

**REPORT
FIELD INTERNSHIP**

**PHARMACEUTICAL WASTEWATER TREATMENT
USING ELECTROCOAGULATION AND
MICROBIOLOGY METHOD IN PT. ETERCON PHARMA**



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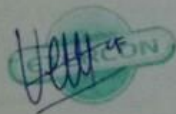
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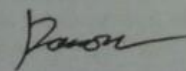
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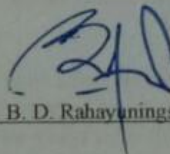


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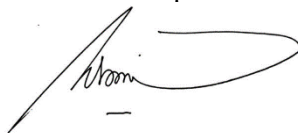
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PREFACE

Praise the author, pray for the presence of Allah SWT, who has bestowed His grace so that the author was able to complete the preparation of this Field Work Practice Report according to the planned time. This report was prepared to complete the requirements for completing the Field Work Practice (PKL) course at the Department of Chemistry, Faculty of Science and Mathematics, Diponegoro University.

This report is the result of practical fieldwork activities that have been carried out at PT. Etercon Pharma on January 4, 2021 to February 4, 2021.

This report can be completed without the help of all parties, so on this occasion the authors would like to thank:

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The author realizes that this work is still lacking and far from perfect. Therefore, all constructive criticism and suggestions from readers will always be expected and accepted as an evaluation for the author towards the direction of improvement for the preparation of other scientific works.

Semarang, 25 May 2021
Author

Safira Aphrodite Ramoza

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CHAPTER I.

INTRODUCTION

1.1. Background

Pharmaceutical drugs play an important role in increasing life expectancy and quality of life for people. Along with the times, the need for medicine is increasing and with the COVID 19 pandemic, the demand for drugs has increased rapidly. This has led to an increase in drug production in the pharmaceutical industry. The increase in the amount of production is directly proportional to the amount of liquid waste produced.

Pharmaceutical industry wastewater is increasing globally as one of the major health problems today, not only for aquatic animals but also for humans and the environment. Highly contaminated wastewater that results from various pharmaceutical manufacturing processes contains a wide variety of toxic compounds that have a negative impact (Verlicchi et al., 2012). Toxic compounds that are frequently detected in different bodies of water and drinking water have non-biodegradable characters that can survive and remain contaminated which leads to potential health and environmental risks (Kanakaraju et al., 2018). It can also pose a potential hazard to aquatic ecosystems and affect animal and human life in the long term so it must be processed efficiently (Klavarioti et al., 2009).

Pharmaceutical waste has a very varied composition of pollutants, even containing very dangerous elements. Some of the hazardous elements contained are heavy metals such as lead (Pb), iron (Fe), chromium (Cr), and mercury (Hg). In addition there are also dissolved solid 2 (TDS), ammonia (NH₃), nitrite (NO₂) and the effect of acidity (pH). The waste water produced from the pharmaceutical industry has a severe color, strong odor, high COD and low BOD (Farhadi et al., 2012).

Low BOD concentrations and high COD concentrations in pharmaceutical wastewater cause problems when treated using biological processes, because the chemical components contained in wastewater can limit the activity of microorganisms (Guieysse and Norvill, 2014). Therefore, chemical and physical

processes other than biological are needed to treat this kind of wastewater. Between physical and chemical processes that are efficient (Brillas and Sirés, 2018) which is commonly used in pharmaceutical industrial wastewater treatment is an electrochemical treatment process that can be applied effectively to remove persistent contaminants (Chen, 2004) from wastewater.

PT. Etercon Pharma uses a batch system with underflow, overflow, and oxidation-reduction methods using H_2O_2 and Ferro Sulfate. However, because the pollutants found in the batch were unstable, the TSS results tended to be unstable. Therefore, the methods that have been applied need to be accompanied by other methods. One of them is by using the electrocoagulation and microbiological methods.

Electrocoagulation is the process of clumping and depositing fine particles in water using electrical energy. Electrocoagulation has a high efficiency in removing contaminants and the operating costs are quite low or economical. In addition, electrocoagulation also helps in removing heavy metals including B3 waste in water. This process is based on the principle where the response of water containing contaminants to electric fields through redox reactions (reduction and oxidation) and can remove some heavy cations and can reduce microorganisms in the water. In addition, this process can also remove some ions and colloids. The success of the electrocoagulation technique is determined by various things including: the type of electrode used, the distance of the electrodes, electrocoagulation time; and the magnitude of the application of direct voltage and electric current, as well as the type and concentration of treated wastewater (Barrera-Díaz et al., 2018).

Microbiology is the study of microbes, their biological properties and activities. Microbes play an important role in cleaning up toxic waste. Microorganisms and their enzymes play a role in the breakdown of organic material in wastewater. Microorganisms play a key role in ecological processes, act as universal catalysts and provide for ecological transformation. Bioremediation is a system that utilizes microorganisms using techniques that convert "biodegradable complex toxic substances" into harmless end products

through cellular metabolism. Suspended colloids are captured and combined as biological flocks and biofilms (Nisha, 2019).

Through this experiment, it is expected to find the most effective method or combination of methods for waste treatment at PT. Etercon Pharma in order to meet the quality standards stipulated in the Minister of Environment and Forestry Regulation No. 5 of 2014

1.2.Objectives

1.2.1. General Instructional Objective

Can apply the theory obtained from lectures on problems that occur in the chemical field or industry.

1.2.2. Specific Instructional Objectives

1. See and get to know the job market firsthand and deepen the theory gained during lectures.
2. Increase insight and understanding of the electrocoagulation method used in industry.
3. Can acquire skills in mastery of work in a company both in the process unit and in the laboratory.
4. Providing opportunities for students to socialize themselves in a real work environment both as employees and as independent workers, especially with regard to work discipline.
5. Obtaining experience input and feedback to reproduce and develop knowledge in accordance with the field being studied.
6. Adding insight into applied chemistry in the PT. Etercon Pharma.
7. Knowing the working principles and the use of instruments used in the work process according to the permits given by the company.
8. Knowing the chemical processes that exist in the work process at PT. Etercon Pharma.

1.3 Benefit

The benefits of carrying out the wastewater treatment using the electrocoagulation and microbiological method are as follows:

1. For students

As a forum for students to apply the knowledge that has been gained during lectures, in this case doing the pharmaceutical industry wastewater treatment with the electrocoagulation method so that it meets environmental quality standards. This is intended so that students have experience in the field of wastewater treatment.

2. For Agencies

The formation of a network of relationships between universities and institutions for the future, where companies need human resources from universities for the advancement of Science and Technology, especially Chemistry

1.4 Rationale

The rationale for implementing Practical Work at PT. Etercon Pharma are as follows:

1. Tridharma Perguruan Tinggi, which includes research education and community service
2. Compulsory courses in the Department of Chemistry, Faculty of Science and Mathematics, Diponegoro University.

CHAPTER II.

LITERATURE REVIEW

2.1.Wastewater

Wastewater is a type of water that has been contaminated from household, industrial, commercial or agricultural activities. Pharmaceutical products are released into wastewater streams from manufacturing sites and drug development bases around the world. Comprehensive production and use of pharmaceutical products produces wastewater with a complex composition(Naddeo et al., 2009).

Pharmaceutical industry waste can be in the form of acids, bases, salts and catalysts, solvents, and various kinds of residual products from the activities of each industry. The characteristics and complexity of the waste depend on the characteristics of the product produced. According to the Regulation of the Minister of Environment of the Republic of Indonesia Number 5 of 2014, the quality standards of waste water for the pharmaceutical industry business and / or activities are as follows

No.	Parameter	Level
1.	pH	6-8
2.	COD	<150 ppm
3.	BOD	<75 ppm
4.	TSS	<75 ppm

(Permen LH RI No.5, 2014)

2.2.pH

pH is a scale used to determine the acidity or alkalinity of a solution. Acid solutions are measured to have a lower pH value than alkaline or basic solutions. The pH scale is logarithmic and inversely shows the concentration of hydrogen ions in solution. The range is from 0 to 14, with 7 being neutral. A pH less than 7 indicates acidity, while a pH greater than 7 indicates alkaline. The pH of water is a very important measure related to water quality.

$$\text{pH} = -\log [\text{H}^+]$$

(Petrucci et al., 2006)

A solution is neutral if it contains the same concentration of hydronium and hydroxide ions; acids if they contain a greater concentration of hydronium ions than hydroxide ions; and bases if they contain a lower concentration of hydronium ions than hydroxide ions (Britannica, 2013).

2.3.BOD

Biological oxygen demand (BOD) is the amount of dissolved oxygen required (used) by aerobic biological organisms to break down organic matter present in a given water sample at a certain temperature over a certain period of time. The BOD value is most often expressed in milligrams of oxygen consumed per liter of sample during 5 days of incubation at 20°C and is often used as an indicator of the degree of organic pollution in water.

The more organic matter there is (for example, in sewage and polluted water bodies), the greater the BOD. If more oxygen is used than is produced, the lower the dissolved oxygen level available. Therefore, BOD is a reliable measuring tool for the organic pollution of a body of water. One of the main reasons for treating wastewater before it is discharged into water resources is to reduce its BOD level (Sawyer et al., 2003).

2.4. COD

Chemical Oxygen Demand or COD is a measure of oxygen needed to oxidize dissolved organic matter and particulates in water. COD is usually expressed in terms of oxygen consumed per volume of solution which in SI units is expressed as milligrams per liter (mg / L). The basis of the COD test is that almost any organic compound can be completely oxidized to carbon dioxide by a strong oxidizing agent under acidic conditions.

COD is often measured using strong oxidants (eg potassium dichromate, potassium iodate, potassium permanganate) under acidic conditions. The excess amount of known oxidant is added to the sample. After the oxidation is complete, the concentration of organic matter in the sample is calculated by measuring the amount of oxidant remaining in the solution. This is usually done by titration,

using an indicator solution. COD is expressed in mg / L, which indicates the mass of oxygen consumed per liter of solution.

In contrast to the BOD test, toxic compounds (such as heavy metals and cyanide) in the sample to be analyzed had no effect on the oxidants used in the COD test. Therefore, the COD test can be used to measure waste that is too toxic for the BOD test(Sawyer et al., 2003).

2.5. TSS

Total suspended solids (TSS) is the dry weight of suspended particles, which are insoluble in the water sample that can be trapped by the filter analyzed using a filtration apparatus. TSS is a water quality parameter that is used to assess the quality of specimens from all types of water or water bodies, or wastewater after being treated in a wastewater treatment plant.

The TSS of a water or wastewater sample is determined by pouring a carefully measured volume of water (usually one liter) through a pre-weighed filter of the specified pore size, then reweighing the filter after a drying process which removes all water in the filter. Filters for TSS measurement usually consist of glass fibers. Weight gain is a measure of the dry weight of the particulates in a water sample expressed in units calculated from the volume of water filtered (usually milligrams per liter or mg / L) (Michaud, 1994).

2.6. Coagulation

Coagulation is the process of mixing coagulants (chemicals) into raw water with a rotation that causes small suspended particles to combine. Coagulants are chemicals needed in raw water to help the coagulation process of small particles that are still suspended in water, examples of coagulants include PAC and alum. The principle of coagulation is that in raw water there are solid particles, most of which are negatively charged. These particles tend to repel each other so that they remain stable in suspended or colloid form in water. Neutralization of the negative charge of solid particles is carried out by adding a positively charged coagulant to water followed by rapid stirring.

