Removal of Hardness Using Ground Water Treatment Plant Sludge

by

Ahmad Fitri Bin Abd Rahim

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Supervisor: AP Dr. Shamsul Rahman Mohamed Kutty

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Civil Engineering Programme
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Approved by,

(AP Dr. Shamsul Rahman Mohamed Kutty)

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TRONOH, PERAK
JAN 2011

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

AHMAD FITRI BIN ABD RAHIM

Civil Engineering Department,
Universiti Teknologi PETRONAS.

ABSTRACT

One of the undesirable characteristics of some water sources is hardness, that can cause many problems including scaling and excessive soap consumption. Therefore several treatment processes have been introduced to remove or reduce the hardness from hard waters. One of the new innovations in this regard is using Ground Water Treatment Plant Sludge by precipitation. Hardness can be removed from hard waters by precipitation of calcium and magnesium. In the cement factory at Tasek, Ipoh, hard water is mostly found in the water recycling treatment where is the source of water is from pond, surrounded by limestone hills. A treatment process which is using Ground Water Treatment Plant Sludge (GWTPS) was conducted in replicate to observe the changes in parameters such as total hardness, calcium hardness, magnesium hardness and pH with varying dosages of GWTPS. A GWTPS dosage range of 500 – 5000 mg/L was chosen for the experiments. Results indicated that the removal of calcium can be achieved to 96% with the dosage of 5000 mg/L by precepitation at pH 9. For instance, GWTPS is not feasible to remove magnesium hardness. Since only calcium is successfuly removed, the total hardness is still in the high range. By comparing to the result of jar test, the soda-ash dosage of 5 ml (5000 mg/L concentration) is the optimum dosage to precipitates $CaCO_3$ and $Mg(OH)_2$ at pH 13 and 12 respectively. The removal of calcium and magnesium by chemical precipitation in jar test is about 69% and 90% respectively.

ACKNOWELEDGEMENT

First and foremost, I would like to express my heartiest gratitude to Allah the Almighty, for that without His grace and mercy, I would not be able to make it through this journey until the very end line.

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LIST OF ABBREVIATION

WHO	World Health Organization
COD	Chemical Oxygen Demand
FBCs	Fluidized Bed Crystallizers
TSS	Total Suspended Solid
GWTPS	Ground Water Treatment Plant Sludge

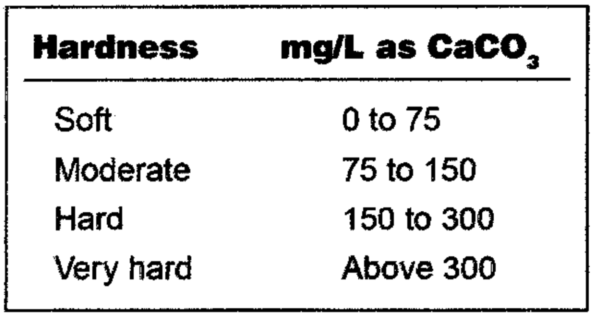
CHAPTER 1

INTRODUCTION

1.1 Background Study

Hardness in water is caused by the ions of calcium and magnesium. Water gets harder if the amount of calcium and magnesium is increased. Strontium (Sr) and barium (Ba) also contribute to water hardness but the amounts of these elements are usually so small that they can be neglected (Gillberg *et al.*, 2003). Calcium usually enters the water as either calcium carbonate ($CaCO_3$), in the form of limestone and chalk, or calcium sulphate ($CaSO_4$), in the form of other mineral deposits. The predominant source of magnesium is dolomite ($CaMg(CO_3)_2$). Hard water is generally not harmful to one's health. **Table 1.1** shows the degree of hardness based on hardness concentration.

Table 1.1: Degree of hardness based on hardness concentration



Hardness	mg/L as $CaCO_3$
Soft	0 to 75
Moderate	75 to 150
Hard	150 to 300
Very hard	Above 300

The reason why water becomes hard is that groundwater often flows through different layers of rock where the water pick up dissolved minerals and particles. Hard water reduces the effects from detergents in laundry machines and consequently more detergents must be used. Water that contains significant amounts of calcium and magnesium could cause a mineral scale build up in pipes which eventually can clog the pipes. Hard water will also cause mineral scale build up in bathtubs, sinks and on everything the water is used (UWSP, 2009).

According to the guidelines from the Worlds Health Organization (WHO) scale deposition will occur when the hardness of Calcium Carbonate ($CaCO_3$) raise above 200 mg/l. The scale deposition will increase if the water is heated. On the other hand, hardness below 100 mg/l will make the water more acidic resulting in corrosion in the pipes. There are no recommended guidelines based on health for hardness, however very hard water can give an unpleasant taste for some (WHO 2009).

1.2 Production of Ground Water Treatment Plant Sludge

Water treatment plant in Chica, Kelantan which is fully owned by Air Kelantan Sdn. Bhd. started its operation on July 2003. This water treatment plant with the capacity of 60MLD, supplies water to the areas around Kota Bharu and Bachok, Kelantan. From the water that is treated in this plant, 87.2% is for domestic use, 11.6% is for industrial use while 1.2% supplies to the mosque.

Chica water treatment plant is fully computer controlled by a system known as System Control and Date Acquisition (SCADA) system which operates 24-hour (real time system). This makes Chica water treatment plant become the most high technology and advanced water treatment plant in Kelantan. The treated water sample is taken every week to be tested to ensure the quality is within the specification. The physical analyses of the treated water are the pH, turbidity and coliform test. The organic and metal content such as iron, manganese and ammonia are also being measured.

The source of water of this treatment plant comes from rivers (70%) and groundwater (30%). The groundwater is taken from 6 well fields (10 acre per well field) with 36 boreholes. The well can be divided into three aquifers which are the first aquifer (15-25m deep), second aquifer and the third aquifer.

The water treatment plant system is shown in the **Figure 1** below.

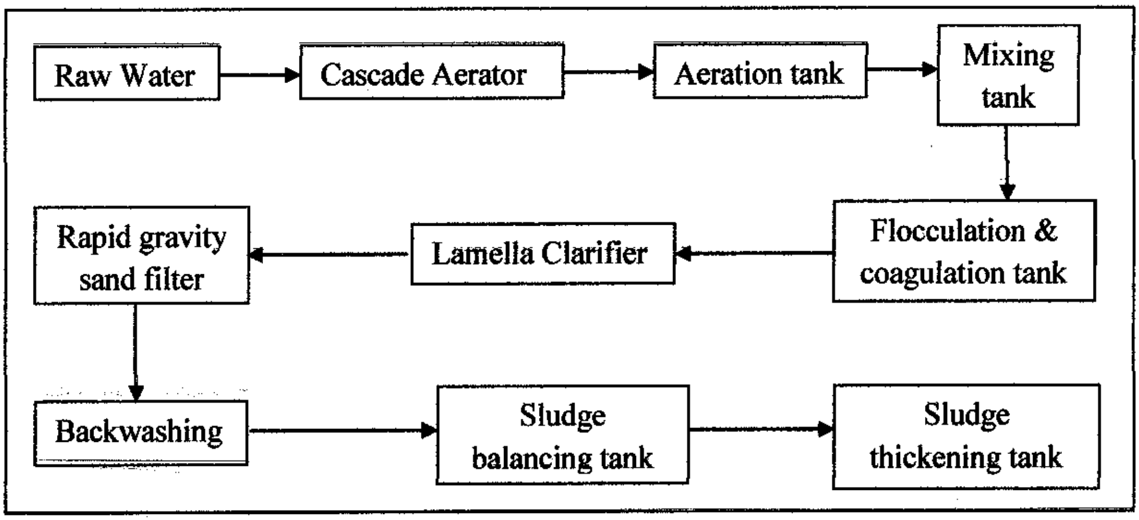


Figure 1.1: Water treatment plant at Chica, Kelantan

The raw water discharge to the inlet of this treatment system is around 2500 m^3/hr. From the inlet chamber, the water is flowed to the **Cascade Aerator**. Cascade Aerator is natural draft gravity aeration for iron oxidation and dissolved oxygen increase. A simple cascade consists of a lateral sequence of basins at various levels, the water spilling over from one basin to the next lower one. It is capable of adding 5 *ppm* oxygen and reducing small amount of free carbon dioxide. This aerator is also applied to iron removal and wastewater dissolve oxygen boost applications.

Figure 1.2: Cascade Aerator

The water then flows into the **aeration tank**. The basic purpose of aeration is the reduction of the content of substances which cause unpleasant tastes and odors as well as discoloration. 6 tonne of lime is added for about 10 times daily. The bubbles can be seen at the surface of this tank as oxygen is added. This oxygen is used to oxidize dissolved iron and manganese and form nearly insoluble hydroxide sludge so that it can be removed in a settling tank or by means of a coarse filter.

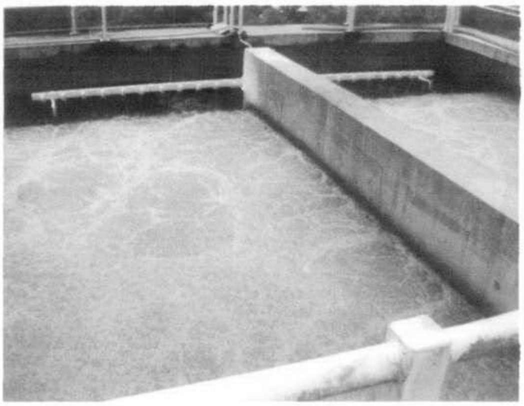


Figure 1.3: Aeration Tank

The aerated water enters the **mixing tank** where it is rapidly mixed with hydrated lime slurry. Speed of the rapid mix mixer is about 1730 *rpm.* 10 *mg/l* of lime is added. The manganese is removed at pH 8 with dose chloride used as oxidation agent. To adjust the pH, polyaluminium chloride (PAC) is added to lower the pH if the pH of raw water is higher than pH 8. While to raise the pH from pH 5 to 7, hydrated lime is added. It can be seen the color of the water inside this tank is brownish as the manganese oxide and iron oxide are formed. Alum is also added which its concentration should be less than 0.2 *mg/l*.

The water then enters the **flocculation and coagulation tank**. Alum liquid and polymer is added to allow the small particles to floc between each other. These flocculants is mixed using mixer with a speed of 1690 *rpm*. Coagulation and flocculation is a clarification method that works by using chemicals which effectively "glue" small suspended particles together, so that they will settle out of the water or stick to sand or other granules in a granular media filter. Many of the suspended water particles have a negative electrical charge. The charge keeps particles suspended because they repel similar particles. Coagulation works by eliminating the natural electrical charge of the suspended particles so they attract and stick to each other.

Next, the water flow into the **lamella clarifier** which is allow vertical flow from the below of the tank. There are five upflow inclined plate clarifier with 15 overflow weirs built in this water treatment plant. This clarifier ensures laminar flow conditions, which results in hydraulic uniformity and high effluent quality. It permits great reduction in the space needed for clarification equipment. In the lamella clarifier, flow enters form both sides of the lamella plate. This allows the feed flow to be equally proportioned, directed and distributed to all settling surfaces without impeding the movement of solids settled. The white layer forming at the water surface of this basin is the iron oxide which is formed during mixing and flocculation process. The slow flow in this large basin allows the floc to settle to the bottom.

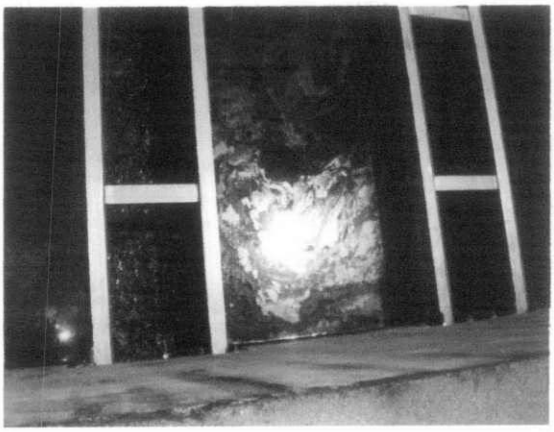


Figure 1.4: Lamella clarifier

After separating most floc, the water is filtered as the final step to remove remaining suspended particles and unsettled floc. In this treatment plant, 10 filtration beds, each consist of 2 beds separated by wash through is used. The filtration rate of this rapid gravity sand filter is 1.5 m^3/hr. **Rapid sand filters** use relatively coarse sand with diameter 1.2 to 2.4 mm, fine sand with diameter 0.85 to 0.95 mm and other granular media to remove particles and impurities that have been trapped in a floc through the flocculation chemicals. Water and flocculants flows through the filter medium under gravity and the flocculated material are trapped in the sand matrix.

