Boron Removal from Produced Water by Chemical Precipitation

by

Nasuha Mohd Zalaludin

19001781

Dissertation submitted in partial

fulfilment of the requirements for

the Bachelor of Engineering (Hons)

Civil

SEPTEMBER 2022

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

Boron Removal from Produced Water by Chemical Precipitation

By

Nasuha Mohd Zalaludin

19001781

A project dissertation submitted to the

Civil Engineering Programme

Universiti Teknologi PETRONAS

in partial fulfilment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

(CIVIL)

Approved by

_____

(Ts Dr Lavania A/P Baloo)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 2022

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.

_____

NASUHA MOHD ZALALUDIN

ABSTRACT

Produced water is the aqueous liquid phase that is simultaneously produced from a producing well along with the oil and/or gas phases during normal production operations. The contaminants in produced water could lead to bad consequences to environment, human health, and aquatic life. Heavy metals found as one of the contaminants in the produced water. Heavy metals are defined as compounds that represent a specific risk to human health due to their potential toxic or carcinogenic effects (Fu and Wang, 2011). The aim of this study is to investigate the boron contaminant removal by conducting chemical precipitation method with the aid of calcium hydroxide, $Ca(OH)_2$ and sodium sulfide (Na_2S). The sample was collected from equalization tank of treatment system at Onshore Gas Terminal. Therefore, the characterization of produced water, determination of optimum pH and dosage of sodium sulfide (Na_2S) and calcium hydroxide, $Ca(OH)_2$ were conducted. The optimum pH and dosage of the precipitant were determined by jar test experiment. The result shows the optimum condition for sodium sulfide was at pH 10 while for calcium hydroxide was at pH 12. The optimum precipitant dosage for sodium sulfide and calcium hydroxide was 2 mL which is 300 mg/L and 200 mg/L respectively. For chemical precipitation using sodium sulfide, boron removal was 36% while by using calcium hydroxide 39% removal was recorded. From these results, it showed that hydroxide was more effective to remove boron compared to sulfide. However, sulfide showed that it can remove heavy metals at wide range of pH.

ACKNOWLEDGEMENT

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1 Background of study

In the oil industry, the phrase "water" is frequently used to refer to water that is drawn from the earth from an oil or natural gas production well, created as a by-product during the extraction of oil and natural gas, or separated from oil or natural gas after the extraction process (Wikipedia, n.d.). Produced water contains suspended solids, dissolved solids, soluble and insoluble oil/organics, and other compounds used in the production process (Abbas, 2020). Produced water is one of the major contributors of wastewater generated in oil and gas industry, with global production estimated at > 70 billion barrels per year in 2009, with the United States alone producing of 21 billion barrels (J.M. Dickhout, 2017).

Heavy metals constantly found in the produced water. According to (Ezerie Henry Ezechi, 2014), the presence of inorganic compound such as boron in produced water makes it harder to use for good purposes. Thus, boron needs to be treated accordingly to the permissible limit set by the regulation. Industrial Effluent Treatment System (IETS) located at Onshore Gas Terminal (OGT) was built in order to comply the regulations established by Department of Environmental (DOE). OGT discharge treated produced water into the sea, thus, Standard B must adhere before disposal. Based on Standard B, Environmental Quality Act 1974, Industrial Effluent Limit 2009 as shown in Table 1, the allowable effluent limit for boron parameters is 4 mg/L.

An appropriate and sufficient treatment much needed to treat the produced water. The boron concentration after being treated at the secondary treatment gave inconsistent result. Occasionally, the boron concentration exceeded the recommended limit by the regulatory. Therefore, this unfavourable condition can cause negative

1

consequences towards the company, community, and environment. Excessive boron concentration in the sea could danger the marine species. Due to this, sustainability cannot be achieved and not comply to the standard.

Table 1.1 shows parameter effluent limits of Standard A and B according to Environmental Quality Act 1974. Standard A and B for boron are 1.00 mg/L and 4.00 mg/L, respectively.

TABLE 1.1: Parameter Effluent Limits of Standard A and B according to EQA 1974

Parameter	Standard		
	Unit	A	B
Temperature	ºC	40	40
pH value	-	6.0-9.0	5.5-9.0
BOD at 20 ºC	mg/L	20	50
COD	mg/L	50	100
Suspended solids	mg/L	50	100
Mercury	mg/L	0.01	0.05
Cadmium	mg/L	0.01	0.02
Chromium, Hexavalent	mg/L	0.05	0.05
Arsenic	mg/L	0.05	0.10
Cyanide	mg/L	0.05	0.10
Lead	mg/L	0.10	0.50
Chromium, Trivalent	mg/L	0.20	1.00
Copper	mg/L	0.20	1.00
Manganese	mg/L	0.20	1.00
Nickel	mg/L	0.20	1.00
Tin	mg/L	0.20	1.00
Zinc	mg/L	2.00	2.00
Boron	mg/L	1.00	4.00
Iron (Fe)	mg/L	1.00	5.00
Phenol	mg/L	0.00	1.00
Free Chlorine	mg/L	1.00	2.00
Sulphide	mg/L	0.50	0.50
Oil and Grease	mg/L	Not Detectable	10

Source: Schedule Standard of Environmental Quality Act 1974

1.2 Problem Statement

The current treatment system, IETS (Industrial Effluent Treatment System) at Onshore Gas Terminal (OGT) cannot reduce the boron contaminants in produced water according to Environmental Quality Act (EQA 1974), Industrial Effluent Regulation (IER 2009), Standard B which is below 4 mg/L. Boron is a micronutrient that is necessary for all living species, including plants, animals, and humans. Boron went through biomagnification processes after entering the food chain, and eventually build up in the human body (Alina Pohl, 2020). On other that, high boron exposure can harm the kidney and reproductive system (Jui et al., 2021). It has been proven that excessive boron intake has negative effects on the human baby during pregnancy (Igra et al., 2016). The boron contamination in sea can endanger the aquatic ecosystem. Aquatic species such as zebrafish (Danio rerio), trout (Oncorhynchus mykiss), and frogs (Xenopus laevis) reproductivity can be affected due to exposure to boron (Tanaka and Fujiwara, 2008). In order to guarantee that the goal of sustainability is attained, the high removal efficiency in industrial wastewater treatment was strongly advised. Thus, sodium sulfide and lime has been used as the aid in the chemical precipitation process to treat and reduce the concentration of boron.

1.3 Objectives and Scope of Study

1.3.1 Objectives

The aim of the study is to determine the optimal condition for the chemical precipitation to occur. Thus, this objective must be achieved:

i. To carry out characterization of produced water from Industrial Effluent Treatment System (IETS).

ii. To determine the optimum pH and dosage of sodium sulfide (Na_2S) and calcium hydroxide $Ca(OH)_2$ for boron removal.

1.3.2 Scope of Study

This study will be conducted by laboratory work. There are three elements involve in this scope of study:

i. Sample collection from Onshore Gas Terminal (OGT). The sample were collected from wastewater treatment plant of OGT, Kerteh, Terengganu.

ii. Experimental work: The jar test experiment to determine the optimum pH and optimum dosage of sodium sulfide (Na_2S) and calcium hydroxide $Ca(OH)_2$ for boron removal.

iii. Analysis of result: The obtained results will be discussed to the analyses purposes.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview of Produced Water

Produced water is the common waste of an oilfield. Jerry M. Neff *et al.,* 2011 stated the produced water is typically generated during the extraction of oil and gas from onshore and offshore wells. Produced water is the largest waste stream in oil and gasindustry which is contained higher concentrations of hydrocarbon, heavy metals, andother contaminants. Around 250 million barrels of produced water are generated globally every day, compared to about 80 million barrels of oil (Yusran Hedar, 2018). By the end of 2020, produced water levels are expected to reach approximately 340 billion barrels, up from over 202 billion barrels in 2014. In recent years, around 300 million standard cubic metres (m^3) of waste were discharged to sea in the oceanic area covered by the Oslo-Paris (OSPAR) conventions; of this, about 130 million m^3 were discharged by Norway (OSPAR, 2019). Consequently, water to oil ratio approximately 3:1, implying 70% of water reduction. According to Fakhru'l-Razia etal., 2009 more than 60% of the produced water generated worldwide comes from oilfields.

