Development of Bio-based Coagulant for the Removal of Fatty Matter from Soapy Wastewater with Electrocoagulation Process

Mebrahtom Gebrerufael Fisha

Thesis Paper Submitted to the Department of Chemical Engineering

School of Mechanical, Chemical, and Material Engineering

Presented in Partial Fulfillment of the Requirement for the Degree of Master's in Chemical Engineering (Specialization in Process Engineering)

Office of Graduate Studies

Adama Science and Technology University

Adama, Ethiopia

September 2022

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Advisors

Dr. Melaku Tesfaye

Co-Advisor

Dr. Tatek Temesgen

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Declaration

I hereby declare that this Master Thesis entitled "**Development of Bio-based Coagulant for the Removal of Fatty Matter from Soapy Wastewater with Electrocoagulation Process"** is my original work. That is, it has not been submitted for the award of any academic degree, diploma, or certificate in any other university. All sources of materials that are used for this thesis have been duly acknowledged through citation.

Mebrahtom Gebrerufael _____ _____

Name of the student Signature Date

Recommendation

We, the advisor(s) of this thesis, hereby certify that we have read the revised version of the thesis entitled **"Development of Bio-based Coagulant for the Removal of Fatty Matter from Soapy Wastewater with Electrocoagulation Process"** prepared under our guidance by Mebrahtom Gebrerufael is submitted in partial fulfillment of the requirements for the degree of Masters of Science in Chemical Engineering (Specialization Process Engineering). Therefore, we recommend the submission of a revised version of the thesis to the department following the applicable procedures.

_____	_____	_____
Major Advisor	Signature	Date
_____	_____	_____
Co-advisor	Signature	Date

Approval Page

We, the advisors of the thesis entitled **"Development of Bio-based Coagulant for the Removal of Fatty Matter from Soapy Wastewater with Electrocoagulation Process"** and developed by Mebrahtom Gebrerufael certify that the recommendation and suggestions made by the board of examiners are appropriately incorporated into the final version of the thesis.

Major Advisor	Signature	Date
Co-advisor	Signature	Date

We, the undersigned, members of the Board of Examiners of the thesis by _____have read and evaluated the thesis entitled **"Development of Bio-based Coagulant for the Removal of Fatty Matter from Soapy Wastewater with Electrocoagulation Process"** and examined the candidate during the open defense. This is, therefore, to certify that the thesis is accepted for partial fulfillment of the requirement of the degree of Master of Science in Chemical Engineering (Specialization in process engineering).

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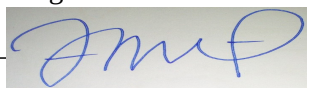
Dr. Alemu Gizaw		28-10-2022
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LIST OF ABBREVIATIONS

ANOVA	Analysis of variance
BBD	Box-Behnken statistical experiment design
BOD	Biological oxygen demand
COD	Chemical Oxygen Demand
DC	Direct current supply
EC	Electro-coagulation
FAO	Food and agricultural organization
FTIR	Fourier transform infrared spectroscopy
GLY-LAC	Glycerol lactate
HRT	Hydraulic retention time
LAOLG	Lactic acid oligomer
MO	Moringa olifera
NTU	Nephelometric Turbidity Unit
PAC	Poly-aluminum chloride
PH	potential of Hydrogen
RSM	Response surface methodology
TDS	Total Dissolved Solid
TFMR	Total fatty matter removal
TSS	Total suspended solid
WHO	World health organization

ABSTRACTS

Soapy wastewater is one of the most difficult environmental issues because it contains high dissolved solids, high suspended solids, chemical oxygen demand (COD), turbidity, and toxic chemicals. The direct discharge of this wastewater without proper treatment into the water bodies (i.e. lakes, rivers, etc.) pollutes the water system directly or indirectly. This indicates the need to develop efficient treatment methods which are environmentally viable. To solve this problem, coagulation/flocculation with the help of electrocoagulation is one of the most effective and simple techniques to remove fatty matter and colloidal and suspended solids from soapy wastewater. The soapy wastewater technically needs adding either synthetic or natural coagulant. However, the use of chemical coagulants has been criticized to have limitations due to environmental impacts. Recently, there is an interest to replace chemical coagulants with natural materials. The overall objective of this study is framed to develop a bio-based coagulant, buildup an electrocoagulation setup, and investigate its removal efficiency of fatty matter from soapy wastewater. The removal efficiency of the coagulant was tested for its removal of fatty matter, COD, TSS, turbidity, and TDS before and after the treatment, and FTIR was used to characterize the specific functional groups. The treatment process was carried out using a jar test at different settling time(1-16hr), coagulant dose(1-5g/l) and initial soap concentration(0.6-1.2) in the bio-based coagulation and at different voltage(10-30v), reaction time(30-120min), enter-electrode distance(2-4cm) and settling time(30-180min) in the electrocoagulation with constant of other parameters. The soap wastewater was characterized before and after treatments. The raw wastewater had a TDS of 1450 mg/l. The TFMR, COD, and turbidity values of the effluent were on average 74.26%, 1500 mg/l, and 165NTU respectively before treatment. The experimental design and data analysis were done using design expert 13 software. The following experimental result was obtained. From the analysis, it was found that the optimal removal efficiency of TFM 93.63, COD 85.03%, TDS 72.43 %, and turbidity 97.503 % recorded at coagulant dose 4.221g/l, initial soap concentration 1.104g/l, voltage 24.07v, reaction time 111.7 min, inter-electrodes distance 2.33 cm and settling time 145.9 min. It was observed that the process of electrocoagulation gives better performance to the synthesized bio-based coagulant in the reduction of TFM. From the study result of characterization of the soap wastewater, it can be concluded that soap waste water pollutants can be efficiently removed using the synthesized bio-based coagulant with the help of electro-coagulation.

CHAPTER ONE

1. INTRODUCTION

1.1. Background

Now a day the increasing needs of society in the growing economy and industrial development cause to increase in the pollution of water. Wastewater from homes and industries is one of these pollution sources. Many industries and laundry consume fresh water and release wastewater as a depletion (OP Sahu, Gupta, Chaudhari, & Srivastava, 2015). All types of water pollution are harmful to the environment, people, and animals(Mickova, 2015). Wastewater contains a variety of pollutants that can be categorized according to their properties: total suspended solids (TSS), inorganic, organic, and microbial particles (Gholami Yengejeh, Jafari Mansoorian, Majidi, Yari, & Khanjani, 2017). Fatty substances, greases, surfactants, oils, insecticides, phenols, and other substances are examples of organic components. Inorganic components may consist of pH, sulfur, chlorides, alkalinity, toxic compounds, etc. (Kulkarni, 2017).

According to WHO, handwashing with soap can be considered a vital public health action for preventing coronavirus spread. Each time you wash your hands, you should spend close to 20 to 30 seconds doing it. A single hand washes with rubbing soap uses almost 2L of water when the tap is closed, and this amount can increase to 4L when the tap is left open. Due to the wastewater's contamination with soap surfactants, this significant rise in wastewater production of 15–18% might result in a decline in water quality. When wastewater is treated in this manner, some issues and difficulties arise, and if water is discharged straight into freshwater bodies without any treatment, there may be negative consequences on the environment and human health(Juela Quintuña, 2020). Large amounts of foam may be produced on the water's surface as a result of the high concentration of soap in freshwater bodies, which can slow down the rate at which oxygen permeates the water. Because of this, aquatic organisms are unable to effectively absorb dissolved oxygen. These elements in the aquatic ecosystem are in charge of changing the pH, turbidity, salinity, and temperature of the water, among other chemical and physical properties (Kim et al., 2021). Fish suffer biochemical and physiological impairments as a result of the deteriorating water quality, which can also have an impact on how much dissolved oxygen they consume and cause serious gill damage (Aditi, Hafizah, & Hermansyur, 2021; Ge et al., 2018).

In addition, aquatic plants may be harmed by these various pollutants in water. Eutrophication can be triggered by detergent discharge into freshwater bodies of water. Corals and algae in particular are thought to be seriously threatened by this process. Sea species that depend on sea plants for spawn, shelter, food, and protection are at risk when their numbers decline. (Bandala et al., 2021). Detergents in the aquatic environment can finally enter the soil and bring adverse effects on flora in the area, especially on the germination of plants(Issayeva, Zh, Zhymadullayeva, & Balgabekova, 2015). The soil structure is destroyed by a high surfactant concentration, endangering the health of the plant (Sawadogo & Mureithi, 2014). Detergents cause an elevation in pH in the soil, which causes the soil's components to separate. Nearly 5 to 9 is the ideal pH range for plants, and any variations, including pH increases and decreases, pose a threat to the soil's biological activity levels(Chirani, Kowsari, Teymourian, & Ramakrishna, 2021).Soap wastewater needs to be treated in particular in order to avoid environmental damage of ground water and surface water resources.

There are many different types of treatment methods, including biological treatment methods, physical treatment methods, coagulation/flocculation treatment methods, electrocoagulation treatment methods, catalytic oxidation, adsorption processes, ion exchange processes, biological processes, membrane separation processes, advanced oxidation processes, ultrafiltration and reverse osmosis, and photo catalysis. Despite being costly and having numerous drawbacks, the majority of these techniques are effective(Aziz & Ali, 2018). Coagulation/flocculation and electrocoagulation were utilized in this investigation.

Coagulation and flocculation are used to separate the suspended solids and dissolved particles portion from the water. Suspended particles vary in source, charge, particle size, shape, and density. The correct application of coagulation and flocculation depends upon these factors. Suspended solids in water have a negative charge and since they have the same type of surface charge, they repel each other when they come close together. Therefore, suspended solids will remain in suspension and will not clump together and settle out of the water, unless proper coagulation and flocculation are used. Coagulation and flocculation occur in successive steps, allowing particle collision and growth of the flock. This is then followed by sedimentation. If coagulation is incomplete, the flocculation step will be unsuccessful, and if flocculation is incomplete, sedimentation will be unsuccessful. Flocculation, a gentle mixing stage, increases the particle size from submicroscopic micro-flock to visible suspended particles. Micro-flock particles

collide, causing them to bond to produce larger, visible flocks called pin-flocks. Floc size continues to build with additional collisions and interaction with added inorganic polymers (coagulants) or organic polymers. Macro-flocks are formed and high molecular weight polymers, called coagulant aids, may be added to help bridge, bind, and strengthen the flock, add weight, and increase the settling rate. Once the flock has reached its optimum size and strength, water is ready for sedimentation (Bratby, 2016). To balance the negative charges on non-settable solids, coagulant chemicals having charges opposite those of the suspended solids are introduced to the water (such as clay and color-producing organic substances). The little dispersed particles might cling together once the charge has been neutralized. Micro-flocks are these slightly larger particles that are invisible to the unaided eye. The water should be clear around the newly generated micro-flocks. If not, the particle's charge has not entirely been neutralized and coagulation has not taken place. In the coagulation-flocculation treatment method, different chemicals such as aluminum sulfate, ferric chloride, and calcium chloride are used for water treatment.

Aluminum sulfate, sometimes known as "alum," has likely been used for water treatment for decades and is the most popular metal coagulant. Simple metal coagulants (conventional) are used widely, in part because they are inexpensive and have an easier application process. They do, however, have several drawbacks, including the requirement for pH adjustment before or after treatment, sensitivity to temperature changes, the need for higher dosages because charge neutralization is typically insufficient, sensitivity to sample-specific characteristics and composition, as well as excessive production of sludge (Gregory, 2005). The two main groups of coagulants that are frequently utilized are organic and inorganic coagulants. This study focuses on bio-based coagulants made from glycerol, a significant byproduct of the biodiesel industry. Glycerol is a polyol molecule with many applications in the renewable industry, it is non-toxic, biodegradable, colorless, and highly stable, with a high density of 1.26 g 3/cm3, a pH of 8 to 10, a melting point of 18.2 °C, and a boiling point of 290 °C under normal atmospheric conditions.

To remove water contaminants, electrocoagulation is a complicated process with numerous synergistic mechanisms at work (metals, anions, organic compounds, etc.). This method's primary benefit is the on-site manufacturing of disinfection chemicals in the same electrochemical cell. Numerous research has suggested using the electro-coagulation approach to reduce the issue with chemical coagulants. An advanced and cutting-edge technique called electrocoagulation involves direct interaction between the ions of the sacrificial metal anode and the water contaminants.

Similar to the conventional approach where coagulation chemicals are used, the dissolved metal ions from the sacrificial anode are released into the wastewater and coagulated with contaminants in the wastewater (Mickova, 2015). Using electric energy and sacrificial metallic plates (electrodes) in place of pricey chemical reagents, the process of electrocoagulation concurrently eliminates heavy metal ions, suspended particles, organic mixtures, and many other water contaminants(Zaleschi, Teodosiu, Cretescu, & Rodrigo, 2012). Additionally, it stops the development of secondary contaminants(DOHARE, JAGWANI, GARG, & KUTIYA). For the treatment of soap wastewater, the Electrocoagulation Process (PEP) was utilized (Benhadji & Ahmed, 2019). The results of their investigation into the electrocoagulation of wastewater at applied voltages of 5, 10, 15, and 20 Volt with varying electrolysis periods revealed that aluminum wire netting electrodes have a higher rate of degrading soap wastewater (Zetriuslita & Athar, 2014). Aluminum plates are used in the electrocoagulation procedure to remediate laundry effluent. The effectiveness of the therapy is examined in several parameters, including the distance between the electrodes, voltage, hydraulic retention time (HRT), and the number of aluminum plates between the anode and cathode(Janpoor, Torabian, & Khatibikamal, 2011). The amount of contaminants in laundry waste water is reduced to a safe level by the aluminum hydroxide produced in the cell. It is found that, under the right circumstances, electrocoagulation is more successful at treating laundry wastewater than other treatment techniques (Janpoor, et al., 2011).

The purpose of this study was to investigate the efficiency of the removal of fatty matter from soap under variable factors such as settling time, the dosage of the coagulant, and initial concentration of soap and with various factors of electrocoagulation such as voltage, inter-electrodes distance and reaction time which may affect the efficiency of the removal of pollutants by using a synthesis coagulant and mono-polar aluminum electrodes of electro-coagulation process.

1.2. Statement of the problem

There are several wonderful cleaning soaps on the market nowadays. The various chemical ingredients that make up these laundry soaps include surfactants, builders, fillers, bleaches, enzymes, optical brighteners, anti-re deposition agents, fragrance, and color. These soaps work by absorbing ultraviolet light and reflecting blue light to give our clothes the appearance of being whiter or brighter. The chemicals employed in manufacturing operations are extremely dangerous, and poisonous, and have a negative impact on both the environment and worker health. Most soaps and detergents such as nonylphenol ethoxylate harm the kidneys and liver and cause hormonal issues, growth and metabolism disruptions, cancer, and other difficulties. In addition to causing skin rashes and diarrhoea, the common detergent ingredient phosphate can also degrade the air quality. Different kinds of wastes, mostly liquid and solid wastes, are produced as a result of manufacturing activities. It was discovered that liquid waste is very soluble in water and hardly biodegrades, making it very difficult to treat. Surfactants are among the most common xenobiotics that considerably increase the contamination level of all types of wastewater and sewage. The increased pollution caused by wastewater from factories that make detergents and soaps during washing operations is one of the main effects of the high level of surfactant production. Because the soap product and its ingredients may be relatively hazardous to aquatic life, soap effluents can cause serious environmental issues.