(Susanto, 2008)

2.7. Flocculation

Foculation is the process of collecting small particles into larger particles called floc. The intermolecular force obtained from agitation is one of the factors that affects the rate at which floc particles are formed. One of the important factors that influence the success of the flocculation process is slow stirring, this condition gives the particles the opportunity to make contact or connection to form a fusion (agglomeration). This slow stirring is carried out with care because the large floc will break easily through high speed mixing.

(Susanto, 2008)

2.8. Electrocoagulation

Electrocoagulation (EC), is a technique used for wastewater treatment, washing water treatment, industrial treated water, and medical care. Electrocoagulation has become a rapidly growing area of wastewater treatment due to its ability to remove contaminants that are generally more difficult to remove by filtration or chemical treatment systems, such as emulsified oil, total petroleum hydrocarbons, refractory organics, suspended solids, and heavy metals.(Al-Shannag et al., 2015).

EC technology is a wastewater treatment process by flowing electricity as the main power source. Typically, the power used in EC is an alternating current (AC) power supply or direct current (DC) power to produce an electric current.

In its simplest form, an electrocoagulation reactor consists of an electrolytic cell with one anode and one cathode. When connected to an external power source, the anode material will experience electrochemical corrosion due to oxidation, while the cathode will experience passivation(Fadhila et al., 2018).

The EC system basically consists of pairs of conductive metal plates in parallel, which act as monopolar electrodes. Furthermore, it requires a direct current power source, a resistance box to adjust the current density and a multimeter to read the current value. Conductive metal plates are commonly known as "*sacrificial electrodes*". *Sacrificial Anodes* decreases the dissolution potential of the anode and minimizes cathode passivation. *Sacrificial anodes* and the cathode can be of the same or different materials (Dindaş et al., 2020).

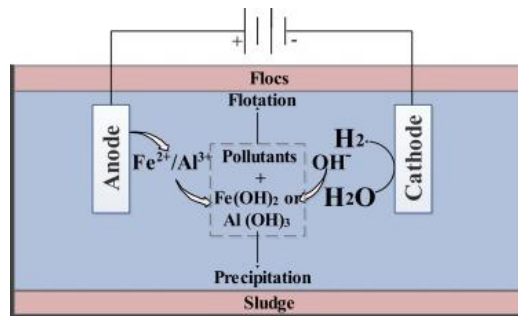
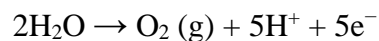
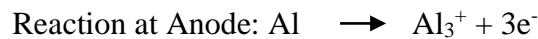
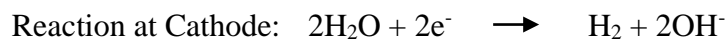


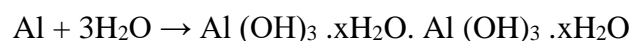
Figure 1. Schematic of the electrocoagulation process

2.8.1. Reaction in Electrocoagulation

Reduction and oxidation reactions occur during the electrocoagulation process. The reduction reaction occurs in the cathode plate and the oxidation reaction occurs in the anode plate. The anode functions as a coagulant in the coagulation-flocculation process that occurs in the cell. While at the cathode a cathodic reaction occurs by forming bubbles of hydrogen gas which function to increase the suspended floc which cannot settle in the cell.



The ions formed in the solution will undergo a hydrolysis reaction, resulting in solid $\text{Al}(\text{OH})_3 \cdot x\text{H}_2\text{O}$ which can no longer dissolve in water.



At the anode, an $\text{Al}(\text{OH})_3$ floc is formed which will then bind the elements in the waste, so the floc will have a tendency to settle. (Hanum et al., 2015).

2.8.2. Factors Affecting Electrocoagulation

- Electric current density, increase in current density will accelerate charged ions to form floc. The amount of electric current that flows is directly proportional to the material produced.
- Time, according to Faraday's law, the amount of charge that flows during the electrolysis process is proportional to the amount of contact time used.

- The current that flows produces chemical changes that flow through the medium (metal or electrolyte) due to the potential difference, because the electrical resistance in the medium is greater than that of metal, what needs to be considered is the medium and the boundary between the metal and the medium.
- The level of acidity (pH), in the electrocoagulation process, a water electrolysis process occurs which produces hydrogen gas and hydroxide ions. The longer the contact time is used, the faster the formation of hydrogen gas and hydroxide ions, if more hydroxide ions are produced, it will increase the pH in the solution.
- The thickness of the plate, the thicker the electrode plate used, the greater its electrostatic attraction in reducing and oxidizing metal ions in solution.
- The distance between the cathodes, the greater the distance the greater the resistance, so that the smaller the current flowing.

(de Santana et al., 2018)

2.9.Filtration

Filtration is a physical, biological or chemical process that separates solids and fluids from a mixture with a filter media that has a complex structure that only fluids can pass through. Solid particles that cannot pass through the filter media are described as large in size and the fluid that passes through them is called the filtrate. Particles that are too large can form a filter screen over the filter and can also block the filter grid, preventing the fluid phase from passing through the filter, which is known as blindness. The largest particle size that makes it through the filter is called the effective pore size of that filter.

The basic requirements for filtration are: (1) filter media; (2) liquid with suspended solids; (3) the driving force such as the pressure difference that causes the fluid to flow; and (4) mechanical devices (filters) that hold the filter media, contain fluid, and allow the application of a force.

(Sutherland and Chase, 2011)

2.10. Microbiology

Microbiology is the study of microbes, their biological properties and activities. The word "microorganism" refers to the word "micro" in Greek which means small. The study of microorganisms including bacteria, algae, fungi, protozoa, and viruses is necessary to investigate the genetic, physiological, and biochemical reactions that occur. The existence of pharmaceutical compounds in aquatic environments resulting from industrial, domestic, and urban waste products causes serious environmental problems. The presence of these contaminants causes various effects on organisms in aquatic ecosystems. This waste changes the value of BOD and COD in water, increasing the toxicity for aquatic organisms (Bhoomika, 2020).

Microbes play an important role in cleaning up toxic waste. Microorganisms and their enzymes play a role in the breakdown of organic material in wastewater. Microorganisms play a key role in ecological processes, act as universal catalysts and provide for ecological transformation. Bioremediation is a system that utilizes microorganisms using techniques that convert "biodegradable complex toxic substances" into harmless end products through cellular metabolism. Suspended colloids are captured and combined as biological flocks and biofilms (Nisha, 2019).

CHAPTER III.

RESEARCH METHODOLOGY

3.1. Internship Date and Location

The field internship was performed from 4 January 2021 to 4 February 2021 in PT. Etercon Pharma.

3.2. Tools and Materials

3.2.1. Tools

- Electrocoagulation reactor (power supply)
- Voltmeter
- Connecting cable
- Al and Fe plate
- 1L Beaker Glass
- Funnel
- Magnetic Stirrer
- Bottle 200mL
- Filter Paper

3.2.2. Materials

- $\text{Fe}_2(\text{SO}_4)_3$
- H_2O_2 30%
- Simethicone
- Flocculan (Alum and Chalk)
- Potassium Dichromate
- Ferro Ammonium Sulfate
- Tryptophan Soya Broth
- Potassium Hydrogen Phthalate
- Potassium Permanganate
- CaCO_3
- Concentrated H_2SO_4
- Ferriin Indicator
- Wastewater inlet
- NaOH 1N

3.3. Work Schemes

3.3.1. Wastewater treatment using electrocoagulation method

3.3.1.1 Fe Electrocoagulation

- **Trial 1**
 1. Add the inlet liquid wastewater sample into a 1L beaker as much as 1000 ml.
 2. Installing the electrode plate on the beaker glass with a distance between the cathodes of 1 cm.
 3. Connect the power supply to the electrode plate.
 4. Turn on the power supply at 6V.

5. Record the changes at the 30th, 90th, and 120th minutes.
 6. Remove the resulting foam and filter the electrocoagulated solution.
- **Trial 2**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.5mL H₂O₂ 30%
 3. Stir for 30 seconds using a magnetic stirrer.
 4. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 5. Connect the power supply to the electrode plate that has been attached to the beaker glass.
 6. Turn on the power supply at 6V.
 7. Record the changes at the 15th and 30th minute.
 8. Discard the resulting foam.
 9. Add 5mL flocculant, stir until macrofloc is formed, record the changes.
 10. Filter the electrocoagulated solution.
 - **Trial 3**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.5mL H₂O₂ 30%
 3. Add 1g Simethicone.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Installing the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Turn on the power supply at 6V.
 8. Record the changes at the 15th and 30th minute.
 9. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 10. Filter the electrocoagulated solution.
 - **Trial 4**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.5mL H₂O₂ 30%
 3. Add 0.3mL Simethicone.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Installing the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Turn on the power supply at 6V.
 8. Record the changes at the 15th and 30th minute.
 9. Add 0.25g CaCO₃ and 5mL flocculant, stir until macrofloc is formed, record the changes.
 10. Filter the electrocoagulated solution.
 - **Trial 5**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.5mL H₂O₂ 30%
 3. Add 1g Simethicone.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.

7. Turn on the power supply at 6V.
 8. Record the changes at the 15th and 30th minute.
 9. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 10. Filter the electrocoagulated solution, store the filtrate in the bottle and leave the sludge in the beaker.
 11. Add 0.5mL 30% H₂O₂, 1g Simethicone, and 800mL of waste water into the beaker.
 12. Stir for 30 seconds using a magnetic stirrer.
 13. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 14. Connect the power supply to the electrode plate that has been attached to the beaker glass.
 15. Turn on the power supply at 6V.
 16. Record the changes at the 15th and 30th minute.
 17. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 18. Filter the electrocoagulated solution, store the filtrate in the bottle and leave the sludge in the beaker.
- **Trial 6 - Trial 9**
 1. Add 0.5mL 30% H₂O₂, 1g Simethicone, and 800mL of waste water to the Trial 5 beaker.
 2. Stir for 30 seconds using a magnetic stirrer.
 3. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 4. Connect the power supply to the electrode plate.
 5. Turn on the power supply at 6V.
 6. Record the changes at the 15th and 30th minute.
 7. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 8. Filter the electrocoagulated solution, store the filtrate in the bottle and leave the sludge in the beaker. Do points 1-8 for trial 7-9.
 - **Trial 10**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.5mL H₂O₂ 30%
 3. Add 1g Simethicone.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Installing the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Turn on the power supply at 6V.
 8. Record the changes at the 15th and 30th minute.
 9. Add 5mL of flocculant, stirring until macrofloc is formed.
 10. Add 2mL of flocculant, stir until macrofloc is formed, record the changes.
 11. Take 200mL, then add 1g Fe₂SO₄
 12. Filter the electrocoagulated solution.
 - **Trial 11**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.5mL H₂O₂ 30%

3. Add 1g Simethicone.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Turn on the power supply at a voltage of 12V.
 8. Record the changes at the 15th and 30th minute.
 9. Add 5mL of flocculant, stirring until macrofloc is formed.
 10. Add 5mL of flocculant, stir until macrofloc forms, record the changes.
 11. Filter the electrocoagulated solution.
- **Trial 12**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.5mL H₂O₂ 30%
 3. Add 1g Simethicone.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Installing the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Turn on the power supply at a voltage of 12V.
 8. Record the changes at the 15th and 30th minute.
 9. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 10. Filter the electrocoagulated solution.
 - **Trial 13**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.07g FeSO₄, add 0.3mL H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 1N NaOH to pH 6-7.
 4. Add 5mL of flocculant, stir until it forms macrofloc.
 5. Add 0.5mL H₂O₂ 30%
 6. Add 1g Simethicone.
 7. Stir for 30 seconds using a magnetic stirrer.
 8. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 9. Connect the power supply to the electrode plate.
 10. Turn on the power supply at a voltage of 12V.
 11. Record the changes at the 15th and 30th minute.
 12. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 13. Filter the electrocoagulated solution.