Rapid sand gravity filter is cleaned every 72 hours, by **backwashing**, which involves reversing the direction of the water. During backwashing, the bed is fluidized and care must be taken not to wash away the media. The backwashing process is at rate of 5 m^3/hr for 20 minutes. The water from the service tank is used for backwashing process.

The contaminated water from backwashing process is disposed, along with the sludge from the sedimentation basin into the **sludge balancing tank**. This wet sludge then is removed into the **sludge thickening tank** to be dried into cake formed. The sludge cake is disposed to the landfill.

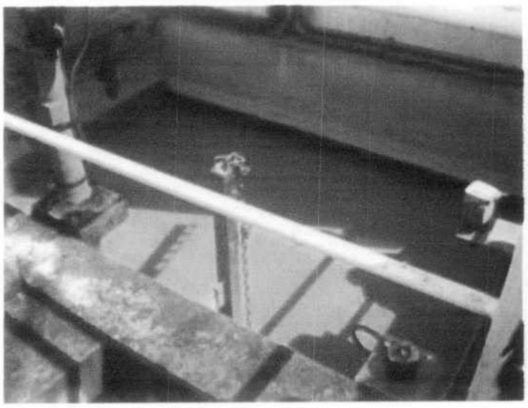


Figure 1.5: Sludge Balancing Tank

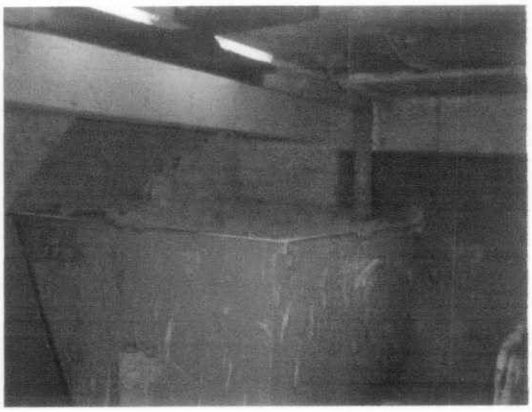


Figure 1.6: Sludge cake

While the clean water from the filtration tank will enter the contact tank where disinfection process is conducted before it is flowed into the clear water tank. Certain amount of chlorine is added to ensure the treated water will not be contaminated during its delivery process.

Table 1.2 below shows the GWTPS constituents.

Table 1.2: GWTPS constituents

Constituent	Percentage by weight (%)
CaO	35.9
Fe_2O_3	29.7
SiO_2	19.5
Al_2O_3	9.43
Eu_2O_3	2.71
P_2O_5	2.10
Mn	0.35
CoO	0.23
Re	0.21
BaO	0.19
K_2O	0.056
Tb_4O_7	0.045
SrO	0.019

1.3 Water Recycling Treatment at Tasek Cement Factory

Figure 1.7 below shows the water recycling treatment that has been conducted at the Tasek cement factory at Ipoh.

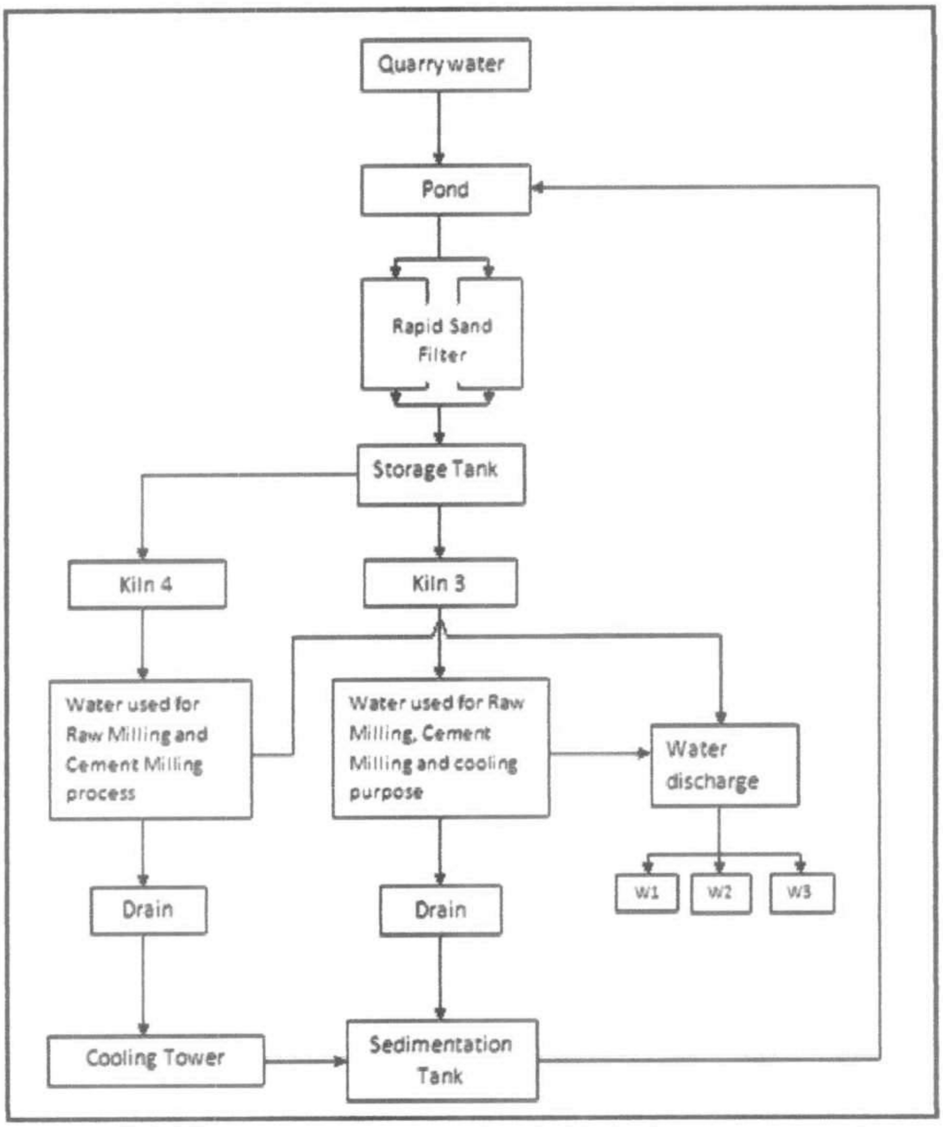


Figure 1.7: Water recycling treatment system at Tasek cement factory

From the main pond, it goes to the rapid sand filter in order to filter the sand contaminant that contained in the water. There are two rapid sand filters available. After that, the water will transferred to Kiln 3 and Kiln 4 tank through the storage tank. From Kiln 3 tank, the water will be using for several processes which are raw milling, cement milling and cooling process. The used water lastly will be sent to two paths which are to provided drain; and releases as water discharge. There are three points to discharge the water which are W1, W2 and W3 (same goes to water that flow through Kiln 4). For water that going through the provided drain, the water will gathered into the sedimentation pond in order to settle the contaminant contains except for drain water that came from Kiln 4. The water will flow to the cooling tower for cooling purpose before entering the sedimentation tank. Then the water will be pumped back to the main pond and will be used again. (Recycling system)

1.4 Problem Statement

At Tasek cement factory at Ipoh, the production of cement demanding huge quantity of water. Therefore, the pond nearby the factory is used as the main source. In the meantime, the pond is surrounded by the limestone hills and also the dusky air due to the cement production itself. In the other hand, high calcium sources were there since the quarry water is the water that contains high level of calcium due to the limestone blasting activities. Since the cement factory is working, there is no hardness removal process done by the management in its water recycling treatment system so that the discharge water containing high amount of hardness will freely flow to the drainage system around the cement factory.

High amount of hardness can cause several problems. In this case, the hardness cause scale problem in the process pipes. Due to that problem, company did spend lot of money in replacing the pipes and maintenance purpose. Therefore, to overcome this particular problem, an improved water recycling treatment system including the hardness removal process is proposed in order to control the limit of hardness contains.

1.5 Objective

The objective of this research study is to determine the feasibility of hardness removal using Ground Water Treatment Plant Sludge (GWTPS).

1.6 Scope of Study

For this study research, the appropriate data/sample is essential in order to establish the findings at the end of the day. Therefore, by collecting water sample from Tasek cement factory and conduct several testing in the laboratory is required. Reviewing the previous study related to the research also recommended for better understanding about the fundamental concept. Although the main idea of the research study is to remove hardness from the wastewater by using GWTPS, other parameters and contaminants also need to be identified in order to understand the characteristic of the water samples. By assumption, water samples from the quarry water will contain high level of hardness and it need to be proven.

CHAPTER 2

LITERATURE REVIEW

2.1 Types of hardness

Two basic types of hardness are associated with the ions causing hardness which are carbonate and noncarbonate hardness. When the hardness ions are associated with the HCO_3^- ions in water, the type of hardness is called carbonate hardness; otherwise, the type of hardness is called noncarbonate hardness. An example of carbonate hardness is $Ca(HCO_3)_2$, and an example of noncarbonate hardness is $MgCl_2$. In practice, when one addresses hardness removal, it means the removal of the calcium and magnesium ions associated with the two types of hardness. (Sincero, 2003)

2.2 Removal of Hardness

2.2.1 Ion Exchange

Hard water can be softened by using water softeners. The water softeners are ion exchangers. Mainly the exchange of cations takes place. Cations are positively charged ions. In this softeners, the exchange medium can be either natural 'zeolites' or synthetic resin beads which are of anionic functional groups. These exchange medium are coated by sodium ions (Na^+) which are supplied from sodium chloride salt, also called brine (NDSU, 1992).

The divalent ions which are more strongly bonded will normally replace the monovalent ions. But at high activities, the monovalent ions can replace divalent ions (Fetter, 2001). So when hard water passes through the water softener, the divalent cations such as Ca^{2+} and Mg^{2+} ions replaces the loosely held monovalent sodium ions

(Na^+) on the exchange media. After softening of large amounts of hard water the exchange media coated with calcium and magnesium ions must be recharged or regenerated. So the exchange medium is regenerated by passing a brine solution of high Na^+ activity through the water softener. The Ca^{2+} and Mg^{2+} ions on the exchange media are replaced by Na^+ ions. During the recharging the back flushed water with high content of calcium and magnesium is taken to wastewater (NDSU, 1992). As can be seen in **Figure 2.1** below the hard water is transported to the resin tank from above and is infiltrating through the resins. By the time the water has reached the bottom of the tank the water is softer and can be transported to the household.