There are several methods for treating wastewater, but most of them are either expensive or harmful to the environment. One of the best and most efficient ways to treat wastewater is by coagulation. Since ancient times, chemical coagulants like aluminum salt, ferric salt, and synthetic polymers have been employed extensively in the purification and treatment of water. However, these chemical coagulants discharge toxic compounds into the environment that are harmful to human health. Additionally, they produce large volumes of sludge, are relatively expensive, ineffective in low-temperature water, and drastically alter the pH value of the treated water. They also induce human diseases like "Alzheimer's"(Werkneh, Beyene, & Osunkunle, 2019). In order to reduce these problems, it is necessary to search for other alternative coagulants. Nowadays, attentions are focused on searching for renewable resources. This study is carried out to develop a new coagulant by mixing lactic acid and glycerol and using an electrocoagulation process to increase the coagulation process.

1.3. Objective

1.3.1. General objective

The general objective of this work is to synthesis bio-based coagulants from glycerol lactate and study the effect of electrocoagulation on the removal efficiency of fatty matter from soapy wastewater.

1.3.2. Specific objective

- Synthesis, purify, and characterize bio-based coagulant.
- Optimize and study the effect of initial concentration, settling time, and dose of the coagulant on the removal efficiency of fatty matter.
- Study the effect of voltage, reaction time, inter electrodes distance, and settling time on the removal efficiency of fatty matter
- Performance evaluation of the bio-based coagulant for the optimized parameters on the reduction of COD, TDS, and turbidity.
- Comparison of the bio-based coagulant with conventional coagulant

1.4. Significance of the Study

Due to the dramatic growth of biodiesel industries today, the Conversion of glycerol into valuable products has been a very interesting area in recent years. Glycerol is bio-based, compatible, non-toxic, and a less costly industrial fluid. This study will introduce the application of bio-based glycerol coagulant as an alternative to conventional coagulants. Generally, once the research is done successfully and implemented, it is expected to:

- Introduce alternative treatment method
- Reducing the pollution of water by wastewater discharged without enough treatment
- It can avoid any conflict that may arise due to discharge limits from the government and society.
- Recycling the wastewater discharged for reuse
- It gives hints for the researchers, who will have plans to study electro-coagulation and bio-based coagulants.

1.5. Scope of the study

This thesis focused on the synthesis and characterization of bio-based coagulants with electrocoagulation for the removal of fatty matter from wastewater. FTIR was used for characterized the synthesized bio-based coagulant and for the interaction of the coagulant and the pollutant. Furthermore, studies of various effects such as adsorbent dose, initial soap concentration, settling time, reaction time, voltage and inter-electrode distance, and contact time on fatty matter removal by the synthesized coagulant.

CHAPTER TWO

2. LITERATURE REVIEW

2.1. Introduction

Water usage and pollution both rise sharply along with the world population's rapid growth. Untreated industrial and municipal wastewater discharges often contaminate rivers, canals, river mouths, and other bodies of water. Water is a naturally occurring resource that is both abundant and vital to both animal and plant life. (Önder, Koparal, & Öğütveren, 2007). It has long been known that waste from industry and urban areas affects the quality of surface and groundwater.

Industrial waste is frequently dumped into rivers and streams around the world. According to (Ezechukwu, Ugochukwu, Egbuonu, & Chukwuka, 2004), the decline in water quality has a negative impact on both humans and the aquatic ecosystem. As a result, pollution of water bodies is constantly rising as a result of industrial use and urbanization. Today, there is significant growth in the manufacture and use of chemical compounds that enter the environment, such as surfactants, on a global scale. According to(Önder, et al., 2007), many of these substances are biologically inert and pose a risk to environmental protection.

Soap was first mentioned as an olive oil by-product in AD 985 by Muslim geographer al-Maqdisī. Records of Holy Land soap being traded in Egypt appear in 11th-century texts from the Cairo genizah. Under the Crusaders, soap was exported out of Jaffa to Europe along with glass and pottery vessels. Soap from the city of Nablus is mentioned on a Mamluk inscription from AD 1285 set in a Muslim hosting and worship facility in Jer- Salem. In approximately AD 1300, Arab geographer al-Dimashqi reports soap export to Egypt, Syria, and the Mediterranean islands. The industry continued to prosper following the Ottoman takeover in 1517. The main production centers in the Holy Land were Nablus and Jerusalem, yet workshops could also be found in several other towns, mostly in the central part of the country(Arbel, 2021). Surfactants have replaced soap in cleaning and laundry formulations in virtually all countries with industrialized societies (Yapijakis & Wang, 2004).

Soaps are used to aid in cleaning equipment and surface by restaurants, dairy farms, and food processing plants and are among the basic constituents of organic pollutants in domestic as well as industrial wastewater causing great environmental damage. Due to their high volume use, detergent chemicals have the potential for broad-scale release into the aquatic and terrestrial

environments. It is quite frequent, especially in rural areas with no sewerage system, and municipal and industrial wastewater containing detergent infiltrates into soil and ground waters from septic tanks. Surfactants can reach humans, animals, and plants from the ground waters used as drinking water supplies. Besides the toxic effects of surfactants, their existence in waters even under the toxic level causes many adverse effects on biological life. They cause pathological, physiological, and biochemical effects on aquatic animals. In aquatic plant species, they have effects such as the break-up of the chlorophyll–protein complex, death of the cell by damaging the membrane, and delay in metabolism and growth(Önder, et al., 2007). To reduce those effects use different methods of waste water. A wide range of wastewater treatment techniques has some associated advantages and disadvantages are prevailing (Ali & Yaakob, 2012). Most commonly wastewater treatments involve biological treatment such as nitrification, denitrification, and phosphorus removal, physio-chemical treatment such as adsorption, ion exchange, precipitation, reverse osmosis, coagulation, and electrocoagulation (Ronke, Saidat, & Abdulwahab, 2016).

Coagulation is a process where the pollutants and suspended particles bring into the sediment by collision with counter particles and get agglomerated to form an insoluble agglomerate complex(Syam Babu, Anantha Singh, Nidheesh, & Suresh Kumar, 2020). Coagulation-flocculation is a commonly used physicochemical method in the treatment of metal-bearing industrial wastewater because it removes colloidal particles, some soluble compounds, and very fine solid suspensions initially present in the wastewater by destabilizing and forming flocs (Burke, Singh, & Theodore, 2000). This process destabilizes the colloidal suspension of the particles with coagulants and then causes the particles to agglomerate with flocculants. After that, it accelerates separation and, thereby, clarifies the effluents (Tripathy & De, 2006). Coagulation is normally accomplished through the use of chemicals known as coagulants. Coagulants used in wastewater treatment can be inorganic (such as aluminum sulfate and poly aluminum chloride), synthetic organic polymers (for example polyacrylamide derivatives and polyethyleneimine), or naturally occurring coagulants (such as chitosan and microbial coagulants). On the other hand, the process of forming large agglomerates of particles in suspension or small agglomerates already formed as a result of coagulation through gentle stirring or agitation is called flocculation. Coagulation-flocculation is a widely used method for wastewater treatment especially if the wastewater is discharged into surface water(Ronke, et al., 2016).

The coagulant produced by the electrochemical process is called electrocoagulation (EC). The usually used electrodes in the process are iron, aluminum, mild steel, and stainless steel(Syam Babu, et al., 2020). When these electrodes are subjected to the electric supply the scarification of the anode leads to coagulant formation in the system, these coagulants further results in coagulation and flocculation. Treatment of wastewater by EC is 10 to 15% higher as compared with chemical coagulation(Zaleschi, et al., 2012) and also sludge generated from the process is less as compared with chemical coagulation. Another advantage of this process is that this can be scaled up to pilot scales(Abdulla et al., 2019; Calvo et al., 2003) as well as to industrial scales (Rech & Paulino, 2019).

2.2. Chemistry and ingredients of soap

2.2.1. Chemistry of soaps and detergents

The first detergent was soap because it is the oldest skin cleanser. Evidence suggests that the first soap-like substances were produced in ancient Babylon in the third millennium BCE(Levey, 1954). In 200 CE, the Greek physician Galenus invented soap for cleansing and healing purposes (Knowles & Partington, 1999). In order to create a soap-like substance for use in medicine, animal and vegetable oils were blended with soda and other alkalies in the Sumerian pharmaceutical tablet, one of the first extant medical records from around 2200 BCE(Konkol & Rasmussen, 2015). Up to the 19th century, technological development revealed various fatty acid structures in neutral fats and oils, resulting in a more thorough grasp of soap-making chemistry. From a purely chemical standpoint, soap is made of water, an alkali, or even an organic base, along with fat or oil (fatty acids). A neutral fat and an alkali react to produce glycerol and soap molecules in a saponification reaction(Chirani, et al., 2021).

2.2.2. Common ingredients of soap

A class of chemically manufactured goods known as soaps as cleaning agents are created to meet agreed-upon performance requirements and satisfy the demands and expectations of consumers. A good soap should also have the following qualities in addition to being effective at cleaning: good lathering performance, low skin irritation, physical and chemical stability, ease of removal from skin and bathtubs, homogeneous and uniform structure with a moderate erosion rate, and good resistance to cracking. Tallow fat, palm kernel oil, olive oil, and coconut oil are the main sources of triglycerides (oils and fats) used to make soap(Hollstein & Spitz, 1982). Toiletry soaps

are made by mixing in certain ingredients, such as colors, perfumes, and other additives(Valeton, Biermann, Reche, & Rosenberg, 1987). To create personal cleaning solutions with various qualities, odors, colors, shapes, and container designs, a variety of ingredients may be used. Soaps are divided into two major categories: natural and synthetic, based on the chemical makeup of the constituents. The chemistry of natural soaps is comparable to that of early soaps. Most of these soaps only contain oils and fats from plants or animals as their main ingredients; they don't contain any parabens, binders, plasticizers, preservatives, or plasticizers (Feagan et al., 2016). Most natural soaps are produced with little energy and produce no harmful waste or byproducts. As a result, they are more in harmony with nature (Maotsela, Danha, & Muzenda, 2019). Natural soaps can be easily made by small businesses and homes using readily available components (Antonić, Jančíková, Dordević, & Tremlová, 2020). Cleaning products are typically formulated from mixtures of synthetic surfactants, plasticizers, binders, and other additives in synthetic soaps, also known as syndetic. Sulfonation, ethoxylation, and esterification techniques are used to create synthetic soaps from fats, petroleum, and oil-based goods (Diaz et al., 2004). Several ingredients are included in formulated soap products. The table below lists the most popular and widely used ingredients as well as their normal ranges in soap.

Table 2. 1. Common ingredients of soap

Ingredient functions	Ingredient	Typical range (%)	Reference
Saponified oils	Tallow, stearic acid, palm kernel oil, olive oil, palm oil	15 – 80%	(Chirani, et al., 2021)
Emolliating agent	Glycerol stearate, glycerine, castor oil, silicone fluids	1 – 3 %	(Chirani, et al., 2021)
colorant	Dyestuff, beta-carotene, chromium hydroxide green, carbazole violet	0.001 – 0.1%	(CHOI, MYUNG, & KOOK, 1984)

Chelating agent	EDTA, hydroxyl ethane phosphonic acid	0.005 -0.2%	(H. Du, Huang, & Wang, 2022)
Bleaching agent	Titanium dioxide	0.1 –2%	(H. Du, et al., 2022)
water	water	5 – 30%	(H. Du, et al., 2022)
Antibacterial agents	Triclosan, trichlorocarbon	0.3- 1.5%	(Chirani, et al., 2021)
Anti-acne compounds	Sulfur, salicylic acid, benzoyl peroxide	0.5 – 10%	(Chirani, et al., 2021)
Secondary surfactants	Sodium lauryl ether sulfate, lauramine oxide, TEA oleate	0.5 – 5%	(Coss, 2016)
pH - buffer	Citric acid monohydrated, polycarboxylates	0.5%	(Coss, 2016)
fragrance	Perfume, vanillin	0.5 – 1%	(Coss, 2016)
Thickening agent	PEG-40 hydrated castor oil, benzoic acid	0.5 – 0.6	(George & Raymond, 2016)
Moisturizing agent	Lactose, propylene glycerol, urea, aloe barbadensis leaf extract	0.1 – 10%	(George & Raymond, 2016)

2.3. Effects of soap wastewater

According to WHO, handwashing with soap can be considered a vital and necessary public health action for preventing coronavirus spread? Each person should wash their hands at least five times per day, each time for almost 20–30 s. One handwashing with soap and a closed tap uses almost 2 liters of water; however, when the tap is left open, this amount might increase to 4 liters. Due to

the wastewater's increased production (which may increase by 15–18%) and contamination with soap surfactants, this significant water consumption might result in a decrease in water quality. If water is dumped straight into freshwater bodies without any treatment in this circumstance, it may have negative consequences on the ecosystem and human health(Juela Quintuña, 2020). This situation also poses difficulties for wastewater treatment. Detergents can be found in high concentrations in freshwater bodies, which can result in the formation of a lot of foam on the water's surface and a slowed uptake of oxygen. This condition prohibits aquatic organisms from effectively absorbing dissolved oxygen. These elements in the aquatic ecosystem are in charge of changing the water's pH, turbidity, salinity, and temperature (Kim, et al., 2021). Fish suffer biochemical and physiological abnormalities as a result of the decline in water quality, which can also have an impact on how much dissolved oxygen they use, seriously damaging their gills(Aditi, et al., 2021; Ge, et al., 2018)

Furthermore, aquatic plants may be harmed by these many waterborne contaminants. Eutrophication may result from detergent discharge into freshwater bodies. In particular, corals and algae are seriously threatened by this process. Sea species reliant on sea plants for spawn, shelter, food, and protection are at risk due to declines in their populations (Bandala, et al., 2021). Aquatic detergents can eventually find their way into the soil and have a negative impact on the local flora, notably on plant germination(Issayeva, et al., 2015). The health of the plant is in danger because a high surfactant concentration causes the soil's structure to be destroyed(Sawadogo & Mureithi, 2014). Detergents cause the pH of the soil to rise, which causes the soil's constituent parts to separate. Nearly 5 to 9 is the ideal pH range for plants, and any adjustments including pH increases and decreases threaten the biological activity levels in the soil(Chirani, et al., 2021).

2.4. Wastewater Treatment Methods

The complex and dynamic structure of the detergent industry's environmental impact is brought on by the range of raw materials, chemicals, procedures, and technological innovations used in those operations. These dynamic structures have an impact on the effectiveness of the treatment and its application. Depending on the wastewater treatment techniques employed and the volume of water utilized, different contaminants have different amounts in home wastewater from detergent. Therefore, putting in place an effective treatment method is of utmost importance. Household wastewater can be treated in a variety of methods. Depending on the type of waste, type of wastewater, and level of treatment required, a different set of procedures is usually used.

General terms used to describe different degrees of treatment, in order of increasing treatment level, are preliminary, primary, secondary, tertiary, and/or advanced wastewater treatment.
(Pala, 2001).

