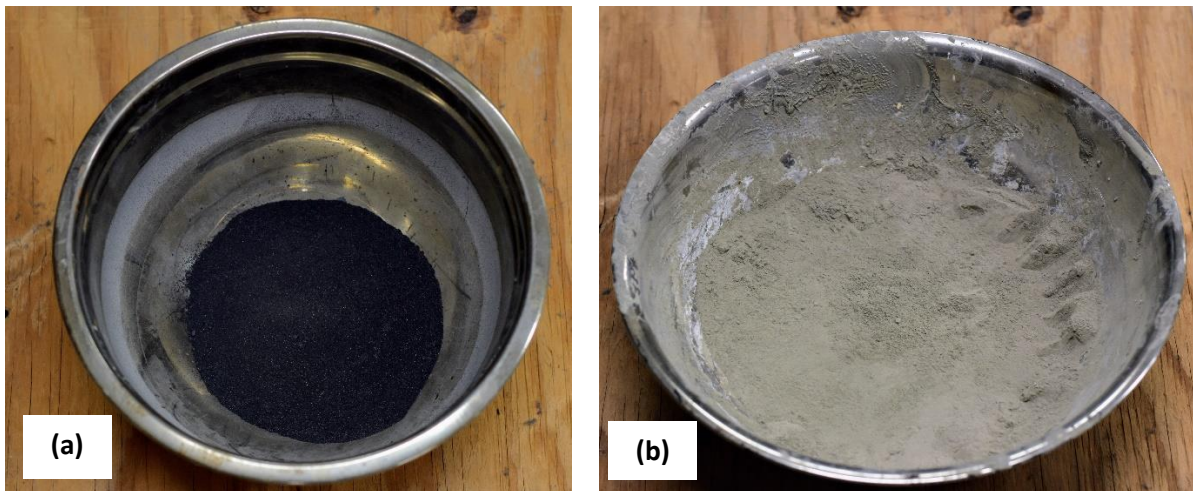


Figure 5: (a) Natural flake graphite used in the experiment. (b) Portland cement used in the experiment.

Table 7: Mix Design of Graphite Concrete Cathodes

Materials	Mix Design of 5% graphite concrete (gm)	Mix Design of 7.5% graphite concrete (gm)	Mix Design of 10% graphite concrete (gm)
Water	25	25	25
Cement	55.55	55.55	55.55
Coarse Aggregate	105.838	105.838	105.838
Fine Aggregate	127.46	127.46	127.46
Graphite	15.57	23.37	31.14

Surface of the molds were lubricated prior and sides of the molds were reinforced with wooden pieces. The cast specimen was kept in room temperature of 25°C for 24 hours. After the concrete was set, it was kept under water for curing for 14 days.

Preparation of the specimen:

Cured specimens were towel dried at first and checked for cracks. It was followed by submerging it in water again for minimum of 24 hours (Fig 6). Saturated concrete was needed to create similar conditions to a concrete pipe or reservoir. Saturated concrete specimen was then coated with PELCO conductive nickel paste (Tedpella, USA). This paste contains high purity nickel flakes (8-13µm) and has fast drying properties with a VOC content of 27.5%. This fast-drying nickel paint forms a thin, conductive, and flexible layer with good adhesion on conductive concrete surface. The conductivity of this nickel cement paint is approximately 20 times better than graphite paint and approximately 10% better than silver paste. On this nickel paste

conductive copper tape is wrapped. This tape is made with copper foil with an acrylic conductive adhesive. After cutting , the ends are bind together to make ample space for electrical connections to be made.



Figure 6: (a) Applying conductive nickel paste on saturated concrete specimen (b) Applying conductive tape over the nickel paste.

Resistivity of the specimen:

Saturated specimens were carefully wrapped with plastic paper to hold the moisture inside. On both sides conductive paint and copper tape were implemented. Both ends were connected with the impedance machine and test was conducted. This procedure measures the resistivity of the specimen in different AC current frequency. It allows to understand the electrical properties of the specimen. For measurement of resistivity from resistant, following equation was used,

$$\rho = \frac{RA}{L}$$

Where, ρ is the resistivity of the specimen, R is the resistant of the specimen, A is the cross sectional area of the specimen and L is the length of the specimen.

Treatment water:

The water used in the experiment was tap water. The source was environmental engineering lab in UTRGV Edinburg campus. Before collecting sample water, the water was kept running for 5 mins. Water was collected in 1 liter glass beaker. The initial hardness of the sample water was between 320-387 mg/L as CaCO_3 . Initial pH was 7.2 on average. The temperature of the water was 23 degrees.

Experimental Setup:

The setup started with connecting the electrodes with the power source. (fig 7) . The power source consisted of 33 W DC power adapter (ASUS, Taipei, Taiwan). The adapter was connected to a buck booster. Buck booster allowed continuous power supply up to 35.5V and 5 A. It also allowed to regulate the voltage and current according to the requirement. Output lines of the buck booster was connected to the electrodes. Conductive concrete specimens were clamped with stands to make a horizontal setup. (Fig 6) Parallely DSA (Dimensionally Stable Anode) is used as the only cathode in the system. This anode is setup parallel with the cathode and the distances between them were .5cm, 1cm and 1.5 cm. The anodes are 2inch X 6inch in size. Crocodile clamps were used to attach 18-gauge electrical wires with the electrodes. The Electrodes were placed within a 1L glass beaker. The beaker was filled with water with predetermined hardness contents. It was made sure that both

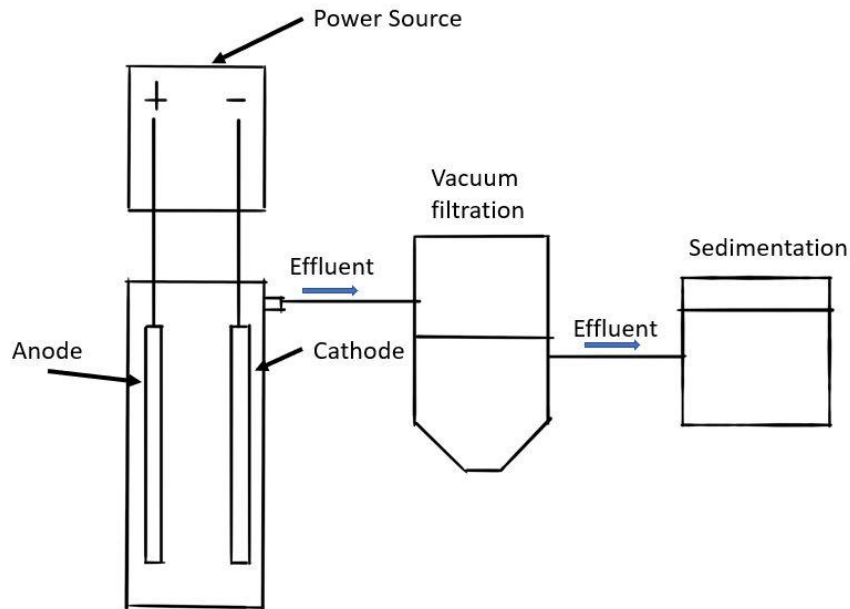


Figure 7: Experimental Setup of the electrochemical water hardness removal cell

electrodes were submerged in water as much as possible. This allows maximum surface area efficiency. The function of the graphite concrete cathode was to generate alkalinity and be the scale deposition surface. No medium was needed for the separation of anodic and cathodic environments. The high pH condition was necessary to promote precipitation near the cathodic surface as a thin layer. As a result, the precipitation reaction occurred exclusively in the thin water film adjacent to the cathodic surface (Tlili et al., 2003a, 2003b).