3.3.1.2 Al Electrocoagulation

3.3.1.2.1 Al-Al Electrocoagulation

- **Trial 14 - Trial 15**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.21g FeSO₄, add 0.5mL H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 1N NaOH to pH 6-7.
 4. Add 5mL of flocculant, stir until it forms macrofloc.

5. Add 0.5mL H₂O₂ 30%
 6. Add 1g Simethicone.
 7. Stir for 30 seconds using a magnetic stirrer.
 8. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 9. Connect the power supply to the electrode plate.
 10. Turn on the power supply at a voltage of 20V.
 11. Record the change at 30 minutes.
 12. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 13. Filter the electrocoagulated solution.
 14. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 15. Connect the power supply to the electrode plate.
 16. Turn on the power supply at a voltage of 20V.
 17. Record the changes at the 45th minute.
 18. Add 3mL of flocculant, stir until macrofloc is formed, record the changes.
 19. Filter the electrocoagulated solution.
 20. Installing the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 21. Connect the power supply to the electrode plate.
 22. Turn on the power supply at a voltage of 20V.
 23. Record the change at the 60th minute.
 24. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 25. Filter the electrocoagulated solution.
- **Trial 16**
 1. Add 80mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.07g FeSO₄, add 0.3mL H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 1N NaOH to pH 6-7.
 4. Add 5mL of flocculant, stir until it forms macrofloc.
 5. Add 0.5mL H₂O₂ 30%.
 6. Stir for 30 seconds using a magnetic stirrer.
 7. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 8. Connect the power supply to the electrode plate.
 9. Turn on the power supply at a voltage of 20V.
 10. Record the change at 15 minutes.
 11. Record the change at 30 minutes.
 12. Record the changes at the 45th minute.
 13. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 14. Filter the electrocoagulated solution.
 - **Trial 17**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.11g FeSO₄, add 0.5mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 0.5mL H₂O₂ 30%.

4. Stir for 30 seconds using a magnetic stirrer.
 5. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Turn on the power supply at a voltage of 20V.
 8. Record the change at 15 minutes.
 9. Record the change at 30 minutes.
 10. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 11. Filter the electrocoagulated solution.
- **Trial 18**
 1. Entering the inlet liquid wastewater sample into a 1L beaker as much as 800 ml.
 2. Add 0.11g FeSO₄, add 0.5mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 0.5mL H₂O₂ 30%.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Installing the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Record the change at 30 minutes.
 - **Trial 19**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.11g FeSO₄, add 0.5mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 0.5mL H₂O₂ 30%.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Turn on the power supply at a voltage of 20V.
 8. Record the change at 15 minutes.
 9. Add 3mL of flocculant, stir until it forms macrofloc.
 10. Record the change at 30 minutes.
 11. Add 3mL of flocculant, stir until macrofloc is formed.
 12. Filter the electrocoagulated solution.
 - **Trial 20**
 1. Add 800mL liquid wastewater sample into a 1L beaker.
 2. Add 0.11g FeSO₄, add 0.5mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 0.5mL H₂O₂ 30%.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Turn on the power supply at a voltage of 20V.
 8. Record the change at 15 minutes.
 9. Record the change at 30 minutes.
 10. Add 5mL of flocculant, stirring until macrofloc is formed.
 11. Filter the electrocoagulated solution.

- **Trial 22**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.11g FeSO₄, add 0.5mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 0.5mL H₂O₂ 30%.
 4. Stir for 30 seconds using a magnetic stirrer.
 5. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 6. Connect the power supply to the electrode plate.
 7. Turn on the power supply at a voltage of 20V.
 8. Record the change at 15 minutes.
 9. Record the change at 30 minutes.
 10. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 11. Filter the electrocoagulated solution.
- **Trial 23**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.11g FeSO₄, add 0.5mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 1N NaOH to pH 6-7.
 4. Add 0.5mL H₂O₂ 30%.
 5. Stir for 30 seconds using a magnetic stirrer.
 6. Installing the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 7. Connect the power supply to the electrode plate.
 8. Turn on the power supply at a voltage of 20V.
 9. Record the change at 15 minutes.
 10. Record the change at 30 minutes.
 11. Add 5mL of flocculant, stir until macrofloc is formed, record the changes.
 12. Filter the electrocoagulated solution.
- **Trial 24**
 1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
 2. Add 0.11g FeSO₄, add 0.5mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
 3. Add 1N NaOH to pH 6-7.
 4. Add 0.5mL H₂O₂ 30%.
 5. Stir for 30 seconds using a magnetic stirrer.
 6. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
 7. Connect the power supply to the electrode plate.
 8. Turn on the power supply at a voltage of 20V.
 9. Record the change at 15 minutes.
 10. Record the change at 30 minutes.
 11. Add 3mL of flocculant, stir until macrofloc is formed.
 12. Filter the electrocoagulated solution.
- **Trial 26**
 1. Add 800mL inlet liquid waste sample into a 1L beaker.
 2. Add 0.6g FeSO₄, add 1mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.

3. Add 1N NaOH to pH 6-7.
4. Add 0.5mL H₂O₂ 30%.
5. Stir for 30 seconds using a magnetic stirrer.
6. Install the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
7. Connect the power supply to the electrode plate.
8. Turn on the power supply at a voltage of 20V.
9. Record the change at 30 minutes.
10. Add 4mL of flocculant, stirring until macrofloc is formed.
11. Filter the electrocoagulated solution.

3.3.1.2.2 Al-Carbon Electrocoagulation

- **Trial 21**

1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
2. Add 0.11g FeSO₄, add 0.5mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
3. Add 0.5mL H₂O₂ 30%.
4. Stir for 30 seconds using a magnetic stirrer.
5. Installing the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
6. Connect the power supply to the electrode plate, Al as the anode and Carbon as the cathode.
7. Turn on the power supply at a voltage of 20V.

3.3.1.2.3 Al-Glass Electrocoagulation

- **Trial 25**

1. Add 800mL inlet liquid wastewater sample into a 1L beaker.
2. Add 0.11g FeSO₄, add 0.5mL 30% H₂O₂, stir until it forms a microfloc using a magnetic stirrer.
3. Add 1N NaOH to pH 6-7.
4. Add 0.5mL H₂O₂ 30%.
5. Stir for 30 seconds using a magnetic stirrer.
6. Installing the electrode plate on the beaker glass with a distance between the electrodes of 1cm.
7. Connect the power supply to the electrode plate, Al as the anode and glass as the cathode.
8. Turn on the power supply at a voltage of 20V.

3.3.2. Microbiology

3.3.2.1 Making Bacterial Culture Media 1

1. Add 30g of Tryptophan Soya Broth (TSB) media to 1000mL of water. Heat and stir until dissolved. Put in 5 bottles, each 200mL.
2. Sterilize in autoclave for 20 minutes.
3. Cool to room temperature.
4. Add 10 mL of bacteria into each bottle, then incubate for 1 day at 30°C.

3.3.2.2 Making Bacterial Culture Media 2

1. Add 30g of TSB media to 1000mL of water. Heat and stir until dissolved. Put in 5 bottles, each 200mL.
2. Sterilize in autoclave for 20 minutes.
3. Cool to room temperature.

4. Add 10 mL of bacteria from bacterial culture 1 into each bottle.
5. Incubation for 1 day at 30°C.
6. Save 1 bottle of bacterial culture 1 for bacterial culture 2

3.3.2.3 Manufacture of Bacterial Culture Media 3

1. Add 18g of TSB media to 600mL of water. Heat and stir until dissolved. Put in 3 bottles, each 200mL.
2. Sterilize in autoclave for 20 minutes.
3. Cool to room temperature.
4. Add 10 mL of bacteria into each bottle.
5. Incubation for 1 day at 30°C.
6. Waste Samples
7. Add 5 mL of waste to the bacterial culture bottle 1 which has been taken 10 mL for bacterial culture 2.
8. Add 5mL of TSB media on the second day.
9. Observe the growth of bacteria every day.

3.3.2.4 Large-Scale Bacterial Culture

1. A suite of tools for large-scale bacterial culture.
2. Pour 4 bottles of bacterial culture 1, and 5 bottles of bacterial culture 2 into a large scale media bucket.
3. Add lactose, oat kernel, NaCl and KH₂PO₄ every day as a food source of bacteria.
4. Add wastewater sample bottles and 500mL of waste to large scale media buckets.
5. Observe the growth of bacteria every day.

3.3.3. COD Analysis

3.3.3.1 Titration Method

- Potassium Dichromate
 - Preparation of Digestion Solution (Potassium Dichromate 0.1N)
Dissolve 4.903g K₂Cr₂O₇ (dry) with 500mL of free organic water into a 1000mL volumetric flask. Add 167mL concentrated H₂SO₄. Add 33.3g HgSO₄, stir until it dissolves completely and adjust to the mark then homogenize.
 - Preparation of sulfuric acid reagent solution
Dissolve 10.12g Ag₂SO₄ crystals in 1000mL concentrated H₂SO₄. Stir until it dissolves
 - Preparation of 0.05N Ferro Ammonium Sulfate standard solution
Weigh 19.6g Fe (NH₄)₂SO₄·6H₂O then dissolve it into a 1000mL volumetric flask containing 300mL of free organic water. Add 20mL of concentrated H₂SO₄, cooled chili sauce and correct it until it is marked, then homogenized.
 - Standardization of FAS
Pipette 1.25mL digestion solution into Erlenmeyer, add 10mL of free organic water, add 1mL of sulfuric acid reagent solution. Add 1-2 drops of the ferroin indicator and titrate with the FAS solution.
 - Analysis of COD sample
 1. Add 10mL of test sample, 6mL of digestion solution, and 14mL of sulfuric acid reagent solution to the digestion vessel.
 2. Close the jar and shake gently until homogeneous.

3. Place the tube on a heater that has been heated at 150°C, reflux for 2 hours.
4. Cool the test sample and the refluxed working solution to room temperature.
5. Transfer the sample into the Erlenmeyer for titration.
6. Add 1-2 drops of the ferroin indicator and titrate with the FAS standard solution until a clear color changes from green-blue to reddish brown, record the FAS standard solution used.
7. Perform Steps 1-6 on organic free water as a blank. Record the volume of the FAS solution used.