2.4.1. Preliminary treatment

The objective of preliminary treatment is the removal of coarse solids and other large materials often found in raw wastewater. Preliminary treatment helps to remove or to reduce in size the large, entrained, suspended, or floating solids. These solids consist of pieces of wood, cloth, paper, plastics, garbage, etc., together with some fecal matter. Removed are heavy inorganic solids such as sand and gravel as well as metal or glass. These objects are called grit and excessive amounts of oils or greases(Sonune & Ghate, 2004).

2.4.2. Primary treatment

Primary treatment is designed to remove organic and inorganic solids by the physical processes of sedimentation and flotation. Approximately 25-50% of the incoming biochemical oxygen demand (BODs), 50-70% of the total suspended solids (SS), and 65% of the oil and grease are removed during primary treatment. Some organic nitrogen, organic phosphorus, and heavy metals associated with solids are also removed during primary sedimentation, but colloidal and dissolved constituents are not affected. The effluent from primary sedimentation units is referred to as primary effluent(Sonune & Ghate, 2004).

2.4.3. Secondary treatment

The objective of secondary treatment is the further treatment of the effluent from primary treatment to remove the residual organics and suspended solids. In terms of the size of the solids, the distribution is approximately 30% suspended, 6% colloidal, and about 65% dissolved solids. The function of primary treatment is to remove as much of the suspended solids as possible. The primary treatment utilizes clarifiers or settling tanks, which remove the settle able organics and settle able inorganic solids from the wastewater. The effluent from primary treatment, therefore, contains mainly colloidal and dissolved organic and inorganic solids. Recent effluent standards and water quality standards require a greater degree of removal of organics from waste-water than can be accomplished by primary treatment alone. Additional removal of organics can be accomplished by secondary treatment. The secondary treatment process consists of the biological treatment of wastewater by utilizing many different types of microorganisms in a controlled

environment. Several aerobic bio-logical processes are used for secondary treatment differing primarily in the manner in which oxygen is supplied to the microorganisms and in the rate at which organisms metabolize the organic matter(Crini & Lichtfouse, 2019).

2.5. Coagulation-flocculation treatment method

Modern technology advancements have paved the way for the adoption of more environmentally friendly conventional wastewater treatment techniques. Growing populations create an exciting need to continually develop preventative measures that take into account both current and future issues. According to (Maryam & Büyükgüngör, 2019), the government grant provides the chance for continuing, collaborative research across higher education, academic institutions, and professional sectors to produce answers that may be put into practice. According to the United Nations' prediction as stated by Goh and Ismail (2018), particularly the half of the countries around the globe in the future will confront water stress and shortages. Water reuse and desalination are among the most favored application to address the issues by offering safe water for daily consumption in many arid, remote and coastal regions. Conjointly, limited land available causes wastewater treatment insufficiency. Thus urge the creation of under-control advanced remediation by accelerating the forces of nature that are compatible in smaller areas(Rajasulochana & Preethy, 2016). Nature has such infinite occasions yet to be developed by present technologies.

Water is utilized in a variety of home, commercial, and agricultural activities that introduce both organic and inorganic materials into the environment. Some of them may be treated quickly, such as particle debris, which is accomplished using the filtration process(Rajasulochana & Preethy, 2016). It is currently not possible to handle the majority of chemicals in a single step. Ion exchange, solvent extraction, reverse osmosis, sedimentation, and filtration are a few well-known conventional methods for treating wastewater effluents, however, they have significant maintenance and operating costs and are less effective at removing pollutants like heavy metals (Simate & Ndlovu, 2015). Since ancient times, notably in the 19th century, the use of conventional methods for the treatment of wastewater has been documented. This process is known as the coagulation-flocculation method. The main objectives of this technique are to remove turbidity and colloidal contaminants from water. Researchers created a substance called a coagulant to stabilize colloidal particles by removing pressures that cause them to hang down(Carolin, Kumar, Saravanan, Joshiba, & Naushad, 2017). The best method to remove heavy metals is coagulation. The non-biodegradable traits of heavy metals can pass through biological wastewater treatment

methods, rendering them ineffective. However, because of the presence of certain heavy metals, the reduction of chemical oxygen demand (COD) is frequently observed to be low in aerobic and anaerobic processes of biological therapy(Rasool et al., 2016). Meanwhile, several different water treatment disciplines have made extensive use of the coagulation-flocculation method. The qualities of the utilized coagulants are the key factor in determining the performance of the coagulation, as they can increase or decrease the effectiveness of the treatment(Meena et al., 2017). An examination of 20 treatment plants revealed that coagulation and flocculation are also popular in water engineering(Z. Du et al., 2012). One of the alleged criteria in the therapy selection is the straightforward operational circumstances. The volume dose of the coagulant, pH, the speed and duration of mixing, temperature, and settling time have all been mentioned as additional elements to consider in order to increase the effectiveness of coagulation (Z. Du, et al., 2012). To achieve an impurities/contaminant reduction that is satisfactory and optimal, these parameters must be regulated. A flocculation procedure is required for effective coagulation. Coagulation by itself offers nothing to aid because adding a coagulant may make more insoluble chemicals present in the treated sample(Al-Sameraiy, 2017). As a result, the succeeding flocculation phase is required, and the key to optimum performance is gentle mixing. The second stage permits the collision, which encourages the gathering of the particles together.

The coagulation-flocculation procedure is applied on a laboratory scale using jar test equipment. Two levels of mixing speed rapid and slow are used in the experiment. Before using the slow mixing speed at the appropriate moment, solitary coagulants are often added during the quick mixing to blend them with the water samples. To ensure that the agglomeration of particles can form during the sedimentation process, which separates the solid or sludge from the treated water and wastewater sample, it is crucial to give both phases enough time to mingle. The treatment can also be used in conjunction with additional polymer flocculants that aid in the quicker accumulation of the laborious settling of tiny flocks, in addition to cationic coagulants such as aluminum or iron salts as primary coagulants (Mishra, Anguera, & Gazzaley, 2016). But excessive usage of chemical coagulants also causes new issues (Mishra, et al., 2016). a significant amount of sludge production, which indicates a desire to use natural coagulants as coagulating agents.

2.5.1. Coagulation/Flocculation Process

Coagulation-flocculation is the process by which solids are physically separated from liquid phases through mixing, flocculation, and the application of chemical reagents to destabilize and enlarge

the particle size. Sedimentation, flotation, or filtration are frequently used to accomplish this separation. By lowering the repellent potential of the electrical double layer, the process of coagulation causes the instability of particles. As a result, particles in suspension aggregate. The process of flocculation, on the other hand, involves bringing destabilized particles together to create aggregates called flocks that are large enough and heavy enough to sediment. This results in the separation of the created flocks and the water (Divakaran & Pillai, 2001).

Particle sedimentation in water can happen naturally, for example when there are huge particles present. However, some particles won't settle due to their chemical interaction with water, as is the case when hydrophilic chemicals are present in the water (Divakaran & Pillai, 2001). In order to diminish the influence of the repellent force electrical potential in the solution and achieve coagulation of the particles, which in turn aggregate and settle, mechanical agitation can also be employed to enhance the velocity of big particles of a colloidal solution. But if particles are too tiny to coagulate readily(Taulis, 2005).

Different substances like suspended, colloidal, and dissolved solids are present in industrial effluent. Colloidal solids between 10-9 mm and 10-6 mm in diameter, suspended solids bigger than 10-6 mm, and dissolved solids less than 10-9 mm. This substance needs to be eliminated before discharge (Metcalf, 2003). Different substances like suspended, colloidal, and dissolved solids are present in industrial effluent. Colloidal solids between 10-9 mm and 10-6 mm in diameter, suspended solids bigger than 10-6 mm, and dissolved solids less than 10-9 mm. This substance needs to be eliminated before discharge (Metcalf, 2003). Due to the characteristics of the colloidal solution, these particles cannot be separated or sedimented by regular physical techniques (such as filtering or settling) unless they are coagulated. One of the most common unit procedures in the treatment of water and wastewater is coagulation. This physicochemical procedure makes it easier to separate suspended and colloidal materials from wastewater. Destabilized particles aggregate as micro floc, then grows into large floccules that can be settled and are referred to as floc. The coagulation-flocculation process typically involves destabilizing colloids, aggregating, and binding them together into flocculates. The resulting flocs can then be removed by settling or flotation. The essential procedures in wastewater treatment systems are coagulation, flocculation, and clarifying, followed by filtration (Freitas et al., 2018).

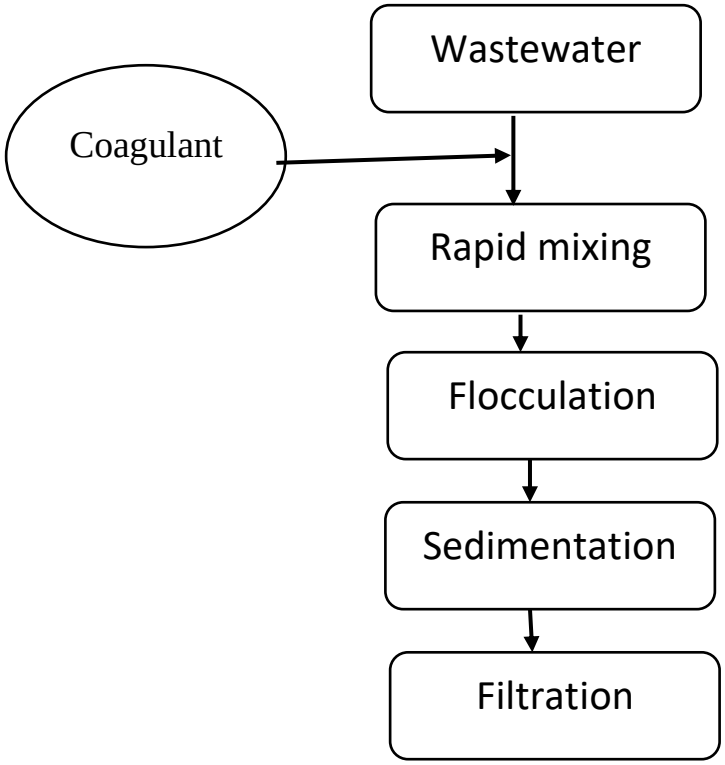


Figure 2. 1. Steps of the coagulation process

A coagulant is dosed into the water during the coagulation process, which causes the water to become unstable. It happens in a series of stages designed to counteract the forces that keep the suspended particles stable, enabling particle collision and floc development. Destabilizing the particle charges in the water is the initial step in the coagulation process. To balance the negative charge on the dispersed non-settable solids, a coagulant of opposing charges was introduced. The smaller particles are capable of adhering to produce somewhat larger particles once the charge has been neutralized. After the coagulant dose, mixing speed is a design parameter. Rapid mixing ensures that the coagulant is evenly distributed throughout the water and encourages particle collision, both of which are necessary for effective coagulation. By increasing particle impacts, slower mixing encouraged floc development and produced larger flocs(Metcalf, 2003). Different destabilization strategies can be used depending on the type of colloidal suspension that needs to go through coagulation, including repression of the double layer, neutralization of colloid charge

by adsorption of counter ions on the colloid surface, bridging of colloidal particles via polymer addition, and entrapment of colloidal particles by sweeping floc. When specific chemicals are added to raw water, the particles become less stable, which allows for floc development and agglomeration. In general, these terminologies are used for this purpose:

- ✓Coagulants, which assist the destabilization of particles (particularly colloidal sizes).
- ✓Flocculants (also known as flocculent aids or coagulant aids), assist in the joining and enmeshing of the particles together.

Coagulation and flocculation are treatment techniques that can be used in soap wastewater treatment to minimize color effluent as well as the overall load of contaminants. These techniques' primary benefit is that they offer treatment options that are affordable, easy to use, and energy-efficient (Zhang & Bishop, 2003). This technique has the inherent drawback of producing significant amounts of chemical sludge, which is classified as hazardous waste and calls for the disposal of hazardous solid waste in protected landfills ((Zhang & Bishop, 2003). For environmental applications, substitute natural materials have been taken into consideration.

In keeping with the general tendency, the term "coagulation" was employed in this study to refer to the entire process, from introducing the coagulant to the wastewater up until their settlement.

Table 2. 2. Difference between Coagulation and Flocculation in Water Treatment

Coagulation	**Flocculation**
Coagulation is an important step in water treatment and it involves the addition of a coagulant to enhance the clotting of suspended particles in water.	Flocculation is another important step in water treatment and it involves the formation of visible flocculation by mechanical or physical mixing
Coagulation is a chemical process.	Flocculation is a physical process.
Coagulation such as inorganic salts of aluminum or iron that neutralizes the suspended particles is added during coagulation.	Flocculants such as an organic polymer that involves bridging and strengthening the flocks are added. It also increases the weight of the flocks and increases the rate of settling
Coagulation does not involve a physical mixing process	Flocculation involves physical mixing

2.5.2. Types of coagulant

Inorganic and organic coagulants are the two main types that are employed. Among the inorganic coagulants are calcium chloride, ferric chloride, and aluminum sulfate. The polyelectrolyte-based organic coagulants, such as those based on polyacrylamide, can be cationic, non-ionic, or anionic. Inorganic coagulants including poly-aluminum chloride, ferric chloride, and alum (aluminum sulfate) are widely used in wastewater treatment due to their effectiveness and simplicity of use. The United States, the second-largest country in the world after China, is said to have employed metal coagulants since the 18th century. While China had been employing alum as an efficient coagulant for water clarity during their reign and had since become one of the first pioneers. These synthetic coagulants have been employed in the drinking water sector in addition to wastewater treatment. The most popular conventional metal coagulants, such as Fe (III) and Al (III), offer greater benefits but are nevertheless limited by several limitations, most notably a lack of green chemistry. The widespread and familiar use of this chemical-based technology is preferable, but there are a few lists of drawbacks that should not be overlooked in earlier literature. When a treatment method is related to neurological disorders like Alzheimer's, particularly when aluminum residue permeates the body and builds up in the brain, there is a small negative perception of the procedure's sustainability. Other risks to human health and the health of other living things have also been identified, most notably energy loss, intestinal constipation, convulsions, and stomach colic.

These inorganic salts and synthetic metals also have a somber past related to their physical characteristics. The price will be obviously greater when it enters the commercial market, influencing the social factor in rural communities, even though the cost of raw materials to make the items is said to be relatively efficient. There is controversy around the post-treatment following coagulation because it produced a significant amount of toxic sludge that made disposal difficult (Rasool, et al., 2016; J. G. Teixeira, Patrício, & Tuunanen, 2019) has been a topic of discussion. Usually, unreacted chemical residues and the interaction of polymer leftovers in treated water are what because the toxic sludge. (Hesami, Ahmadi, & Nematzadeh, 2014; Subramonian, Wu, & Chai, 2014). Since larger flocks are generated by smaller ones approaching one another during the coagulation process, the resulting sludge cannot be avoided (Douillard et al., 2013). Since coagulation is only intended to be used as a pre- and primary treatment, the produced sludge needs to go through another stage of the treatment process. Furthermore, as effective coagulation only

functions under specific pH and environmental conditions, the pH of the effluent must also be changed before it can be processed. Low temperatures make it difficult for them to work effectively (Douillard, et al., 2013) (F. P. Teixeira, Gomes, & de Andrade Silva, 2019). More advances in natural polymers and organic coagulants have been made in recent years as a result of a review of the shortcomings of these hydrolyzable cations. It is constantly advised to look for environmentally friendly and sustainable treatment methods because they assist so many people. While preventing the chemical and physical aftereffects in the treated medium, the decrease or substitution of these controversial coagulating substances will increase the value of the sources of origin(Essig Jr et al., 2013).