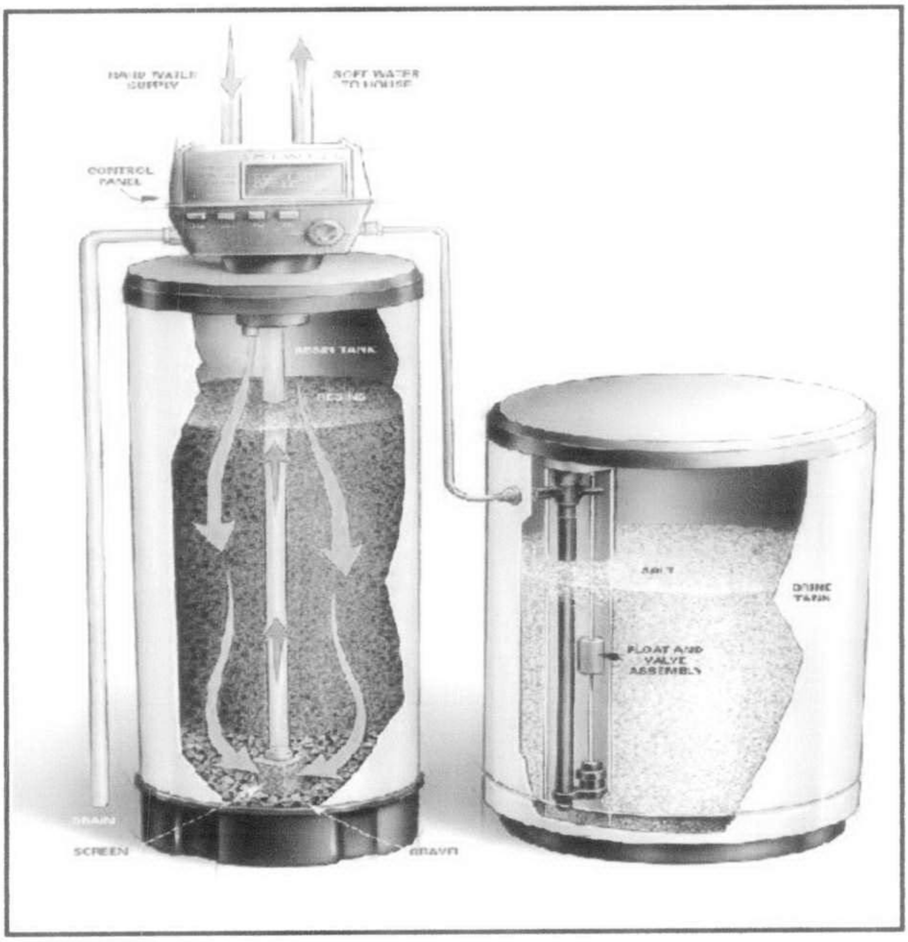


Figure 2.1: Basic structure of a water softener

The general reaction during water softening process and recharging process is as shown below (NDSU, 1992):

Softening Process

$NaZeolite + Ca^{2+} \rightarrow CaZeolite + Na^{+}$

and

$NaZeolite + Mg^{2+} \rightarrow MgZeolite + Na^{+}$

Recharging Process

$NaC1 + CaZeolite \text{ --> } NaZeolite + CaC1_2$ and

$NaC1 + MgZeolite \text{ --> } NaZeolite + MgC1_2$

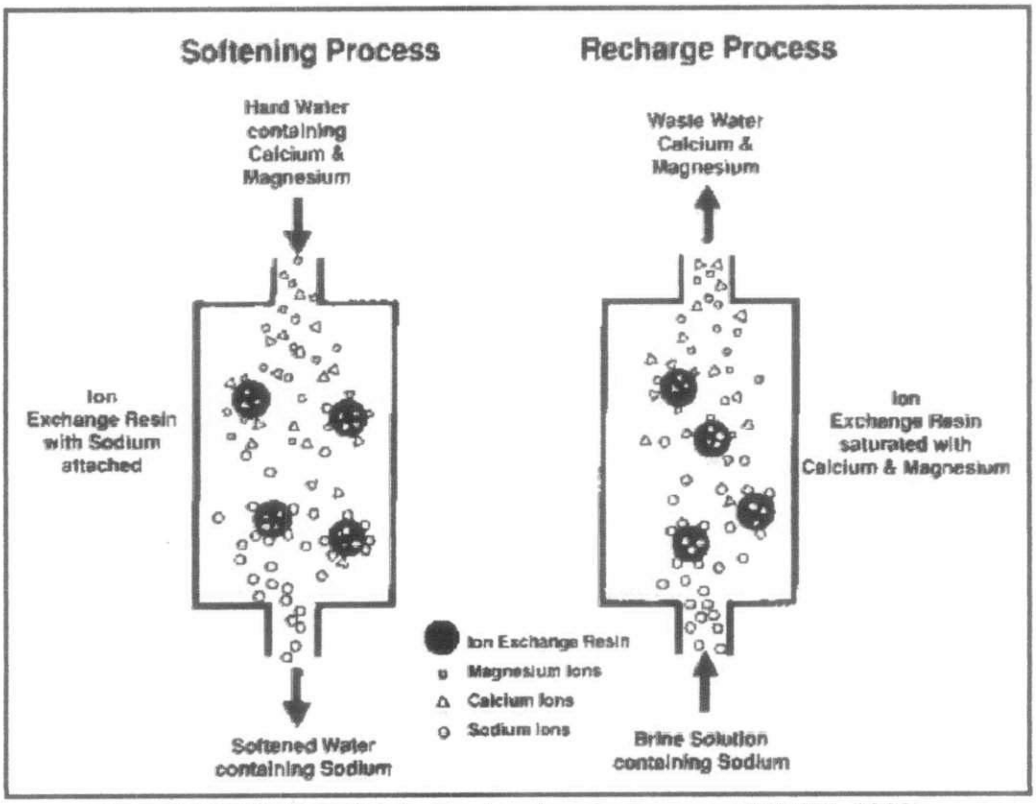


Figure 2.2: Water softening and recharging process (NDSU, 1992)

The size and selection of water softener depends on the measured hardness of water to be treated and the use and need of treated water supply based on the calculated value. Also the time between recharging cycles depends on the hardness of the water, the amount of water used, the size of the unit, and the capacity of the exchange media to remove hardness etc (Hometips, 2009).

Ion exchange can be defined as the reversible interchange of ions between a solid and a liquid phase in which there is no permanent change in the structure of the solid. Typically, in water softening by ion exchange, the water containing the hardness is passed through a column containing the ion-exchange material. The hardness in the water exchanges with an ion from the ion-exchange material. The alkalinity remains unchanged. The exchange results in essentially 100 percent removal of hardness from the water until the exchange capacity of the ion-exchange material is reached, as shown in **Figure 2.3** below. (Mackenzie, 2007)

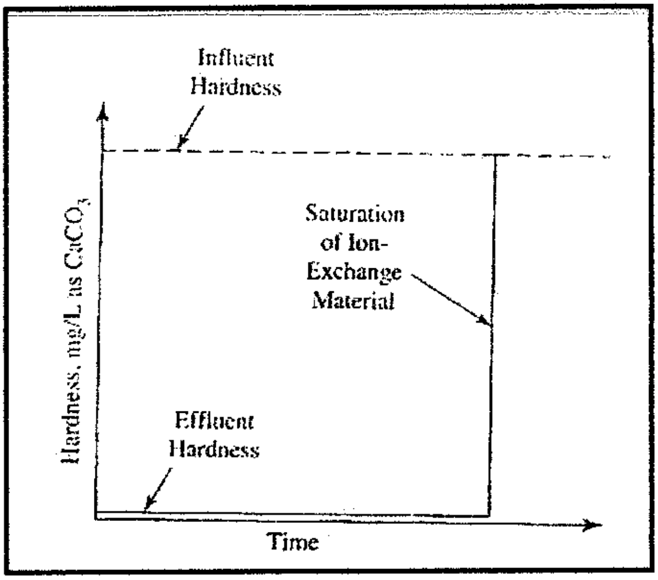


Figure 2.3: Hardness removal in ion-exchange column.

When the ion-exchange material becomes saturated, the column is taken out of service and the ion-exchange material is regenerated. The hardness is removed from the material by passing water containing high concentration of Na^+ through the column.

Ion-exchange resin beds sit inside the tanks and contain natural or synthetic granular materials. Multiple tanks are used to allow the regeneration of one without disrupting household water supply. The substances attract sodium cations (Na^+) courtesy of a slight electrical charge. As the treatment water flows through the tank the Na^+ are displaced (exchanged) by calcium or magnesium hardness ions (Ca^{2+} or Mg^{2+}). The Na^+ is not damaging to water hardware and is subjected to removal by the flowing water (Newman,2007).

Regeneration of the resin beds is required when they are completely saturated with hardness ions. This can be done manually with clean water, or by an automatic device that flushes the tank with concentrated Na^+ brine. An unspecified contact time is required to allow the regenerant to effectively replace the hardness ions with Na+ ions (Genders, 1997). The used regenerant and rinse water flow into a holding tank. This used water should be disposed of off-site or to a sewer (Genders, 1997).

2.2.2 Lime Soda Softening

Lime-soda softening is the most commonly method in removing hardness. The objective of using Lime-soda softening is to precipitate the calcium as $CaCO_3$ and the magnesium as $Mg(OH)_2$. In order to precipitate $CaCO_3$, the pH of the water must be raised to about 10.3. To precipitate magnesium, the pH must be raised to about 11. If there is not sufficient naturally occurring bicarbonate alkalinity (HCO_3^-) for the $CaCO_3(s)$ precipitate to form, adding up the CO_3^{2-} to the water is needed. **Figure 2.4** below shows the summary of stages in the removal of hardness (Mackenzie, 2007).

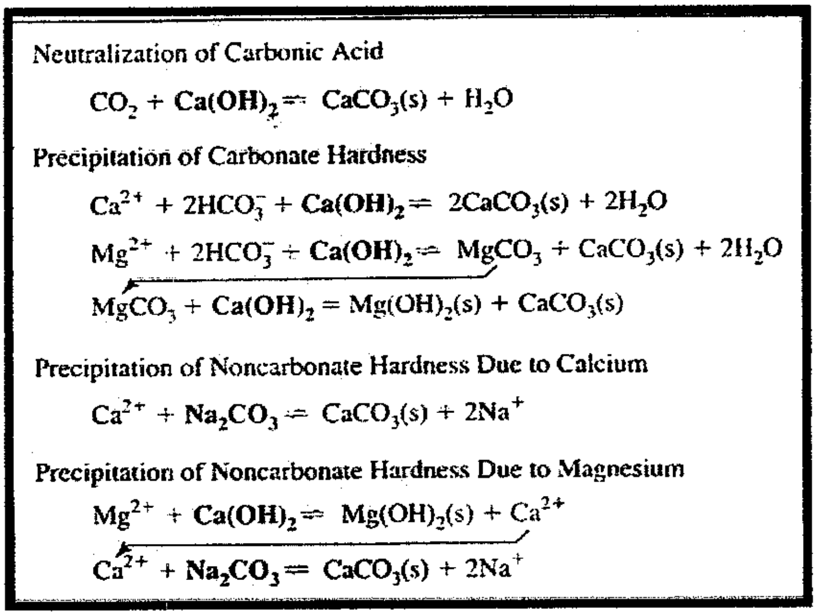


Figure 2.4: Summary of softening reactions. (Note: The chemical added is printed in bold type. The arrow indicates where a compound formed in one reaction is used in another reaction.)

Above pH 8.3, the bicarbonates are converted to carbonate ions. Hydroxide appears at a pH greater than 9.5 and reacts with carbon dioxide to yield both carbonates and bicarbonates. The maximum CO_3^{2-} concentration for dilute solution is in the pH range 10-11 (Viessman *et al*, 2005). Calcium and magnesium ions are soluble when associated with bicarbonate anions. But if the pH of a hard water is increased, insoluble precipitates of $CaCO_3$ and $Mg(OH)_2$ are formed. This is accomplished in water treatment by adding lime to raise the pH level. At a value of about 10, hydroxyl ions convert bicarbonates to carbonates to allow the formation of calcium carbonate precipitate.

Lime is used in pH adjust to 7.5. It was observed that the addition of lime increases hardness level significantly. Therefore, soda was used to avoid elevating hardness levels. In precipitation purpose, soda ash was used to increase wastewater alkalinity but elevated pH (above 9) partly destroyed the flakes. So, this result makes simultaneous precipitation and coagulation unfeasible (Viero *et al*, 2002).

2.2.3 Crystallization Process in Pellet Reactor

Fluidized bed crystallizers (FBCs), also called pelletizers or pellet reactors, are an attractive alternative to conventional water softening process (Harms *et al*, 1992). In addition, the study also concludes that, calcium removal is very rapid within the reactor, leaving a large portion of the bed unused at any one time; the calcium removal rate can be described by the Reddy-Nancollas crystallization model; high supersaturation levels due to poor mixing at the point of chemical mixing promotes microfloc production and solids carryover in the effluent; classification of the pellets within the fluidized bed does not occur; the sampling techniques appear to be adequate for characterizing FBC reactors; and operation of FBCs would be aided by the installation of a pressure gauge on the FBC influent to monitor bed growth.