Figure 8: Placement of electrodes in experiment.

In this experiment, effect of treatment time, voltage, inter electrode distance and cathode material had been investigated. The water was treated for 100 seconds, 15 mins, 30 mins and 60 mins. Voltage variations were 25V, 30V and 35 V. Inter electrode distances were 1cm, 1.5 cm and 2 cm (Fig:8). The concretes used as cathode had 5%,7.5% and 10% graphite content by total volume.

For each experiment, the hardness of water was checked with EDTA (Ethylenediamine Tetraacetic Acid) titration method according to the standard method. After that pH multimeter HACH HQ40d (HACH, Colorado, USA) was used to measure the initial pH of water. Following that LaMatte Alkalinity DRT kit was used to measure the alkalinity of the water.

After recording initial measurements of the sample, water experiment was conducted. In each experiment, power was turned on while circuit was closed. On interval of 10 mins, applied current and temperature of the water sample were checked. After completing the treatment for

specified time, precipitation was removed by vacuum filtration on 1 μm glass fiber filter (Whatman, UK) connected to a vacuum pump (WELCH 2534B-01A, , Louisiana, USA) .After 30 mins the water is taken from the tank and hardness, pH and alkalinity are measured.

Specimen containing 10% graphite by volume was tested for compressive strength. Cylinder of 10 cm diameter and 20 cm length was used as mold. The mold was filled with 10% graphite concrete. The specimens were compacted and maintained according to ASTM standards. After 24 hours, the molds were taken off. The specimens were then cured for 28 days. The specimens were named 10T1, 10T2 and 10T3. (Fig 9)

ASTM C39M-21 was implemented for this standard test. Forney F250 compressive strength machine (Forney Corporation, Texas, USA) was used to perform the compressive strength. For input, weight was measured using a standard scale machine. The specimen was set in between the load providing pistons (Fig 10). Safety measures were taken to make sure debris do not harm the machine or experimenters. The loading stopped when there was visual crack in the specimens and peak compressive strength was noted. (Fig 11)



Figure 9: Conductive concrete cylinders containing 10% graphite by volume. Specimens are 10 cm by diameter and 20 cm by length . (a) side view of the specimens and (b) top view of the cylindrical specimens.

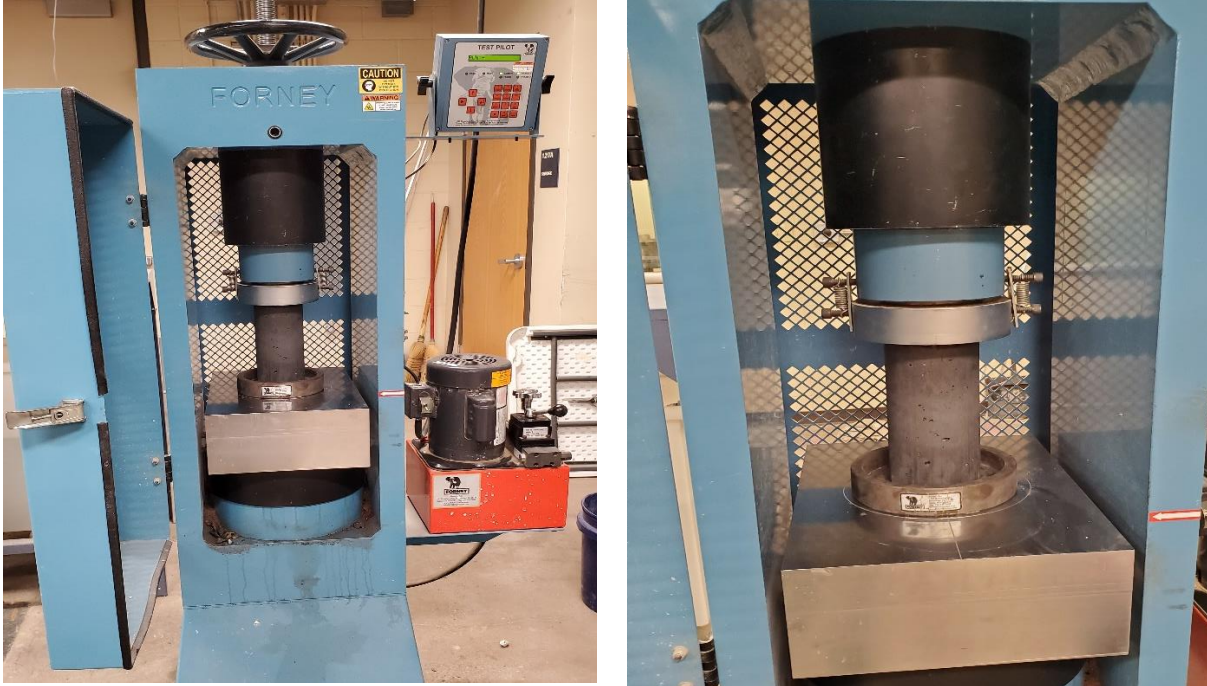


Figure 10: (a) Conducting compressive strength test on FORNEY F250. (b) specimen setup for the compressive strength test.



Figure 11: Failure of specimen 10T1, 10T2 and 10T3 under lateral loading.

CHAPTER V

RESULTS AND DISCUSSION

Resistivity

Resistivity is an important property of electrodes used in electrochemical processes. It not only gives a firm understanding of the conductivity of the material but helps to understand the capability of the material. All three specimens were tested for resistivity. Specimens containing 10% graphite by volume were marked as 10A1, 10A2 and 10A3. Specimens containing 7.5% graphite by volume 7.5B1, 7.5B2 and 7.5B3. Specimens containing 5% graphite by volume were marked as 5C1, 5C2 and 5C3.

In Fig 12, specimen 5C1, had a resistivity starting from 83.81 ohm-cm and piqued at 208.68 ohm-cm when frequency was 3.1623Hz. In Fig 13, it was observed that resistivity of specimen 5C2 ranges between 105.60 ohm-cm to 400.961ohm-cm. The highest pique was seen when frequency was 1.7783 Hz. In Fig 14, 5C3 showed resistivity between 87.83 ohm-cm and 168.13 ohm-cm. Frequency of 3.1623 showed highest resistivity in this material.