Sustainable water management has received a lot of focus, but it can be difficult to create effective procedures, particularly in the wastewater business. Current research always looks forward to natural materials again for effective approaches to wastewater treatment, starting from natural ingredients to the laboratory-produced synthetic chemical-based. Natural coagulants, particularly those derived from biological sources, are widely recognized as being free of toxins (Awang & Aziz, 2012; F. P. Teixeira, et al., 2019). The use of natural coagulants or coagulant aids derived from plants, animals, or agricultural wastes, such as Moringa oleifera (MO) seeds, chitosan, and cassava peel, has been the subject of numerous investigations (Choy, 2014; Mohd-Asharuddin, Othman, Zin, & Tajarudin, 2017). These organic coagulants, such (MO) seeds and chitosan have been the most popular types of bio-based coagulants over the years (Zehir et al., 2017). Since they can be produced from renewable resources and are practical, especially in remote places, the trending application of these coagulants is generally speaking in favor of environmentally beneficial technology (Hossain & Yin, 2010). Before the invention of chemical salts to clarify wastewater, natural coagulants with biodegradable properties that are believed to be harmless for ecological systems and living things already existed. Initially, it was discovered that the primary cause of less effective coagulation was the decreased molecular weight of natural polymers. As a result, it is typically used as a coagulant aid rather than as a first choice. When used as a coagulant aid, natural polymer facilitates the creation of the chemical bridge with the suspended solids during the coagulation process. The growing amounts of organic matter from coagulants, which may lead to steric problems between polymer molecules at high concentrations, are the only cause of the performance drawbacks documented. Additionally, this promotes further study into the use of natural materials as alternatives to synthetic coagulants. The growing interest in using natural

materials has prompted in-depth research into the features and behavior of these materials during the coagulation process, including the widely used MO seed coagulant. According to the Food and Agricultural Organization of the United Nations (FAO), it comes from a tropical tree with many health benefits that are widely distributed, especially in the sub-Himalayan valleys, Asia, Africa, and Latin America. According to earlier research, the seed can be utilized to produce coagulants as well as edible oils that are used in health and beauty products due to their nutrient content. The remaining residual rind can be used as fertilizer, animal feed, and activated carbon(Ghebremichael, Gunaratna, Henriksson, Brumer, & Dalhammar, 2005) Additionally, it is claimed to produce less sludge by volume and have similar effects to alum in the coagulation of highly turbid water. It has been discovered that the MO seed extract includes the cationic protein of polymerization, which aids in the low-cost techniques for clarity and is frequently utilized in rural communities to clear hazy water. Another example is chitosan, a natural polyelectrolyte that has attracted significant interest due to its high molecular weight, charged density, and origin as a biodegradable polymer. Arthropods, insects, crustaceans, fungus, and other marine organisms all include chitosan, which is produced when chitin is DE acetylated Chitosan is chosen when employed as a coagulant because, upon contact with an acidic solution, it emits positively charged particles that interact with the anionic suspended colloidal. Due to the presence of amine functional groups in nature, chitosan has a positive charge along its chain (Mohd-Salleh, Mohd-Zin, & Othman, 2019). Coagulants' natural components may also be obtained from organic waste, such as agricultural waste. Agricultural wastes are among the waste types that contribute the most to landfill volume because of their lack of economic value and barriers to commercialization. For instance, cassava peel is a readily available waste-derived coagulant. Since only its cassava starch is used for further commercialization, the usage of cassava peel raises serious concerns because the industries involved in cassava processing produce a tremendous amount of waste (Howeler et al., 2001; Leon et al., 2016). A cassava starch processing machine produces 100 tons of fresh by-products every day, or roughly 47 tons, which, if improperly disposed of, could have a significant influence on the environment. Cassava peels contain a significant amount of cyanogenic glucosides, in addition to having the potential to release carbon dioxide and a potent odor of rotting trash. Cassava peel has the potential to be chosen as a waste-based coagulant because of its capacity to remove nutrients and heavy metals. This potential is supported by the presence of Si, Ca, Mg, and Fe, as well as by the abundance of functional groups and surface porosity of the media. Considering its

bio-polymers, which are made up of proteins and polysaccharides, this natural-polymer coagulant appears to be a viable option for water coagulants(Mohd-Asharuddin, et al., 2017) (Othman, Abd-Kadir, & Zayadi, 2016).

Table 2. 3. Types of bio-based coagulant

Coagulant	**Type of wastewater**	**Removal parameter (%)**	**Reference**
MO	Textile industry	Heavy metal; Cd,Cr,Mn(100%), turbidity(98.6%) and BOD (11.7%)	(Mi et al., 2017)
Banana pith	Polluted river water	Cr,Cu,Pb (100%), Fe (92%), Zn (81%), Mn (60%)Turbidity (98.5%), COD (54.3%), Suspended solids (96.03%), sulphates (98.9%), nitrate	(Kakoi, Kaluli, Ndiba, & Thiong'o, 2016)
Jatropha curcas seed	Kaolin synthetic wastewater	Turbidity (96%)	(Abidin, Shamsudin, Madehi, & Sobri, 2013)
Nirmali seed	Laundry waste	TSS (75.5%), turbidity (95.5%)	(Mohan, Sarswat, Ok, & Pittman Jr, 2014)
Pectin of orange peel pith	surfactant	Dual coagulants with nirmali seed; Turbidity (89.5%), TSS (81.5%)	(Mohan, et al., 2014)
bio-based	Soap waste	The gap in this study	

2.5.3. Factors Affecting the Effectiveness of Coagulation

The kind of wastewater, the initial concentration of the waste, the amount and type of coagulants/flocculants used, the temperature, the physical and chemical properties of the coagulant, the pH of the solution, the rate of mixing, and other parameters all affect how well the coagulation works (Rozainee et al., 2008). One of the most crucial factors taken into account while figuring out the ideal circumstances for the coagulation-flocculation process performance is the coagulant dose. Essentially, a dosage that is either low or too high will lead to subpar performance.

There are a number of optimum doses for coagulants at which maximum settling and the most efficient elimination of contaminants are accomplished. Below this point, the amount of coagulant injected is insufficient to cause the particles to become unstable. The coagulant effectively acts as a chemical coating that re-stabilizes the particle above this threshold (Patel & Vashi, 2013). Therefore, it is crucial to establish the ideal dose in order to reduce dosing costs and sludge formation as well as to achieve the best results possible during the treatment process. This research focused on the dose because the spectrum of coagulant doses differs depending on the type of coagulant. The pH is a second crucial factor that has been taken into account while determining the ideal setting for the administration of medication (Singh, Verma, & Singh, 1970). The degree of stabilization of the suspension will also be impacted by the pH of the coagulation process, in addition to the surface charge of the coagulants. Additionally, pH appears to be a fascinating factor that has a higher impact on the effectiveness of removing contaminants. In order to reduce the production of sludge and get the best treatment results, one of the elements essential to determine the ideal level was pH influence. Thus, to provide the ideal environment for the coagulation process, pH must be regulated by adding either strong acid or basic. In the coagulation-flocculation process, mixing time has a significant impact on flocs' creation and proliferation in addition to coagulant dose and ph. (Sabur, Khan, & Safiullah, 2012). In any water and wastewater treatment facility that uses coagulation, one of the operating factors that are given careful consideration is the time of macro floc development (flocculation time). Therefore, it's crucial to establish the treatment system's ideal mixing time. In a coagulation process, there are normally three phases of mixing: quick mix, gradual mix, and no mix. A brief period of extremely turbulent mixing known as the quick mixing phase permits coagulants to come into contact with suspended particles.

The following stage is flocculation time, which is characterized by sluggish mixing and enables destabilized particles to coalesce into bigger particles (Majure et al., 2012). This period of mixing is crucial for the production of flocs. Due to flocs' extreme fragility, abrupt motions cause floc breakup, which reduces their effectiveness. The last phase doesn't involve any mixing. It enables flocculated particles to leave the system by settling. Poor particle agglomeration results from insufficient slow mixing times. However, prolonged durations of agitation cause floc fragments to rupture and cause the deagglomeration of particles. The effectiveness and efficiency of coagulation are generally greatly increased by optimizing the coagulant dose, mixing duration, and ph.

2.6. Electro-coagulation

Electro-coagulation is a process involving chemical and physical phenomena, which uses sacrificial electrodes for the generation of coagulants. Electrocoagulation involves the in situ generations of coagulants by dissolving electrically either aluminum or iron ions from aluminum or iron electrodes, respectively but Aluminum electrodes showed better performance than iron(El Sayed, El Shehawy, & Afify, 2021). At the anode, metal ions are produced, and at the cathode, hydrogen gas is liberated. The flocculated particles would also be helped to float out of the water by the hydrogen gas. The anode is where metallic cations are produced, whereas the cathode is where H2 is created. Coagulants are made when suitable anode materials, such as iron or aluminum, are electrolytically oxidized. This process creates highly charged polymeric metal hydroxyl species, which are then used to make coagulants. These species aid in an agglomeration that leads to separation from the aqueous phase and balances the electrostatic charges on the suspended particles.

By injecting highly charged polymeric hydroxide species, this approach eliminates metals, colloidal particles, and soluble organic contaminants from aqueous systems.

2.6.1. Mechanism of EC Process

EC is a separation technique in which both physical and chemical mechanisms for pollutant removal are involved(Kobya, Ciftci, Bayramoglu, & Sensoy, 2008). This process is mainly destabilizing the suspended, dissolved, or emulsified pollutants in the aqueous medium by supplying electricity(Omprakash Sahu, Mazumdar, & Chaudhari, 2014). Fig. 2.2 shows the schematic portrayal of the mechanism involved in the EC process. The EC process generally consists of the following mechanisms (i) scarification of anode material when it gets exposed to electric supply, which leads to the generation of metal cations; (ii) hydrolysis at cathode generates hydroxyl ion, (iii) The metallic cations interact with OH^- ion and forms metal hydroxides, which has a good adsorption ability to bind the pollutants; (iv) oxidation of pollutants to lesser toxic species; (v) metal oxides react with pollutant, and make the formation of neutralized matter; (vi) neutralized matter get aggregated and adsorption on metal hydroxides and followed by sweep coagulation and removal; (vii) some of the neutralized matter interacts with the gas generated in the system and entrapment of gas in the flocculated strictures and follows sweep flotation(Syam Babu, et al., 2020). The electrolytic oxidation of anode metal dissociated to di or tri metallic ions and discharges a proportional number or electrons (Eq. 1) and also water reacts with the anode and

releases hydrogen ions along with oxygen gas in the system (Eq. 2). The water reacts on the cathode region generates hydrogen ion and hydroxyl ions (Eq. 3), the generated two hydrogen ions combine to form hydrogen gas. Metal ion formed from the anode reacts with hydroxyl ions from the cathode combined to form metal oxides, which act as very good adsorbent(Aboubaraka, Aboelfetoh, & Ebeid, 2017).

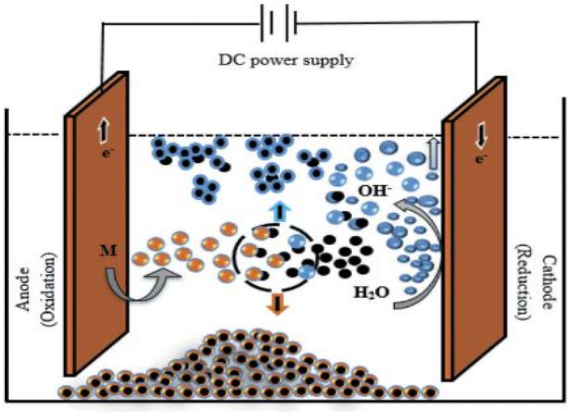


Figure 2. 2. Pollutant removal mechanism by Electrocoagulation process (Syam Babu, et al., 2020)

At the anode

$$M_{(S)} \rightarrow M^{n+}_{(aq)} + ne^- \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

$$H_2O \rightarrow 4H^+_{(aq)} + O_{2(g)} + 4e^- \ldots\ldots\ldots\ldots\ldots\ldots....(2)$$

At the cathode

$$nH_2O + ne^- \rightarrow (n/2)H_{2(g)} + nOH_{(aq)} \ldots\ldots\ldots\ldots(3)$$

Due to the creation of their multivalent ions and diverse hydrolysis products, iron and aluminum are the most commonly employed anode materials (Duan & Gregory, 2003; Nariyan, Sillanpää, & Wolkersdorfer, 2017). According to the pH of the solution and the potential for the creation of mono and poly nuclear hydroxides, the metal cations liberated during the scarification of anodic material go through a series of hydrolysis processes (Anantha Singh & Ramesh, 2013; Vickers, 2017). As a result, the performance of EC is significantly influenced by the pH of the solution. According to equation (Eq. 4), the hydrolysis scheme will proceed from left to right with an increase in pH, resulting in doubly and singly-charged cationic species before Me (OH)3, an

uncharged metal hydroxide. As pH rises higher, the soluble anionic Me (OH)4 form predominates(Duan & Gregory, 2003).

$$Me^{3+} \rightarrow Me(OH)^{2+}$$
$$\rightarrow \ Me(OH)^{2+} \rightarrow Me(OH)_3 \rightarrow Me(OH)^{-}_4 \ \ldots\ldots\ldots(4)$$

EC process has been utilized effectively for the removal of color(Modirshahla, Behnajady, & Kooshaiian, 2007), turbidity(Mohammadi et al., 2017; Zhao, Huang, Cheng, Wang, & Fu, 2014), hardness(Zhao, et al., 2014), phosphate(Hashim et al., 2019; Omwene & Kobya, 2018), fluoride(Khatibikamal, Torabian, Janpoor, & Hoshyaripour, 2010; Vickers, 2017), mercury(De, Hazra, & Dutta, 2019; Subramanyan Vasudevan, Jothinathan Lakshmi, & Ganapathi Sozhan, 2012), lead(Mansoorian, Mahvi, & Jafari, 2014), copper(Beyazit, 2014), nickel(Mansour & Hasieb, 2012), chromium(Vickers, 2017), cobalt(Subramanyan Vasudevan, Jothinathan Lakshmi, & Ganapathy Sozhan, 2012), boron(Kartikaningsih, Shih, & Huang, 2016), cadmium(Xu et al., 2017), iron(Xu, et al., 2017), cesium(Kamaraj & Vasudevan, 2015), strontium(Kamaraj & Vasudevan, 2015) and treatment of industrial effluents generated from agro industries(Drogui, Asselin, Brar, Benmoussa, & Blais, 2008), paper and pulp industry(Mahesh et al., 2016; Sridhar, Sivakumar, Immanuel, & Maran, 2011), textile industry(Can, Kobya, Demirbas, & Bayramoglu, 2006; Zongo et al., 2009), battery industry(Mansoorian, et al., 2014), distillery industry(Yavuz, 2007), semiconductor fabrication industry(Lai & Lin, 2003), mechanical polishing industry(Drouiche, Ghaffour, Lounici, & Mameri, 2007), poultry processing industry(Eryuruk, Un, & Ogutveren, 2018), chicken processing industry(Gomes, Atambo, Das, Cocke, & Das, 2018) , meat industry(Vickers, 2017), dairy industry(Aitbara, Cherifi, Hazourli, & Leclerc, 2016), edible oil industries, vegetable oil industry(Inan, Dimoglo, Şimşek, & Karpuzcu, 2004), olive oil industry(Inan, et al., 2004), palm oil industry(Nasrullah et al., 2017), almond oil industry(Valero et al., 2011), egg processing industry(Azarian, Rahmani, Atashzaban, & Nematollahi, 2018), train industry(Ozyonar, 2016), machinery industry(Pantorlawn, Threrujirapapong, Khanitchaidecha, Channei, & Nakaruk, 2018), metal cutting and plating industry(Oden & Sari-Erkan, 2018), petrochemical industry(Yavuz, Koparal, & Öğütveren, 2010), pistachio processing industry(Ozay et al., 2018), potato chips manufacturing industry(Kobya, Hiz, Senturk, Aydiner, & Demirbas, 2006), sugar industry(O Sahu, Mazumdar, & Chaudhari, 2019), tannery industry(Espinoza-

Quiñones et al., 2009), pasta and cookie processing industry(Vickers, 2017), acrylic fibers manufacturing industry(Amato et al., 2014), marble processing industry(Solak, Kılıç, Hüseyin, & Şencan, 2009), can manufacturing industry(Kobya & Demirbas, 2015), paint manufacturing industry(Akyol, 2012), hospital wastewater(Ahmadzadeh & Dolatabadi, 2018), bio diesel wastewater(Tanattı, Şengil, & Özdemir, 2018), restaurant wastewater(Chen, Chen, & Yue, 2000), laundry wastewater, printing ink wastewater(Papadopoulos et al., 2019)etc.