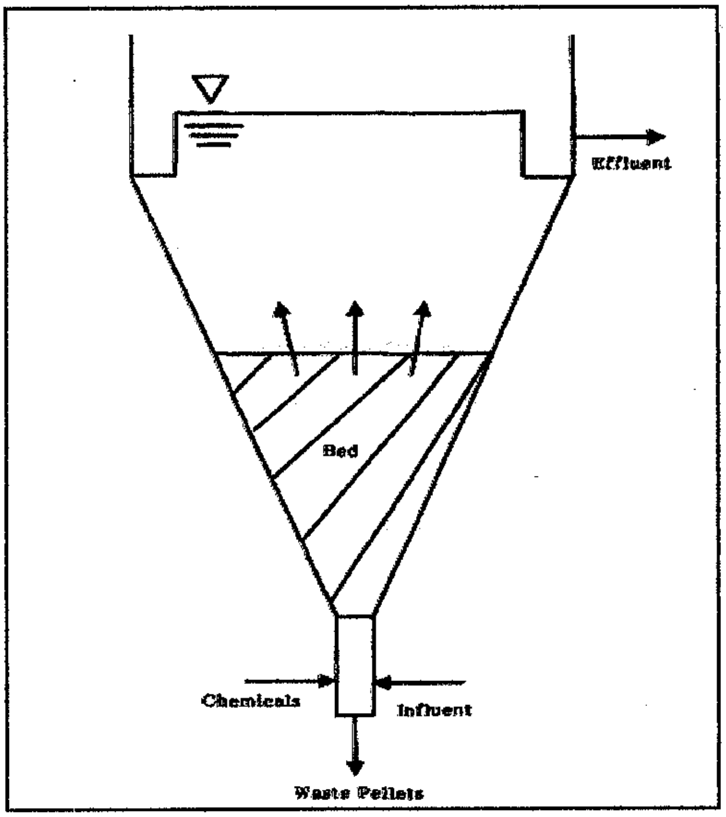


Figure 2.5: Typical Spiractor-Type Fluidized Bed Crystallizer

The reactor is fluidized by the upward flow of water, as shown in **Figure 2.5**. Chemicals such as lime or sodium hydroxide are added near the bottom of the reactor to cause the crystallization of $CaCO_3$ onto the fluidized pellets. Pellets are wasted from the bottom of the reactor periodically. Alternatively, the entire bed can be wasted after several weeks and the reactor recharged with silica sand.

From the study of FBCs, the initial calcium removal rate was very rapid. The reactors precipitate more than 90% of the applied calcium in about 30 sec of mean detention time. Removal of calcium hardness in raw wastewater was about 75%.

For method comparing purpose, Mahvi *et al* (2005) has the same point of view in the issue of crystallization process. According to them, the chemistry of pellet softening process is essentially the same as conventional softening process, instead of precipitation of calcium carbonate which does not have any useful consumption and must be removed once in a while some how, crystallization process in a fluidized bed reactor produces pure solid grain of calcite. These pellets can be used in some industries and are in fact an economic commodity. For this purpose a pilot was set up as a crystallization reactor and water with total hardness ranging from 150 up to 500 mg/l as $CaCO_3$ was fed to the system with a flow rate of 24 l/hr. To remove hardness, caustic and soda, lime milk was fed to the system separately at first stage at then together. The formation of $CaCO_3$ crystals on the surface of sands represented the reduction of hardness.

In this process very tiny spherical crystal beads are produced with calcite nature (Rankin and Sutcliffe, 1999). With the use of sands and grains as seeds the removal efficiency of hardness can be increased (Todd, *et al*, 1994). In pellet reactors there is a chance to remove other cations such as Sr, Fe and Mn as well as calcium and manganesium (Todd, *et al*, 1994). It is also possible to reduce sulfate concentration of over 600 mg/l to lower than 70 mg/l. with the addition of calcium aluminates and lime, in such a condition Ca, and Mg have been removed almost completely.

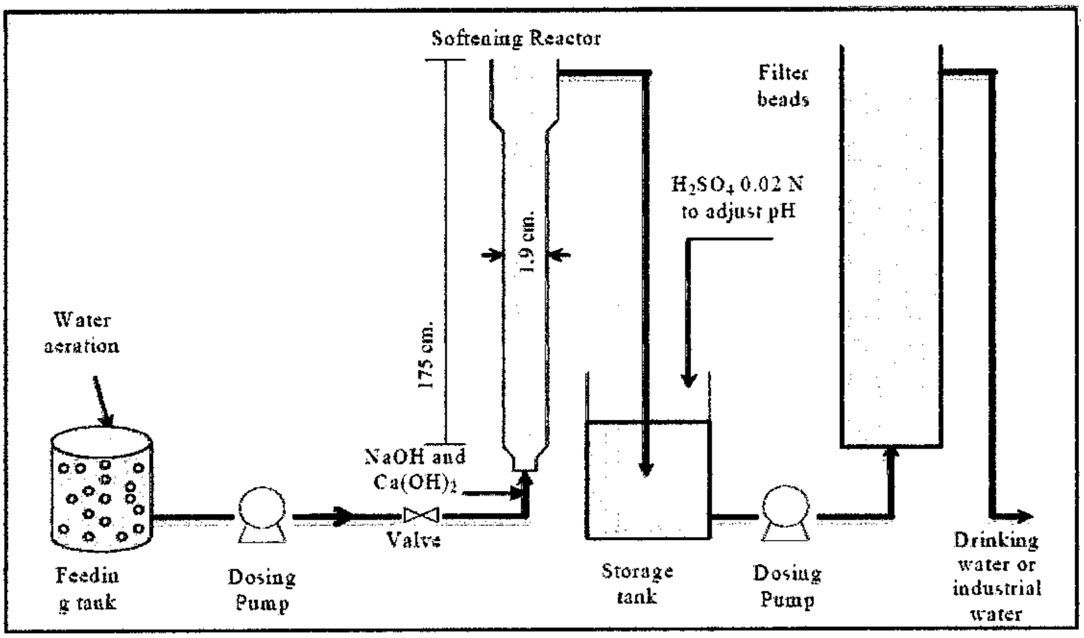


Figure 2.6: Water softening technology in Pellet Reactor

The result on the removal of hardness with influent concentration of 150, 300 and 500 mg/l as $CaCO_3$ is about 50%. These results showed that the ability of pellet reactor for hardness removal is independent of hardness concentration.

Since the total surface area for crystal formation is the same, therefore the efficiency is almost equal. For any of the influent concentration of hardness and a flowrate of 24, l/hr the efficiency was around 50%.The result of hardness removal for influent concentration 300 mg/l as $CaCO_3$ with addition of $Ca(OH)_2$ and NaOH were 20 and 52% respectively. These figures for influent concentration of 150 m/gl as $CaCO_3$ were 57 and 67% respectively. These results showed that NaOH addition has more effect on hardness removal.

CHAPTER 3

METHODOLOGY

3.1 Methodology Flow Chart

There are several procedures that need to be completed in order to ensure the project flow is smooth and accomplish in the given period. The flow chart is as below:

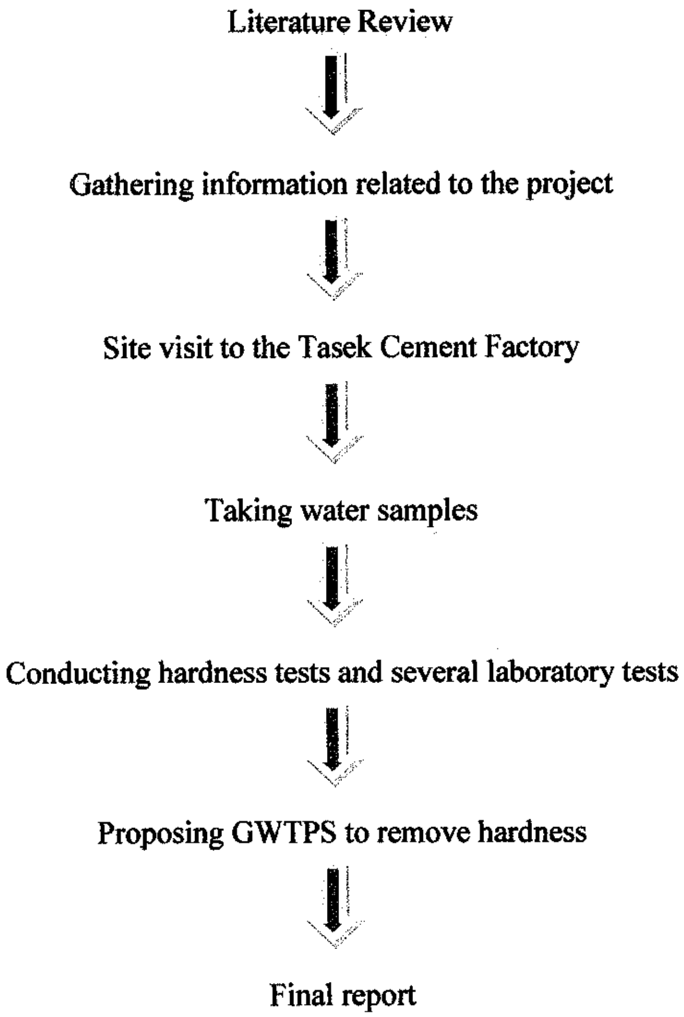


Figure 3.1: Flow Chart activities

3.2 Literature Review

To have an understanding of this project, the research has been done by referring the journals, reference books and websites. The research also has been carried out by getting details explanation from supervisor and post graduate's student that has been experienced on related topic of this project.

3.3 Site Visit to the Tasek Cement Factory

In order to understand the concept and design of the water recycling treatment plant, visiting the research area is vital. By deep explanation given by the person in-charge of the treatment plant, it enhanced the understanding of the researcher about the process and treatment that has been carried out. At the same time, water samples were collected in the desired amount in order to be tested in the laboratory.

3.4 Total Suspended Solids (TSS) Test Procedure

The objective is to calculate the non-filterable residue in water/wastewater using gravimetric method. The apparatus that being used in this experiment are 47 mm filter paper, filtering flask, watch glass, drying oven, desiccators and measurement cylinder. To ensure the filter paper is dry enough, filter paper will be put into the oven at 103°C for 24 hours. After that, the filter paper should be weighted as initial weight. Then, by using the tweezers, put the filter paper on the filter holder with the wrinkled surface upward. Filter 400 ml of well-mixed, representative water sample by applying vacuum to the flask. Follow with three separate 10ml washings of deionized water. After that, remove the filter paper by using tweezers and put back into the aluminium pan. Then, dry the filter paper at 103°C for 1 hour in the oven. In order to make the filter paper in the room temperature after the dry process, put the filter paper into the desiccators. Lastly, weight the filter paper as the final weight. Calculation on TSS value should be conducted.

3.5 pH Measurement Procedure

The objective is to determine the pH of sample either it acid, neutral or alkali. In the experiment, pH meter is used. Putting the electrode in the sample is conducted by pressing the Dispenser Button. The pH value is recorded since the pH value displayed is stable.

3.6 Nitrogen, Ammonia Test using Nessler Method

The objective of this test is to measure the Nitrogen, Ammonia contain in the wastewater sample by using Nessler method. Nessler reagent is a toxic and corrosive liquid because it contains of mercuric iodide. Therefore, the solution cannot be poured into the drain. Carefully conduct the experiment is highly encouraged. The 25 ml of sample will be used for preparing the sample and blank solution. Add three drops of Mineral Stabilizer to each cylinder and continued by three drops of Polyvinyl Alcohol Dispersing Agent. Let the solution mix properly by stopper and invert several times. After that, pipette 1.0 ml of Nessler Reagent into each cylinder. Take 1 minute for reaction purpose. Pour 10 ml of each solution into a square sample cell. Insert the sample into the Spectrophotometer and read the value of Nitrogen, Ammonia. Remember that, always use blank solution to zero the readings.

3.7 Nitrate Test using Cadmium Reduction Method

The objective of conducting this test is to determine the value of nitrate contain in the wastewater sample by using NitraVer 5 Nitrate Reagent Powder Pillow. First of all, setting up the spectrophotometer is most appropriate. Select the 355 N,Nitrate HR PP as test requirement. Fill the square sample cell with 10 ml of sample. Add the content of one NitraVer 5 Nitrate Reagent Powder Pillow. Take 1 minute for reaction time. Shake the cell vigorously until the time expires. After that, leave the cell for 5 minutes as the reaction period. Then, insert the sample cell into the spectrophotometer and record the reading. For blank preparation, just use 10 ml of wastewater sample. Remember that, always use blank solution to zero the readings.

3.8 Alkalinity Test Procedure

The objective of conducting this test is to determine the alkalinity of water samples. All forms of alkalinity (hydroxide, carbonate and bicarbonate) can be measured by titration with a standard solution of an acid (0.02N H_2SO_4) and represented in terms of equivalent $CaCO_3$/L. For samples with initial pH above 8.3, the titration is made in two stages. First titration is carried out until the pH is lowered to 8.3 (phenolphthalein end point). This is followed by titration of the aliquot to pH 4.5 (methyl orange end point). When the pH of sample is less than 8.3, single titration to pH 4.5 is made. The calculation of alkalinity according to the usage of H_2SO_4 in the titration process should be done in the proper equation.