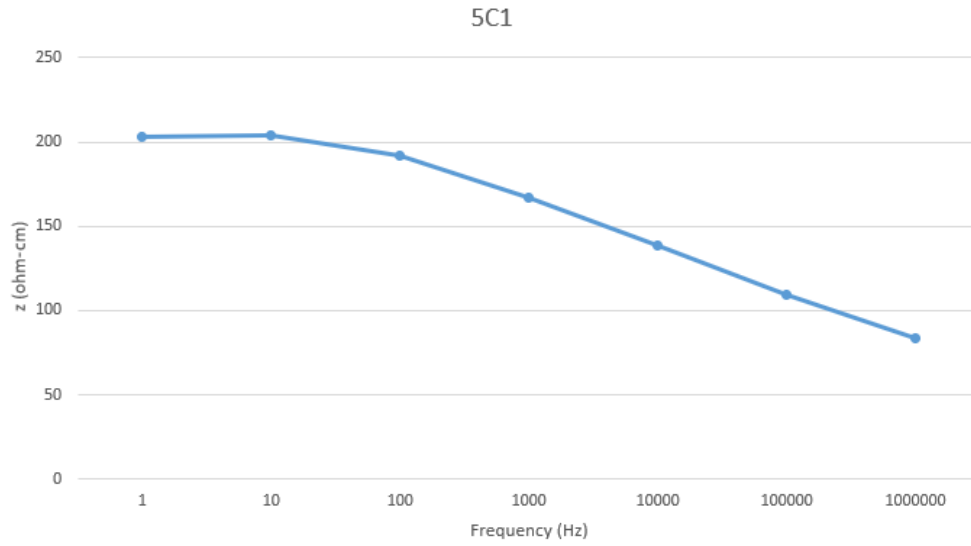


Figure 12: Resistivity graph of 5C1 graphite concretes in AC impedance Spectroscopy in different frequencies

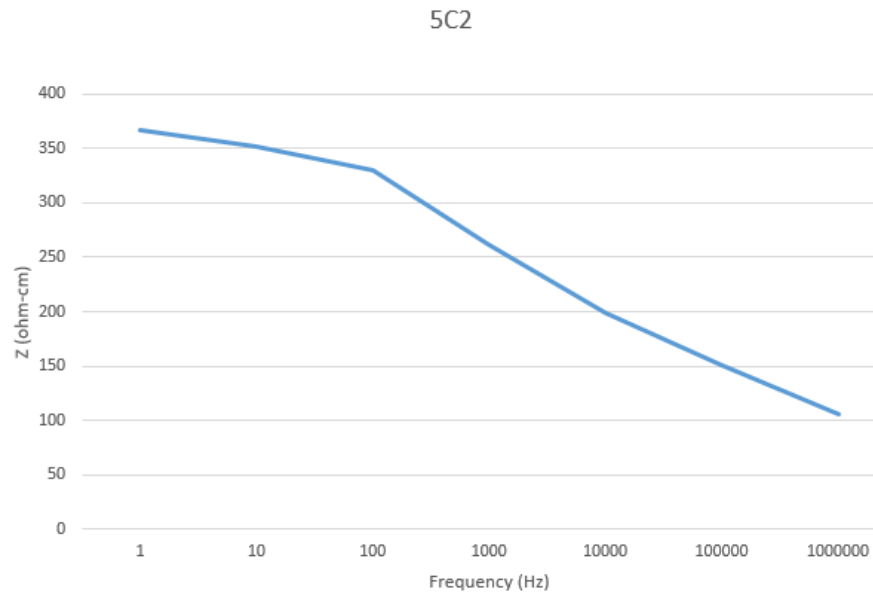


Figure 13: Resistivity graph of 5C2 graphite concretes in AC impedance Spectroscopy in different frequencies

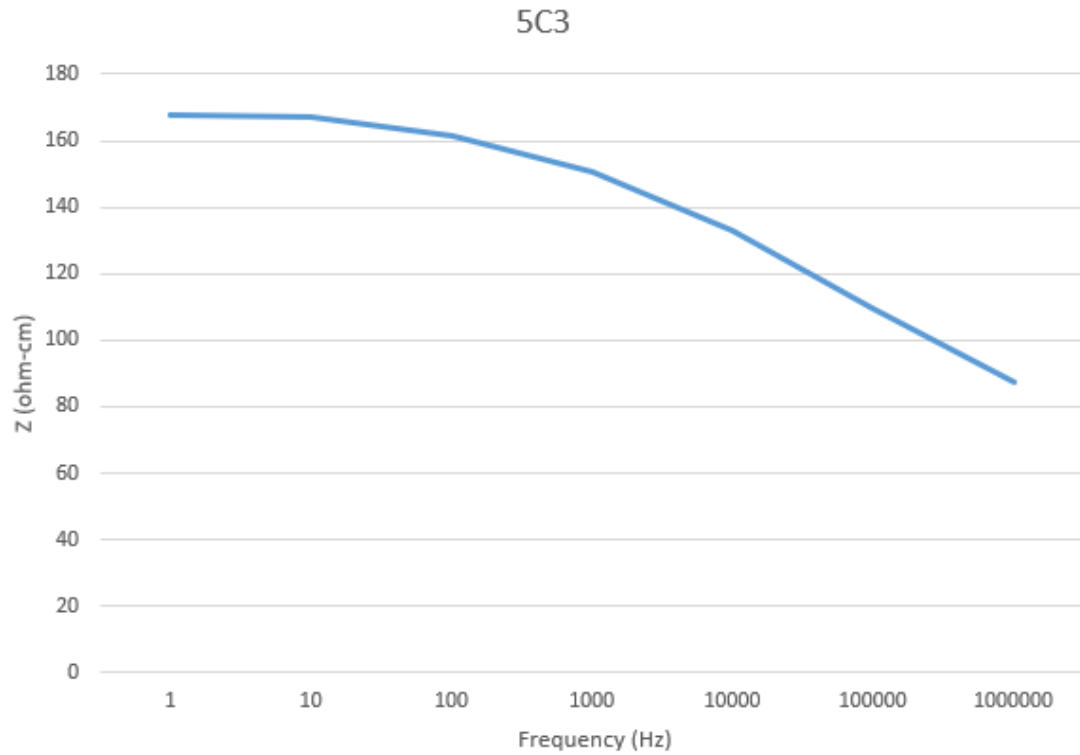


Figure 14: Resistivity graph of 5C3 graphite concretes in AC impedance Spectroscopy in different frequencies

In Fig 15, specimen 7.5B1 showed a resistivity of 38.89 ohm-cm at lowest and 68.061 ohm-cm at highest. Highest pique was available during 25.119 Hz frequency. Similarly, in Fig 16, specimen 7.5B2 showed a resistivity of 44.33 ohm-cm initially. During 2.8184 Hz it showed resistivity of 56.225 ohm-cm. Specimen 7.5B3 started with a resistivity of 53.09 and peaked at 67.39 ohm-cm 1 Hz in Fig 17.