The nature of the producing/storage formation from which they are extracted, the environmental conditions, and the chemicals employed in processing facilities all have an impact on the properties of generated water. The produced water compositionis quite similar to oil and gas production even though the composition from various sources might vary due to different level of magnitude (Yusran Hedar, 2018). Toxic contaminants in the produced water have a negative impact on the environment and drive up field costs (Yu Liang, 2018). The composition of produced water can be divided into two (2) categories: organic and inorganic substances, which include dissolved and dispersed oils, grease, heavy metals, radionuclides, treatment chemicals,

formation solids, salt, dissolved gases, scale products, waxes, microorganisms, and dissolved oxygen (A Fakhru'l-Razi, 2009).

2.2 Overview of Boron Compound

Boron is non-metal compound in Group 12 of the periodic table. According to Ezechi et al., (2012), boron is major contributor in the chemical contaminant in oil produced water. As a vital component of the oil and gas industry for more than a century, borax has a high concentration in produced water. Reproduction of living organisms can be interrupted by the presence of boron contaminants (Dydo, P. et al., 2005). Long term ingestion of boron-enriched water can cause dysfunction of the human and animal systems, changes to blood composition, handicapped children physical and mental, and the increase the likelihood of having a low birth (Bryjak, M et al., 2008). The negative consequences depend on dose consumed, duration and frequency of exposure.

Boron usually appeared as boric acid (H_3BO_3) and borax, ($Na_2B_4O_7.10H_2O$) in nature (Ezerie et al., 2012). It primarily exists in aquatic systems as dissolved boric acid and borate ions (Bryjak et al., 2008). Borax is one of boron compound. In the oil and gas sector, borax, also known as sodium borate, sodium borate decahydrate, or sodium tetraborate decahydrate, is widely utilised. It is usually in the form of a powder or granular substance that dissolves in water. Borates are multipurpose additives used in the exploration, drilling, and production of oil and gas (David M.Schubert et al., 2015). The usage of borates makes the process easier for the water to carry crude for the oil production. In the extraction process, the hydrocarbon is breaking down into smaller which is more easily retrievable molecules. Furthermore, borate also used in oilfield applications such as neutron absorption, lubricant effects, corrosion inhibition, cement set retarding, etc. As summary, borates can be used in many aspects in oil and gas industry.

2.2.1 Impacts of Boron

Boron consuming can affect the organ system of human body. The stomach, intestines, liver, kidney, brain, and other organs can be damaged by large quantities of boron (approximately 30 g of boric acid) consumed quickly; in the worst circumstances, death may result (M Hardi et al., 2019). Dissolved boron in water may be hazardous to aquatic organisms, with fish being more sensitive to chronic boron exposure than aquatic invertebrates (Topala A et al., 2016). Acute effects on fish have been reported in the range of 10-20 mg/L in the most sensitive tests (Gulsoy N et al., 2015).

Table 2.1 shows the effects of boric acid.

TABLE 2.1: Effects of boric acid (Bryjak et al., 2008)

Acute health effects	Eye irritation might happen when direct contact with the solution.
Inhalation exposure	Might irritate the respiratory tract
Eye contact	Long term exposure may cause mild irritation
Contact with skin	No effect to skin. Only may be absorbed through damaged skin and cause poisoning
Oral consumption	May cause headache, followed by nausea, vomiting, diarrhea. Kidney damage
Long term health consequences	Reproduction: Inorganic borates showed effects toward reproductive effects in males of animal studies.

2.3 Discharge Limit of Boron Contaminants

Treated industrial produced water must comply with the regulatory requirements before being dispose to the environment. Any facility that handles the discharge or release of industrial effluent or mixed effluent onto or into any soil or into any inland waters must comply with the Industrial Effluent Regulations 2009, which are a part of the Environmental Quality Act of 1974. Additionally, these regulations are used to monitor any spills or accidental releases of industrial or mixed effluent, the prohibition of the release of industrial effluent containing compounds, the restrictions on the release of sludge onto land or into inland water, license fees, penalties, and more.

According to EQA 1974, discharge of industrial effluent can be categorized to two standards which are standard A and standard B. Technically, standard A is used when the point of discharge into the river is upstream while standard B used when the point of discharge is downstream from a water intake points for consumption or water catchment areas. As for the effluent discharge limit of boron compounds, Malaysian standard A has recommended permissible limit of 1 mg/L into any inland water within the catchment area and 4 mg/L for standard B into any other inland water or Malaysian waters. Failure to comply with the regulations will result in fine or imprisonment or both by the Department of Environmental (DOE). According to Regulation 32 of IER 2009, anybody who violates Regulations 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 16, 17, 18, 19, 20, 21, 22, 23, 24, 27, 28, 29 and 30 is guilty of an offence and is subject to a fine of up to 100,000 ringgits, a term of imprisonment of up to five years, or both, as well as a daily fine of up to 1,000 ringgits for each day the offence continues after the initial conviction.

2.4 Treatment System of Boron Removal

Table 2.2 shows the current treatment system for boron removal.

TABLE 2.2: Treatment system to remove boron

Treatment	Advantages	Limitation	References
Ion exchange	Environment friendly; Fast removal efficiency; Efficient removal for low boron contaminated water	Low selectivity and reactivity for heavy metal (Zeolites and organic resins as medium); High costs for regeneration and disposal	Bashir et al., 2019
Electrocoagulation	High removal efficiency; Less waste of sludge; Cost friendly; Less treatment time	Greater energy consumption is used as charge loading increased; The increased charge loading caused greater amount of metal precipitant production.	Ezerie et al., 2014; Xu and Jiang, 2008; Xu et al., 2009
Adsorption	Cost effective; Environment friendly; Efficient for small scale operation	Expensive to manufacture; Limited removal efficiency (require high temperature and specific pH)	Husain et al., 2021; Guan et al., 2016; Lin et al., 2021
Chemical precipitation	Simple and fast method; Cost friendly	Large amount of sludge production; High cost for sludge management	Alina Pohl, 2020

Constructed wetland	Environment friendly; Cost effective; No energy consumption	Highly depend on environmental and operational conditions; Formation of toxic contaminants of microorganisms; Pollutants can affect the growth of plants;	Turker et al., 2014
Coagulation-flocculation	Easy to operate; Applicable for small scale operations; Low cost for maintenance and operational cost;	Need adequate controls; Great formation of sludge; High disposal costs; Need additional chemical for pH adjustments; Need filtration system	Lin et al., 2021

From Table 2.2, each methods have their own limitations. The treatment chosen depends on the quality of produced water and the concentration of contaminant. In this study, the produced water contained various types of heavy metal. Therefore, there is seems potential for the chemical precipitation for the treatment process. Sodium sulfide (Na2S) is a highly effective precipitation that removes heavy metal cations from wastewater with great efficiency. When compared to hydroxide precipitation, sulfide precipitation has advantages such as the ability to recover additional metals and substantially lower end metal concentrations (Hanna et al., 2020). Sulfide precipitation can be accomplished using an HS solution adjusted to pH 8, and the pH of the precipitating solution can be adjusted to around 5.5 (Syifa, 2016). According to Hanna et al., (2020), due to the acid solubility of sulfide precipitates, only a portion of heavy metals can be precipitated at extremely low pH. Thus, the pH adjustment is very crucial to ensure the effectiveness of the treatment process.

Chemical precipitation is the process of turning the dissolved material into solid-state (Saif et al., 2021). Chemical precipitation is a process of pH adjustment mainly used for removal of dissolved metals from aqueous wastes. A solution is pH-adjusted using an acid or base to achieve the lowest solubility of the components to be removed. As pH rises, metal ions become less soluble and precipitate as hydroxide, decreasing the solubility of the metals (M.N Rao, 2017). Hydroxide precipitation and sulphide precipitation are the most common chemical precipitation processes (Pohl, 2020).

Table 2.3 shows the comparison between hydroxide and sulfide reagent for chemical precipitation.