2.6.2. Parameters affecting EC

A) Reaction time: The EC time has a significant impact on the effectiveness of pollutant removal. Longer periods result in a better rate of pollutant removal because more metal coagulants and flocs are produced at a constant current density (Alaba). However, the EC efficiency reaches a plateau at certain time intervals because occupied active sites produce coagulant pollutant flocs, which cause the pollutant removal rates to stabilize(Lekhlif, Oudrhiri, Zidane, Drogui, & Blais, 2014). To prevent energy and resource waste, electrolysis time must be optimized because it affects the pace at which pollutants are removed from the environment.

B) Inter electrode distance: In an EC operation, the distance between the electrodes is crucial because it affects the electrostatic field that exists between the anode and the cathode. The inter-electrode distance is at its smallest when the electrostatic field is at its maximum. Because of the significant collisions caused by the strong electrostatic attraction, the metal hydroxides help form flocs to enable coagulation to degrade(Khandegar & Saroha, 2013). As a result, at a minimum inter-electrode distance, EC efficiency is low. On the other hand, a wider inter-electrode gap delays the development of metal hydroxide-flocs because electrostatic forces are reduced (S. Verma, Khandegar, & Saroha, 2013). In order to compensate for the released ions' slower journey between the anode and cathode, increased power consumption is required when electrode spacing is above the optimal level (Mollah et al., 2004). Consequently, it is crucial to run EC at the ideal inter-electrode spacing. For diverse types of wastewater, a number of studies employed a minimum spacing of at least 10 mm (Lekhlif, et al., 2014). In addition, many investigations had also used an inter-electrode spacing between 20 and 30 mm (A. K. Verma, 2017).

C) Current density: Current density, which is the amount of current provided per effective electrode surface area, is a critical factor impacting EC efficiency. As a result of the dissociation of metal ions from the electrode, current density controls the rate at which electrons are released (Vickers, 2017).

In actuality, the electrode dissociation and applied current density are intimately related (Moussa, El-Naas, Nasser, & Al-Marri, 2017). For various forms of wastewater, the current density applied ranges greatly. The nature of the contaminants contained in the wastewater plays a major role in the variety of ionic interactions that cause the discrepancies.

Typically, the current density applied for EC may range from as low as 0.01 Am^{-2} (Ghosh, Solanki, & Purkait, 2008) to as high as 880 Am^{-2}(Larue, Vorobiev, Vu, & Durand, 2003). Even though the applied current greatly corresponds to the metal ion dissociation and release of ions in the solution, an excess of current may negatively affect the efficiency of EC by enabling secondary reactions, and overdosage of coagulants can cause charge reversal of the colloids. This phenomenon may reduce the electrode life span while deteriorating the efficiency of EC and management of resources (energy supply, electrodes, etc.). Therefore, the current density is an important parameter that requires optimization to carry out EC for the desired wastewater/water treatment.

D) Electrode material and arrangement: The performance, efficiency, and cost of the EC are significantly impacted by the electrode material. The kind of electrochemical reaction that occurs depends on the material. The electrode dissociation rate, the amount of pollutant removed, and the amount of coagulant required are the primary metrics used to assess efficiency. The release of the metallic ion coagulants in the electrolytic solution is closely correlated with these factors. Because they can improve the coagulation of pollutants thanks to a higher level of electrical double-layer compression, coagulants with a higher charge valence are preferred(Garcia-Segura, Eiband, de Melo, & Martínez-Huitle, 2017). Al and Fe electrodes have been widely used for EC with successful removal of specific pollutants due to their capacity for effective coagulation as well as their affordability, availability, dependability, and non-hazardous qualities(Vickers, 2017).

Numerous studies have also demonstrated that the COD% removal rates for both Fe and Al were roughly similar, with Al generally being more effective than Fe while Fe excels due to its relatively low power consumption, which lowers the overall process cost(Linares-Hernández, Barrera-Díaz, Roa-Morales, Bilyeu, & Ureña-Núñez, 2009). Moreover, in terms of decolorization of acid dye, Fe once again surpassed Al by removing 98 % of the dye, unlike 53 % by Al Chafi et al(Chafi, Gourich, Essadki, Vial, & Fabregat, 2011). Besides maximum decolorization yield, the energy requirements and costs were minimized with Fe. Therefore, a fine line cannot be drawn as to which electrode combination gives the best outcome. Both Fe and Al have their distinct properties with

respective mechanisms in their dissociation and chemical properties. More factors such as optimum working pH and reaction time must be considered as well in parameter optimization as they vary for different electrodes, along with the chemistry of the targeted wastewater. However, a number of works concluded that Al-Al is the best electrode combination for maximum COD removal(Demirbas & Kobya, 2017).

E) Temperature: Most EC studies are generally carried out at room temperature. However, some researchers had investigated the effect of temperature on pollutant removal efficiency in EC. For instance (Lammi et al., 2018) concluded that heated solutions result in a more efficient and cost-effective

colloidal separation. The higher temperature increased the generation of Al (OH) 2+ ions which is one of the most effective coagulant species. Raising the temperature from 25 °C to 85 °C, not only enhanced the turbidity removal percentage from colloidal calcareo-argillaceous suspension significantly but also reduced power consumption(Lammi, et al., 2018). Corresponding to increased effectiveness, operating EC at higher temperatures, lowers current density requirements, therefore, reducing process costs notably.

F) Stirring speed: Stirring speed is an important factor for EC to ensure homogeneity in the reactor mixture and enhance pollutant removal rate by imparting velocity through agitation. In general, the stirring speed is kept constant in most EC studies for wastewater treatment, typically within 80–300 rpm(Keshmirizadeh, Yousefi, & Rofouei, 2011). However, some researchers investigated the influence of stirring speed on the EC process in pollutant removal efficiency. Increasing mixing speed enhances the movement of ions and flocs produced within the EC reactor increasing the overall efficiency of pollutant removal. However, beyond the optimum range, the agitation can impact negatively the EC efficiency by breaking down the flocs that trap the pollutants within by possibly restabilizing the colloids (Khandegar & Saroha, 2013).

G) Supporting electrolytes. : Supporting electrolytes boost the conductivity of the EC mixture by enhancing electron transfer in anode dissolution. Consequently, a larger current can pass through the electrolytic solution at a smaller voltage, reducing the power consumption of the overall EC process, and making it economical by saving time and resources to achieve the same efficiency. The most common electrolyte used is sodium chloride (NaCl) as it is inexpensive and readily available. Besides, polyaluminium chloride (PAC), potassium chloride (KCl), and sodium nitrate (NaNO3) supporting electrolytes were compared along with NaCl for EC by

Keshmirizadeh(Keshmirizadeh, et al., 2011). It was found that KCl worked as effectively as NaCl in the total removal of chromium IV from wastewater. Therefore, the conductivities of the EC mixture are adjusted by the addition of appropriate supporting electrolytes at optimum concentrations.

However, more studies should be carried out to optimize supporting electrolyte concentration in EC as it affects the total dissolved solids (TDS) of the final effluent which is crucial for operations that require feed water of strictly low TDS value.

Wastewater from all laundry sources accounts for approximately 10% of municipal sewer discharges. In addition to high suspended solids and biological oxygen demand (BOD) loading, the levels of oil and grease, heavy metals, and other organics exceed municipal discharge standards. Commonly, laundry effluents contain more than 1000 ppm suspended solids, 5000 ppm chemical oxygen demand(COD), 1100 ppm fats, oil and grease(FOG), and 1300 ppm BOD, in addition to metals and organic solvents such as toluene, benzene, and perchloroethylene(Janpoor, et al., 2011).

CHAPTER THREE

3. MATERIALS AND METHODS

3.1. Equipment and apparatus

The major equipment and apparatus that were used in this study are a vacuum pump, filter paper, separator funnel, round bottom flask, analytical balance, beakers, aluminum electrodes, copper wire(used to connect the electrodes and DC power supply), heater, ph meter, temperature controlled oven, DC power supply, turbidity meter, and Fourier transform infrared spectroscopy (FTIR).

3.2. Chemicals and reagents

The chemicals that were used in this study are Glycerol, lactic acid, chloroform, aluminum sulfate, Nitric acid (HNO_3), sulfuric acids (H_2SO_4), ethyl acetate, hydrochloric acid (HCl), sodium hydroxide (NaOH), and distilled water was used throughout the experiment.

3.3. Methodology

The following methods were followed to synthesize and purify the bio-based coagulant from glycerol and lactic acid, study the effect of different parameters on the removal of fatty matter, evaluate the effect of bio-based coagulant chain length and effect of electrocoagulation on the removal efficiency of fatty matter.

3.3.1. Synthesis and purification of the bio-based glycerol coagulant

With varying molar concentrations of lactic acid and glycerol, the esterification of glycerol (99%) and lactic acid (88%) through a condensation process was take place in a temperature-controlled heating oven. The two reactants were combined into a single-necked round bottom flask (RBF) with a horizontal condenser for water segregation, and heated to 190 °C for an 8-hour reaction duration in a temperature-controlled oven (Tsion, 2021).

Table 3. 1. Reaction conditions for bio-based coagulant synthesis

Temperature (°C)	Time (hr)	Molar ratio	Product
190	8	3	GLY-LAC 1:3
190	8	6	GLY-LAC 1:6
190	8	9	GLY-LAC 1:9
190	8	12	GLY-LAC 1:12
190	8	16	GLY-LAC 1:16

Esterification reaction: Glycerol + Lactic acid ⟶ Glyceryl lactate (bio-based coagulant)

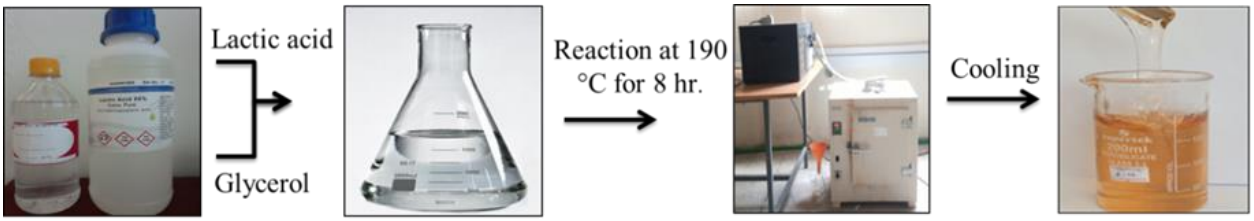


Figure 3. 1. Synthesis of the bio-based coagulant

A purification step was conducted before we obtain the pure bio-based micro-molecule products. The solution was poured into a separator funnel with half of the produced product amount of ethyl acetate to extract the residual glycerol and lactic acid from the solution (because of the better solubility of lactic acid in water than in ethyl acetate and the insolubility of glycerol in ethyl acetate)(Yuan et al., 2016). All of the clear liquid of the upper layer, which contains ethyl acetate and bio-based micro-molecule coagulant was collected. By reducing-pressure distillation was carried out to remove the Ethyl acetate and water from the clear liquid, and then, the pure bio-based coagulant was obtained.

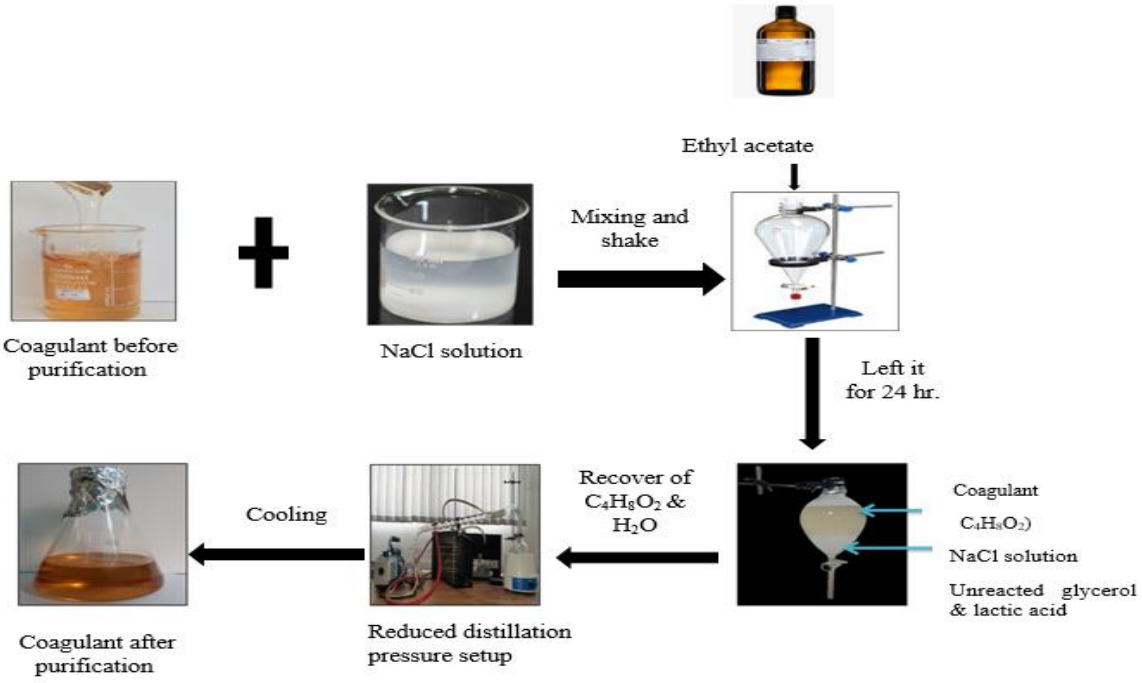


Figure 3. 2. Purifications of the bio-based coagulant

Acid value determination: this experiment was done according to the ASTM D974-12 method(Knothe, 2006). In this case, one gram of the sample was weighed and dissolved in 20 milliliters of chloroform. In the presence of 10 droplets of penophtaline (indicator) solution, the solution was then titrated with 0.1 N KOH solution (Murillo, Vallejo, & López, 2011). The acid value was calculated according to equation 3.1.

$$AV = \frac{(\mathrm{V} - \mathrm{Vo}).\ NKOH.\ 56.1}{\mathrm{m}}.100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{Eq 3.1}$$

Where; AV is an acid value (mg/g sample) of the conjugate, Vo is the volume of 0.1 N OH solution used to titrate the blank solution, V is the volume of 0.1 NKOH solution used to titrate the sample and m is mass of the sample (1 g).