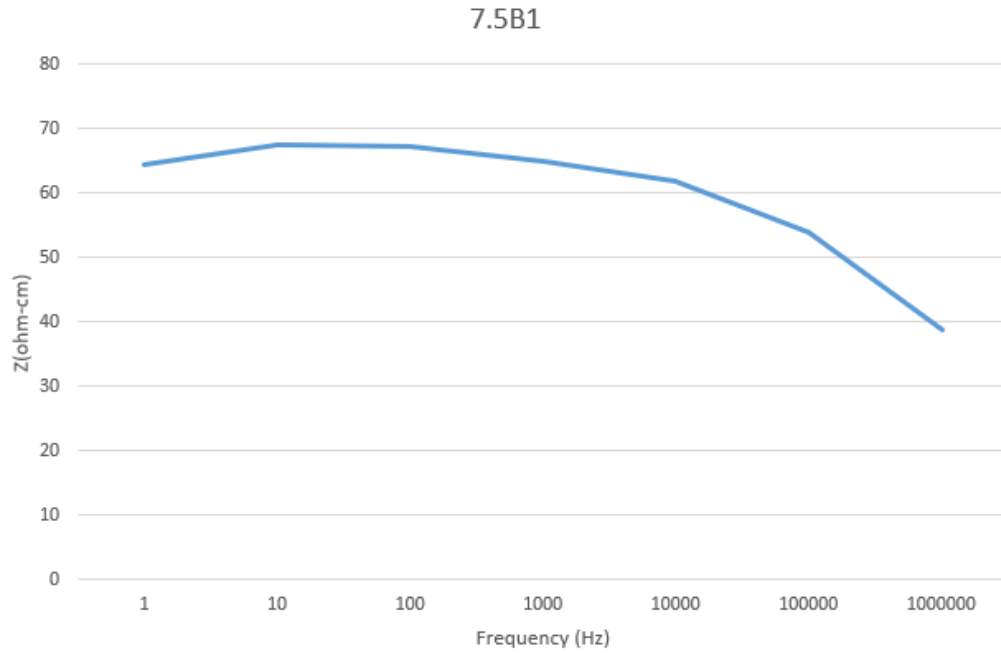


Figure 15: Resistivity graph of 7.5B1 graphite concretes in AC impedance Spectroscopy in different frequencies

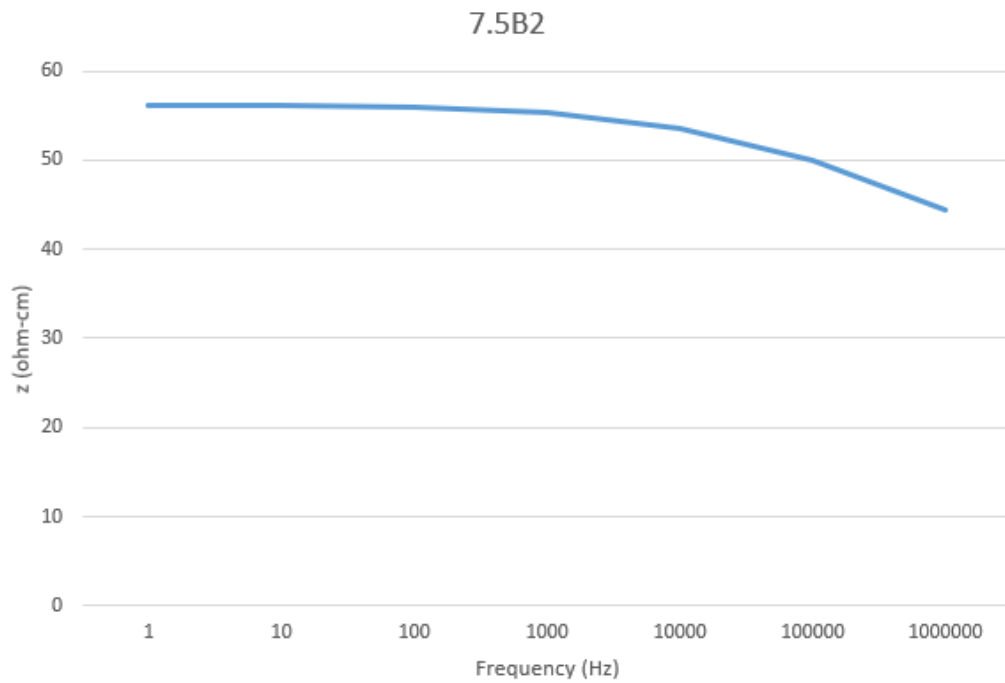


Figure 16: Resistivity graph of 7.5B2 graphite concretes in AC impedance Spectroscopy in different frequencies

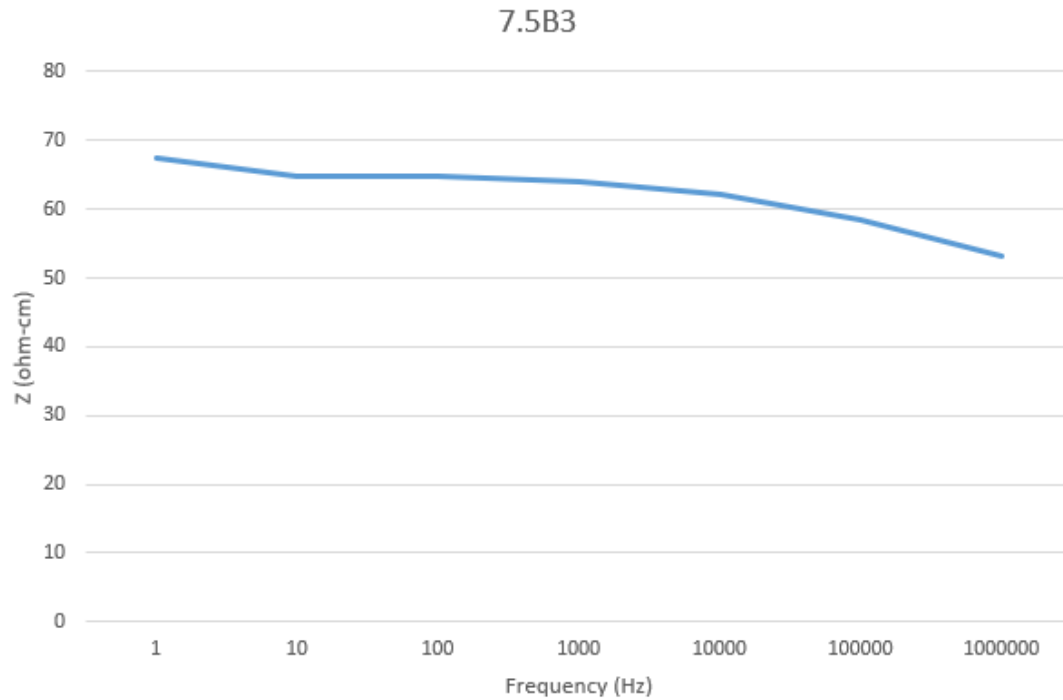


Figure 17: Resistivity graph of 7.5B3 graphite concretes in AC impedance Spectroscopy in different frequencies

In Fig18, specimen 10A1 started with a resistivity of 26.0 ohm-cm to 46.68 ohm-cm. Highest resistivity was seen on 5.019 Hz. Specimen 10A2 showed resistivity from 33.93 ohm-cm to 43.71 ohm-cm in Fig.19 . Highest resistivity was seen on 3.981 hz. Specimen 10A3 showed resistivity from 34.14 ohm-cm. to 44.91 ohm-cm. Highest pique was seen on 3.9811Hz in Fig 20.