3.9 Sulfate Test using SulfaVer 4 Method

The objective of conducting this test is to determine the sulfate contain in the water sample. First of all, select the specified test in the spectrophotometer which is 680 Sulfate. Prepare a sample by fill in the 10 mL sample into the square sample cell. Add the contents of one SulfaVer 4 reagent powder to the sample cell. Swirl vigorously to dissolve powder. Note that, white turbidity is form if sulfate is present. Let the solution takes 5 minutes to react. Besides that, prepare a blank solution by pouring 10 mL of sample into the second square sample cell. When the timer expires, insert the blank into the cell holder in the spectrophotometer. Set up the device with zero reading. Then, insert the prepared sample into the cell holder and press the read button. Record the data.

3.10 Preparation of GWTPS

Since the GWTPS is in the cake form and in wet condition, it needs to be in powder form and dry. Therefore, the wet GWTPS is dried in the oven for 24 hours at 105°C of temperature. After that, the GWTPS is ready to be used for experimental purpose.

3.11 Experimental procedure of removal of hardness by using GWTPS

The objective of this experiment is to determine the feasibility of using GWTPS to remove hardness in water sample. At the beginning of the experiment, the initial value of hardness is measured by conducting hardness determination tests. Since the value of hardness is too high in the water sample, it needs to be dilute to 1:100 so that the spectrophotometer can read the hardness contain. 54 conical flasks has been used in providing three samples for each dosage where is three difference dosages of GWTPS are going to be tested in six variations of time. The three dosages are 500 mg/L, 1000 mg/L and 5000 mg/L. The time variation is from 30 minutes to 300 minutes (5 hours). In the other hand, for each time variation, the water sample must contain of three dosages in nine conical flasks where is triplicate method is implemented (30 minutes: 3 samples contains 500 mg/L dosage, 3 samples contains 1000 mg/L dosage and 3 samples contains 5000 mg/L dosage of GWTPS, same goes to each time variation).

After preparing the samples, initial pH measurement is conducted for each sample. After that, all the samples will shakes using the orbituary shaker for the respective time variation. After 30 minutes, the samples are taken out and ready to be filtered and measurement of final pH. After filtering process, the filtered sample will be test for hardness contain. The final reading of hardness contain is compared with the initial reading (before shake) of hardness. The process is repeated for another time variations.

3.12 Hardness Determination Tests – Magnesium and Calcium using Calmagite Colorimetric Method

The objective of conducting this test is to determine the value of hardness contain in the sample. There are several equipments and items are needed to conduct this experiment which are alkali solution for magnesium and calcium test, Calcium and magnesium indicator solution, EDTA solution 1M, EGTA solution, Cylinder 100 ml, dropper 1 ml and sample cell 1 inch square. First of all, 100ml of sample was poured into the 100 ml graduated mixing cylinder. Then, 1.0 ml of Calcium and Magnesium indicator solution was added using a 1.0 ml measuring dropper. The solution was shaken well. After that, 1.0 ml of alkali solution for calcium and magnesium test was added by using a 1.0 ml measuring dropper. The solution also was shaken well. 10 ml of the solution was poured into each of three square sample cells. For blank preparation, 1 drop of 1M EDTA was added to the first cell. Swirl to mix the solution. For magnesium sample, 1 drop of EGTA solution was added to the second cell. Swirl to mix the solution. Blank sample was placed into the spectrophotometer in the right position. **222,magnesium** program has been selected to determine the value of magnesium. Then, zero buttons was pressed. Second cell was inserted into the cell holder. READ button was pressed. Record the result and the results are in mg/L magnesium as calcium carbonate. By using the second cell without removing it from the spectrophotometer, **220,calcium** has been selected. ZERO button was pressed. After remove the second cell, the third cell was placed into the cell holder and read the result for calcium hardness. Results are in mg/L calcium as calcium carbonate. The test was repeated using the same wastewater sample for comparing purpose.

3.13 Milestone of the project

Timeline (Week) / Activities and Progress	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6	Week 7	Week 8	Week 9	Week 10	Week 11	Week 12	Week 13	Week 14	Week 15
Preparing manuals and laboratory apparatus	🟨						MID SMESTER BREAK								
Laboratory works and research on literature review		🟨	🟨	🟨	🟨	🟨									
Completion of progress report								🟨							
Laboratory works									🟨	🟨	🟨	🟨			
Poster Preparation											🟨				
Finalizing dissertation													🟨		
Oral presentation															🟨

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Characteristic Tests Result

There are some results on several tests/experiments that have been conducted regarding determining the characteristic of water sample.

Tests	Result	Description
pH measurement	7.65	Slightly alkaline
Nitrogen, Ammonia tests using Nessler Method	-0.61 mg/L NH_3-N	No nitrogen,ammonia contain in the wastewater
Nitrate tests using Cadmium Reduction Method	1.0 mg/L NO_3-N	Nitrate contain is very low
Total Suspended Solid	8.28 mg/L	Solid contain in the wastewater is very low
Alkalinity Tests (total alkalinity)	260 mg $CaCO_3$/L	High alkalinity
Sulfate tests using SulfaVer 4 Method	24.3 mg/L SO_4^{2-}/L	Sulfate contain is in the medium condition
Calcium Hardness Concentration	234 mg/L as $CaCO_3$	Total hardness concentration is 316 mg/L as $CaCO_3$ – CLASSIFIED AS VERY HARD WATER
Magnesium Hardness Concentration	82 mg/L as $CaCO_3$	

Table 4.1: Result of several tests conducted to determine the characteristic of water sample

4.2 Removal of magnesium hardness using Ground Water Treatment Plant Sludge (GWTPS)

After conducting the experiment, below are the results.

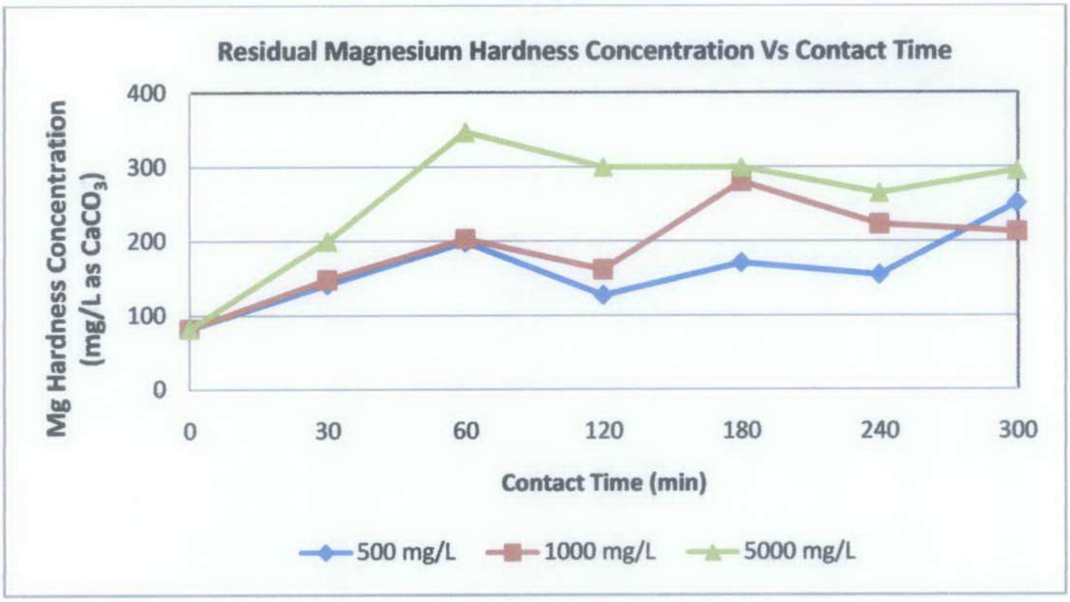


Figure 4.1: Graph of Residual Magnesium Hardness Concentration Vs Contact Time

The above graph shows that from the initial value of magnesium concentration which is 82 mg/L as $CaCO_3$, it's going increased by time. For dosage of 500 and 1000 mg/L, at 1 hour of shakes, the value is slightly the same but after 60 minutes, the value is differ until 300 minutes of shakes. At the end of experiment, the value of magnesium concentration falls within 200 and 300 mg/L. The pattern of the three lines is also slightly the same showing that each sample has the same reaction between sludge, but differs in value of concentration.

It is because of the magnesium hardness concentration is increasing; another test is conducted using distilled water mixed with the fixed dosage of GWTPS (1000 mg/L) and shakes for various contact time. The result is in the **Figure 4.2** below.

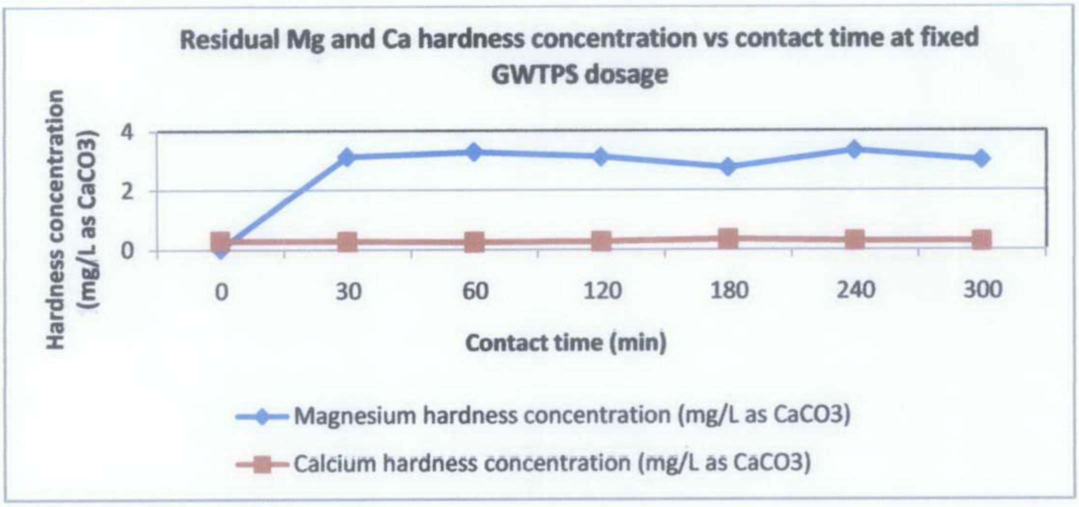


Figure 4.2: Graph of residual Mg and Ca hardness concentration vs contact time at fixed GWTPS dosage

From **Figure 4.2** above, the result shows that the magnesium hardness concentration is increased after 30 minutes of shake. For calcium hardness concentration, the value is remaining the same from initial until the final shakes. By this obtained result, it does conclude that the GWTPS have contained magnesium ion. That's the reasons why the magnesium hardness concentration is increasing when the GWTPS is shakes with the water sample contained high concentration of hardness.

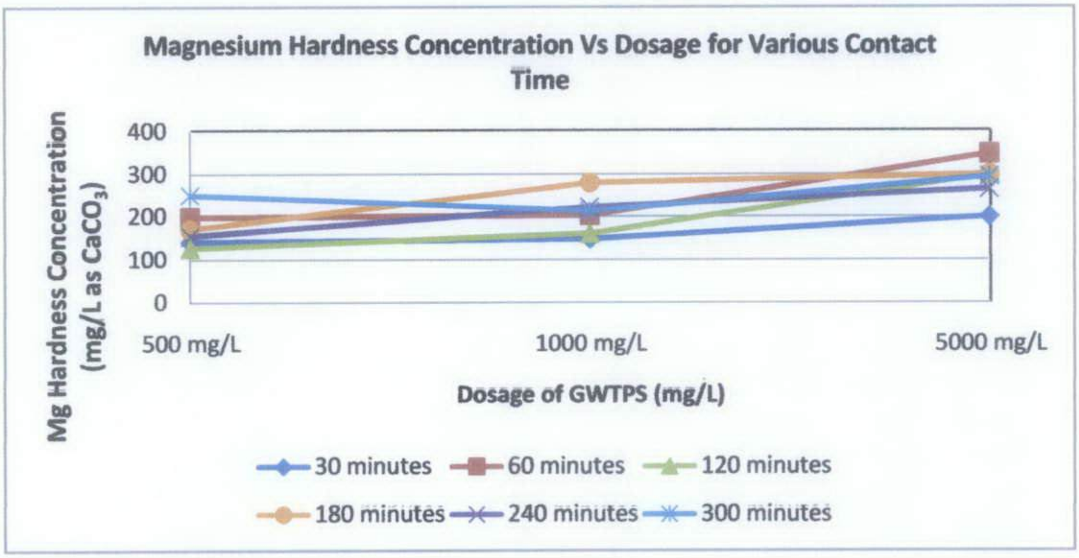


Figure 4.3: Graph of Magnesium Hardness Concentration Vs Dosage for Various Contact Time

The above result shows that for 1000 mg/L of dosage, the concentration of magnesium is within the range of 150 and 230 mg/L for all time variation. In the other hand, starting from 30 minutes of shakes until 300 minutes, the magnesium concentration for 1000 mg/L dosage is not vary too much but not for another 2 dosages.