- **Potassium Permanganate**

- Preparation of a digestion solution (Potassium Permanganate)
Dissolve 4.9g KMnO_4 with 500mL of free organic water into a 1000mL volumetric flask. Add 167mL concentrated H_2SO_4 . Add 33.3g HgSO_4 , stir until it dissolves completely and adjust to the mark then homogenize.
Preparation of sulfuric acid reagent solution
Dissolve 10.12g Ag_2SO_4 crystals in 1000mL concentrated H_2SO_4 . Stir until it dissolves
- Preparation of 0.05N Ferro Ammonium Sulfate standard solution
Weigh 19.6g $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ then dissolve it into a 1000mL volumetric flask containing 300mL of free organic water. Add 20mL of concentrated H_2SO_4 , cooled chili sauce and correct it until it is marked, then homogenized.
- Standardization of FAS
Pipette 1.25mL digestion solution into Erlenmeyer, add 10mL of free organic water, add 1mL of sulfuric acid reagent solution. Add 1-2 drops of the ferroin indicator and titrate with the FAS solution.
- Analysis of COD sample
 1. Add 10mL of test sample, 6mL of digestion solution, and 14mL of sulfuric acid reagent solution to the digestion vessel.
 2. Close the jar and shake gently until homogeneous.
 3. Place the tube on a heater that has been heated at 150°C, reflux for 2 hours.
 4. Cool the test sample and the refluxed working solution to room temperature.
 5. Transfer the sample into the Erlenmeyer for titration.
 6. Add 1-2 drops of the ferroin indicator and titrate with the FAS standard solution until a clear color changes from green-blue to reddish brown, record the FAS standard solution used.
 7. Perform Steps 1-6 on organic free water as a blank. Record the volume of the FAS solution used.

3.3.3.2 Spectrophotometry Method

- High Digestion Solution Manufacturing (100-900nm)
Add 1.0216 g of potassium dichromate (dry) to 50mL of free organic water in a 100mL volumetric flask. Add 16.7 H_2SO_4 . Add 3.33g HgSO_4 , stirring until it dissolves completely and adjust to the mark then homogenize.
- Low Digestion Solution Manufacturing ($\leq 90\text{nm}$)

Add 0.1022 g of potassium dichromate (dry) to 50mL of free organic water in a 100mL volumetric flask. Add 16.7 H₂SO₄. Add 3.33g HgSO₄, stirring until it dissolves completely and adjust to the mark then homogenize.

- Preparation of Potassium Hydrogen Phthalate Standard Solution (500ppm)
Dissolve 0.425 g of Potassium Hydrogen Phthalate (dry) into 1000mL of free organic water, then homogenize it.
- Preparation of Sulfuric Acid Reagent Solution
Dissolve 1.012 g AgSO₄ into 100mL H₂SO₄
- Preparation of KHP Standard Solution
 - Dilute the standard solution of KHP 500 ppm into standard solutions of 0, 15, 30, 45, 60, 75, 90ppm for low COD values.
 - Dilute 1000 ppm KHP standard solution into 0, 150, 300, 450, 600, 750, 900 ppm standard solutions for high COD values.
- Calibration curve creation
 - Low COD
 1. Add 2.5 ml of 0 ppm standard solution into the test tube, add 1.5ml of low digestion solution, add 3.5mL of H₂SO₄. Do the same for the concentrations of 15, 30, 45, 60, 75, 90 ppm. Cover all tubes using aluminum foil. Heat for 2 hours.
 2. Measure the absorbance of each standard solution.
 3. Create a calibration curve from the absorbance values obtained from each concentration.
 - High COD
 1. Add 2.5 ml of 0 ppm standard solution into the test tube, add 1.5ml high digestion solution, add 3.5mL H₂SO₄. Do the same for the concentrations of 150, 300, 450, 600, 750, 900 ppm. Cover all tubes using aluminum foil. Heat for 2 hours.
 2. Cool for ± 20 minutes at room temperature.
 3. Measure the absorbance of each standard solution.
 4. Create a calibration curve from the absorbance values obtained from each concentration.
- Sample analysis
 - Low COD
 1. Add 2.5mL of sample into the test tube, add 1.5mL of low digestion solution, add 3.5mL of H₂SO₄. Cover all tubes using aluminum foil. Heat for 2 hours.
 2. Cool for ± 20 minutes at room temperature.
 3. Measure the absorbance of each sample at a wavelength of 420 nm.
 4. Calculate COD levels based on the linear equation of the calibration curve
 - High COD
 1. Add 2.5mL of sample into the test tube, add 1.5mL of high digestion solution, add 3.5mL of H₂SO₄. Cover all tubes using aluminum foil. Heat for 2 hours.
 2. Cool for ± 20 minutes at room temperature.
 3. Measure the absorbance of each sample at a wavelength of 600 nm.
 4. Calculate COD levels based on the linear equation of the calibration curve.





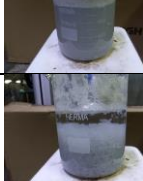


CHAPTER IV.

RESULT AND DISCUSSION














4.1.Fe Electrocoagulation

This experiment aims to treat wastewater by forming filterable sludge and by oxidizing pollutants in the wastewater so that it can meet the standards set by the Minister of Environment and Forestry Regulation No. RI No. 5/2014. The electrocoagulation experiment of wastewater using Fe-Fe electrodes was carried out based on the principle of the reduction-oxidation reaction. One of the Fe acts as an anode which will undergo oxidation and the other Fe acts as a cathode which will experience reduction. In the electrocoagulation experiment using Fe electrode, 13 experiments were carried out with different working methods.

Table 1. Results of Waste Treatment Using the Fe-Fe Electrocoagulation Method

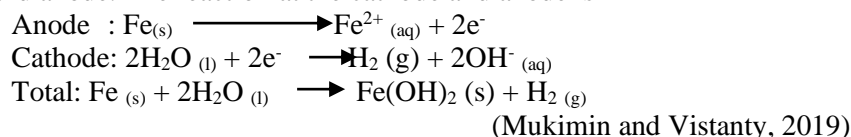
Trial	Treatment	Time	Information	Photo
1	1L Wastewater + Electrocoagulation	30 minutes	Formed foam, formed microfloc, water was not clear	
		90 minutes	Formed foam, the Microflok is getting bigger, the water is not clear yet	
		120 minutes	Formed foam, the Microflok is getting bigger, the water is not clear yet	
2	0.5mL H ₂ O ₂ + 800mL Wastewater	15 minutes	Formed foam, Formed microfloc, Water was not clear	
		30 minutes	Formed foam, Microflok getting bigger, Water is not clear yet	
	+ 5mL Flocculant		Formed Macroflok, clear water	
3	0.5mL H ₂ O ₂ + 1g Simethicon + 800 mL Wastewater	15 minutes	Little foam, formed microfloc, water is not clear	

		30 minutes	There is no foam, the microfloc is getting bigger, the water is not clear yet	
	+ 5mL Flocculant		Formed Macroflok, clear water	
4	0.5 mL H ₂ O ₂ + 0.3mL Simethicon + 800 mL Wastewater	15 minutes	Little foam, formed microfloc, water is not clear	
		30 minutes	There is no foam, the microfloc is getting bigger, the water is not clear yet	
	+ 0.25g CaCO ₃ + 5mL flocculant		Macrofloc is formed, the water is not clear (there is still a suspension)	
5	0.5 mL H ₂ O ₂ + 1g Simethicon + 800 mL Wastewater	15 minutes	Little foam, formed microfloc, water is not clear	
		30 minutes	There is no foam, the microfloc is getting bigger, the water is not clear yet	
	+ 5mL flocculant		Formed macrofloc, clear water	
	Sludge + 0.5 mL H ₂ O ₂ + 1g Simethicon + 800 mL Wastewater	15 minutes	Little foam, formed microfloc, water is not clear	
		30 minutes	There is no foam, the microfloc is getting bigger, the water is not clear yet	
	+ 5mL flocculant		Formed macrofloc, clear water	
6	Sludge + 0.5 mL H ₂ O ₂ + 1g Simethicon + 800 mL Wastewater + Electrocoagulation	15 minutes	Little foam, formed microfloc, water is not clear	

		30 minutes	There is no foam, the microfloc is getting bigger, the water is not clear yet	
	+ 5mL flocculant		Formed macrofloc, clear water	
7.	Sludge + 0.5 mL H ₂ O ₂ + 1g Simethicon + 800 mL Wastewater + Electrocoagulation	15 minutes	Little foam, formed microfloc, water is not clear	
		30 minutes	There is no foam, the microfloc is getting bigger, the water is not clear yet	
	+ 5mL flocculant		Formed macrofloc, clear water	
8.	Sludge + 0.5 mL H ₂ O ₂ + 1g Simethicon + 800 mL Wastewater + Electrocoagulation	15 minutes	Little foam, formed microfloc, water is not clear	
		30 minutes	There is no foam, the microfloc is getting bigger, the water is not clear yet	
	+ 5mL flocculant		Macrofloc forms (settles), clear water	
9.	Sludge + 0.5 mL H ₂ O ₂ + 1g Simethicon + 800 mL Wastewater + Electrocoagulation (6V)	15 minutes	Little foam, formed microfloc, water is not clear	
		30 minutes	There is no foam, the microfloc is getting bigger, the water is not clear yet	
	+ 5mL flocculant		Formed macrofloc, clear water	
10.	0.5 mL H ₂ O ₂ + 1g Simethicon + 800 mL Clear Water (5-9 trial accumulation) + Electrocoagulation (6V)	15 minutes	Little foam, formed microfloc, water is not clear	
		30 minutes	There is still foam, the microfloc is getting bigger (settles), the water is clear enough	

	+ 5mL flocculant		Formed macrofloc (soft & settles), clear water	
	+ 2mL flocculant		Formed macrofloc (soft & settles), clear water	
	+ 1g Fe ₂ SO ₄		The color becomes orange, not clear	
11.	0.5 mL H ₂ O ₂ + 1g Simethicon + 800 mL Wastewater (yellow color) + Electrocoagulation (12V)	15 minutes	Little foam, formed microfloc, water is not clear	
		30 minutes	No foam, microfloc getting bigger (floating), clear water (yellow)	
	+ 5mL flocculant		Macrofloc forms (more clumpy), clear water (yellow color)	
	+ 5mL Flocculant		Macrofloc forms (more clumpy), clear water (yellow color), slimy	
12.	0.5 mL H ₂ O ₂ + 1g Simethicon + 800 mL Wastewater + Electrocoagulation (12V)	15 minutes	Little foam, formed microfloc, water is not clear	
		30 minutes	No foam, bigger microfloc, clear water	
	+ 5mL flocculant		Macrofloc (more clumpy) formed, clear water	
13.	0.07g FeSO ₄ + 0.3ml H ₂ O ₂ + NaOH (pH 6-7) + Flocculant		The color turns black, sludge forms, the water is not clear yet	
	+ 0.5mL H ₂ O ₂ + 1g smethicone + Electrocoagulation of Fe (12 V)	15 minutes	Formed microfloc, little foam, clearer water	
		30 minutes	No foam, larger microfloc (floating), clear water	
	+ 5mL Flocculant		Macrofloc forms (more clumpy), clear water, slimy	

In Trial 1, wastewater treatment was only carried out using the electrocoagulation method. Electrocoagulation is carried out by connecting the Fe-Fe plate to the power supply at a voltage of 6V. During the electrocoagulation process, a redox reaction occurs at the cathode and anode. The reaction at the cathode and anode is



The Fe plate which acts as an anode undergoes oxidation from $\text{Fe}_{(s)}$ to $\text{Fe}^{2+}_{(aq)}$ while the Fe plate which acts as a cathode undergoes reduction in which water is reduced from $\text{H}_2\text{O}_{(l)}$ to $\text{H}_{2(g)}$ and $2\text{OH}^{-}_{(aq)}$, so the redox reaction as a whole gives $\text{Fe}(\text{OH})_{2(s)}$.