TABLE 2.3: Comparison of hydroxide and sulfide

Reagent type	Advantages	Limitation	References
Hydroxide	Cost-friendly; Well-proven	Changing pH tends to dissolve precipitates; Slimy sludge production; Great volume of sludge; Cadmium cannot be precipitated by lime	Svetlana, 2018
Sulfide	Higher degree of reduction can achieve in shorter time; Lower solubility than hydroxide; May achieve higher degree of removal over wide range of pH	Highly sensitive to dosage; Risk of H_2S gas formation (poisonous, corrosive)	Svetlana, 2018

According to Table 2.3, it shows the advantages and disadvantages of hydroxide and sulfide as the reagent or precipitant in chemical precipitation. According to Svetlana, 2018, by using hydroxide as the reagent in the chemical precipitation process, it can reduce the cost and well proven. As for the limitation of hydroxide, it is very sensitive towards pH which is the changing pH tends to dissolve precipitates. Hydroxide also tend to produce a great volume and sludge and have a slimy texture. Besides that, calcium hydroxide is not applicable in precipitating the cadmium. Sulfides have better advantages than hydroxide. As compared to hydroxide, sulfide has lower solubility than hydroxide. It can achieve higher degree of reduction in shorter time and higher degree of removal over wide range of pH. However, the pH needs to be maintained in alkaline condition in order to prevent H2S gas formation which is poisonous and corrosive. Dosage plays such an important role in chemical precipitation by using sulfide as it is highly sensitive to dosage.

Table 2.4 shows the optimum pH and dosage of sodium sulfide and calcium hydroxide for heavy metal removal.

TABLE 2.4: Optimum pH and dosage of precipitants for heavy metal removal

Precipitant	Heavy metal	Optimum pH	Optimum Dosage	Temp (ºC)	Removal efficiency (%)	Reference
Sodium sulfide, Na_2S	Zn	10.6	735 mg/L		99.89	
	Cu	10.4	557 mg/L		99.89	Chen et al., 2018
	Pb	10	174 mg/L		99.75	
Calcium hydroxide , $Ca(OH)_2$	Zn	11.2	170 mg/L		99.65	
	Cu	11.3, 11.5	292, 350 mg/L		99.99	
	Pb	10.9	89 mg/L		76.14	
Calcium hydroxide , $Ca(OH)_2$	B	12.4 ± 0.1	12 g/L	60 ºC	90.00	C. Irawan et al., 2011
	B	11.2	50 g/L	90 ºC	92.85	Remy et al., 2004
Calcium hydroxide, $Ca(OH)_2$	B	13	$Ca(O/H)_2$/B = 7.4	95 ºC	78.00 in 1h	Tsai and
		13	$Ca(O/H)_2$/B = 7.4	130 ºC	95.00 in 1 h	Lo, 2015
		5.7	$Ca(O/H)_2$/B = 1.9	77 ºC	93.00 in 1 h	Bilen et al,
			*Molar dosage			2018

Based on Table 2.4, sodium sulfide showed better removal than calcium hydroxide. Sodium sulfide was effective in removing heavy metal of zinc, copper, and lead. According to Chen et al., 2018, the optimum pH range in between 10 until 11.5 and the removal efficiency was 99.99 %. The efficiency of boron removal by using calcium hydroxide depends on the temperature. According to Lo, 2015, the higher temperature at 130 ºC showed better removal which is 95 % with the reaction time of 1 hour.

CHAPTER 3

METHODOLOGY

3.1 Introduction

Sample of produced water was used for the characterization including the measurement of biochemical oxygen demand (BOD), chemical oxygen demand (COD), total suspended solid (TSS), ammonia, turbidity and heavy metal concentration which is boron. These measurements were taken before the experiments. Sodium sulfide and lime were used as the precipitant of the chemical precipitation. By doing jar test, the optimum pH and optimum dosage of precipitant were achieved.

3.2 Research Methodology

3.2.1 Sampling Characterization

The produced water sample used for the study was collected from the sampling point one, equalization tank of Onshore Gas Terminal (OGT), Kerteh, Kuala Terengganu. The characterization of produced water needs to conduct to identify and determine the components and properties of water samples. Through this process, the organic and inorganic compounds of water samples can be defined. Parameters for the produced water characterization:

    a) Biochemical oxygen demand, BOD

    b) Chemical oxygen demand, COD

    c) Total suspended solids, TSS

    d) Ammonium, NH_4-N

    e) Turbidity

    f) Boron

3.3 Experimental Design

3.3.1 Influent Characterization Analysis

i) Biochemical oxygen demand (BOD)

BOD is the quantity of oxygen utilized by bacteria to decompose the organic matter contained in the generated water. Bacteria will use organic materials as a food source when in contact. The organic matter will be oxidized and produce end products such as carbon dioxide and water. The BOD bottle with bacteria, nutrients and dissolved oxygen were placed and incubated for 5 days at temperature of 20 °C. The 5 days biochemical oxygen demand is the amount of dissolved oxygen consumed under these conditions. The value of BOD was determined by Equation 1:

$$BOD = \frac{Initial\ DO - Final\ DO}{\left(\dfrac{Volume\ of\ sample}{300}\right)}$$

(1)

ii) Chemical oxygen demand (COD)

COD is defined as the oxygen required for organic and in-organic content in wastewater or produced water to be oxidized. It was measured by using spectrophotometry. The influent sample was diluted to 1:10 factor in a volumetric flask and transfer to the vial. The prepared vial was inserted into the reactor at 150 °C for 120 minutes. Then, the samples were cool to the room temperature. The blank sample was inserted into the spectrophotometry in order to ZERO reading for calibration. After that, the step was repeated as the sample was cleaned and inserted into the spectrophotometry to get COD reading (mg/L). Equation 2 was used to determine COD value.

$$COD = \frac{COD\ reading}{Dilution\ factor}$$

(2)

iii)   Total suspended solid (TSS)

The amount of total solids in a water or wastewater sample that are retained after filtering using a glass fiber filter is known as total suspended solid. The retained residues on the filter paper were dried at 103°C to 105°C to evaporate the moisture. The sample was cooled down before weighed. The weight of sample was obtained from the stated formula below according to APHA 2549 D Method. TSS was determined by using Equation 3.

$$Total\ suspended\ solids, mg/L = (A - B) \times \frac{1000}{C}$$

(3)

Where: A = weight of filter and dish + residue in mg

B = weight of filter and dish in mg

C = volume of sample filtered in mL

iv)   Ammonia, NH₄

Ammonia is one of the inorganic compounds found in the air, soil, and water. It acts as a nutrient in biological wastewater treatment. During gaseous phase, the chemical formula is NH_3 and called as ammonium ion (NH_4^+) when dissolved in water. The compound needs to be removed due to toxicity. Therefore, before dispose the effluent, the ammonia levels need to be monitored to prevent the negative impact to marine life. 25 mL of sample was measured using the measuring cylinder and transferred to the conical flask. 3 drops of minerals stabilizer were added to the sample and followed by 3 drops of polyvinyl alcohol dispersing agent. The flask inverted for few seconds to mixed it. Then, about 1 mL of Nessler reagent was added and swirled slowly to mix. As for blank preparation, same step was repeated by using the distilled water. Dilution might be needed if the spectrophotometer can't read the ammonia.

v)   Turbidity

Turbidity is used to measure the cloudiness or milkiness of the waste or produced water caused by the suspended solids. The quality of water can be evaluated by turbidity. The unit used to measure the turbidity of water or the presence of suspended solids in water is Nephelometric Turbidity Unit (NTU). The higher turbidity indicates the higher concentration of suspended solids in the water. According to Malaysia Water Quality Standards, the turbidity is classified by Class I, IIA/IIB, III, IV and V. The value of turbidity can refer in the table below.

TABLE 3.1: Malaysia Water Quality Standard for Turbidity

Parameter	Unit	Class				
		I	IIA/IIB	III	IV	V
Turbidity	NTU	5	50	50	-	-

vi)   Determination of Boron Concentration

The water sample were analyzed by DR3900 using Carmine Method according to HACH.

The experiment will be conducted as below:

1. 100 mL of graduated cylinder was used to measure 75 mL of concentrated sulfuric acid. The acid was poured into 250 mL conical flask.
2. One BoroVer 3 Reagent Powder Pillow was added into the flask under the fume hood.
3. The conical flask was swirled for 5 minutes to dissolve the powder completely.
4. 0.2 mL of deionized water was added into 16 mm tube for blank preparation.
5. As for sample, 0.2 mL of sample was added into 16 mm tube.
6. 3.5 mL of BoroVer 3 solution from step 3 was added into the prepared sample tube.
7. The tube was closed with the cap and inverted for mixing.
8. 3.5 mL of BoroVer 3 solution from step 3 was added into the blank sample tube.