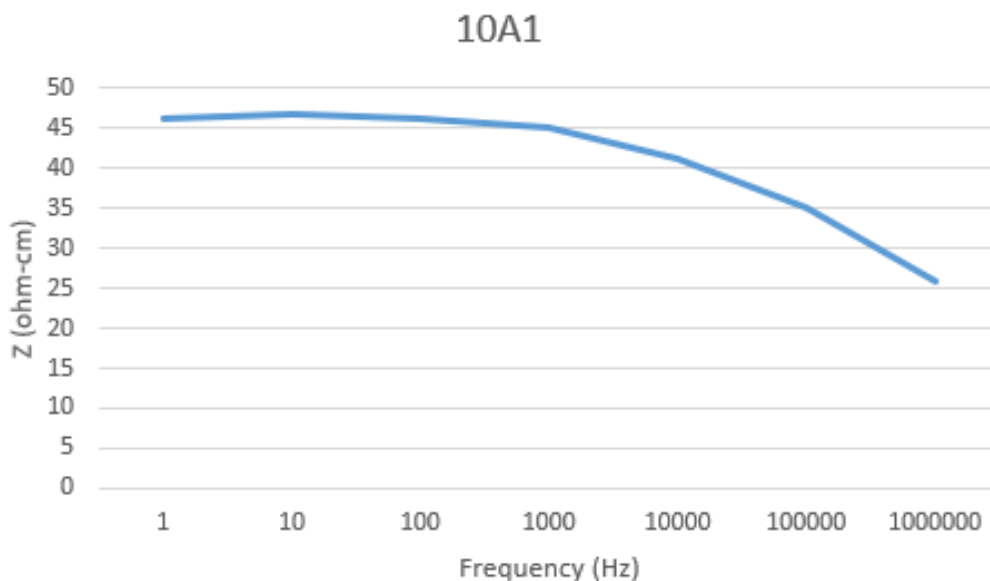


Figure 18: Sample 10A1 showing resistivity in different frequencies in AC impedance spectroscopy

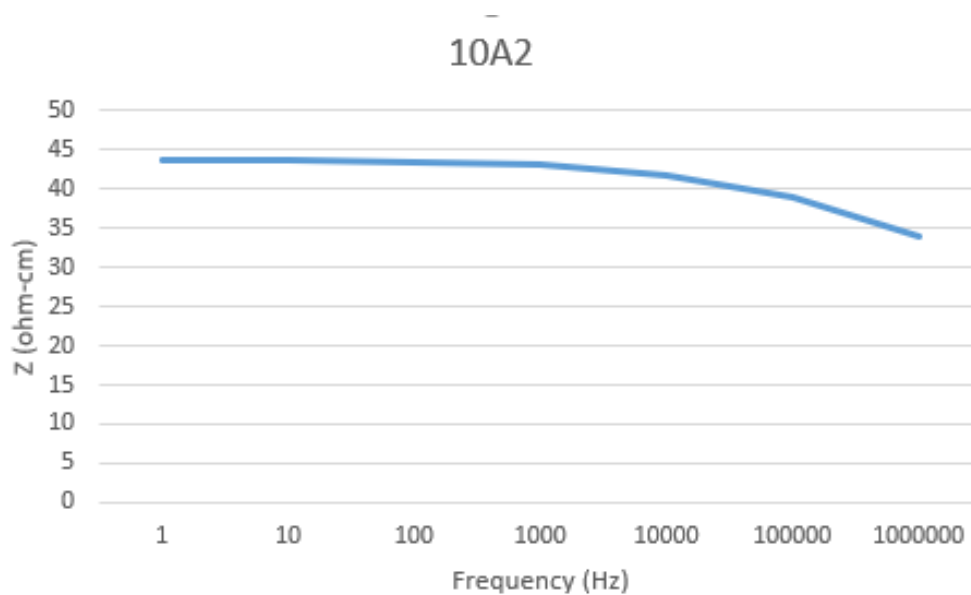


Figure 19: Sample 10A2 showing resistivity in different frequencies in AC impedance spectroscopy

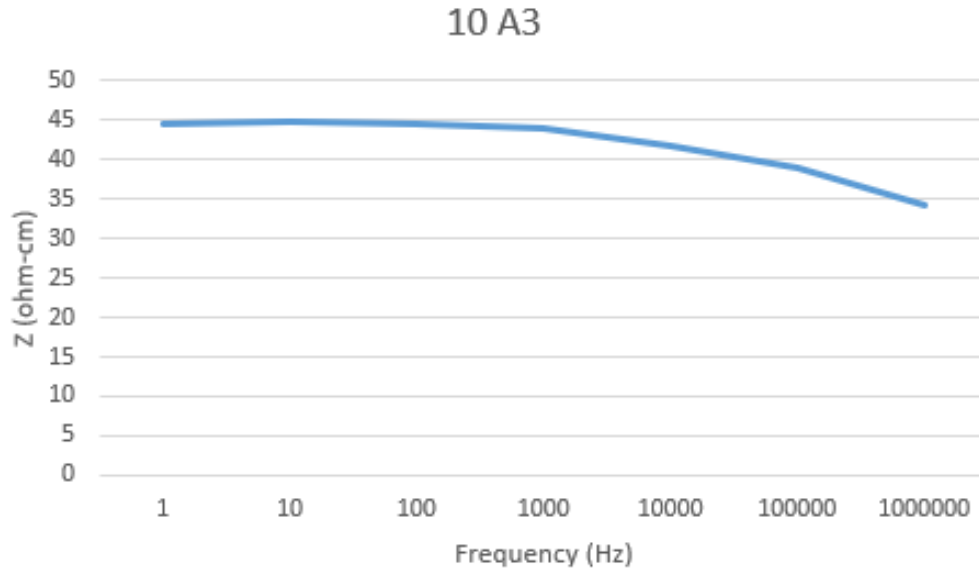


Figure 20: Sample 10A3 showing resistivity in different frequencies in AC impedance spectroscopy

Retention Time

Retention time provides a meaningful impact on the performance of the novel electrochemical precipitation time. Under operational parameters of 1cm electrode gap, 36.5V power supply, and same electrode configuration, a significant change in hardness removal was observed in the experiment. While being treated for 15 minutes, the hardness removal rate ranged from 3.27 to 7.75%; this rate increased with the retention time.