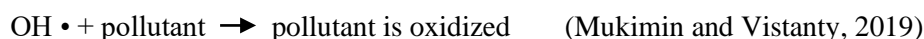
After 30 minutes of electrocoagulation, a large amount of foam was formed. This shows that there are many surfactants dissolved in liquid waste at PT. Ethercon Pharma. Dissolved surfactant interacts with H_2 gas generated from the electrocoagulation process to form foam. It can also be seen that microfloc has begun to form.

Microfloc can be formed due to the coagulation process that occurs between $\text{Fe}(\text{OH})_2$ and the pollutants contained in the waste. $\text{Fe}(\text{OH})_2$ acts as a coagulant. The positively charged Fe^{2+} cation can destabilize the colloid or suspended particles by neutralizing the negatively charged colloids in the wastewater, causing these particles to gather together and form a microfloc (Mbaeze et al., 2017). After 90 minutes and 120 minutes, the microfloc became bigger, indicating that more and more suspended particles became aggregates. However, even though it could be filtered on a lab scale using filter paper, it could not be filtered when it is applied in the field.

In Trial 2, waste treatment was carried out by adding H_2O_2 to the wastewater sample first. It aims to oxidize $\text{Fe}(\text{OH})_2$ formed in the electrocoagulation process to $\text{Fe}(\text{OH})_3$ because Fe^{3+} is a better coagulant than $\text{Fe}(\text{OH})_2$. In addition, the reaction between H_2O_2 and $\text{Fe}(\text{OH})_2$ produces OH^\bullet . The reaction that occurs between H_2O_2 and $\text{Fe}(\text{OH})_2$ is as follows.



OH^\bullet is a strong oxidizing agent, so it can oxidize the pollutants contained in the wastewater. The reaction between OH^\bullet and the material in the waste is as follows.



In trial 2, stirring was also carried out using a magnetic stirrer so that all particles contained in the wastewater could interact and react with $\text{Fe}(\text{OH})_3$ and OH^\bullet .

Microfloc has started to form since the 10th minute, which means that the formation of microfloc is faster when H_2O_2 is added and stirred. The formation of microfloc in Trial 2 was faster than Trial 1, this proves that after adding H_2O_2 , the coagulation process occurs faster and maximally due to the oxidation of Fe^{2+} to Fe^{3+} . After adding the flocculant, the microfloc undergoes flocculation and turns into macrofloc. This happens because the flocculants cause the microflocs that have been formed to form bonds with one another, causing agglomeration.

In Trial 3, simethicone was also added after the addition of H_2O_2 . The addition of simethicone aims to inhibit the formation of foam during the electrocoagulation process. Foam or bubbles form when surfactants interact with water and gas. The hydrophilic side of the surfactant will bind to water and also to the hydrophilic side of the other surfactants

while the hydrophobic side will face outward, forming a layer that is shaped like a bag, so that the gas can be trapped in the bag to form bubbles.

Simethicone is an anti-foaming agent. The anti-foaming agent is hydrophobic, so that when it comes into contact with foam or bubbles, the anti-foaming agent will interact with the hydrophilic part of the surfactant and form a bridge on the hydrophilic layer of the surfactant, causing the bubbles to burst and gas trapped inside to escape. At 15 to 30 minutes, it was found that almost no bubbles were formed during the electrocoagulation process.

In Trial 4, CaCO_3 was added after electrocoagulation. The purpose of adding CaCO_3 is to make the sludge that originally floated, to settle. The result obtained after the addition of CaCO_3 is that the water becomes cloudy, even after adding flocculants and forming macrofloc, the resulting water is still cloudy. This is possible because the simethicone added in this trial is too much, thus forming suspensions. Moreover, the presence of CaCO_3 which is insoluble in water and does not interact with the formed macrofloc, made the mixture became cloudier.

In trials 5 - 9, the sludge was not discarded to determine whether the sludge could still be formed when the sludge formed from the previous trial was added with new wastewater. On trial 5; 6; 7, the macrofloc formed is in a floating state, while in trial 8, the macrofloc formed is in a sedimentary state. This is possible because the macrofloc formed so much that the density of the macrofloc became heavier than water.

In trial 10, the water used in the electrocoagulation process was clear water accumulated from trials 5-9. This was done to determine whether there are suspended particles and pollutants that can be oxidized and coagulated. After electrocoagulation and flocculation, it was found that the macrofloc was white and very soft. This proves that there are fewer pollutants in clear water than wastewater, which means that less can be coagulated and oxidized.

In Trial 11, electrocoagulation was carried out at a voltage of 12V. This aims to increase the quantity of Fe(OH)_2 produced during electrocoagulation, the more Fe(OH)_2 produced, the faster the floc formation will occur. At the 5th minute, microfloc is starting to form. Microfloc formation is faster than electrocoagulation at a voltage of 6V. This proves that more Fe(OH)_2 produced in 12V electrocoagulation compared to 6V electrocoagulation. At 15 minutes, the microfloc starts to float and the water is clear enough compared to 6V electrocoagulation.

At 30 minutes, the resulting microfloc was larger than the microfloc in 6V electrocoagulation. After adding 5 mL of flocculant, the formed macrofloc was denser and clumpy. This is possible because the bonds formed between the macrofloc become stronger and denser. However, when 5mL of flocculant was added again, the water became slimy. This means that the macrofloc can no longer be aggregated, which means that the addition of flocculants was too much. The clear water produced is also yellow, because the inlet waste water is yellow.

In Trial 12, the resulting clear water was colorless and only 5mL of flocculants were added, so the resulting clear water was not slimy. This means that the addition of flocculants cannot be more than 10mL and also proves that the inlet wastewater of PT. Etercon has a different content every day.

In Trial 13, FeSO_4 and H_2O_2 were added to wastewater. This aims to carry out coagulation and chemical oxidation so that wastewater treatment can be maximized. After FeSO_4 was added, the wastewater began to turn black. After adding H_2O_2 , microflocases begin to form because Fe^{3+} can destabilize colloids or suspended particles by neutralizing the negatively charged colloids in the wastewater, causing these particles to gather together to form microfloc. The reaction that occurs between FeSO_4 and H_2O_2 is



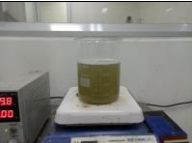
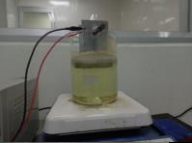


After that, NaOH was added. The addition of NaOH was carried out to adjust the pH to pH 6-7. At neutral pH, the colloid surface is uncharged, and the combination of charge neutralization with complex reactions causes a larger floc formation and pollutant removal to become more efficient.(Cao et al., 2010). After adding the flocculant, the microfloc undergoes flocculation and turns into macrofloc. After electrocoagulating for 15 minutes, the microflocases formed were larger and the water produced was clear enough compared to the chemical coagulation. This is possible because more Fe^{3+} ions are produced than in chemical coagulation, so the coagulation that occurs is more optimal.


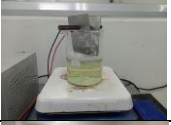

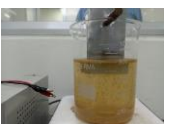
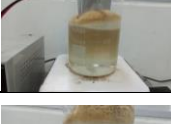




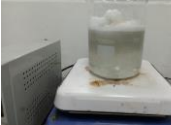


In the 30th minute, the microfloc is bigger and the water is clear. After adding the flocculant, the macrofloc that is formed coagulates and floats. Then filtration was carried out and the filtrate was stored in two different bottles, one added with H_2SO_4 and the other one not being added with anything. After that, the TDS measurement was carried out using a conductivitymeter and the TDS value obtained was 2.81 ppt; 1.87 ppt; and 4.41 ppt, for J1; J2; and Wastewater respectively. At the end of electrocoagulation, it was seen that the parts of the Fe plate that had contact with wastewater during electrocoagulation were blackish in color and eroded. This can be explained because oxidationreaction occur on the Fe plate which acts as the anode, some of the solid form of Fe has turned into water-soluble Fe^{2+} .

4.2.A1 Electrocoagulation

This experiment aims to treat wastewater by forming filterable sludge and by oxidizing pollutants in the waste so that it can meet the standards set in the Minister of Environment and Forestry Regulation No. RI No. 5 of 2014. The electrocoagulation was done by using Al-Al, Al-Carbon, Al-Glass electrodes were carried out based on the principle of the reduction-oxidation reaction. In the electrocoagulation experiment using Al electrodes, 13 experiments were carried out with different working methods.

Table 2. Results of Waste Treatment Using Al-Al Electrocoagulation Method

Trial	Treatment	Time	Information	Photo
14.	0.21g FeSO_4 + 0.5 mL H_2O_2 + NaOH (pH 6-7) + Flocculant		The color becomes orange, the microfloc is very small	
15.	+ 0.5mL H_2O_2 + 1g smethicone + Electrocoagulation Al (20V)	30 minutes	microfloc formed, foam formed, clearer water (yellow)	
	+ 5mL Flocculant		Macrofloc formed, clumping sludge + floating, water is quite clear (yellow)	
		45 minutes	microfloc formed, foam formed, clearer water (yellow)	

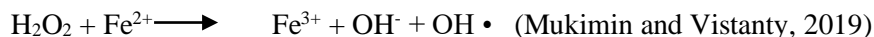
	+ 3mL Flocculant		Macrofloc formed, clumping sludge + floating, clear water (yellow)		
		60 minutes	formed microfloc, no foam, clear water (yellow), slimy		
	+ 5mL flocculant		Macrofloc formed, clumping sludge + floating, clear water (yellow)		
16.	0.07g FeSO ₄ + 0.3ml H ₂ O ₂ + NaOH (pH 6-7) + Flocculant		The color turns from black to brownish orange, sludge was formed, the water was not clear yet		
	+ 0.5mL H ₂ O ₂ + Electrocoagulation (20 V)	15 minutes	formed microfloc, lots of foam, clear water floating sludge		
		30 minutes	formed microfloc (bigger + darker color), lots of foam, clear water, floating sludge		
		45 minutes	formed microfloc (bigger + darker color), lots of foam, clear water, floating sludge		
	+ 5 mL flocculant		Forms of macrofloc, soft sludge + floating + gray, clear water		
17.	0.11g FeSO ₄ + 0.5ml H ₂ O ₂		orange color, formed microfloc, water is quite clear (yellow)		
		15 minutes	formed microfloc (gray), formed foam, water is clearer		
		30 minutes	formed microfloc (darker + drift), more foam		
	+ 5mL flocculant		formed macrofloc, clear water		

18.	0.11g FeSO ₄ + 0.5ml H ₂ O ₂		orange color, formed microfloc		
		30 minutes	microfloc formed, foam formed, clearer water (yellow)		
19.	0.11g FeSO ₄ + 0.5ml H ₂ O ₂		orange color, formed microfloc, water was quite clear (yellow)		
		15 minutes	microfloc formed, foam formed, clearer water (yellow)		
	+ 3mL Flocculant		Macrofloc formed, sludge floats + clots, water was clearer (yellow)		
		30 minutes	formed microfloc (floats) + clearer water (yellow) + formed foam		
	+ 3mL Flocculant		formed macrofloc, sludge floats + clots, clear water (yellow)		
20.	0.11g FeSO ₄ + 0.5ml H ₂ O ₂		orange color, formed microfloc		
	+ Electro Al 20 V	15 minutes	formed foam, formed microfloc, water is quite clear (yellow)		
		30 minutes	formed foam, formed microfloc, clearer water (yellow)		
	+ 5mL flocculant		formed macrofloc, sludge clumping + float, clear water		
22.	0.11g FeSO ₄ + 0.5mL H ₂ O ₂		orange color, formed microfloc		
	+ electro Al-Al 20V	15 minutes	microfloc forms, foam forms, clearer water (yellow)		
		30 minutes	formed foam, formed microfloc, clearer water (yellow)		
	+ 5mL flocculant		formed macrofloc, sludge clumping + floats, clear water (yellow)		