9. The tube was closed with the cap and inverted for mixing.

10. An instrument timer was started for 30 minute reaction time.

11. After the reaction time ended, the blank sample cell was cleaned and inserted into the cell holder and ZERO button was pushed.

12. The prepared sample cell was cleaned and inserted into the cell holder and READ button was pushed.

13. The results were recorded.

vii)   Preparation of Synthetic Boron Solution 1000 mg/L

5.716 g of boric acid (H3BO3) was weighed, transferred in 1 L volumetric flask, and diluted with deionized water. 20 mL of the 1000 mg/L boron solution was pipetted into 1 L volumetric flask and diluted with deionized water to get 20 mg/L of boron concentration.

3.3.2 Jar Test Experimental Procedure for Boron Removal

• **Optimum pH of Sodium Sulfide for Removal of Boron**

1. 3% of sodium sulfide was prepared. 30 g/L stock of sodium sulfide was dissolved into 1000 mL of distilled water.
2. The jars were filled with 200 mL of 20 mg/L of boron solution.

TABLE 3.2: Variation of pH for Sodium Sulfide

Jar	pH	Sodium Sulfide (mL)	Dosage of Sodium Sulfide (mg/L)
1	4		
2	6		
3	8	2.0	300
4	10		
5	12		
6	14		

3. The pH was varied from 4 – 14 to get the optimum pH.
4. The jars were placed under the paddle of the jar test apparatus and the paddles were lowered.
5. The samples went the process of rapid mix at 100 rpm for 1 min and slow mix at 30 rpm for 20 min.
6. The stirrer was turned off after the slow mixing and settling was allowed for 30 minutes.
7. The boron concentration was measured by using Carmine Method.

- **Optimum dosage of Sodium Sulfide for Removal of Boron**

1. The jars were filled with 200 mL of sample water.
2. By using the optimum pH of 10, the dosage of sodium sulfides were varied.

TABLE 3.3: Variation of Dosage for Sodium Sulfide

Jar	Dosage of Sodium Sulfide (mg/L)	Sodium Sulfide (mL)	Optimum pH
1	100	0.7	
2	150	1.0	
3	200	1.3	10
4	250	1.7	
5	300	2.0	
6	350	2.3	

3. The jars were placed under the paddle of the jar test apparatus and the paddles were lowered.
4. The samples went the process of rapid mix at 100 rpm for 1 min and slow mix at 30 rpm for 20 min.
5. The stirrer was turned off after the slow mixing and settling was allowed for 30 minutes.
6. The boron concentration was measured by using Carmine Method.

- **Optimum pH of Calcium Hydroxide for Removal of Boron**

1. 2% of sodium sulfide was prepared. 20 g/L stock of sodium sulfide was dissolved into 1000 mL of distilled water.
2. The jars were filled with 200 mL of 20 mg/L of boron solution.

TABLE 3.4: Variation of pH for Calcium Hydroxide

Jar	pH	Calcium Hydroxide (mL)	Dosage of Calcium Hydroxide (mg/L)
1	4		
2	6		
3	8	2.0	200
4	10		
5	12		
6	14		

3. The pH was varied from 4 – 14 to get the optimum pH.
4. The jars were placed under the paddle of the jar test apparatus and the paddles were lowered.
5. The samples went the process of rapid mix at 100 rpm for 1 min and slow mix at 30 rpm for 20 min.
6. The stirrer was turned off after the slow mixing and settling was allowed for 30 minutes.
7. The boron concentration was measured by using Carmine Method.

- **Optimum dosage of Calcium Hydroxide for Removal of Boron**

1. The jars were filled with 200 mL of 20 mg/L of boron solution
2. By using the optimum pH of 12, the dosage of calcium hydroxides were varied.

TABLE 3.5: Variation of dosage for Calcium Hydroxide

Jar	Dosage of Calcium Hydroxide (mg/L)	Calcium Hydroxide (mL)	Optimum pH
1	100	1.0	
2	150	1.5	
3	200	2.0	12
4	250	2.5	
5	300	3.0	
6	350	3.5	

3. The jars were placed under the paddle of the jar test apparatus and the paddles were lowered.
4. The samples went the process of rapid mix at 100 rpm for 1 min and slow mix at 30 rpm for 20 min.
5. The stirrer was turned off after the slow mixing and settling was allowed for 30 minutes.
6. The boron concentration was measured by using Carmine Method.

3.4 Project Activities

The project activities during this study are defined as below in Figure 1:

```
┌─────────────────────────────┐
│   Collection of produced    │
│        water samples        │
└─────────────────────────────┘
              ↓
┌─────────────────────────────┐
│     Characterization of     │
│       produced water        │
└─────────────────────────────┘
              ↓
┌─────────────────────────────┐
│  Jar Test Experiment to find│
│   optimum pH and dosage     │
└─────────────────────────────┘
              ↓
┌─────────────────────────────┐
│   Produce more effluent     │
│ using achieved optimum      │
│        parameters           │
└─────────────────────────────┘
              ↓
┌─────────────────────────────┐
│   Analysis and discussion of│
│        the results          │
└─────────────────────────────┘
              ↓
┌─────────────────────────────┐
│   Conclusion and future work│
└─────────────────────────────┘
```
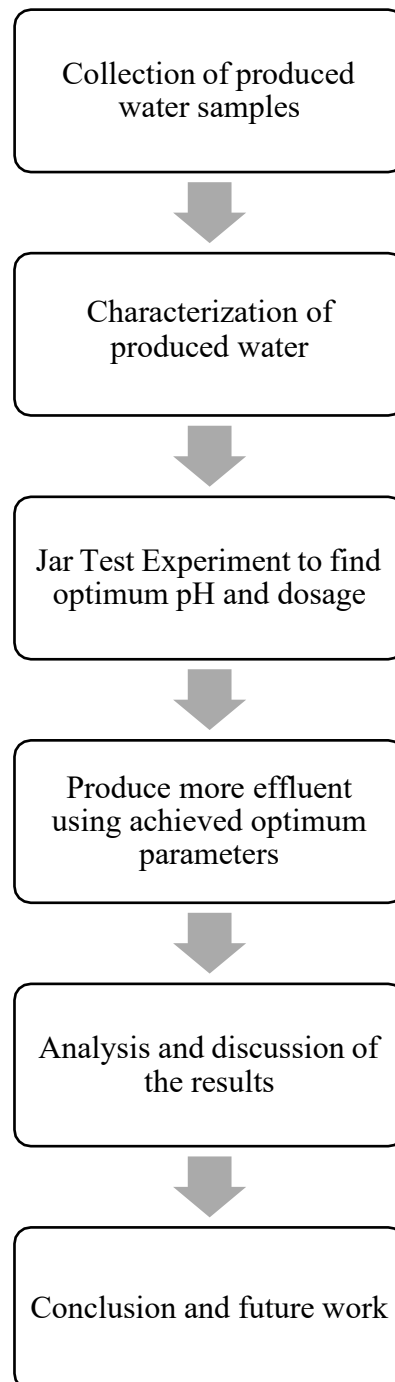
FIGURE 3.1: Project Activities

3.5 Tools

The tool needed in this study are listed as below:

Laboratory apparatus

➢ Apparatus and equipment for experiment are provided in laboratory facilities located at Environmental Laboratory of Civil Engineering Department in Universiti Teknologi PETRONAS. The apparatus is used to define the characteristic of produced water and to determine the optimum parameters for chemical precipitation.

List of equipment to be used:

      i.   Measuring cylinder

     ii.   Beaker

   iii.   Filter paper

   iv.   TSS filter pump

     v.   Pipette

   vi.   BOD bottle

  vii.   Spectrophotometer

 viii.   Refluxing unit

   ix.   Cuvette

     x.   Volumetric flask

   xi.   Vial

  xii.   pH meter

 xiii.   Jar Test Equipment

CHAPTER 4

RESULT & DISCUSSION

4.1 Introduction

The section analyzed the characteristics of produced water and the comparison of boron removal by using sodium sulfide and calcium hydroxide.

4.2 Characterization of Produced Water

The pH value of raw influent water collected from the equalization tank is 8.14.

4.2.1 Biochemical Oxygen Demand (BOD)

According to Standard B, the limit of BOD is 50 mg/L. The obtained result from the experiment were 62.17 mg/L for influent with inhibitor and 61.93 mg/L for influent without inhibitor. This shows the results of BOD exceeded the limitation of 50 mg/L.