4.3 Removal of calcium hardness using Ground Water Treatment Plant Sludge (GWTPS)

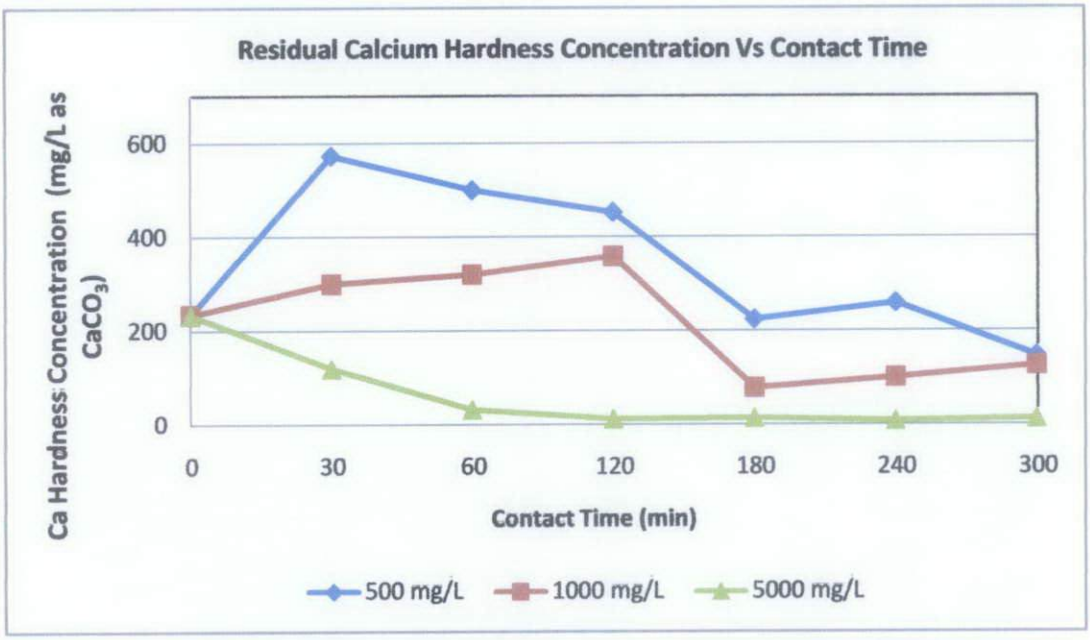


Figure 4.4: Graph of Residual Calcium Hardness Concentration Vs Contact Time

From **Figure 4.4** above, the calcium is effectively removed by using the dosage of 5000 mg/L. For the dosage of 1000 mg/L and 500 mg/L, at 300 minutes of shakes, the calcium contain in the samples also partly removed. By comparing the 3 dosages, the dosage of 5000 mg/L is an effective dosage to remove calcium. In terms of pH, by referring to the **Figure 4.8**, the initial and final pH of the three dosages is nearly 9. So, in this case, the precipitation of calcium occurs in the water sample at pH 9. The percentage of calcium removal is about 96%.

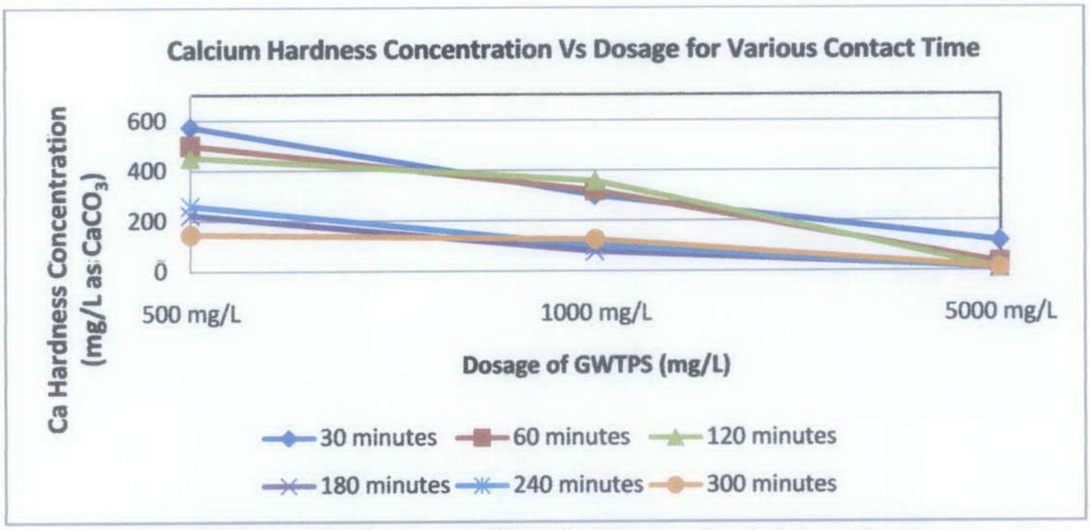


Figure 4.5: Graph of Contact Time Vs Dosage for Calcium Hardness

From **Figure 4.5**, the result shows that for 5000 mg/L of dosage, all time variations brings the calcium concentration to the low amount at the end of the experiment. In the other hand the graph also shows that for the 3 dosages, the calcium concentration is decreasing when dosage is increased.

4.4 Removal of total hardness using Ground Water Treatment Plant Sludge (GWTPS)

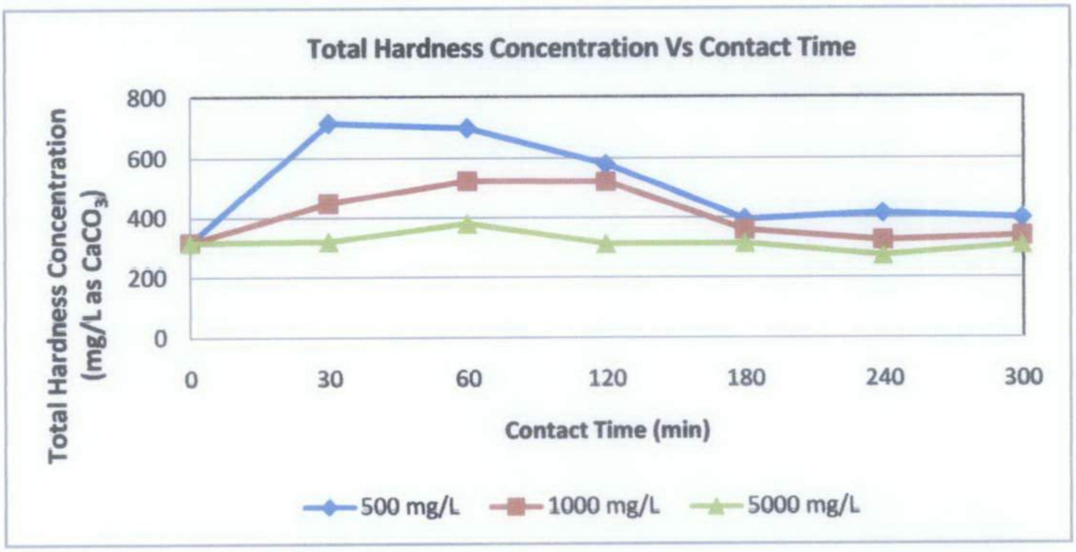


Figure 4.6: Graph of Total Hardness Concentration Vs Contact Time

From **Figure 4.6,** since the magnesium concentration has not been removed, the total hardness is still in high condition although the calcium hardness is effectively removed. It is because; total hardness is the total of calcium and magnesium concentration. From the graph, the pattern of line of the dosage of 5000 mg/L shows that the total hardness concentration is maintained from initial to the final shakes.

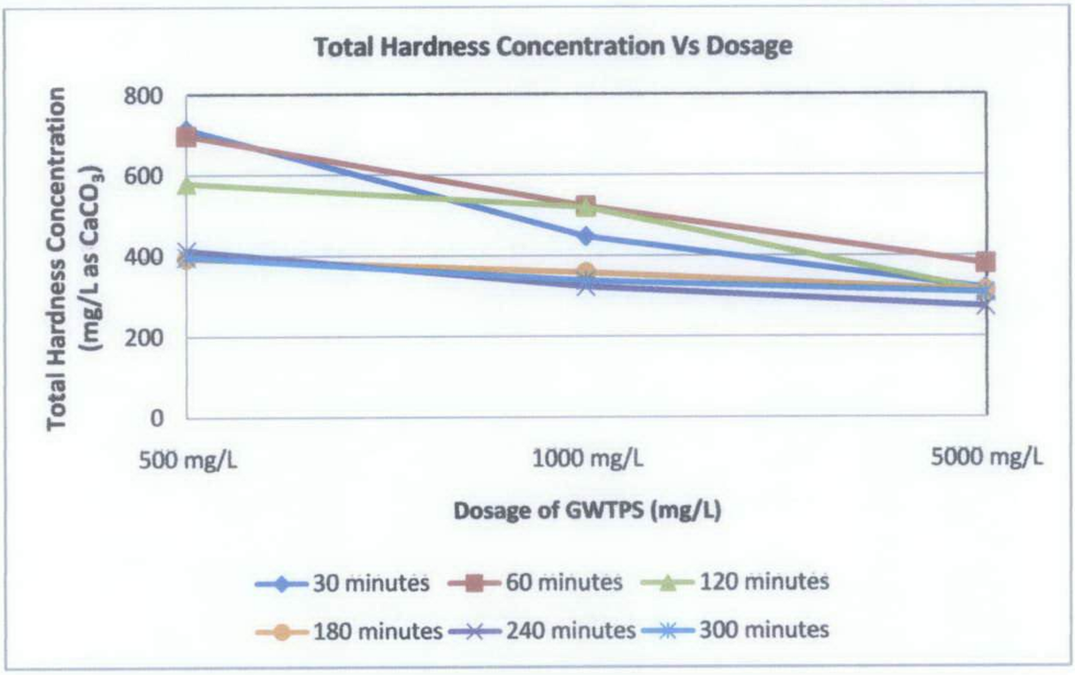


Figure 4.7: Graph of total hardness concentration vs dosage

From **Figure 4.7**, the result shows that for 5000 mg/L dosage, the total hardness concentration is fall into small range (300 – 400 mg/L) which is better for all time variations. By comparing to the dosage of 500 mg/L, the total hardness concentration is falls between wide ranges (400 – 700 mg/L) and it shows that the dosage is not appropriate to be an optimum dosage for all time variations.

After the data analysis is conducted using t-*test,* by comparing the dosage between 1000 mg/L and 5000 mg/L, the t_{stat} is equal to 2.35. The bounds of the t-value is $1.89 < P < 2.36$. Therefore, the null hypothesis can be accepted. In the other side, by comparing the dosage between 500 mg/L and 1000 mg/L, the bounds of the t-value is out of range. Therefore, the null hypothesis is rejected. So, there is evidence to conclude that mean total hardness concentration of 1000 mg/L is different from the mean total hardness concentration of 5000 mg/L. Furthermore, the mean total hardness concentration of 1000 mg/L is higher than 5000 mg/L. (See table 16 and 17 at appendix for details)

4.5 Effect of variation of pH vs. contact time for various dosages of GWTPS

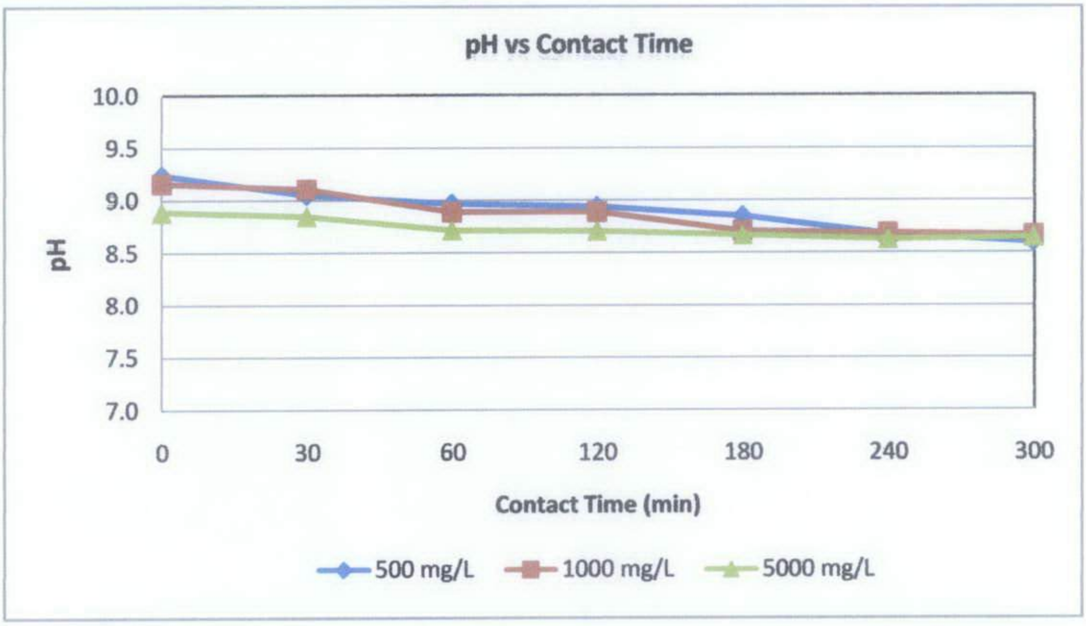


Figure 4.8: Graph of pH vs contact time

The **Figure 4.8** shows that the pH in the six variations of time. From the graph, the starting point of the shaking shows that the pH is exceeding 9. But before that, the initial pH before adding the sludge is 6.37. Thus, adding the sludge is increase the pH value of the sample. Furthermore, the increasing of pH in the sample makes the calcium tend to precipitate.