At the end of 60 minutes of treatment in all configurations, the hardness removal rate also increased. From Fig.15, it can be observed that 10% graphite concrete cathode showed 16.61% hardness removal in 30 minutes, and it increased to 53.46% in 60 minutes. Similar results were seen with all other cathodes. It could be observed that with increased time of treatment, the

hardness removal efficiency was growing as well. Similar results were found by (Clauwaert et al., 2020; Kalash et al., 2015a; Sanjuán et al., 2019b; Yu et al., 2018a). From *Faraday's law*, it is known that as the electrolysis time increases, the OH^- in the solution also increases. This increased OH^- generation is the primary reason behind increased Ca^{2+} removal from water.

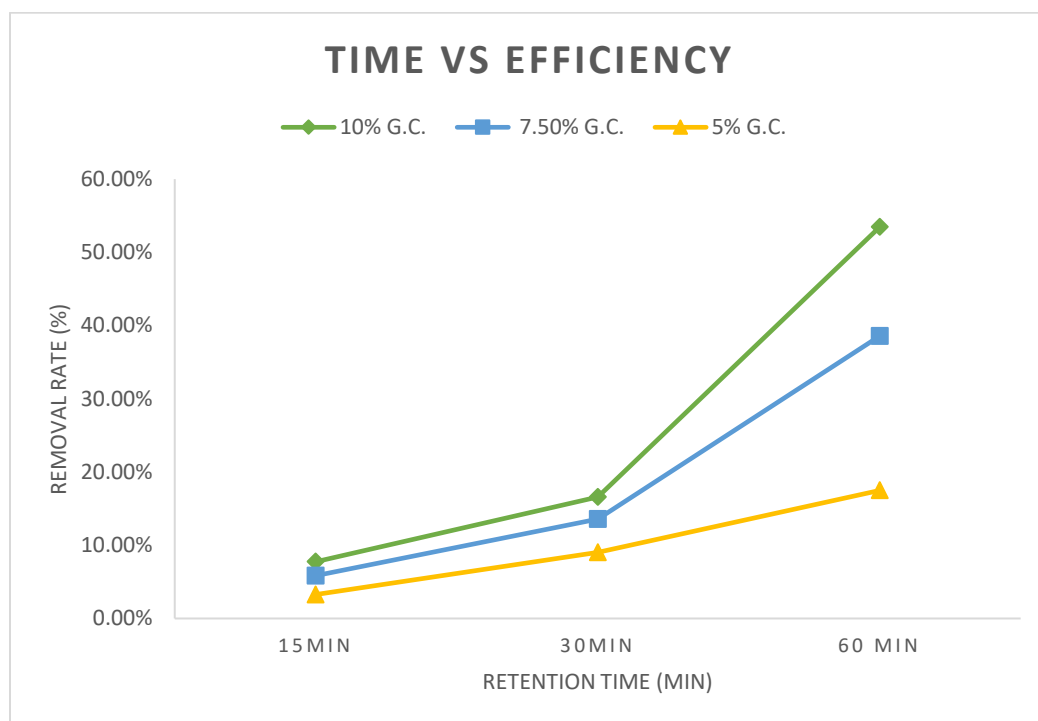


Figure 21: Effect of retention time on hardness removal ratio

Voltage Variation

Electrical potential difference or voltage variation is a major factor in efficient electrochemical hardness removal process. In this electrochemical precipitation process, the result of voltage variation on final hardness removal efficiency was checked. 10% graphite -concrete

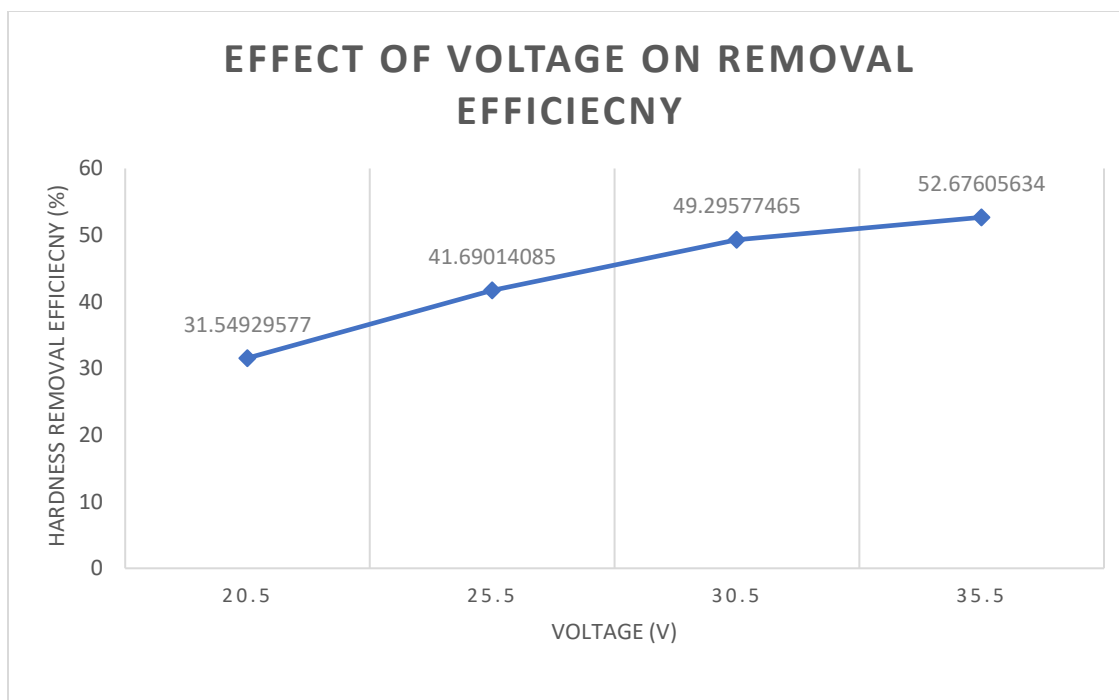


Figure 22: Effect of voltage on hardness removal efficiency of the electrochemical precipitation cell

electrodes were used. The inter electrode distance was 1cm, retention time was 60 mins and initial hardness was 355 mg/l as CaCO_3 . In this experiment it was seen that the lowest voltage of 20.5 V showed hardness removal of 31.45% on average. The efficiency rises sharply to 41 % when voltage was also increased to 25.5V. It was also observed that efficiency rose to 49% in 30.5V and reached to 52.67% when 35.5 V current was applied. The results were similar to (Kalash et al., 2015). These results can be explained by the fact that, increasing potential allowed more electricity to pass through the system. As a result more electrons could pass through the water in the same time. This process increased the density of the water bubbles adjacent to the cathode, while decreasing the size of them. Since the effective surface and retention time of smaller bubbles were more than the bigger ones, in the same retention time hardness removal efficiency was improved. This also explains the sharp increase of efficiency at the initial

voltages, where change in the higher voltages showed less increase. In the early stages increased voltage caused the phenomena explained before and increases efficiency. But in the higher voltages, bubbles were already at their smallest size, which explains the less increase in efficiency.