TABLE 4.1: Results of Biochemical Oxygen Demand (BOD)

Sample	Bottle ID	Note	Vol. of sample (ml)	Initial DO (mg/L)	Final DO (mg/L)	DOi – DOf (mg/L)	BOD (mg/L)	Avg. BOD
Influent	1	With inhibitor	30	7.88	1.73	6.15	61.5	62.17
	2	With inhibitor	30	7.92	1.68	6.24	62.4	
	3	With inhibitor	30	7.93	1.67	6.26	62.6	
Influent	1	Without inhibitor	30	7.93	1.91	6.02	60.2	61.93
	2	Without	30	7.95	1.76	6.19	61.9	

	inhibitor				
3	Without inhibitor	30	8.02	1.65	6.37	63.7

4.2.2 Chemical Oxygen Demand (COD)

According to Standard B, the limit of COD is 200 mg/L. The result of COD achieved from the produced water was 270 mg/L. It was slightly high, and treatment is needed before being disposed into sea.

TABLE 4.2: Results of Chemical Oxygen Demand (COD)

COD (mg/L)	Average COD (mg/L)
280	
271	270
258	

4.2.3 Turbidity

The obtained turbidity of the produced water was 14.4 NTU and classified as Class I according to DOE Water Quality Index. As for water supply, practically no treatment needed, and it was very sensitive to aquatic species.

TABLE 4.3: Results of Turbidity

Turbidity (NTU)	Average Turbidity (NTU)
13.7	14.4
12.6	
16.9	

4.2.4 Ammonia

According to Standard B, the limit for ammonia was 20 mg/L. The ammonia concentration of influent in equalization tank was 12 mg/L. Thus, the ammonia is within the required standard.

TABLE 4.4: Results of Ammonia

Ammonia (mg/L)	Dilution factor	Ammonia (mg/L)	Average Ammonia (mg/L)
0.24	1:50	12	
0.22	1:50	11	12
0.25	1:50	12.5	

4.2.5 Total Suspended Solid (TSS)

The required total suspended solid according to Standard B limit was 100 mg/L. According to the table below, the total suspended solid is 10 mg/L. It showed that the parameter met the standard set by the regulatory.

TABLE 4.5: Results of Total Suspended Solid (TSS)

Vol. of sample (ml)	Initial weight of foil + filter paper (mg)	Final weight of foil + filter paper after 105 ºC (mg)	TSS (mg/L)	Avg. TSS
50	1421.2	1421.8	12	10
50	1424.1	1424.6	10	
50	1420.4	1420.8	8	

Overall results of the produced water characteristic were met the Standard B. Based on the results of BOD, the values were significantly low due to degradation. The sample supposedly to be tested by 48 hours after collection and need to be store at 4º C after being collected. Due to some conditions, the water sample can't be store at optimum conditions. Therefore, obtained result were not accurate.

4.3 Comparison of Boron Removal by Sodium Sulfide and Calcium Hydroxide

This section describes the results of boron removal by chemical precipitation using sodium sulfide Na2S and calcium hydroxide Ca(OH)2. The efficiency of the removal was determined by obtaining the optimum pH and dosage of the precipitant.

4.3.1 Effect of Optimum pH

The pH value was significantly affected the efficiency of heavy metals removal. pH range between 4, 6, 8, 10, 12 and 14 were chosen to conduct the jar test experiment for choosing the optimum pH for the sulfide precipitation and hydroxide precipitation. Each experiment was conducted at constant dosage of 2 mL (precipitant) and 200 mL of synthetic boron solution.

Based on the line graph for the sulfide precipitation in Figure 4.1, it showed a decrease at pH 4, 6, 8 and 10. There was a slightly increases at pH 12 and 14. The highest boron removal was 34% which at pH 10 while the lowest boron removal was 26% at pH 4. Based on the results of the final boron concentration, there were only slightly changes. As for pH value of 4 until 14, the final boron concentrations were 14.8, 14.2, 13.9, 13.3, 13.5 and 13.7 mg/L. It showed that the efficiency of precipitation by sulfide as the precipitant increases as the pH increased. There was no solubility curve for heavy metal of boron removal using sulfide and hydroxide. According to Chen et al, 2018, sodium sulfide can treat zinc, copper, and lead with removal efficiency of 99.89 %. The optimum pHs were 10.6, 10.4, 10 and 11.2. It was proven that pH of range 10 and above is the most optimum pH for the precipitation to occur.
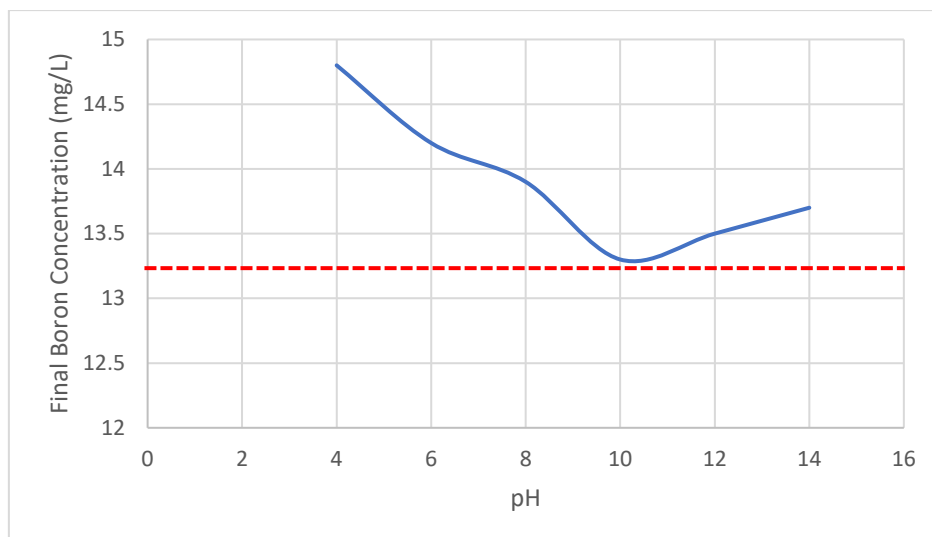
FIGURE 4.1: Result of pH value vs Boron Concentration (mg/L) by
Sodium Sulfide

Figure 4.2 shows the result of pH value vs boron concentration (mg/L). The initial boron concentration was 20 mg/L. For the calcium hydroxide precipitation, the graph showed a slightly decrease at pH 4 and 6. It showed the highest boron removal at pH 8 until 12, but it increases again at pH 14. The lowest boron removal was 1% which at pH 4 while the highest removal was 38% at pH 12. The highest percentage of boron removal was pH 12 which is in alkaline condition. The final boron concentration at the highest removal was 12.5 mg/L. Therefore, pH 12 was selected as the optimum pH.

As stated by C. Irawan et., 2011, the chemical precipitation by calcium hydroxide can treat the boron with removal of 90 % with pH 12 and temperature of 60 ºC. Furthermore, Remy et al., 2004 stated that with pH 11.2, boron can be removed at 92.85 % with temperature of 90 ºC. The removal efficiency increased when the temperature increase.
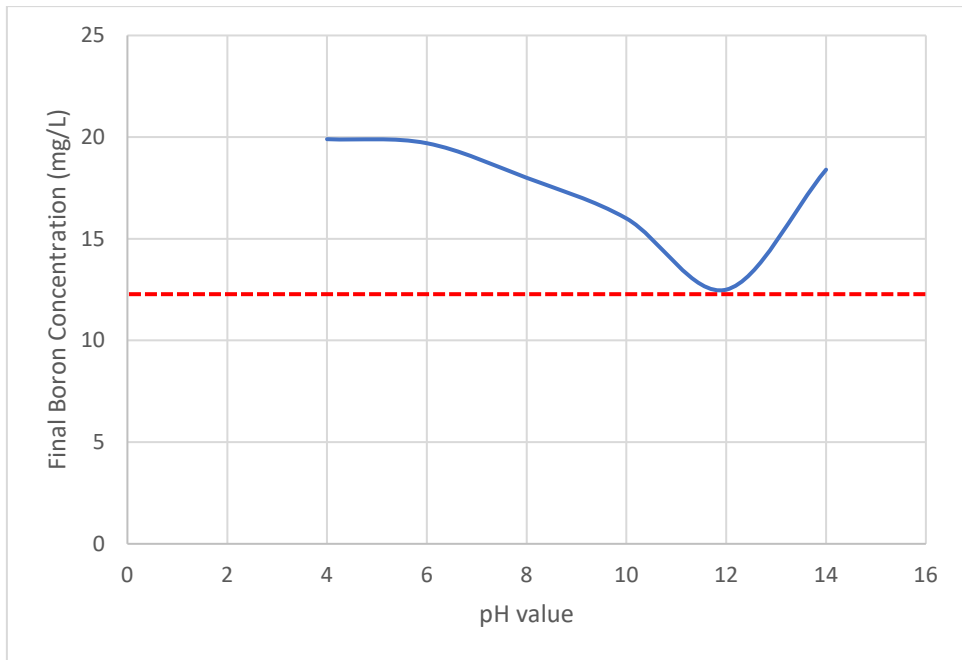
FIGURE 4.2: Result of pH value vs Final Boron Concentration
(mg/L) by Calcium Hydroxide