4.6 Chemical Precipitation

4.6.1 Determining the optimum pH to remove magnesium at various dosages of soda-ash (Na_2CO_3) and fixed pH

It is because of the calcium removal using GWTPS is from precipitation process, jar tests is conducted in order to compare the result in terms of pH. Initially, the pH is fixed to 9 while the dosage of soda-ash is varying from 1 ml to 3.5 ml. The concentration of soda-ash is about 5000 mg/L.

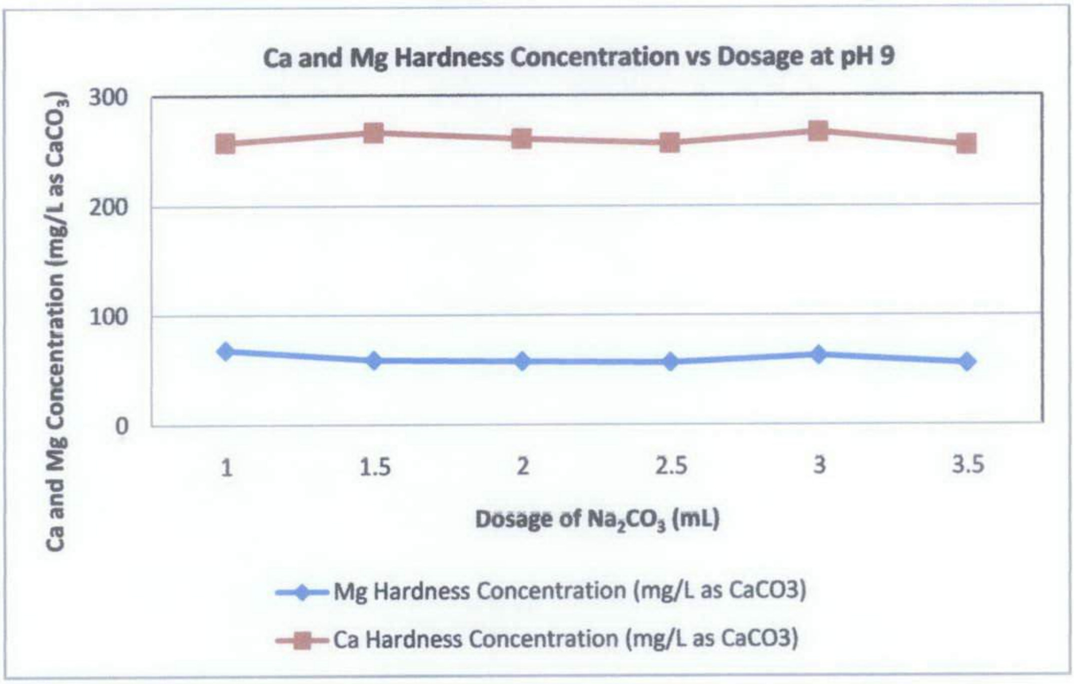


Figure 4.9: Graph of Ca and Mg Hardness Concentration vs dosage of Na_2CO_3 at pH 9

From the obtained result in **Figure 4.9**, at pH 9, the hardness concentration of magnesium is slightly reduced from the initial value of 82 mg/L to the range between 50 – 70 mg/L. For hardness concentration of calcium, there is no reducing value at pH 9. In terms of varying dosage, there is no big difference in reducing the hardness concentrations. Therefore, the pH is up to 12 and the same dosages are used in order to see the changes of the result. The result is in the **Figure 4.10** below.

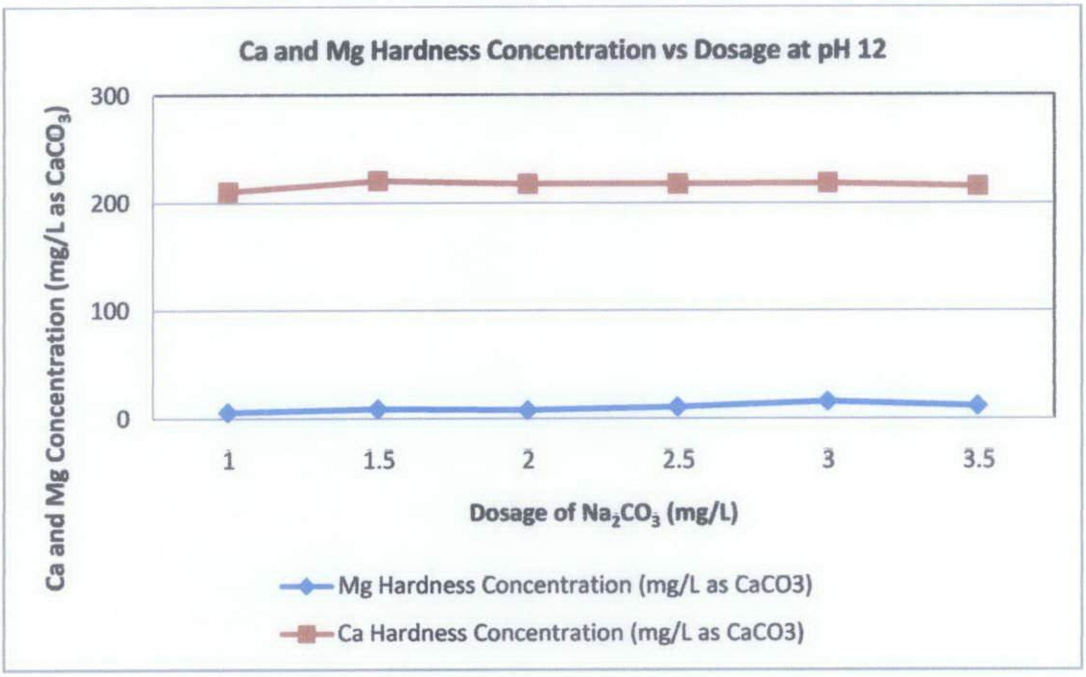


Figure 4.10: Graph of Ca and Mg Hardness Concentration vs Dosage of Na_2CO_3 at pH 12

From **Figure 4.10**, the result shows that the hardness concentration of magnesium is effectively removed at pH 12 with the dosage varied from 1 to 3.5 mL. For hardness concentration of calcium, the result shows that there is no difference between the results obtained from **Figure 4.9**. So, for instance, from both graph, the conclusion that can be made is, the optimum pH for removing magnesium is at pH 12.

In order to remove the calcium, the optimum pH should be determined. For that purpose, the next step is varying the pH at fixed dosage.

4.6.2 Determining the optimum pH to remove calcium at fixed dosage of soda-ash (Na_2CO_3) and various pH

By analyzing the results obtained from above graph, the dosage used should be more than 3.5 mL. So, by conducting try and error method, the dosage of soda-ash is fixed at 5 mL and the pH is varying from 4 to 13 in order to find the optimum pH to remove the calcium. The result is shown in **Figure 4.11** below.

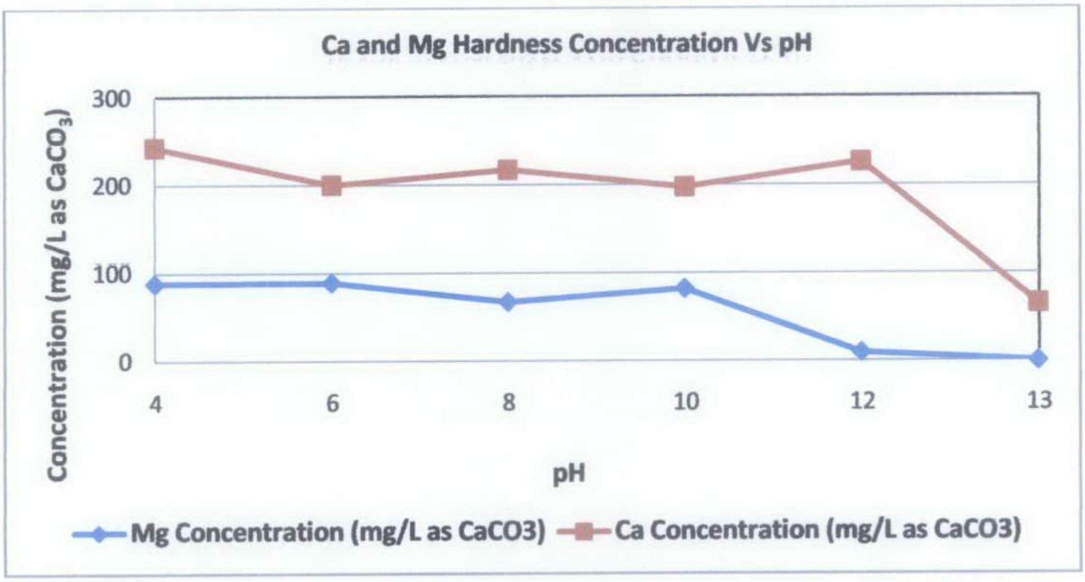


Figure 4.11: Graph of Calcium and Magnesium vs pH

From **Figure 4.11** above, the tests is varying the pH while the dosage of soda-ash is fixed. The dosage is fixed at 5 mL for each jar. From the result, it shows that at pH 13, the calcium is effectively removed. At the same time, the removal of magnesium also shows the better result where the magnesium is totally removed.

As conclusion, the optimum pH to precipitate magnesium and calcium is 12 and 13 respectively. In this result also shows that the calcium concentration has been removed at 73% at the pH 13 while the magnesium concentration has been removed at 90% at pH 12.

After finding the optimum pH to remove calcium and magnesium, the optimum dosage also should be determined. For that purpose, the next step is varying the dosage at fixed pH.

4.6.3 Determining the optimum dosage to remove calcium and magnesium at fixed pH and various dosages of soda-ash (Na_2CO_3)

In order to find the optimum dosage in removing calcium and magnesium, the dosage of soda-ash is vary from 3 to 8 mL at pH 13. The result is shown in **Figure 4.12** below.