23.	0.11g FeSO ₄ + 0.5mL H ₂ O ₂ + NaOH (pH 6-7)		orange color, formed microfloc		
	+ electro Al-Al 20V	15 minutes	formed foam, formed microfloc, clearer water (yellow)		
		30 minutes	formed foam, formed microfloc, water was clearer (clear)		
	+ 5mL Flocculant		formed macrofloc, sludge clumping + float, clear water		
24.	0.11g FeSO ₄ + 0.5mL H ₂ O ₂ + NaOH (pH 6-7)		The color turned from black to brown, microfloc formed, the water was quite clear (yellow)		
	+ electro Al-Al 20V (1-3 A)	15 minutes	formed microfloc (bigger + milk chocolate + float), clear water		
	+ 3mL Flocculant		Macrofloc formed, yellow sludge + settles		
	Al electro filtrate 15 minutes	30 minutes	formed microfloc (bigger + gray + floating), clear water		
	+ 5mL Flocculant		formed macrofloc, sludge gray + floating, clear water		
26.	0.6g FeSO ₄ + 1mL H ₂ O ₂ + NaOH (pH 6-7)		orange color, formed microfloc, water is quite clear (yellow)		
	+ electro Al-Al 20V	30 minutes	formed microfloc (larger, milk chocolate, floating), clear water		
	+ 4mL flocculant		formed macrofloc, floating sludge, clear water		

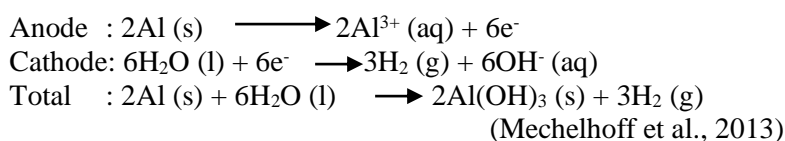
In Trial 14, 0.21g FeSO₄ and 0.5mL H₂O₂ were added, FeSO₄ as a coagulant and H₂O₂ as an oxidizing agent. This aims to carry out coagulation and chemical oxidation so that the treatment can be maximized. After adding FeSO₄, the wastewater starts to change color to orange. After adding H₂O₂, microflocases begin to form because Fe³⁺ can destabilize colloids or suspended particles by neutralizing the negatively charged colloids in the waste, causing these particles to gather together to form microfloc. However, the

microfloc that is formed is very small and fine. The reaction that occurs between FeSO_4 and H_2O_2 is



After that, NaOH was added. The addition of NaOH was carried out to adjust the pH to pH 6-7. At neutral pH, the colloid surface is uncharged, and the combination of charge neutralization with complex reactions causes a larger floc to be formed and pollutant removal became more efficient. After adding the flocculant, the microfloc will flocculate and turn into macrofloc. This happens because the flocculants cause the microflocases that have been formed to form bonds with one another, causing agglomeration. However, in Trial 14, macrofloc was not formed at all and the microflocases formed were still very small and fine. Trial 14 was left overnight hoping for further coagulation and flocculation, but there was no change at all.

In Trial 15, electrocoagulation was performed using an Al-Al plate. The wastewater samples used were the results of Trial 14. Before electrocoagulation, H_2O_2 was added to oxidize Al^{2+} produced in electrocoagulation to become Al^{3+} . Electrocoagulation is carried out by connecting the Al-Al plate to the power supply at a voltage of 20V. During the electrocoagulation process, a redox reaction occurs at the cathode and anode. The reaction at the cathode and anode is



The Al plate which acts as an anode undergoes oxidation from Al (s) to $\text{Al}^{3+} \text{ (aq)}$ while the Al plate which acts as the cathode undergoes reduction in which water was reduced from $\text{H}_2\text{O (l)}$ to $\text{H}_2 \text{ (g)}$ and $2\text{OH}^- \text{ (aq)}$, so the redox reaction as a whole gives $\text{Al(OH)}_3 \text{ (s)}$.

At the 5th minute, microfloc was starting to form. The microfloc was larger than the electrocoagulation of Fe. At the 15th minute, the microfloc immediately floats and the water starts to be clear even though it is still yellow due to the initial yellow color of the wastewater. At 30 minutes, the microfloc gets bigger, the water gets clearer, and foams are still present even though simethicone has been added. This shows that the surfactants are more dissolved than the previous trials. Then, flocculant was added, and filtered to separate the sludge with the filtrate. A part of the filtrate was electrocoagulated again until the 45th minute and some of the filtrate was stored for COD analysis, before that, the TDS measurement was carried out and the TDS value was 809 ppm.

At the 45th minute, not as many microfloc as it was formed when the 30th minute. This shows that the less suspended particles are still present in the sample. Then it was filtered, part of the filtrate was electrocoagulated again until the 60th minute and part of the filtrate was stored for COD analysis. Beforehand, the TDS was measured and the TDS value was 778 ppm.

coagulated again until the 60th minute. In the 60th minute, the formed microfloc was not as much as the 45th minute, the water that was formed was clearer but still yellow with a TDS value of 410 ppm.

In Trial 16, electrocoagulation was performed without adding simethicone. This is intended to allow surfactants dissolved in water to form foam so that they can be removed. In the 15th minute of electrocoagulation, a brownish microfloc was formed due to the presence of dissolved Fe^{3+} as a result of chemical coagulation. At 30 minutes, the microfloc becomes bigger and the color turns gray which is probably due to the Al^{3+}

coagulate the pollutants dissolved in the wastewater. By the 45th minute, the microfloc was bigger and darker and doesn't float. After 5mL of flocculant was added, the microfloc turned into macrofloc and floated but did not clot. Then filtered, part of the filtrate is stored for COD analysis. Beforehand, the TDS was measured and the value obtained was 1.67 ppt.

In Trial 17, 0.11g FeSO₄ and 0.5mL H₂O₂ were added because the microfloc did not form when only 0.07g FeSO₄ was added. This is one of the weaknesses of chemical coagulation where the addition of reagents is very dependent on the content of the wastewater, in which pharmaceutical wastewater varies greatly so that is why it is not easy to predict the quantity of reagent that should be added. The water that results from chemical coagulation is quite clear. Then it was filtered and the filtrate was coagulated until the 15th and 30th minutes. The sludge formed was gray, does not clot and is still relatively soft. The filtrate from trial 17 has a TDS value of 5.5 ppt.

In Trials 18-20, no NaOH was added. This aims to maximize the oxidation process. The average sludge produced is lumpy and yellow-orange, while the water produced is clear but still yellowish because the inlet wastewater is indeed yellow-green in color with a TDS value of 2-5 ppt. In Trial 22, the addition of NaOH was not carried out because the pH of the sample had reached 6.7.

In Trial 23, NaOH was added after chemical coagulation using 0.11g FeSO₄ and 0.5mL H₂O₂. This was done because electrocoagulation works maximally at pH 6-7. Previously, the pH of the wastewater was measured and the value obtained was in the range of pH 5-6. After electrocoagulation, clear water was obtained with a TDS value of 2.28 ppt and a pH of 7-8. The increase in pH value is due to the production of OH⁻ ions during the electrocoagulation process.

In Trial 24, electrocoagulation was carried out at a voltage of 20V but the resulting current reached 1-3 Ampere. However, in the previous trials, the ampere value was never more than 1. This is possible because the pollutants contained in the wastewater in Trial 24 were not as many as previous trials which resulted in a small resistance value and a larger electric current. This is in line with Ohm's law which states that the amount of electric current flowing through a conductor is always proportional to the voltage applied to it and is inversely proportional to the value of its resistance.

$$V = IR$$

V = large voltage (V)

I = magnitude of electric current (A)

R = the amount of resistance (Ω) (Britannica, 2013)

But on the other hand, because of the large current that flows, the Al³⁺ produced is also increasing. This is in line with Faraday's law which states that the mass produced in an electrolysis cell system is directly proportional to the electric charge flowing in the cell (Britannica, 2013).

$$m = ZIt$$

m = mass (g)

Z = the equivalent mass

I = Electric current (A)



t = time (s) (Britannica, 2013)

Thus, the Al³⁺ produced exceeds the need for coagulation of pollutants, the Al³⁺ contained in water becomes excessive. After electrocoagulation for 15 minutes, part of the filtrate was taken for electrocoagulation for up to 30 minutes and part of the filtrate was added

with 3 mL of flocculant to coagulate the macrofloc. After 30 minutes, a portion of the filtrate was added with 5mL of flocculant to agglomerate the macrofloc. Each filtrate was used as a sample for the COD analysis.



In Trial 26, 0.6g FeSO_4 and 1mL H_2O_2 was added to maximize coagulation and chemical oxidation. Then electrocoagulation was carried out at a voltage of 20 volts and a current of less than 1A. After 30 minutes, a fairly large, floating, gray microfloc was formed. After adding 4mL of flocculant, the sludge clotted and the water looked clean and clear. Then it is filtered and the filtrate is used as a sample for the COD analysis

Table 3. Results of Waste Treatment Using Al-Carbon Electrocoagulation Method

Trial	Treatment	Time	Information	Photo
21.	0.11g FeSO_4 + 0.5mL H_2O_2		orange color, formed microfloc	
	+ Electro Al-Carbon 20V	-	No changes	

In Trial 21, Al plate was used as the anode and carbon sheet as the cathode. This is so that the oxidation of the Al plate can take place optimally because the carbon is inert. However, when connected to the power supply, nothing changes. This is because the carbon sheets used cannot conduct electricity. Thus, the electrocoagulation process cannot take place.


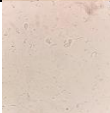
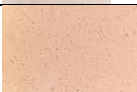
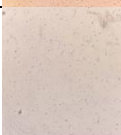
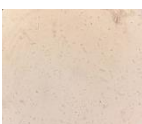



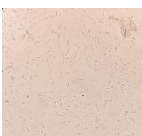

Table 4. Results of Waste Treatment Using Al-Glass Electrocoagulation Method



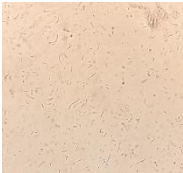

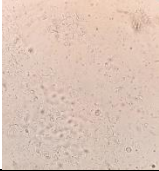
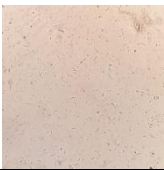
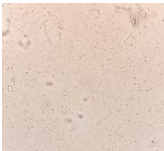


Trial	Treatment	Time	Information	Photo
25.	0.11g FeSO_4 + 0.5mL H_2O_2 + NaOH (pH 6-7)		orange color, formed microfloc	
	+ electro Al-Glass 20V	-	No changes	


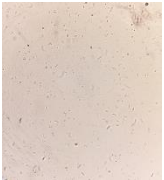
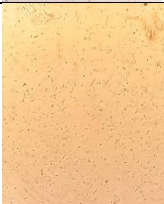

In Trial 25, an Al plate was used as the anode and a petri dish as the cathode. This is so that the oxidation on the Al plate can take place optimally because the petri (glass) dishes are inert. However, when connected to the power supply, nothing changes. This is because the petri dish used cannot conduct electricity, so the electrocoagulation process cannot take place.