4.3.2　Effect of Precipitant

The optimum pH by sodium sulfide was pH 10 while for calcium hydroxide was pH 12. In this experiment, the pH was set to the optimum value. Figure 4.3 showed the result of precipitant dosage for sodium sulfide. From the graph, the chemical precipitation using sodium sulfide showed increases at 0.7 mL until 2.3 mL of Na2S with dosage of 100 mg/L, 150 mg/L, 200 mg/L, 250 mg/L, 300 mg/L and 350 mg/L. The highest percentage of boron removal was 36% while the lowest was 31%. Therefore, the optimum precipitant dosage for Na2S was 2.0 mL which is 300 mg/L. The showed that sulfide precipitation can remove heavy metals at broad range of dosage.

Based on Table 2.4, Chen et al., 2018 stated different optimum dosage for different heavy metal. The optimum dosage in treating zinc, copper and lead were 735 mg/L, 557 mg/L and 174 mg/L. Thus, it can be concluded that different heavy metal needs different amount of dosage concentration to treat the contamination.
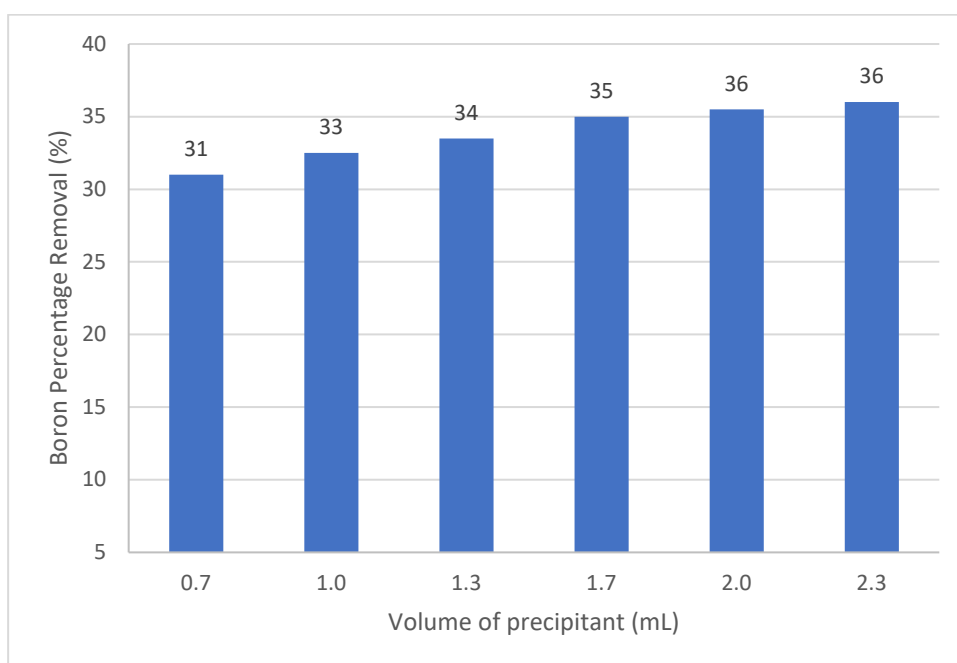

FIGURE 4.3: Volume of precipitant (mL) vs Percentage of Boron removal % for Sodium Sulfide

The result of volume of precipitant (mL) vs percentage of boron removal (%) for calcium hydroxide was shown in Figure 4.4. The bar graph showed a slightly increase of boron removal at 1.0 mL (100 mg/L) and 1.5 mL (150 mg/L) of calcium hydroxide. At 2.5 mL (250 mg/L), 3.0 mL (300 mg/L) and 3.5 mL (350 mg/L) of calcium hydroxide, the result decreased. The highest boron removal was at 2.0 mL (200 mg/L) of $Ca(OH)_2$ which is 39 % while the lowest boron removal was at 1.0 mL (100 mg/L) which is 20.5 %.

According to C.Irawan et al., 2011, with optimum dosage of 12 g/L and 50 g/L, boron can be removed with the removal efficiency of 90 % at 60 ºC and 92.85 % at temperature of 90 ºC. As for the experiment, it was carried out at the room temperature. The highest removal efficiency was 39 % with dosage of 200 mg/L. Lime at room temperature was inefficient at removing the heavy metal. Thus, the removal efficiency at room temperature was low rather than removal at higher temperatures.

Thus, the result showed that the increment of sulfide precipitant dosage can enhance the boron removal. As for the hydroxide precipitant, when the precipitant was exceeding the optimum dosage, the boron removal was decreases. This indicated no reaction occur between the ions. As to conclude, the result showed that the chemical precipitation using sodium sulfide showed a better result for the boron removal compared to calcium hydroxide.
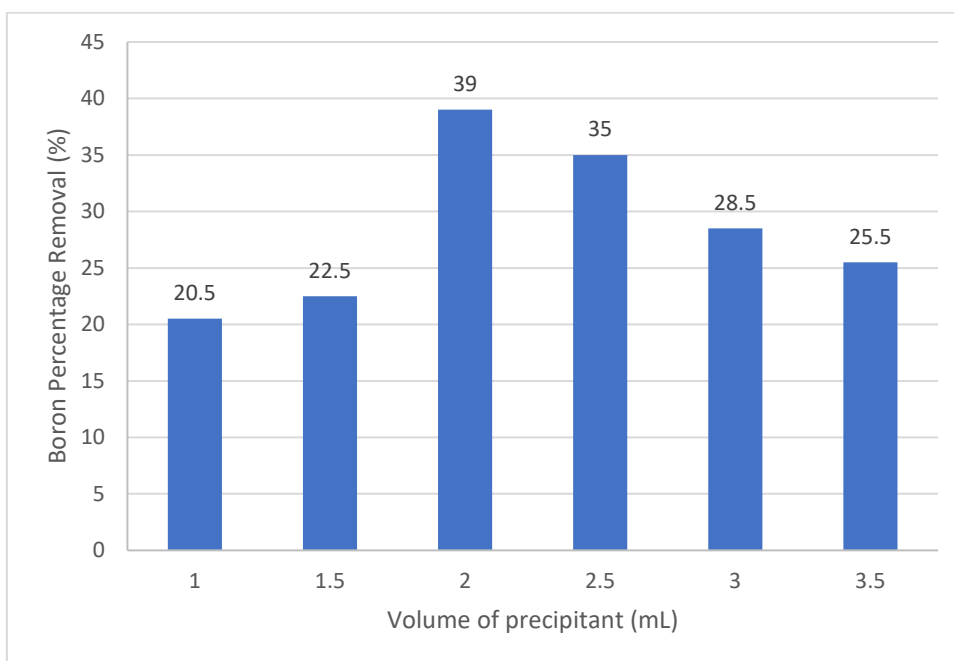
FIGURE 4.4: Volume of precipitant (mL) vs Percentage of Boron
removal % for Calcium Hydroxide

CHAPTER 5

CONCLUSION & RECOMMENDATIONS

5.1 Conclusion

The optimum pH and dosage of precipitant were determined through a few sequences of jar test experiment. The performance of sodium sulfide and calcium hydroxide was compared under those optimum conditions. Sodium sulfide as the precipitant showed better result than the calcium hydroxide. The result shows that for sodium sulfide under optimum conditions of pH 10 and 2.0 mL (200 mg/L), 36% of boron was removed. Sodium sulfide can remove the boron at broad range of precipitant dosage. The calcium hydroxide only can remove the boron at certain dosage. Therefore, sodium sulfide as the precipitant was found to remove heavy metal boron more efficient than calcium hydroxide. In the industry, calcium hydroxide is more preferred than sodium sulfide due to cost. Calcium hydroxide is cheaper than sodium sulfide. However, the final boron concentration failed to meet the Standard B requirement which is 4 mg/L. Sodium sulfide can effectively remove other heavy metals such as zinc, copper, and lead with more than 90 % of removal.