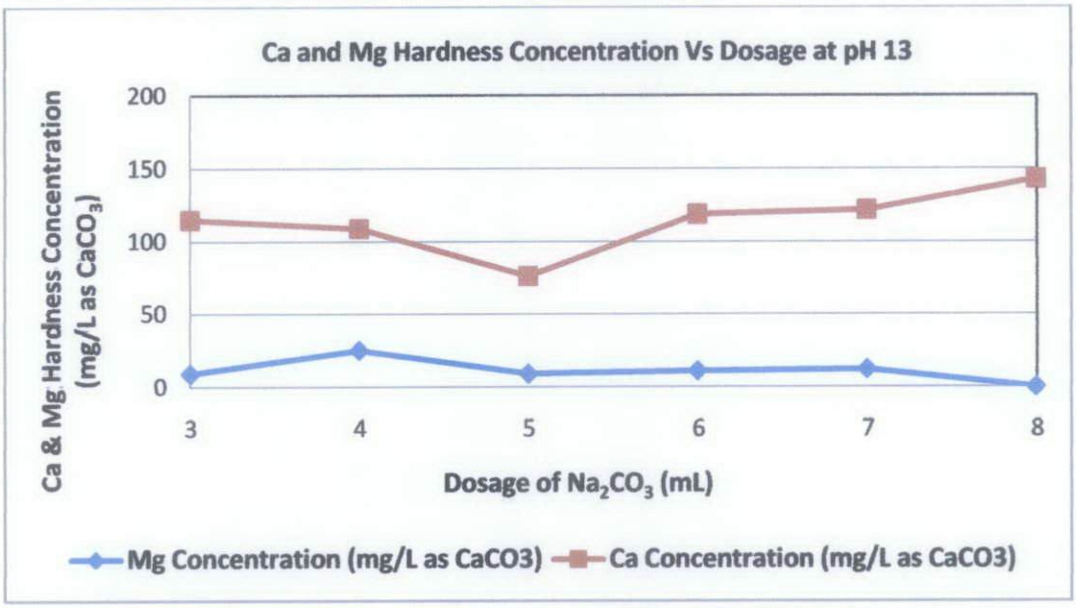


Figure 4.12: Graph of Calcium and Magnesium Hardness Concentration vs Dosage of Na_2CO_3 at pH 13

From the result obtained in the **Figure 4.12**, the optimum dosage to remove calcium and magnesium is 5 mL where is the removal of calcium concentration is 69% and removal of magnesium concentration is about 90%.

CONCLUSION

From the obtained result, calcium is effectively removed using the GWTPS but not for magnesium. For instance, GWTPS managed to remove calcium about 96% at 5000 mg/L dosage at pH 9. Therefore, the total hardness is still in high condition, classified as very hard water after the removal process. By comparing the result with the jar test, the calcium can be removed at pH 13 while magnesium can be removed at pH 12 with the optimum dosage of 5 mL of Na_2CO_3 (5000 mg/L concentration). As conclusion, the GWTPS is not an effective and feasible method to remove hardness.

RECOMMENDATION

For future works, the research can be expanded by varying more dosages and time. For that purpose, more time is needed to accomplish the desired objectives. The method of hardness removal can also be expanded to the other materials such as Microwave Incinerated Rice Husk Ash (MIRHA) and other activated carbon materials.

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APPENDIX

Appendix 1: pH measurement

READING	**pH VALUE**
First reading	**7.65**
Second reading	**7.64**
Third reading	**7.65**

Table 1: Result of the pH experiment

Appendix 2: Nitrogen, Ammonia Tests using Nessler Method

Solution: 10ml (water sample + Nessler Reagent + Mineral Stabilizer + Polyvinyl Alcohol Dispersing Agent)

Sample	**Reading**	**Value (mg/L NH_3-N)**	**Average Value (mg/L NH_3-N)**
Sample 1	1	-0.45	**-0.49**
	2	-0.51	
	3	-0.52	
Sample 2	1	-0.59	**-0.60**
	2	-0.6	
	3	-0.62	
Sample 3	1	-0.75	**-0.75**
	2	-0.75	
	3	-0.75	

Table 2: Result of Nitrogen, Ammonia Test

Appendix 3: Nitrate Tests using Cadmium Reduction Method

Solution: 10ml (water sample + NitraVer 5 Nitrate Reagent Powder Pillow)

Sample	**Reading**	**Value (mg/L NO_3^--N)**	**Average Value (mg/L NH_3-N)**
Sample 1	1	1.0	**1.0**
	2	1.0	
	3	1.0	
	1	0.8	

Sample 2	2	0.8	**0.8**
	3	0.8	
Sample 3	1	1.3	**1.2**
	2	1.1	
	3	1.2	

Table 3: Result of Nitrate Tests

Appendix 4: Total Suspended Solids (TSS)

Sample volume: 400ml

Sample	Sample size (ml)	Weight of pan + filter paper before drying (mg)	Weight of pan + filter paper after drying (mg)	TSS (mg/L)	Average TSS (mg/L)
1	400	1277.9	1281.1	8.0	**8.5**
			1281.4	8.75	
			1281.4	8.75	
2	400	1347.2	1349.8	6.5	**7.67**
			1350.4	8	
			1350.6	8.5	
3	400	1347.9	1351.0	7.75	**8.67**
			1351.3	8.5	
			1351.8	9.75	

Table 4: Result of Total Suspended Solid (TSS)

Appendix 5: Alkalinity Tests

	Initial reading, mL	Final reading, mL	Total, mL
Phenolphthalein alkalinity	82.0	83.4	1.4
Total alkalinity	83.4	96.4	13.0

Table 5: Result of Alkalinity

Calculation:

Phenolphthalein alkalinity =

((mL H_2SO_4 titrant used) x Normality of H_2SO_4 x 50,000)/ mL sample

Total alkalinity =

((total mL H_2SO_4 titrant used) x Normality of H_2SO_4 x 50,000)/ mL sample

Appendix 6: Sulfate Test using SulfaVer 4 Method

Sample	Reading	Value (mg/L SO_4^{2-})	Average Value (mg/L SO_4^{2-})
Sample 1	1	24.0	**24.0**
	2	24.0	
	3	24.0	
Sample 2	1	23.0	**23.0**
	2	23.0	
	3	23.0	
Sample 3	1	26.0	**26.0**
	2	26.0	
	3	26.0	

Table 6: Result of Sulfate Tests

Appendix 7: Removal of hardness using Ground Water Treatment Plant Sludge (GWTPS)

	Dosage								
time (min)	**500 mg/l**			**1000 mg/l**			**5000 mg/l**		
	1	2	3	1	2	3	1	2	3
0	-	-	-	-	-	-	-	-	-
30	121	277	162	129	167	202	-	200	-
60	126	214	183	119	219	187	281	351	344
120	111	143	207	151	172	239	288	301	312
180	162	117	179	284	242	277	293	299	308
240	153	112	156	279	227	219	273	292	258
300	192	232	271	209	268	217	300	294	292

Table 7: Result of magnesium concentration after experiment

	Dosage								
time (min)	500 mg/l			1000 mg/l			5000 mg/l		
	1	2	3	1	2	3	1	2	3
0	-	-	-	-	-	-	-	-	-
30	574	-	-	300	-	-	-	120	-
60	500	-	-	460	240	320	119	22	44
120	480	425	293	391	326	219	35	14	11
180	222	348	226	35	94	63	18	-2.68	11
240	256	349	263	39	113	89	24	12	6
300	177	116	54	135	32	117	15	11	29

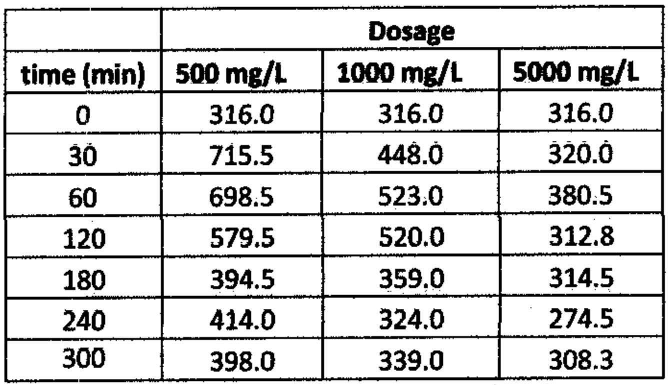
Table 8: Result of calcium concentration after experiment

	Dosage		
time (min)	500 mg/L	1000 mg/L	5000 mg/L
0	82.0	82.0	82.0
30	141.5	148.0	200.0
60	198.5	203.0	347.5
120	127.0	161.5	300.3
180	170.5	280.5	300.0
240	154.5	223.0	265.5
300	251.5	213.0	295.3

Table 9: Average value of magnesium concentration

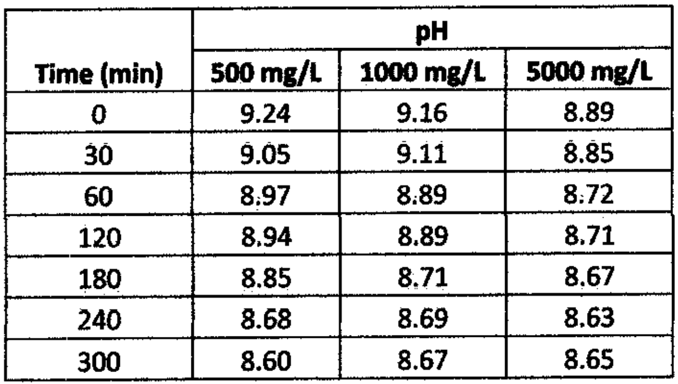
	Dosage		
time (min)	500 mg/L	1000 mg/L	5000 mg/L
0	234.0	234.0	234.0
30	574.0	300.0	120.0
60	500.0	320.0	33.0
120	452.5	358.5	12.5
180	224.0	78.5	14.5
240	259.5	101.0	9.0
300	146.5	126.0	13.0

Table 10: Average value of calcium concentration



	Dosage		
time (min)	500 mg/L	1000 mg/L	5000 mg/L
0	316.0	316.0	316.0
30	715.5	448.0	320.0
60	698.5	523.0	380.5
120	579.5	520.0	312.8
180	394.5	359.0	314.5
240	414.0	324.0	274.5
300	398.0	339.0	308.3

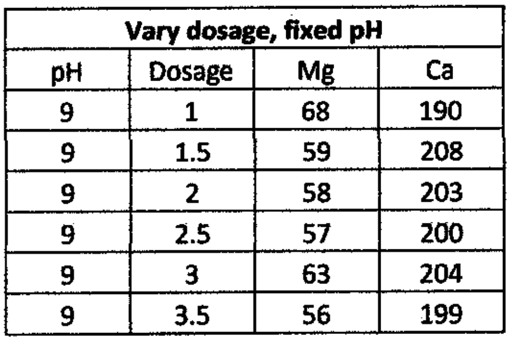
Table 11: Total hardness value



Time (min)	pH		
	500 mg/L	1000 mg/L	5000 mg/L
0	9.24	9.16	8.89
30	9.05	9.11	8.85
60	8.97	8.89	8.72
120	8.94	8.89	8.71
180	8.85	8.71	8.67
240	8.68	8.69	8.63
300	8.60	8.67	8.65

Table 12: pH value of the sample of the dosage

Appendix 8: Chemical precipitation Result (Jar Test)



Vary dosage, fixed pH			
pH	Dosage	Mg	Ca
9	1	68	190
9	1.5	59	208
9	2	58	203
9	2.5	57	200
9	3	63	204
9	3.5	56	199

Table 13: Result of magnesium and calcium concentration for determining the optimum pH to remove magnesium

Vary dosage, fixed pH			
pH	Dosage	Mg	Ca
12	1	6	205
12	1.5	9	212
12	2	8	210
12	2.5	11	207
12	3	16	203
12	3.5	12	204

Table 14: Result of magnesium and calcium concentration for determining the optimum pH to remove magnesium

Vary pH, fixed dosage			
pH	Dosage (mL)	Mg	Ca
4	5	88	243
6	5	89	200
8	5	67	217
10	5	82	197
12	5	9	226
13	5	0	65

Table 15: Result of magnesium and calcium concentration for determining the optimum pH to remove calcium

Fixed pH, vary dosage			
Ph	Dosage (mL)	Mg	Ca
13	3	9	115
13	4	25	109
13	5	9	76
13	6	11	119
13	7	12	122
13	8	0	143

Table 16: Result of magnesium and calcium concentration for determining the optimum dosage to remove calcium and magnesium

	Dosage	
	1000 mg/L	500 mg/L
Mean	404.1428571	502.28571
Variance	8324.47619	25818.738
Observations	7	7
Hypothesized Mean Difference	0	
df	10	
t Stat	-1.405256217	
P(T<=t) one-tail	0.095121032	
t Critical one-tail	1.812461102	
P(T<=t) two-tail	0.190242063	
t Critical two-tail	2.228138842	

Table 17: t-test of comparison between 2 GWTPS dosages (1000 and 500 mg/L)

	Dosage	
	1000 mg/L	5000 mg/L
Mean	404.1428571	318.09524
Variance	8324.47619	989.80423
Observations	7	7
Hypothesized Mean Difference	0	
df	7	
t Stat	2.358920059	
P(T<=t) one-tail	0.025210667	
t Critical one-tail	1.894578604	
P(T<=t) two-tail	0.050421334	
t Critical two-tail	2.364624251	

Table 18: t-test of comparison between 2 GWTPS dosages (1000 and 5000 mg/L)