Cathode Material

Electrode materials, especially cathode materials, are vital for hardness removal efficiency (Yu et al., 2018c). In the same operational condition of 36.5V, 1 cm electrode distance, and similar retention time, from (Fig.15), different hardness removal was seen from the three types of cathodes. 10% graphite concrete cathode contained graphite powder equivalent to 10% of the total volume of the specimen. As it had the highest amount of conductive graphite among the three, it was also the most conductive. This enhanced capability to conduct current allowed more electrons to enter the water solution and create an increased amount of OH^- ions. This phenomenon eventually resulted in more Ca^{2+} removed from the water. In all three-retention time of 15,30 and 60 minutes, the removal rate was 7.75%, 16.6%, 53.46% consecutively. 7.5% graphite concrete was the cathode with the second highest conductivity. It produced 5.85%,13.58%, and 38.57% of hardness removal when retention time was 15,30 and 60 minutes. 5% graphite concrete performed 3.27%, 9.018%, and 17.49% in relative retention time. It was clearly seen that the cathode materials played an impactful part in the total hardness removal rate.

Inter-Electrode distance

The distance between the cathode and anode is also a significant parameter. In the experiment, it was seen that the closest distance showed the best result. Under the same operating conditions of 30 min retention, 36.5V, and 10% graphite concrete cathode, inter-electrode distance .5cm showed best performance of 16.61% (Fig.16) performance gradually dropped while increasing the distance and reached 10% hardness removal at 1.5 cm distant. When the inter-electrode distance was reduced, calcium and HCO_3 in the bulk phase diffused to the cathode reaction zone faster (Hasson et al., 2011, 2010). As a result, reaction (3) and (4) were amplified, and Ca^{2+} deposition increased. Also, the hydrogen on the cathode and oxygen in the anode caused more disturbance to the solution. As the distance between the electrodes was close, it covered the whole cathodic reaction zone. It enhanced the mass transfer of ions from the solution to both poles. When the inter-electrode distance was increased, gas disturbance only affected the area near one electrode and could not reach the other plate, which had an adverse effect on the calcium removal.

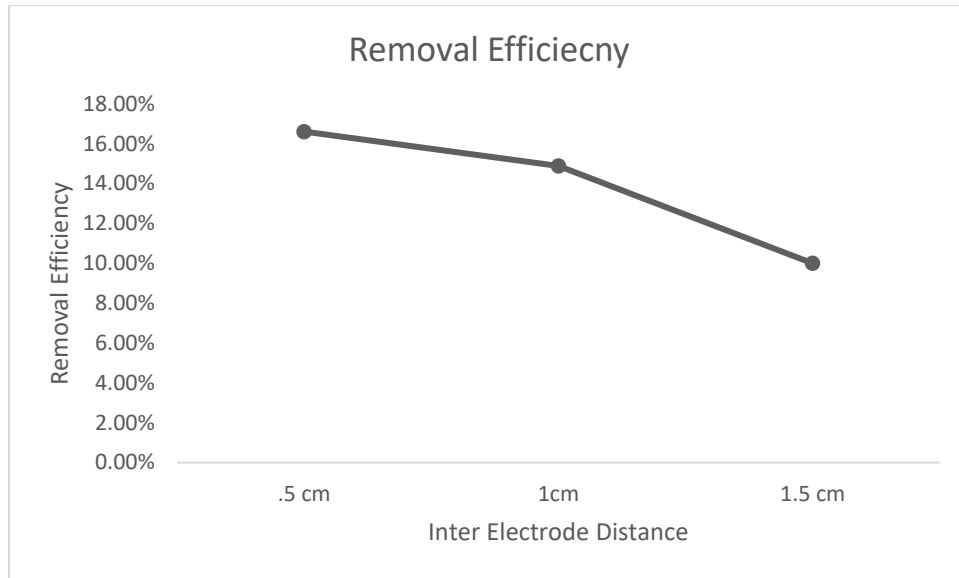


Figure 23: Effect of inter-electrode distance on hardness removal rate

Water Temperature

Temperature increases electrical conductivity of water (Hayashi, 2004). This temperature is directly related to the resistance of the electrodes. Though the cathodes used in the experiment were conductive enough to proceed with electrochemical precipitation treatment, their resistances were higher than metals. 10% graphite cathodes have resistance of .90 ohm on average. This increased resistance creates heat and gradually increases the temperature of the water medium. This heat enables increased current flow in the water. For each degree of temperature increase, electrical conductivity of water increases up to 2-3% (Clauwaert et al., 2020; Hasson et al., 2011; Yu et al., 2018c). Which increases hardness removal efficiency in this experiment. It was also observed that in the configuration centering 10% graphite concrete cathodes, temperature increases from 25.83 to 40.33 degree Celsius after 30 min of treatment on 35.6V. At the end of the treatment the temperature increases up to 51.16 degree Celsius (Fig.17).

During this period 1.055 amp current was passed through the medium. It was the highest conductivity recorded in the experiment. It removed 53.46% total hardness from the tap water.

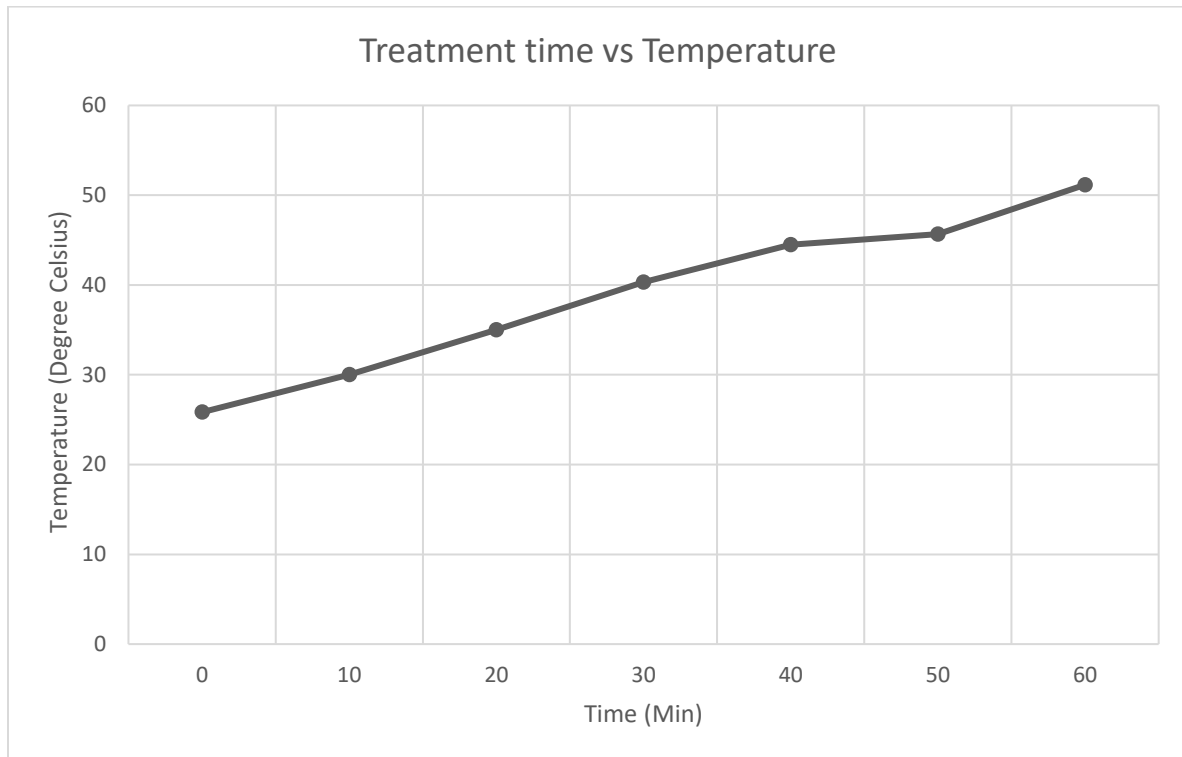


Figure 24: Effect of electrochemical precipitation on the treatment water temperature