5.2 Recommendations

In future, further analysis of chemical precipitation using another chemical should be done. Since there is no solubility curve for boron, it is recommended to do further analyses on this. Furthermore, another treatment of boron removal such as constructed wetland should be consider. Only several studies on constructed wetland in Malaysia for the industry wastewater. Thus, this can be considered as the stepping stone for trying the new method in order to treat the produced water according to Environmental Quality Act 1974.

REFERENCES

Abass A. Olajire, Recent advances on the treatment technology of oil and gas produced water for sustainable energy industry-mechanistic aspects and process chemistry perspectives, *Chemical Engineering Journal Advances*, Volume 4, 2020, 100049, ISSN 2666-8211, https://doi.org/10.1016/j.ceja.2020.100049.

A.M. Igra, F. Harari, Y. Lu, E. Casimiro, M. Vahter. Boron exposure through drinking water during pregnancy and birth size. *Environ. Int*., 95 (2016), pp.54-60.

Bashir, A., Malik, L.A., Ahad, S. et al. Removal of heavy metal ions from aqueous system by ion-exchange and biosorption methods. *Environ Chem Lett 17*, 729–754 (2019). https://doi.org/10.1007/s10311-018-00828-y

Fu, F., & Wang, Q. (2011). Removal of heavy metal ions from wastewaters: a review. *Journal of Environmental Management, 92*, 407 – 418. https://doi.org/10.1016/j.jenvman.2010.11.011.

Guan, Z., Lv, J., Bai, P., & Guo, X. (2016). Boron removal from aqueous solutions by adsorption—A review. Desalination, 383, 29-37.

Gülsoy N, Yavas C and Mutlu O 2015 Excli J. 14 890–899.

Hardi, M., Siregar, Y. I., Anita, S., & Ilza, M. (2019). *Determination of heavy metals concentration in produced water of oil field exploration in siak regency*. In Journal of Physics: Conference Series (Vol. 1156, No. 1, p. 012009). IOP Publishing.

Henry Ezechi, Ezerie & Isa, Mohamed Hasnain & Mohamed Kutty, Shamsul Rahman. (2012). *Removal of Boron from Produced Water by Electrocoagulation*.

Hussain, A., Madan, S., & Madan, R. (2021). Removal of Heavy Metals from Wastewater by Adsorption. In M. K. Nazal, & H. Zhao (Eds.), Heavy Metals Their Environmental Impacts and Mitigation. IntechOpen.

https://doi.org/10.5772/intechopen.95841

Irawan, C., Kuo, Y. L., & Liu, J. C. (2011). Treatment of boron-containing optoelectronic wastewater by precipitation process. *Desalination*, *280*(1-3),146-151.

Jui-Yen Lin, Nicolaus N.N. Mahasti, Yao-Hui Huang. Recent advances in adsorption and coagulation for boron removal from wastewater: A comprehensive review, *Journal of Hazardous Materials*:Volume 407, 2021, 124401, ISSN 0304-3894, https://doi.org/10.1016/j.jhazmat.2020.124401

Li, P., Liu, C., Zhang, L., Zheng, S., & Zhang, Y. (2017). *Enhanced boron adsorption onto synthesized MgO nanosheets by ultrasonic method*. Ultrasonics sonochemistry, 34, 938–946. https://doi.org/10.1016/j.ultsonch.2016.07.029

M. Tanaka, T. Fujiwara. *Physiological roles and transport mechanisms of boron: perspectives from plants*. Pflug. Arch. - Eur. J. Physiol., 456 (2008), pp. 671- 677.

Najid, N., Kouzbour, S., Ruiz-García, A., Fellaou, S., Gourich, B., & Stiriba, Y. (2021). Comparison analysis of different technologies for the removal of boronfrom seawater: A review. *Journal of environmental chemical engineering*, 9, 105133.

Nasiri, Masoud & Jafari, Iman. (2016). *Produced Water from Oil-Gas Plants: A Short Review on Challenges and Opportunities*. Periodica Polytechnical Chemical Engineering. 61. 10.3311/PPch.8786.

Pohl, A. *Removal of Heavy Metal Ions from Water and Wastewaters by Sulfur-Containing Precipitation Agents*. Water Air Soil Pollut 231, 503 (2020). https://doi.org/10.1007/s11270-020-04863-w

Prokkola, Hanna & Nurmesniemi, Emma-Tuulia & Lassi, Ulla. (2020). *Removal of Metals by Sulphide Precipitation Using Na$_2$S and HS − -Solution*. ChemEngineering. 4. 51. 10.3390/chemengineering4030051.

Remy, P., Muhr, H., Plasari, E. and Ouerdiane, I. (2005), *Removal of boron from wastewater by precipitation of a sparingly soluble salt*. Environ. Prog., 24: 105-110. https://doi.org/10.1002/ep.10058

Svetlana B. Zueva, 9 - Current legislation and methods of treatment of wastewater coming from waste electrical and electronic equipment processing, Editor(s): Francesco Vegliò, Ionela Birloaga, In Woodhead Publishing Series in Electronic and Optical Materials, Waste Electrical and Electronic Equipment Recycling, Woodhead Publishing, 2018, Pages 213-240, ISBN 9780081020579, https://doi.org/10.1016/B978-0-08-102057-9.00009-3.

H.C. Tsai, S.L. Lo, *Boron removal and recovery from concentrated wastewater using a microwave hydrothermal method*, J. Hazard. Mater. 186 (2011) 1431–1437.

Türker, O.C., Vymazal, J., & Türe, C. (2014). Constructed wetlands for boron removal: A review. Ecological Engineering, 64, 350-359.

Topala A, Orucb E, Altunb S, Ceyhunc S B and Atamanalpc M 2016 J. Appl. Anim Res. 44(1), 297–302.

Veil J 2015 US Produced Water Volumes and Management Practices in 2012 report prepared for the Groundwater Protection Council.

Zukhri, Syifaa (2016) *Boron Removal from Model Wastewater Using Adsorption Process*: A Case Study. Universiti Teknologi PETRONAS.
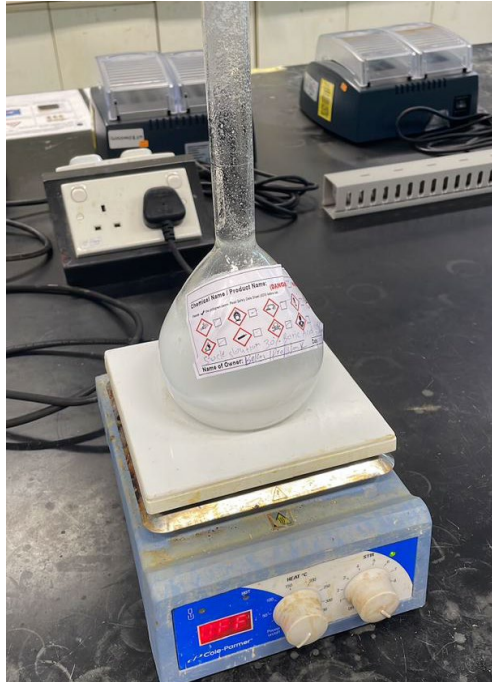
APPENDIXES

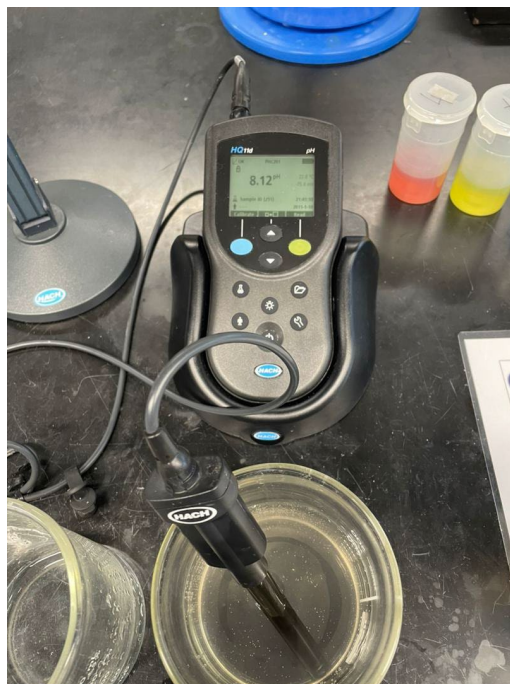FIGURE 6.1: Preparation of Boron Solution