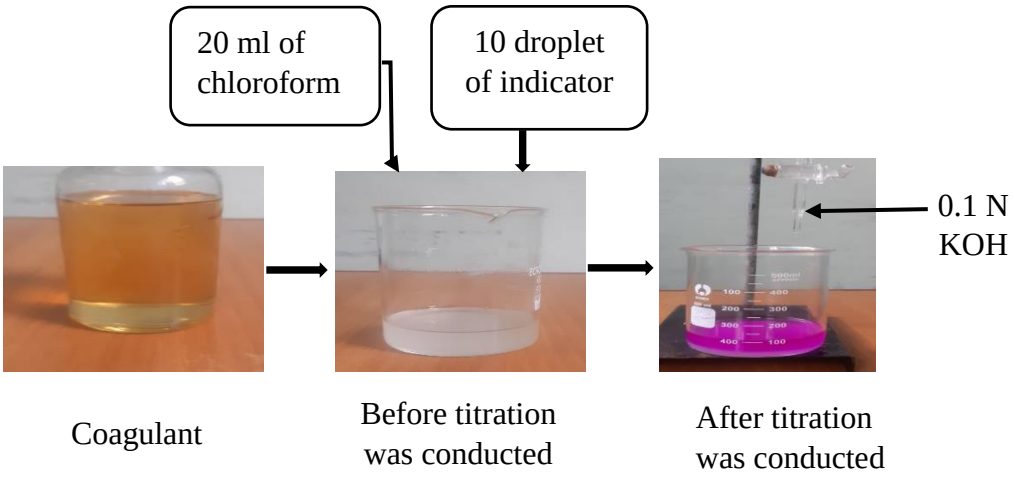


Figure 3. 3. Measurement of the Conjugate acid value

Viscosity determination: The bio-based coagulant viscosity was determined by VK 2000 viscometer types based on the modified method of KREBS operating methods. The sample to be tested was transferred to a clean and dry 250 ml beaker, and the viscosity of the sample was determined by using a spindle N° 5 at speed of rotated 25 rpm, near 100% torque.

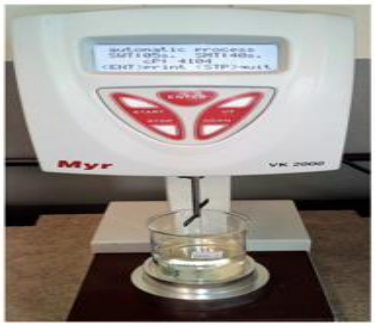


Figure 3. 4. Measurement of the Conjugate Viscosity

3.3.2. Coagulation Experiments

In this study, the batch test was conducted using a set of six beakers and six spindle steel paddles in a jar test apparatus, as illustrated in figure 3.5. The wastewater samples were homogeneously mixed before the jar test, and their initial pH was corrected by adding either 1M strong acid (HCl) or a strong base (NaOH). The 300 ml of the sample was then added to each beaker.

Figure 3. 5. Experimental setups of Jar test apparatus

Following the addition of the various synthesis coagulant doses (1–5 g/l), the beakers conducted constant speed quick mixing for 30 minutes at 100 rpm. The coagulant is evenly distributed across the beakers due to the quick mixing. The suspension was allowed to settle for various amounts of time after the agitation ended (from 1hr to 16 hr.). The supernatant was filtered using filter papers after settlement. The removal of the total fatty matter was then determined for the samples of the supernatant obtained from each beaker. The ultimate concentration results of the wastewater sample were compared to the beginning concentration values. In this study, the experimental setup was carried out by changing some operating parameters, such as coagulant dose, settling time, and initial concentration, while holding other parameters constant to study their impact on the coagulation-flocculation process. This also helped to achieve the optimal condition of parameters.

3.3.3. Electro-coagulation procedure

The experiments were carried out using two aluminum electrodes in a batch reactor. To investigate the effects of various electrocoagulation (EC) process parameters, various EC tests were conducted. Inter-electrode distance, voltage, reaction time, and settling time are used in this study as several elements that may affect the efficacy of pollutant removal.

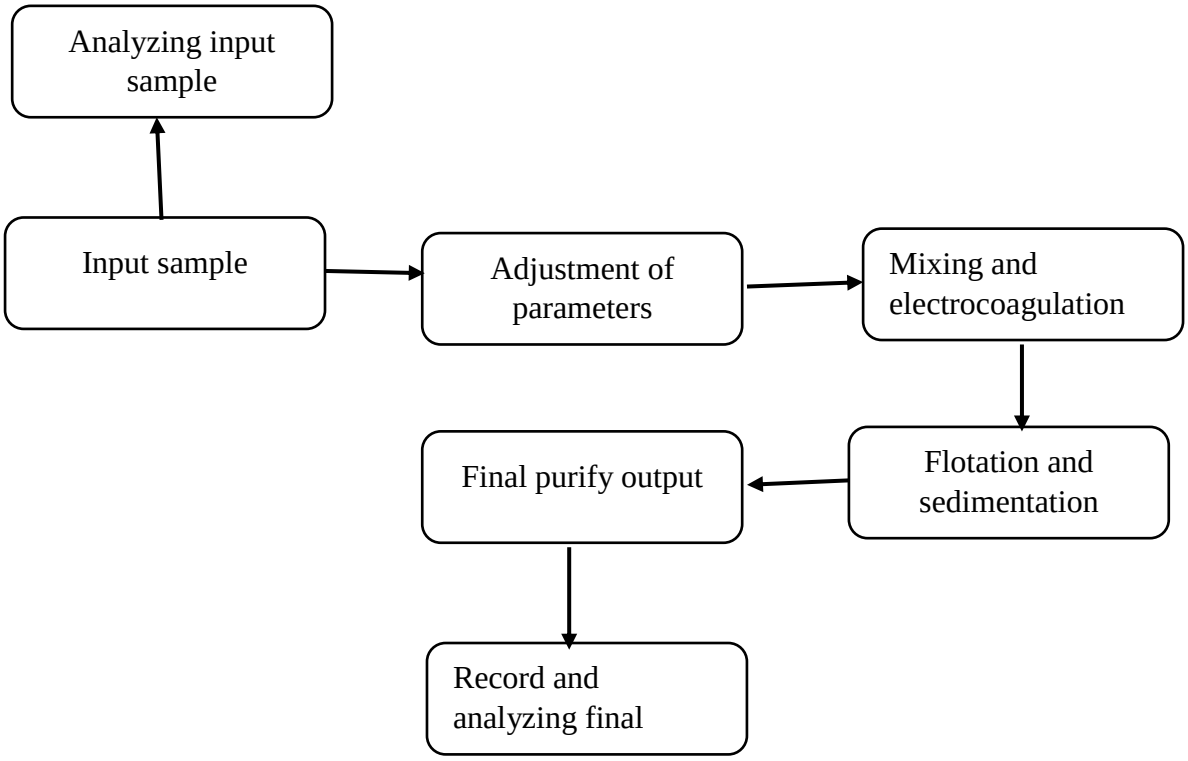


Figure 3. 6. The Bach experiment of electrocoagulation

After adding the solution to the electrocoagulation cell, the electrodes were set at the appropriate distance and linked to the DC power source with copper wire. To create a homogeneous solution, sample mixing was done using a magnetic stirrer with a constant speed of 100 rpm applied throughout all tests(Mohammadi, et al., 2017). A digitally controlled DC power supply (0-30V, 1A) was in charge of controlling the current and voltage (Janpoor, et al., 2011). The solution was filtered through filter paper at the end of each experiment. The samples were made by the parameters after filtering, and the values were then recorded.

3.3.4. Experimental Set up of electrocoagulation

The lab-scale batch study approach was used to treat the samples. Aluminum electrodes measuring 12 cm in length, 5 cm in width, and 1.16 mm in thickness were employed for this experiment(Takdastan et al., 2017). The electrodes were positioned vertically and parallel to one another in the undivided reactor electrochemical cell that was used for this experiment. The experimental set-up provides a length adjustment between 2 cm and 4 cm between the two electrodes. Because the electrostatic field is dependent on the distance between the anode and the

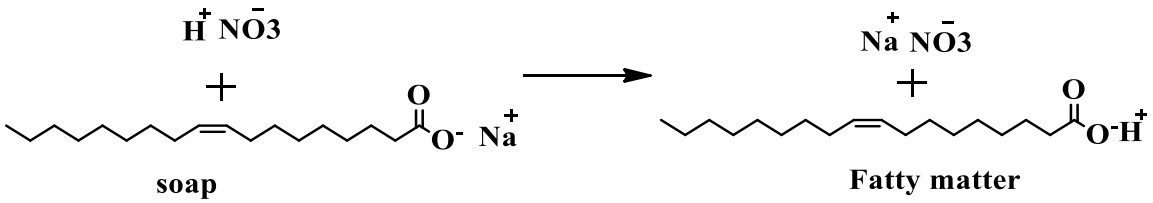
cathode, the inter-electrode distance plays a significant effect on EC strength, which is why 2 cm and 4 cm were chosen.

Figure 3. 7. Experimental setups of electrocoagulation

Maximum pollutant removal effectiveness is provided by an optimum distance between electrodes. Low pollutant removal effectiveness is provided by a small inter-electrode spacing. The produced ions move more slowly as inter-electrode distance increases. Ions have more time to produce the floc needed for the coagulation of contaminants because of their slower mobility (Mallesh, 2018). (Mallesh, 2018). The electrodes were adjusted to lengths of 2 cm and 4 cm to get the optimum inter-electrode distance. An unstable power source was employed to deliver consistent current (between 0 and 2 A) at a fluctuating voltage (0–30 V). Different settling times (30min, 60min, 90min, 120min, and 180min), voltages (10V, 20V, 30V), and reaction times were used during the electrolysis (30min,60min,90min, and 120min). The setup was finished after adjusting the voltage and current. In all studies, samples were mixed using a magnetic stirrer at a constant speed of 100 rpm.

3.3.5. Determination of the Total Fatty Matter (TFMR)

Adding nitric acid (HNO3) to sodium soap gives sodium chloride (NaCl) and fatty acid.



The Na^+ and H^+ trade places. The Na^+ leaves the fatty anion, so it can go be friends with NO_{3-} in solution, and at the same time, the H^+ joins with the fatty anion, making a molecule with a COOH group on one end, a fatty acid. The fatty acid is not soluble in water at low (acidic) pH, so it "crashes out". It gathers together into droplets and floats on top of the heavier aqueous (water) solution.

About 40ml of 0.5N HNO3 was added to the sample to make it acidic. The mixture is heated until the fatty matter was floating as a layer above the solution. It is cool in ice water to solidify the fatty matter. The fatty matter was separated and the aqueous solution was treated with 50ml chloroform to remove the remaining fatty matter. The separated fatty matter was a mix, the solvent was evaporated and the yield is noted.

The total fatty matter can be calculated using the following method

% of fatty matter = (**A– B**)/ (Weight of soap sample) x 100

Where **A** = Soap after drying (mg) + weight of dish (mg), **B** = weight of dish (mg)

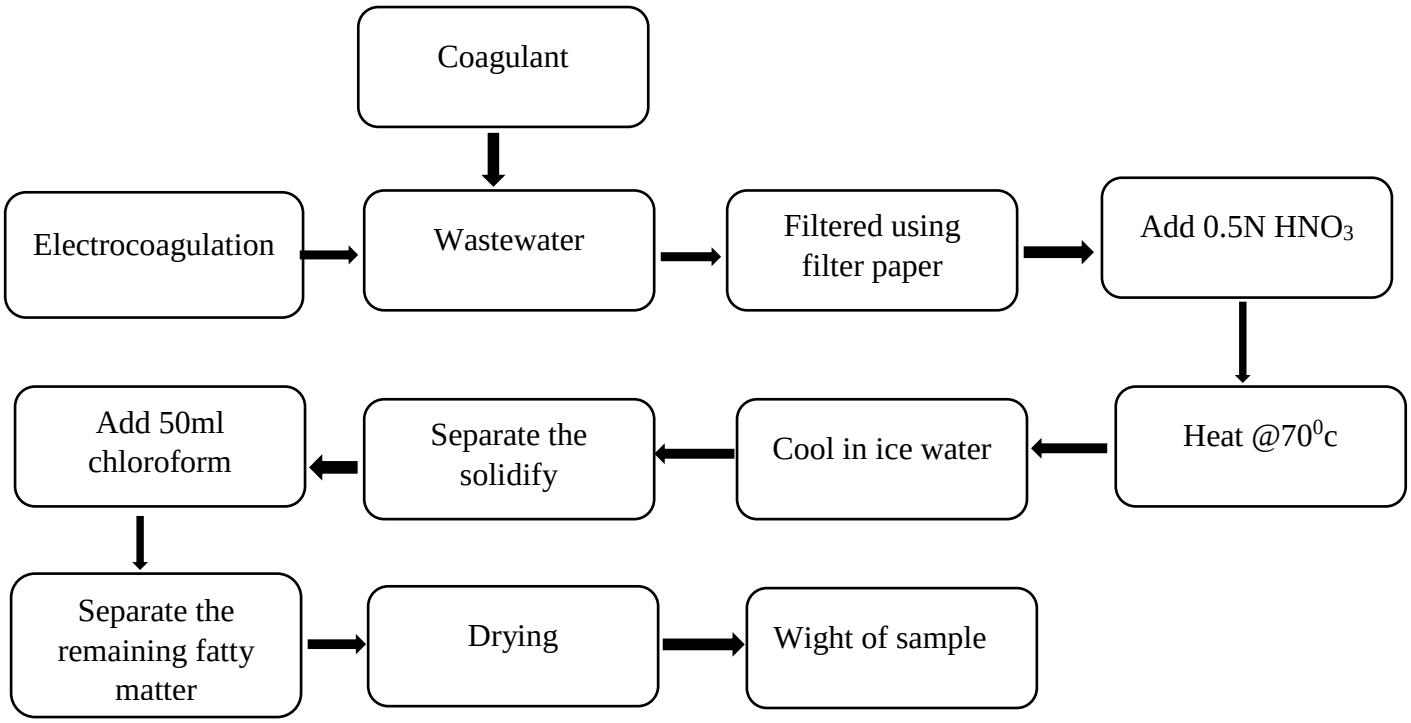


Figure 3. 8**.** Determination of total fatty matter

3.3.6. Determination of chemical oxygen demand (COD)

The chemical oxygen demand (COD) was determined using the open reflux method. Accordingly, most types of organic matter will oxidize by boiling in a mixture of chromium and sulfuric acids. Thus, the wastewater soap sample was re-flux in a strongly acid solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). The oxidation reaction time has been 2 hours, and it was held under standard conditions. At the end of the reaction, the remaining unreacted $K_2Cr_2O_7$ was treated with ferrous ammonium sulfate to determine the consumed amount of $K_2Cr_2O_7$. Thereafter, the oxidizable organic matter amount of the wastewater soap sample was calculated in terms of oxygen equivalent.