4.3. Microbiology

Table 5. Microbial Growth Table

Trial	Day / Date	Development	Visible Microbes
Big Tank	2 / 14-01-21		Aerobacter, Nitrosomonas, Saccharomyces.
	3 / 15-01-21		Aerobacter, Nitrosomonas, Saccharomyces.
	4 / 16-01-21		Aerobacter, Nitrosomonas, Saccharomyces,
	6 / 18-01-21		Aerobacter, Nitrosomonas, Saccharomyces.
	7 / 19-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus
	8 / 20-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus
	9 / 21-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus
	10 / 22-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa
	11 / 23-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus
	13 / 25-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus

	14 / 26-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus
	15 / 27-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus
	16 / 28-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus
	17 / 29-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus
	18 / 30-01-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus
	20 / 01-02-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus
	21 / 02-02-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus
	22 / 03-02-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus, worms
	23 / 04-02-21		Aerobacter, Nitrosomonas, Saccharomyces, Staphylococcus, Bacillus, Protozoa, Streptococcus, worms

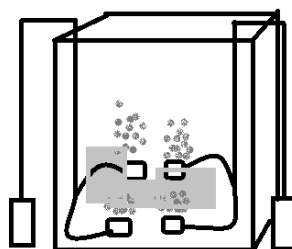
1 bottle of TSB 1 + 5mL of waste	1 / 15-01-21		Aerobacter, Nitrosomonas, Saccharomyces.
	2 / 16-01-21		Aerobacter, Nitrosomonas, Saccharomyces., Staphylococcus, Bacillus, colloidal particles
	3 / 18-01-21		Aerobacter, Nitrosomonas, Saccharomyces., Staphylococcus, Bacillus, colloidal particles
	4 / 19-01-21		Aerobacter, Nitrosomonas, Saccharomyces., Staphylococcus, Bacillus, Streptococcus, colloidal particles

At the time of manufacturing the bacterial culture, Tryptophan Soya Broth media was used as the initial medium full of nutrients for the growth of bacteria. Then sterilization was carried out in an autoclave for 20 minutes to sterilize the bottles filled with the TSB media that would be used for culture. Then the incubation was carried out for 24 hours at 30°C so that the bacteria could grow in the media.

Aeration on a large bacterial culture (bucket) is carried out to supply oxygen so that the bacteria can live. The addition of oat kernels is intended as a source of protein because it contains nitrogen, lactose as a carbon source for bacterial food intake, NaCl and KH_2PO_4 as a source of Na^+ and K^+ ions so that bacteria can grow normally.

On the 6th day, wastewater was added so that the bacteria can adapt to the substances contained in the wastewater. After the wastewater was added, new bacteria, protozoa and worms began to emerge.

Figure 2. Design of Large Bacterial Culture Model



4.4.COD Analysis

Chemical Oxygen Demand or COD is a measure of the amount of oxygen needed to oxidize the dissolved organic matter and particulates in water. COD is usually expressed in terms of oxygen consumed per volume of solution which in SI units is expressed as milligrams per liter (mg / L). The basis of the COD test is that almost any organic compound can be completely oxidized by strong oxidizing agents under acidic conditions.

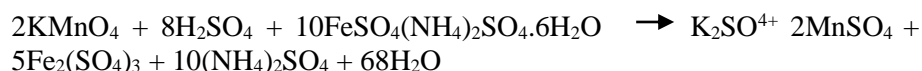
COD is often measured using strong oxidants (e.g. potassium dichromate, potassium iodate, potassium permanganate) under acidic conditions. The concentration of organic matter in the sample is calculated by measuring the amount of the remaining oxidant in the solution. Measurements can use the titration method or spectrophotometry.

4.4.1. Titration

The principle of COD analysis using the titration method is the oxidation of solutes and particulates in water using potassium dichromate or potassium permanganate as an oxidizer and sulfuric acid as a catalyst using reflux. Then a redox titration is performed using ferro ammonium sulfate (FAS) in the principle of the oxidation of FAS by the remaining potassium dichromate or potassium permanganate. The amount of reduced potassium dichromate or potassium permanganate indicates the COD value in milligrams of oxygen consumed per liter of sample.

4.4.1.1 Potassium Permanganate

A total of 1.25 ml of digestion solution (potassium permanganate) was added to 10 ml of free organic water and added with 1 ml of H₂SO₄ and 1-2 drops of ferroin indicator to carry out FAS standardization. The addition of digestion solutions is carried out to oxidize organic substances in water, the addition of H₂SO₄ acts as a catalyst, the addition of FAS was to reduce the remaining potassium permanganate, and the ferroin indicator acts as an indicator. After adding the reagent, the water changes color to purple-pink. After the titration is carried out, the color of the solution should change from purple-pink to colorless indicating the equivalence point. The redox reaction that occurs is



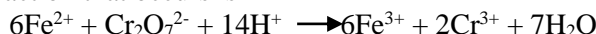
(Huckaba and Keyes, 1948)

However, after titration using FAS, there was still no change in color from purple-pink to clear. This is possible because there is no addition of AgSO₄ and HgSO₄ which functions to bind the Cl⁻ intruder, which means that the Cl⁻ intruder is still present in the solution which results in the disruption of the titration process.

4.4.1.2 Potassium Dichromate

A total of 1.25 ml of digestion solution (potassium dichromate) was added to 10 mL water and added with 1 ml of H₂SO₄ and 1-2 drops of ferroin indicator to carry out the FAS standardization. The addition of the digestion solution was to oxidize the organic substances in water, the

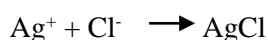
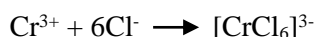
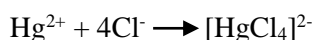
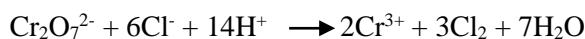
addition of H₂SO₄ functions as a catalyst, the addition of FAS was to reduce the remaining potassium dichromate, and the ferroin indicator acts as an indicator. After adding the reagent, the organic free water changes color to green-blue. After the titration is carried out, the color of the solution should change from green-blue to brick red which indicates the equivalence point. The redox reaction that occurs is



(Government of Great Britain et al., 1980)

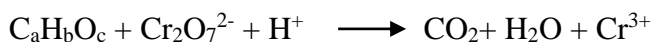
However, after titrating using FAS, there was still no change in color from green-blue to brick red even though the ferroin indicator was added up to 5 drops. This might be due to no addition of AgSO₄ and HgSO₄, which function was to bind the Cl⁻ intruder. Thus, the Cl⁻ intruder is still present in the solution which results in disruption of the titration process.

Cl⁻ intruding reaction



(Government of Great Britain et al., 1980)

A total of 6 ml of digestion solution (potassium dichromate) and 14 ml of H₂SO₄ were added to each 10 ml of free organic water, wastewater samples, electrocoagulated wastewater samples, and water samples treated with microbes. After all reagents are added and closed heating (reflux) is carried out for one night, the free organic water is still yellow which is the color of potassium dichromate, the wastewater sample turns dark green, the electrocoagulated wastewater sample turns green, and the microbe-treated wastewater sample turns green-black. The color change (green) indicates that the potassium dichromate has been reduced since it oxidizes the dissolved organic matter in the sample, which means that there is a lot of organic matters that are still dissolved in the sample. The redox reaction of potassium dichromate with organic substances is



(Juliasih and Amha, 2019)

In water samples treated with microbes, the color of the sample changes to green-black which is most likely due to the presence of microbes contained in the sample which are then oxidized by the potassium dichromate.

The refluxed sample is then added with 1-2 drops of the ferroin indicator. Then the titration was carried out using FAS. However, there was no change in color from green-blue to brick red. This is possible because prior to analysis, the samples were added with HCl instead of H₂SO₄. The addition was done to prevent oxidation by air. However, this cause an increase in the dissolved Cl⁻ intruder in the sample, which interfered with COD analysis. In

addition, the failure of the analysis can also be caused by the potassium dichromate that has completely oxidize the organic substances contained in the sample, so there is no more potassium dichromate left to oxidize FAS. Therefore, the addition of potassium dichromate was carried out until the color of the solution turned yellow. However, the analysis was still unsuccessful, which might happen due to the presence of Cl^- intruder.

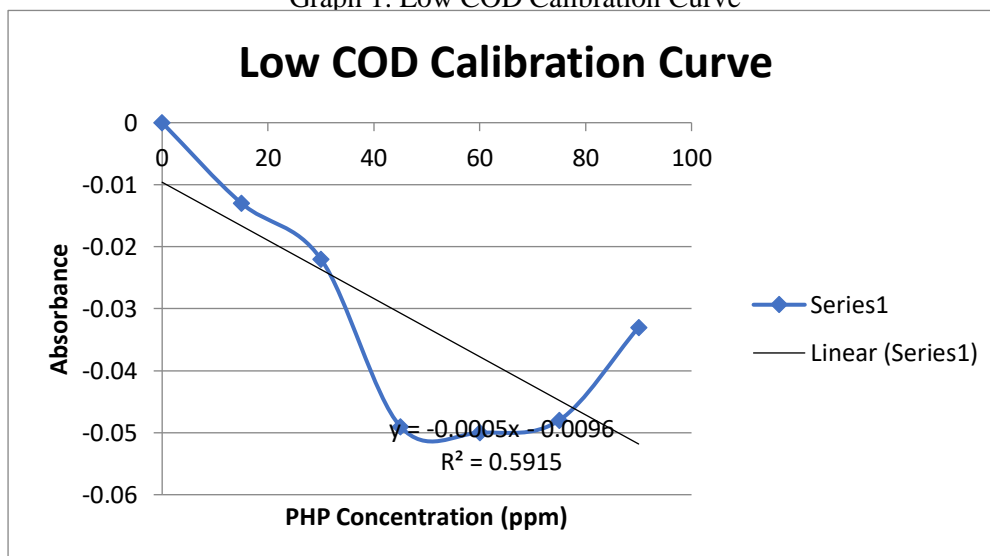
4.4.2. Spectrophotometry

The principle of COD analysis using the spectrophotometric method is the oxidation of solutes in the sample by $\text{Cr}_2\text{O}_7^{2-}$ and the absorption of visible light at the wavelength of 420nm or 600nm. Samples with COD values <90 ppm can absorb light at a wavelength of 420nm, in which the wavelength is the absorption wavelength of $\text{Cr}_2\text{O}_7^{2-}$, while samples with a COD value of 100-900 ppm can absorb at a wavelength of 600nm where the wavelength is the absorption wavelength of Cr^{3+} .