FIGURE 6.2: Taking pH of produced water

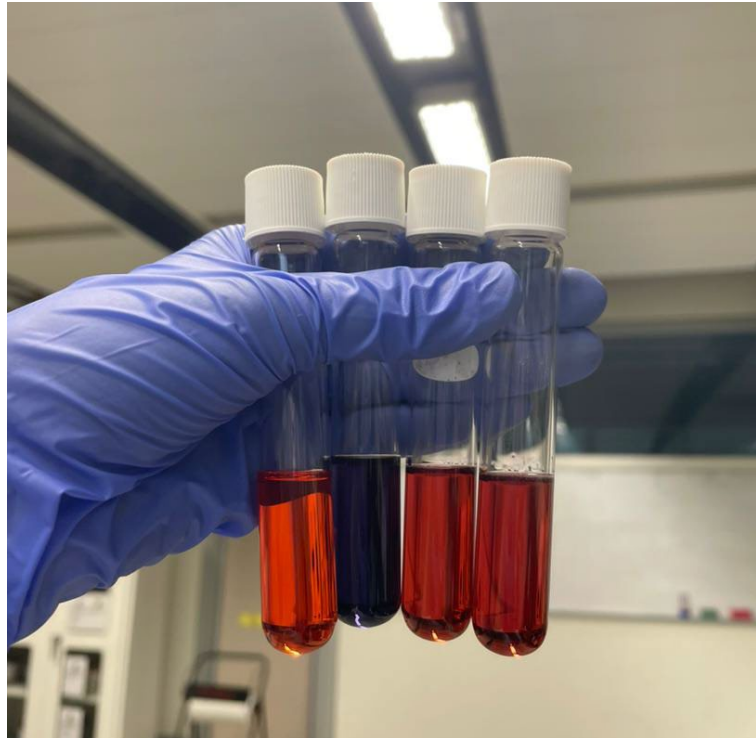FIGURE 6.3: Low Concentration Boron Solution

FIGURE 6.4: Set of Jar Test Experiment

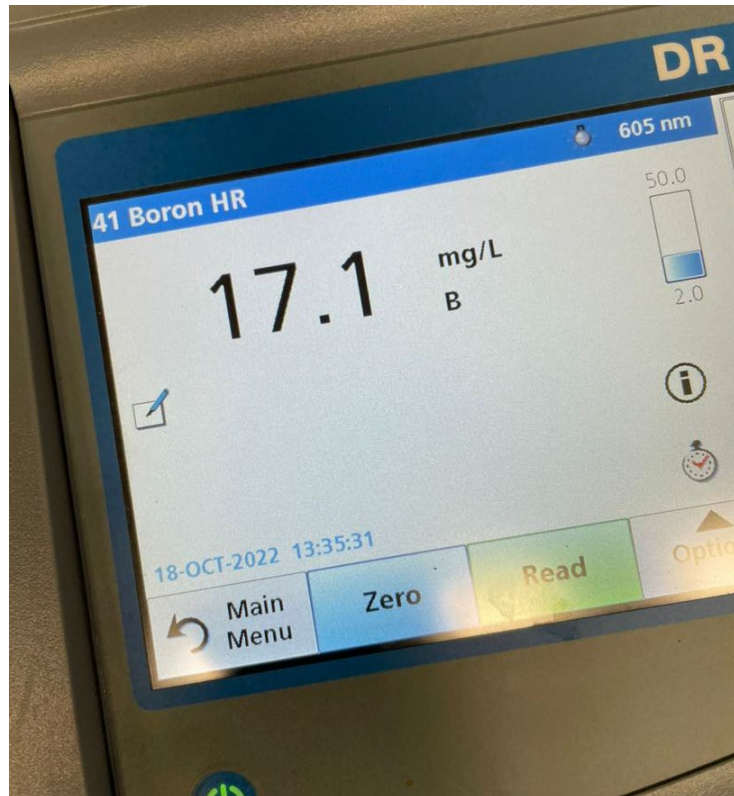FIGURE 6.5: High Concentration Boron Solution

FIGURE 6.6: Reading of boron concentration (mg/L)

FIGURE 6.7: Close up of jar test using calcium hydroxide

FIGURE 6.8: Set up of Jar Test Experiment

FIGURE 6.9: Set of boron solution after jar test experiment

FIGURE 6.10: Jar Test Experiment by Calcium Hydroxide

TABLE 6.1: Dosage of Calcium Hydroxide

Dosage of calcium hydroxide (mg/L)	in 200 mL
100	1.0
150	1.5
200	2.0
250	2.5
300	3.0
350	3.5
400	4.0

Example of calculation:

Equation 4 was used to determine the dosage of calcium hydroxide.

2 % = 20 000 mg/L

$$C1V1 = C2V2$$

(4)

$$20\,000\,\frac{mg}{L} \times ?\,mL = 100\,\frac{mg}{L} \times 200\,mL$$

$$?\,mL = 100\,\frac{mg}{L} \times 200\,mL \div 20\,000\,\frac{mg}{L}$$

$$?\,mL = 1.0\,mL$$

TABLE 6.2: Dosage of Sodium Sulfide

Dosage of sodium sulfide (mg/L)	in 200 mL
100	0.7
150	1.0
200	1.3
250	1.7
300	2.0
350	2.3
400	2.7

Example of calculation:

Equation 5 was used to determine the dosage of sodium sulfide.

3 % = 30 000 mg/L

$$C1V1 = C2V2$$

(5)

$$30\,000\frac{mg}{L} \times ?\,mL = 100\frac{mg}{L} \times 200\,mL$$

$$?\,mL = 100\frac{mg}{L} \times 200\,mL \div 30\,000\frac{mg}{L}$$

$$?\,mL = 0.7\,mL$$

TABLE 6.3: Data for optimum pH of Calcium Hydroxide

Jar	Vol of sample (mL)	Type of precipitant	Volume of precipitant (mL)	pH raw	pH adjustment	Initial Boron Conc (mg/L)	Final Boron Conc (mg/L)	Boron Percentage Removal (%)
1	200				4		19.9	1
2	200				6		19.7	2
3	200	CALCIUM HYDROXIDE	2	9	8	20	18.0	10
4	200				10		16.0	20
5	200				12		12.5	38
6	200				14		18.4	8

TABLE 6.4: Data for optimum dosage of Calcium Hydroxide

Jar	Vol of sample (mL)	Type of precipitant	Volume of precipitant (mL)	pH raw	pH adjustment	Initial Boron Conc (mg/L)	Final Boron Conc (mg/L)	Boron Percentage Removal (%)
1	200		1				15.9	20.5
2	200		1.5				15.5	22.5
3	200	CALCIUM HYDROXIDE	2	9	12	20	12.2	39
4	200		2.5				13.0	35
5	200		3				14.3	28.5
6	200		3.5				14.9	25.5

TABLE 6.5: Data for optimum pH of Sodium Sulfide

Jar	Vol of sample (mL)	Type of precipitant	Volume of precipitant (mL)	pH raw	pH adjustment	Initial Boron Conc (mg/L)	Final Boron Conc (mg/L)	Boron Percentage Removal (%)
1	200			9	4		14.8	26
2	200			9	6		14.2	29
3	200	SODIUM SULFIDE	2	9	8	20	13.9	31
4	200			9	10		13.3	34
5	200			9	12		13.5	33
6	200			9	14		13.7	32

TABLE 6.6: Data for optimum dosage of Sodium Sulfide

Jar	Vol of sample (mL)	Type of precipitant	Volume of precipitant (mL)	pH raw	pH adjustment	Initial Boron Conc (mg/L)	Final Boron Conc (mg/L)	Boron Percentage Removal (%)
1	200		0.7				13.8	31
2	200		1.0				13.5	33
3	200	SODIUM SULFIDE	1.3	9	10	20	13.3	34
4	200		1.7				13.0	35
5	200		2.0				12.9	36
6	200		2.3				12.8	36