3.3.7. Turbidity Measurements

The primary purpose of the coagulation/flocculation process is the removal of turbidity from the wastewater. The cloudiness of water is referred to as turbidity and has its origin in particles suspended in the water. Turbidity is a measure of the extent to of light is either absorbed or scattered by suspended material in water. Turbidity measurements were conducted by using the turbid meter. First, the turbid meter was calibrated with Stable Cal stabilized formazin primary standards of 0.01, 15, 100, 750, and 2000 NTU before starting the analysis. The sample was filled into a sample cell (test tube) and put into the cell holder for measurement and the turbidity of the samples was displayed on the LCD panel of the instrument in NTU.

3.3.8. Determination of Total dissolved solids (TDS)

A Buchner funnel with a rubber bung and fitting, a conical filtration flask, filter paper, a drying oven, a recorder, an analytical balance, a graduated cylinder, and a dish is among the instruments used to conduct the analysis. The method of analysis is a gravimetric method.

The procedure was as follows:

- Take a sample and add 100ml with filter paper and filtrate
- Weight the washed dish carefully
- After filtration is complete transfer the total filtrate sample to a weighted evaporating dish
- Keep drying in a drying oven (104 to 105 o C) for at least 1 hour then weigh.
- After drying weight and calculate the result by using the following formula.

$$Total\ dissolved\ solid(mg/l) = \left(\frac{A-B}{Sample\ volume(ml)}\right) * 1000$$

Where **A** = initial weight

B = final weight

3.4. Characterization

The Physico-chemical characteristics of the prepared coagulant/flocculants materials for the removal of the soap from wastewater were characterized by the following techniques. FTIR spectroscopy was conducted to gain the elemental group present in the prepared bio-coagulant before and after purification and, before and after wastewater treatment. FTIR spectra of the prepared coagulant/flocculants material before and after treatment was recorded in the range of 4000–400 cm−1 using an FTIR spectrometer.

3.5. Design experiments and optimization of process variables

Response surface methods were used to evaluate how the primary parameter affects removal efficiency as well as to optimize all of the affecting parameters collectively through statistical experimental design. The experiments have been carried out using the Box-Behnken statistical experiment design (BBD) of the RSM, which includes a three-factor and three-level for the coagulation process and a four-factor and three-level for the electrocoagulation patterns. The experimental results were studied and interpreted according to design. Statistical program Expert.11.1.0.1 is used to estimate the response of the dependent variable.

3.5.1. Design experiments for the coagulation process

i. Selection of response variable: % total fatty matter removal

ii. Choice of factors, levels, and range

Factors: The potential design factors that have a prime effect on the coagulation of soap wastewater are:-

a. Time

b. Initial concentration

c. Coagulant dose

The variables and their levels in the coagulation process were depicted in Table 3.2

Table 3. 2. The variables and their levels in the coagulation process

No	Parameters	unit	Range and level		
			-1	0	1
1	Dose	g/l	1	3	5
2	Settling time	hr	1	8.5	16
3	the initial concentration of soap	g/l	0.6	0.9	1.2

Utilizing the Response surface (Box-Behnken) design with the factors of time, coagulant dose, and initial concentration with three levels of each factor was selected to see each treatment's effect on the response (Fatty matter), and analysis of variance (ANOVA) was used for statistical significance, the data collected from the treatment of soap wastewater at various times, doses, and initial concentration values were analyzed.

Table 3. 3. Experimental run of the coagulation process

		Factor 1	Factor 2	Factor 3	Response 1
Std	Run	A: time	B: initial concentration	C: dose	FMR
		hr	g/l	g/l	%
12	1	8.5	1.2	5	
13	2	8.5	0.9	3	
3	3	1	1.2	3	
5	4	1	0.9	1	
8	5	16	0.9	5	
4	6	16	1.2	3	
2	7	16	0.6	3	
15	8	8.5	0.9	3	
1	9	1	0.6	3	
6	10	16	0.9	1	
16	11	8.5	0.9	3	
9	12	8.5	0.6	1	
11	13	8.5	0.6	5	
7	14	1	0.9	5	
10	15	8.5	1.2	1	
17	16	8.5	0.9	3	
14	17	8.5	0.9	3	

3.5.2. Design experiments and optimization electrocoagulation

i. Selection of response variable: % total fatty matter removal

ii. Choice of factors, levels, and range

Factors: The potential design factors that have a prime effect on the coagulation of soap wastewater are:-

a. Voltage

b. Reaction time

c. Inter electrode distance

d. Settling time

The variables and their levels in the coagulation process were depicted in Table 3.4

Table 3. 4. The variables and their levels in the electrocoagulation process

NO	Parameters	unit	Range and level		
			-1	0	1
1	Voltage	V	10	20	30
2	Reaction time	min	30	75	120
3	Inter electrode distance	cm	2	3	4
4	Settling time	min	30	105	180

The collected data from the treatment of soap wastewater at different reaction times, voltage, the distance between the electrode, and settling time value were analyzed using the Design-expert version 11 portable software by the Response surface (Box-Behnken) design with factors of reaction time, voltage, the distance between the electrode and settling time with three levels of each factor was selected to see each treatment effect on the response (Fatty matter) was recorded and analysis of variance (ANOVA) used for statistical significance of the experimental data.

Table 3. 5. Experimental run of the electrocoagulation process

Std	Run	Factor 1	Factor 2	Factor 3	Factor 4	Response 1
		A: Voltage	B: reaction time	C: distance	D: settling time	FMR
		V	min	cm	Min	%
24	1	20	120	3	180	
25	2	20	75	3	105	
3	3	10	120	3	105	
14	4	20	120	2	105	
23	5	20	30	3	180	
17	6	10	75	2	105	
22	7	20	120	3	30	
26	8	20	75	3	105	
5	9	20	75	2	30	
21	10	20	30	3	30	
28	11	20	75	3	105	
20	12	30	75	4	105	
12	13	30	75	3	180	
1	14	10	30	3	105	
29	15	20	75	3	105	
18	16	30	75	2	105	
7	17	20	75	2	180	
4	18	30	120	3	105	
15	19	20	30	4	105	
11	20	10	75	3	180	
9	21	10	75	3	30	
16	22	20	120	4	105	
27	23	20	75	3	105	
19	24	10	75	4	105	
8	25	20	75	4	180	
6	26	20	75	4	30	
13	27	20	30	2	105	
2	28	30	30	3	105	
10	29	30	75	3	30	

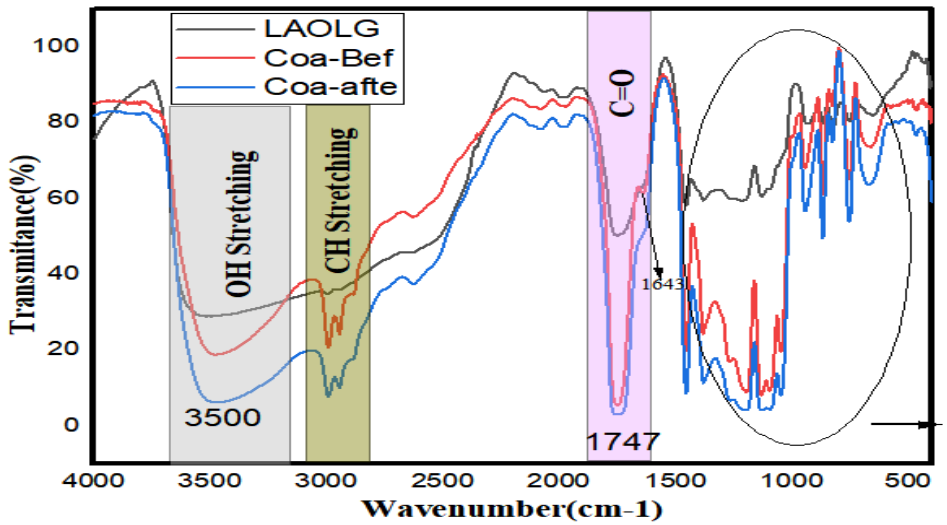
CHAPTER FOUR

4. Result and Discussion

4.1. Synthesis, purify, and characterize bio-based coagulant

4.1.1. Functional group analysis of synthesized coagulant and purify coagulant using FTIR

Figure 4-1 the results of the Fourier transform infrared (FTIR) for the lactic acid oligomer (LAOLG), synthesized and purified coagulants are shown below. The end groups of the various chains in the conjugate are compared using IR analysis as a qualitative technique. At 3100-3600 cm-1, a large transmittance peak is seen. A broad peak found at about 3464 cm−1 is evidence of the presence of OH end groups in the synthesized glycerol lactate conjugate (Valerio, Pin, Misra, & Mohanty, 2016). The samples show a distinctive transmittance peak at 1747 cm-1 for an ester functional group, 2951 cm-1 for a CH2 group, and 2988 cm-1 for a CH3 group. When seeing the transmittance peak at 1643 cm-1, which represents the carbonyl stretching of the remaining free lactic acid after the condensation reaction, the removal of unreacted reactant from the synthesis coagulant increases the percentage intensity of the ester functional group around wave number 1746 cm-1 and the percentage intensity of OH functional group at the wave number 3100 -3600. In another case, a transmittance peak is observed at 995 cm-1that is for the C-O stretching of the primary hydroxyl group in glycerol, and this peak is observed in the synthesized bio-based coagulant but not found on the LAOLG spectra. The other peak found at 1044 cm-1 is for C-O stretching in the secondary OH group of glycerol and this peak is also found on the LAOLG spectra



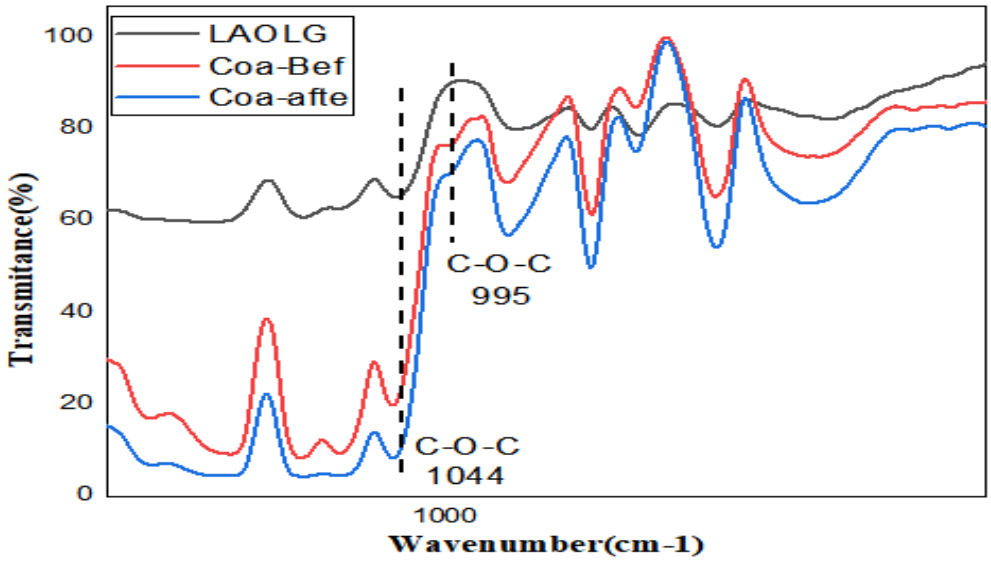


Figure 4. 1. FTIR graph of the LAOLG, synthesized coagulant, and purify coagulant

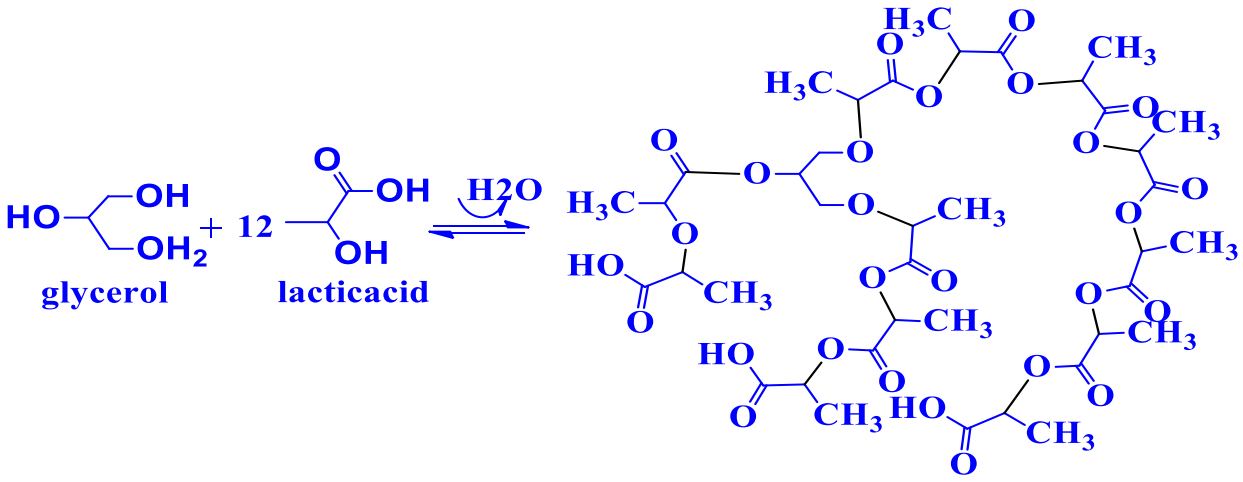


Figure 4. 2. Reaction Scheme for the synthesis of Glycerol lactate 1:12 Bio-based coagulant

In the condensation reaction glycerol core lactic acid oligomer was synthesized from 1 mole of glycerol and 12 moles of lactic acid by removing water (OH from lactic acid and H from glycerol). The first 3 moles of lactic acid grow on the OH end of glycerol and the remaining lactic acid grows on the OH end of the lactic acid which is grown on the glycerol.

Finally, it can be concluded that the product is made of a glycerol core and lactic acid chain unit, where the lactic acid is attached to the primary and secondary hydroxyl groups of glycerol with

the presence of a reactive OH end functional group at the end of the chain and after purification was conducted unreacted raw material were removed from synthesis coagulant.