Hardness Removal Efficiency

Implementing all the parameters of best performance for example, distance 1cm, voltage 35.5 V, retention time 1-hour, highest hardness removal was achieved at 53.46%. 10% graphite concrete specimen achieved that milestone. 7.5% removed 32.57% and 5% removed 17.49% hardness from tap water. Details on the hardness removal efficiency is presented in Table 7.

Table 8: Hardness removal efficiency in different conductive concrete electrochemical precipitation setups

Specimen	Initial Hardness	Final hardness	Removal
5% graphite Concrete	377	311	17.5%
7.5% graphite concrete	369	226.7	38.6%
10% graphite Concrete	347	161.5	53.5%

Hardness removal efficiency observed in this research is compared with previous studies with similar processes and presented in Table 8. These studies implemented (Yu et al., 2018), Segregated flowing setups like (Clauwaert et al., 2020). (Kalash et al., 2015a) experimented with novel setup of using graphite cathodes in this process.

Table 9: Comparison of hardness removal efficiency of previous research with this research

Research	Hardness Removal Efficiency (g/h/m ²)	Electrodes	Retention Time (Hour)
This experiment	53.46%	G.C. cathode, DSA anode	1
(Clauwaert et al., 2020)	78%	Stainless steel cathode, DSA Anode	24
(Xie et al., 2015)	99%	DSA Electrodes	1
(Kalash et al., 2015a)	85%	Graphite Electrodes	1
(Yu et al., 2018)	22.8%	DSA Electrodes	.1

PH and ALKALINITY

In previous studies, effect of pH on the performance of the electrochemical precipitation process were observed differently. In (Malakootian et al., 2010) it is stated that pH has a significant impact on the performance of the electrochemical precipitation. It is also stated that higher initial pH gives better hardness removal efficiency. However, in (Kalash et al., 2015a) it is shown that pH did not have significant or no impact on the hardness removal performance of an electrochemical precipitation cell. But all the references agreed on the point that, in a successful electrochemical precipitation cell, pH of the water increases significantly. Similar results can be seen in this research (Table 9) for 10% graphite concrete cathode setup, for 60 mins of treatment final pH reached up to 8.76 from 7.19. Other test results showed similar results regardless of the retention time and graphite contents in cathodes. The lowest pH increase from 7.0 to 7.12 was observed from 5% graphite concrete for 60 mins of retention.

Also, the alkalinity change of the water had been checked in this research. Carbonate alkalinity as CaCO_3 was found in these waters. After hardness removal significant drop in alkalinity could be seen. But this hardness drop in the alkalinity was far less than actual hardness removed from the water. 10% graphite concrete removed 184.3 mg/L as CaCO_3 , where alkalinity removal was 23.16 mg/L as CaCO_3 . Similar results were seen in all the retention time and setups.

Table 10:pH and alkalinity change in electrochemical precipitation setups

Electrode	Treatment time	Initial pH	Final pH	Initial Alkalinity	Final Alkalinity	CaCO ₃ removed as alkalinity
10%graphite	60	7.19	8.76	76.6	38	23.16
10%graphite	30	7.2	7.72	78	52	15.6
10%graphite	15	7.2	7.63	78	62	9.6
7.5% Graphite	60	7.2	7.6	78	46	19.2
7.5% Graphite	30	7.2	7.61	78	54	14.4
7.5% Graphite	15	7.22	7.56	78	60	10.8
5% Graphite	60	7.12	7.0	84	57.3	16.02
5% Graphite	30	7.19	7.58	78	56	13.2
5% Graphite	15	7.22	7.55	78	60	10.8

Compressive Strength

Compressive strength of the 10% graphite concrete specimen was measured to check its structural integrity. Three specimens of cylindrical size, 10cm diameter and 20cm length were tested. As lateral load was applied peak load bearing capacity was noted. In table (10) the detailed result of the specimen can be observed. Ideally compressive strength of normal concrete stands around 2500-4000 psi. The lack of compressive strength in these specimens can be of two reasons, first one is the size of the aggregate. Size of coarse aggregate is a huge influence on the performance of the concrete. (Mihashi et al., 1991; Tasdemir et al., 1996). Because of having heterogenous behavior, concrete does not show linear fracture. So it becomes extremely difficult to apply linear fracture mechanics in concrete. So, fracture energy and fracture toughness have been found as proper fracture mechanisms. These helps to describe the resisting properties of the

Table 11: Compressive strength test of 10% graphite concrete cylinder

Specimen	Weight (lb)	Load	Area (Sq. Inch)	Compressive Strength (psi)
10 T1	7.55	11920	12.566	948.59
10 T2	7.55	10855		863.83
10 T3	7.60	9960		792.614

concrete. It was seen in experiment that increasing coarse aggregate in the concrete increases fracture energy up to 2.5 times (Chen and Liu, 2004). In this experiment aggregates of 9.5mm was used. Size of this aggregate can play a vital role in low strength concrete. The second reason behind the low compressive strength is graphite content of the specimen. It has been seen that increasing graphite content in conductive concrete reduces compressive strength significantly. (Sun et al., 2021)

CHAPTER VI

CONCLUSION

A series of experiments were performed to find the optimal operating condition for the novel electrochemical precipitation system. The experiments were conducted in lab-scale batch operations. It was observed that operations were affected by retention time, inter-electrode distance, cathode material, and water temperature. The hardness removal efficiency was observed from 4.85% to 53.46%. The highest hardness removal was achieved at the operational condition of 36.5V, .5cm of inter-electrode distance, 60 minutes of treatment, and 10% graphite concrete cathode. Under these conditions, total energy consumption was .0368 kWh/l. Thus, it was demonstrated that this process may be used as a pretreatment step for water hardness removal. Future studies on the conductive concrete's structural integrity are necessary to implement it in large scale treatment plans. Also, further study on the economic efficiency of the treatment facility is essential. Because in the electrochemical treatment technologies, the real-life application's success depends on efficient and reasonable energy consumption and operational costs. More pilot-scale studies and large practical scale studies are required.

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