4.4.2.1 Low COD

Potassium Hydrogen Phthalat (PHP) which is equivalent to 500 ppm is diluted into standard solutions with different concentrations, namely 0; 15; 30; 45; 60; 75; 90 ppm, in which absorbance will be used as the standard curve. Then, 1.5 ml of low concentration digestion solution (potassium dichromate) and 3.5 ml of H_2SO_4 were added in each 2 ml of standard solution under closed reflux for 2 hours. The digestion solution was added as an oxidizer for the solute in the sample and H_2SO_4 acts as a catalyst. Reflux is carried out for 2 hours so that the reaction occurs completely. In samples with low COD levels, less $\text{Cr}_2\text{O}_7^{2-}$ were reduced to Cr^{3+} than those that were still in the form of $\text{Cr}_2\text{O}_7^{2-}$. Therefore, absorbance was carried out at a wavelength of 420nm where the wavelength was the absorption wavelength of $\text{Cr}_2\text{O}_7^{2-}$.

Graph 1. Low COD Calibration Curve

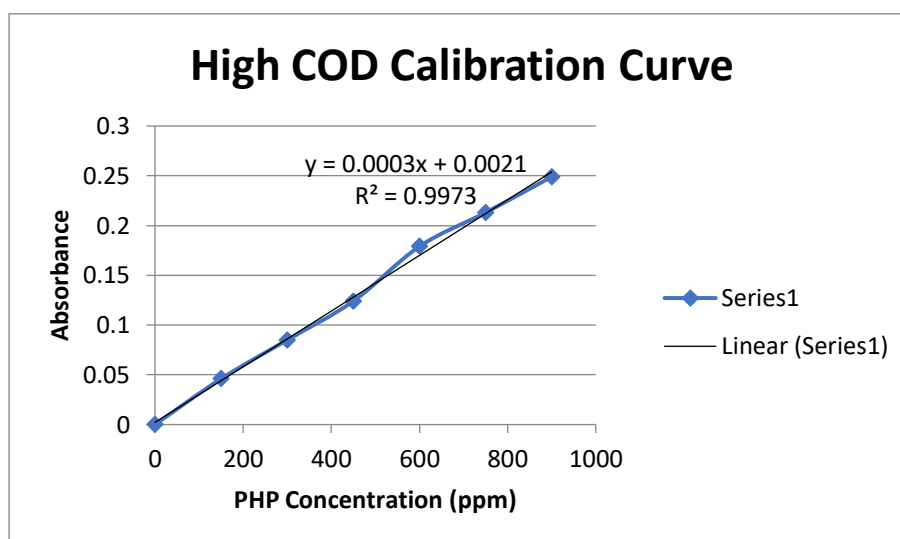


After spectrophotometry, it was found that all absorbances had negative values and decrease with increasing PHP concentration. This is because the higher the PHP concentration, the more $\text{Cr}_2\text{O}_7^{2-}$ is reduced to Cr^{3+} . However, the regression results are still less than 0.995, which indicates that the relationship between absorbance and PHP concentration is not linear. This can be due to the absence of the addition of AgSO_4 and HgSO_4 so there are still Cl^- intruders present. The line equation is $y = 0.0005x + 0.0095$.

4.4.2.2 High COD

Potassium Hydrogen Phtalat (PHP) which is equivalent to 1000 ppm is diluted into standard solutions with different concentrations, namely 0; 150; 300; 450; 600; 750; 900 ppm, in which the absorbance will be used as the standard curve. Then 1.5 ml of high concentration digestion solution (potassium dichromate) and 3.5 ml of H_2SO_4 were added in each of 2 ml of standard solution under closed reflux for 2 hours. The digestion solution was added as an oxidizer for the solute in the sample and H_2SO_4 acts as a catalyst. Reflux is carried out for 2 hours so that the reaction occurs completely. In samples with high COD levels, more $\text{Cr}_2\text{O}_7^{2-}$ were reduced to Cr^{3+} than those that were still $\text{Cr}_2\text{O}_7^{2-}$. Thus, the absorbance was carried out at a wavelength of 600nm where the wavelength was the absorption wavelength of Cr^{3+} .

Graph 2. High COD Calibration Curve



After spectrophotometry, it was found that all absorbances had positive values and increased with increasing KHP concentrations. This is because the higher the KHP concentration, the more $\text{Cr}_2\text{O}_7^{2-}$ is reduced to Cr^{3+} . The regression result of the calibration curve was 0.9973 which indicates that there is an accurate linearity relationship between the absorbance and the KHP concentration. The line equation is $y = 0.0003x + 0.0021$.

COD analysis was carried out on Trial 15, Trial 23, Trial 24, Trial 26, morning inlet wastewater, and evening inlet wastewater.

Table 6. COD Analysis Results

Trial	COD value
15	236.33 ppm
23	209.67 ppm
24 (15 minutes)	646.33 ppm
24 (30 minutes)	573 ppm
26	246.33 ppm
Morning Inlet Wastewater	329.67 ppm
Evening Inlet Wastewater	490 - 800 ppm

The best COD value obtained was 209 ppm with the percentage of reduction in COD values of 59% -70%. The COD value of trial 24 was higher than that of the inlet wastewater. This is possible because during the electrocoagulation process, the electric current that flows is 1-3 A. This causes too much Al^{3+} being dissolved in the sample.

Table 7. Conditions of PT. Etercon Pharma

No.	Parameter	Levels
1.	pH	6-7
2.	COD	1500-3000 ppm
3.	BOD	150-300 ppm
4.	TSS	50-90 ppm

The COD value of the morning inlet wastewater is lower than the afternoon inlet wastewater because the production process happened in the morning is not as much as that in the afternoon and evening. Thus, the dissolved materials in the morning inlet wastewater tends to be less than that of the afternoon inlet wastewater. The COD value of the afternoon inlet waste is smaller than usual, 1500 ppm, because at the time of taking the waste sample, the condition was moderate or after raining, so the inlet wastewater was diluted by the rainwater.

4.5. Fe Electrocoagulation and Al Electrocoagulation Comparison

Table 8. Comparison of Fe-Fe Electrocoagulation Results with Al-Al Electrocoagulation

Electrocoagulation	Voltage	Ampere	TDS	Sludge	COD
Fe-Fe	6V	0.2–0.5 A	-	<ul style="list-style-type: none"> 15 minutes: microfloc Soft sludge 	-

Fe-Fe	12V	0.2-0.5 A	-	<ul style="list-style-type: none"> • 5 minutes: Microfloc • Clumped sludge 	-
Al-Al	20V	0.5-0.8 A	400ppm - 2.28 ppt	<ul style="list-style-type: none"> • 5 minutes: microfloc floats • 10-30 minutes: clear water, floating microfloc • Clumped sludge 	200-300 ppm
Al-Al	20V	1-3 A	-	<ul style="list-style-type: none"> • 5 minutes: Microfloc floats • 10-30 minutes: clear water, floating microfloc • Soft sludge 	573 ppm

In electrocoagulation using Al-Al plates, the coagulation process takes place earlier than the electrocoagulation using Fe-Fe plates. This is because Al is more easily oxidized than Fe. Thus, in the same period of time, more Al ions are produced than Fe ions. However, this results in an increase in the COD value when the current is too high.

When the current is below 1 A, the amount of Al ions produced is still in balance with the number of dissolved pollutants that need to be oxidized. However, when the current is higher than 1 A, the amount of Al ions produced is more than the amount of dissolved pollutants that need to be oxidized. Thus, when using the Al-Al plate, make sure that the electric current is lower than 1 A. In table 7, it is shown that the value of the Total Dissolved Solids is not linear with the COD value. This is because the dissolved solids could be in the form of salts such as NaCl or other compounds that can't be oxidized, which therefore does not affect the COD value.

CHAPTER V.

CLOSING

5.1 Conclusion

- The electrocoagulation method is always stable in purifying the wastewater.
- Al-Al electrocoagulation is more effective than the Fe-Fe electrocoagulation.
- AgSO_4 and HgSO_4 are indispensable in COD analysis.
- The linearity of the High COD calibration curve is 0.9973 with the line equation $y = 0.0003x + 0.0021$.
- The COD after the electrocoagulation treatment was in the range of 200-300 ppm.
- The decrease in COD value is in the range of 59% -70%.
- Wastewater of PT. Etercon Pharma contains a lot of surfactants and dissolved materials.
- Waste of PT. Etercon Pharma varies depending on the ongoing production process.
- The TDS value does not affect the COD value.

5.2 Recommendation

- When performing electrocoagulation using an Al-Al plate, keep the electric current below 1 A.
- Testing must be carried out carefully and carefully so that the results obtained are maximum.
- Try to do a COD analysis on the same day or the next day for maximum results.
- Add NaOH to keep the pH in the range of 6-7 when doing electrocoagulation.

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ATTACHMENT

1. Calculation Appendix

- **COD Trial Value 15**
Abs: 0.073
 $y = 0.0003x + 0.0021$
 $0.073 = 0.0003x + 0.0021$
 $x = 236.33 \text{ ppm}$
- **COD Trial Value 23**
Abs: 0.065
 $y = 0.0003x + 0.0021$
 $0.065 = 0.0003x + 0.0021$
 $x = 209.67 \text{ ppm}$
- **COD Trial Value 24**
 - **15 minutes**
Abs: 0.196
 $y = 0.0003x + 0.0021$
 $0.196 = 0.0003x + 0.0021$
 $x = 646.33 \text{ ppm}$
 - **30 minutes**
Abs: 0.174
 $y = 0.0003x + 0.0021$
 $0.174 = 0.0003x + 0.0021$
 $x = 573 \text{ ppm}$
- **COD Trial Value 26**
Abs: 0.076
 $y = 0.0003x + 0.0021$
 $0.076 = 0.0003x + 0.0021$
 $x = 246.33 \text{ ppm}$
- **Morning Inlet Wastewater COD Value**
Abs: 0.101
 $y = 0.0003x + 0.0021$
 $0.101 = 0.0003x + 0.0021$
 $x = 329.67 \text{ ppm}$
- **Evening Inlet Wastewater COD Value**
 - **Rain**
Abs: 0.151
 $y = 0.0003x + 0.0021$
 $0.151 = 0.0003x + 0.0021$
 $x = 496.33 \text{ ppm}$
 - **Rain**
Abs: 0.158
 $y = 0.0003x + 0.0021$
 $0.158 = 0.0003x + 0.0021$
 $x = 519.67 \text{ ppm}$

- **Not raining**
 Abs: 0.242
 $y = 0.0003x + 0.0021$
 $0.242 = 0.0003x + 0.0021$
 $x = 799.67 \text{ ppm}$
- **The percentage of COD reduction**

$$\%COD = \frac{COD \text{ Wastewater} - COD \text{ Clear}}{COD \text{ Wastewater}} \times 100\%$$
- **Rain**

$$\%COD = \frac{519.67 - 209.67}{519.67} \times 100\%$$

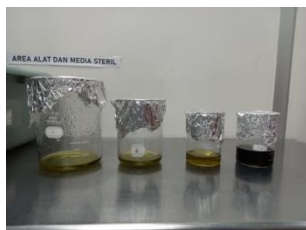
$$\%COD = 59.7\%$$
- **Not raining**

$$\%COD = \frac{799.67 - 236.33}{799.67} \times 100\%$$

$$\%COD = 70.45\%$$

2. Image attachment

Sample Analysis COD



Sample Titration (blank)



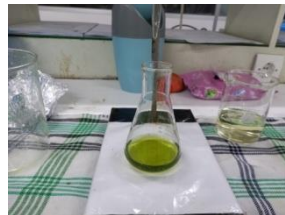
Sample Titration
(after addition of ferroin)



Sample Titration Results



Sample Titration



Spectroscopy Sample



Inlet Wastewater
Condition