4.1.2. Effect of mixing ration and purification on viscosity

As indicated in table 4-1 it is evident that the purification and mixing ratio has an impact on viscosity. Due to the presence of sufficient numbers of reactive groups in the system, the conjugate's viscosity increases as the molar ratio increases. The more product is produced the higher the reactant concentration. We may obtain five distinct viscosity levels by adding varying amounts of lactic acid while maintaining the same mole of glycerol. The viscosity decreased as the amount of glycerol core increased, but it increased as the number of lactic acids increased. This indicates that the glycerol arm's chain length affects the conjugates' viscosity. After purification, the viscosity of the conjugate increases. This is due to the higher amount of unreacted Lactic acid monomers that were eliminated from the final product during the purification process, increasing the viscosity of the products.

Table 4. 1. Viscosity before purification and after purification

Glycerol to lactic acid ratio	1:3	1:6	1:9	1:12	1:16
Before purification	263	295	356	371	421
After purification	>5270	>5270	>5270	>5270	>5270

4.1.3. Effect of mixing ration and purification on acid value

The acid value is *a* measure of potassium hydroxide (mg) that is required to neutralize the free fatty acid contained in the unit mass (g) of the chemical substance. It is shown in table 4-2 that the mixing ratio and purification both affect the acid value of the conjugate. As the molar ratio of lactic acid to glycerol increases, the acid value also increases. This indicates that the acid value of the conjugates is influenced by the amount of lactic acid. The conjugate's acid value decreases after purification. This is because a larger amount of unreacted lactic acid monomers were removed from the finished product during the purification process, lowering the product's acidity.

Table 4. 2. Acid value before purification and after purification

Glycerol to lactic acid ratio	**1:3**	**1:6**	**1:9**	**1:12**	**1:16**
Before purification	72.34	133.56	151.21	159.02	164.76
After purification	31.51	87.52	103.18	117.02	125.61

4.2. Characteristics of Raw soap Wastewater

The initial step was to determine the physic-chemical composition of the first sample of the soap wastewater (Ph., TDS, Fatty matter, COD, and turbidity). The wastewater from the laundry appears to be more alkaline as a result of the average pH of the sample effluent wastewater being 9.07, as shown in Table 4.3 below. The wastewater sample included 1450 mg/l of total dissolved solids as well. Average values for fatty matter, COD, and turbidity in the effluent were 74.26%, 1500 mg/l, and 165 NTU, respectively. Overall, the physical-chemical characterization of the wastewater effluent (Ph., TDS, fatty matter, COD, and turbidity) results indicate that treatment is essential to prevent environmental pollution degradation of groundwater and surface water resources. This is done by using the synthesized coagulant and electrocoagulation process, which significantly reduces the values of COD, fatty matter, TDS, and turbidity.

Table 4. 3. Raw soap wastewater Physic-chemical characteristics

parameters	value
PH	9.07
TDS	1450 mg/l
Fatty matter	74.26%
COD	1500 mg/l
Turbidity	165 NTU

4.3. Effect of coagulation parameters on percentage removal of TFMR

4.3.1. Effect of settling time

As shown in figure 4.3 and table 4.4 in the coagulation process, the settling time is an important factor that can influence the overall removal efficiencies of fatty matter in wastewater treatment. To determine the effective settling time, experiments were done at different settling times of (1hr-16hr) while other parameters such as agitation time (30 min), agitation speed(100rpm), temperature(25^0c), initial soap concentration (0.9g/l), and coagulant dose (3 g/l) were kept constant. The results indicate that the percentage of fatty matter removal was increased with increasing settling time up to 8 hr and then decrease for 1:3 glycerol lactate coagulant and the percentage of fatty matter removal was increased with increasing settling time up to 16 hr for those 1:6, 1:9, 1:12, 1:16 this is because as the settling time increases the contact surface between the coagulant and pollutant was high due to this reason the floc formation over the system was increased.

Table 4. 4. Effect of settling time on TFMR

Time(hr.)	**Glycerol to lactic acid ratio**				
	1:3	**1:6**	**1:9**	**1:12**	**1:16**
1	2.1	24.6	37.1	31.4	12.8
4	32.5	27.2	39.9	69.7	23.5
8	23.2	38.4	62.5	81.3	32.3
12	6.7	56.9	83.8	91.1	38.6
16	4.6	57.9	84.1	88.4	46.9

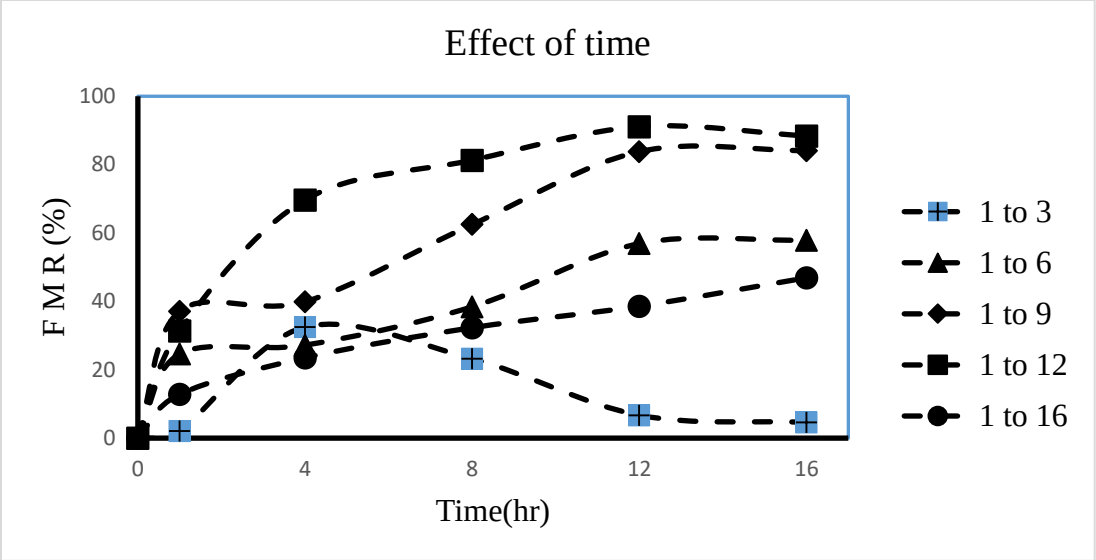


Figure 4. 3. Effect of settling time on TFMR

From the FTIR results as shown in the figure below there is an O-H stretching within the range (3100 – 3600 cm-1 wave number. It shows that as the O-H broadness and intensity increase the interaction (bond) of the coagulant (glycerol lactate) and pollutant is low in strength because as the O-H peak intensity of the conjugate increase the product is more hydrophilic and easily dissolve in water and easily break the bond between the coagulant and pollutant. The first 4 (1:3, 1:6, 1:9, 1:12) coagulant (glycerol lactate) increase their effectiveness as the hydrophobicity of the product decrease but for 1:16 the effectiveness of coagulant (glycerol lactate) decreases because the glycerol lactate conjugate was interacted with another glycerol lactate rather than interact with the pollutant.

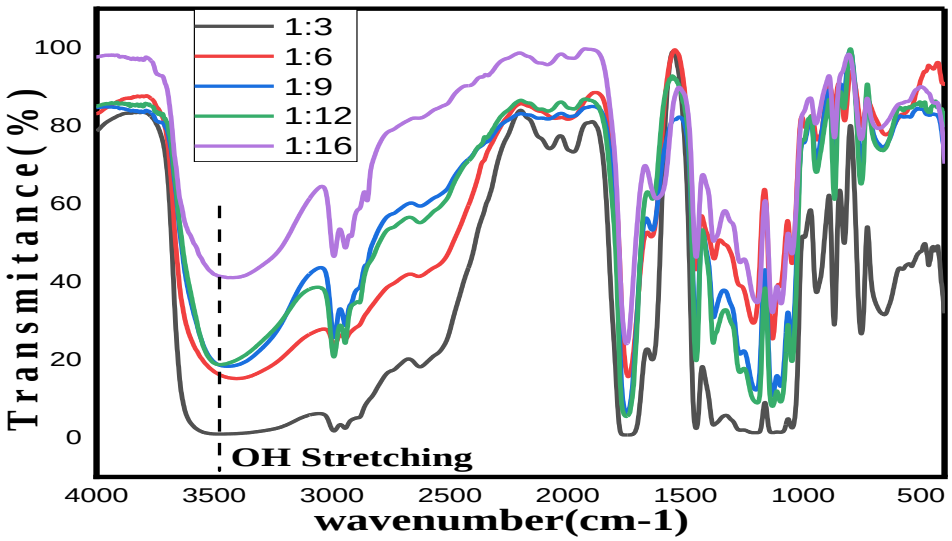


Figure 4. 4. FTIR graph of the synthesized bio-based coagulant conjugates

As indicated in table 4.5 the peak intensity of ester peak to OH peck decrease as the glycerol to lactic acid increase, this result indicates the OH intensity decrease with an increased mixing ratio of glycerol to lactic acid.

Table 4. 5. Peak intensity ration of the synthesized bio-based coagulant

Glycerol to LA ratio	**Intensity (%)**		**Peak intensity ratio**
	C=O(baseline)	OH	C=O/(OH)
1:3	1.766	1.12	1.58
1:6	16.54	15.38	1.075
1:9	26.54	19.6	1.35
1:12	10.23	16.8	0.609
1:16	25.5	43.19	0.5904

4.3.2. Effect of coagulant dose

Coagulant dose is one of the most significant parameters that has been taken into consideration to estimate the performance of coagulants in coagulation and flocculation as Figure 4.6 shows the

influence of coagulant dose on the percentage removal of fatty matter. In essence, a dosage that is either minimal or excessive will lead to weak performance. Therefore, it is important to determine the optimum dose in order to reduce dosing costs and sludge formation as well as to achieve the best results possible during the treatment process. The experiments to test the effect of coagulant dose ranging from 1 – 5g, while mixing time of 30 minutes consisting of rapid mixing at 100 rpm , at constant initial soap concentration which is 0.9g/l and settling time (12hr). Accordingly, there was continuous removal of fatty matter with increases in coagulant doses up to 3g and thereafter no significant increase was observed for 1:9 and 1:12. This is because of two reasons I) at low coagulant doses the soap uptake capacity is high because of the better utilization of the available active sites and at high coagulant dose, too many sites are available for a limited quantity of soap. ii) At a high coagulant dose, the lower equilibrium concentration of soap.

Table 4. 6. Effect of coagulant dose on TFMR

Dose(g/l)	**Glycerol to lactic acid ratio**				
	1:3	**1:6**	**1:9**	**1:12**	**1:16**
1	4.1	26.8	39.3	49.6	33.8
3	5.4	57.5	84.7	90.6	38.1
5	12.8	62.1	87.3	91.7	42.7

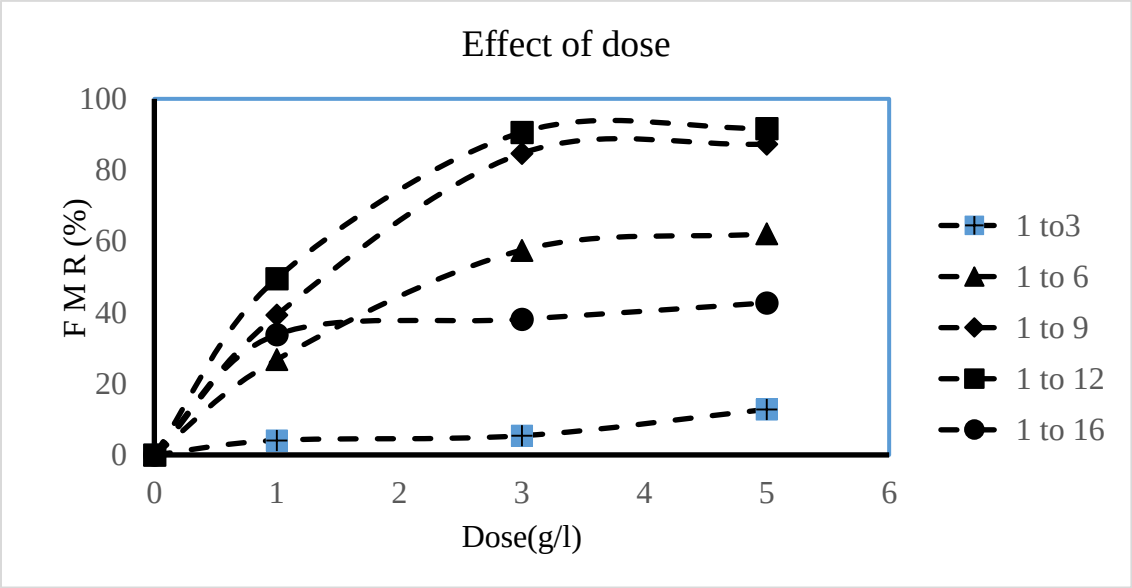


Figure 4. 5. Effect of coagulant dose on TFMR

But for 1:3, 1:6, and 1:16 increase the removal of the fatty matter when increasing the coagulant dose from 3- 5g because the removal percent of those coagulants was very low (5.4%, 57.5%, 38.1%respectively for 1:3, 1:6, 1:16) at 3g of coagulant dose the amount of coagulant was not had enough active site to remove the dissolved soap particles so, it needs additional coagulant dose to have a more active site. The minimum removal efficiency of the fatty matter was observed at a coagulant dose of 1g which is around (4.1, 26.8, 39.8, 49.6, and 33.8 % respectively for 1:3, 1:6, 1:9, 1:12, and 1:16 and the maximum was observed at around 5g which is (12.8, 62.1, 87.3, 92.7 and 42.7% respectively for 1:3, 1:6, 1:9, 1:12 and 1:16)

4.3.3. Effect of initial concentration of soap

The effect of initial concentration on the removal of the fatty matter was studied at different initial concentrations of soaps by keeping other parameters constant. The effect of initial concentration on the removal of the fatty matter is shown in Fig 4.6 and table 4.7 it was found that with the increase in initial concentration of soap from 0.6g/l to 0.9 g/l, the removal capacity of fatty matter also increases. The observation is obvious due to the availability of a higher number of fatty matter ions resulting in a higher concentration gradient. With the increase of the concentration of soap in solution, the availability of fatty matter ions also increases at the interface of the coagulant, increasing removal performance, and the maximum removal efficiency was about 90.3%. After reaching 90.3 % removal of fatty matter it decrease to 87.4% this was happening because of the increase in the initial concentration of soap increase from 0.9g/l to 1.2g/l. for 1:9 and 1:12, as the initial soap concentration increased, fatty matter removal efficiency decreased, so the efficiency for soap concentrations of 0.9g/l and 1.2g/l was 84.9% and 82.1% for 1:9 and (90.3% and 87.4%) for 1:12, respectively. In the case at the first, the removal efficiency increased slightly with the increase of soap concentration from 0.6 g/l to 0.9 g/l. After that, the efficiency decreased slowly with the increase in soap concentration.

Table 4. 7. Effect of initial soap concentration on TFMR

Initial soap concentration(g/l)	Glycerol to lactic acid ratio				
	1:3	**1:6**	**1:9**	**1:12**	**1:16**
0.6	3.5	50.2	72.3	78.2	35.2
0.9	5.3	57.6	84.9	90.3	37.8
1.2	4.4	59.9	82.1	87.4	37.8

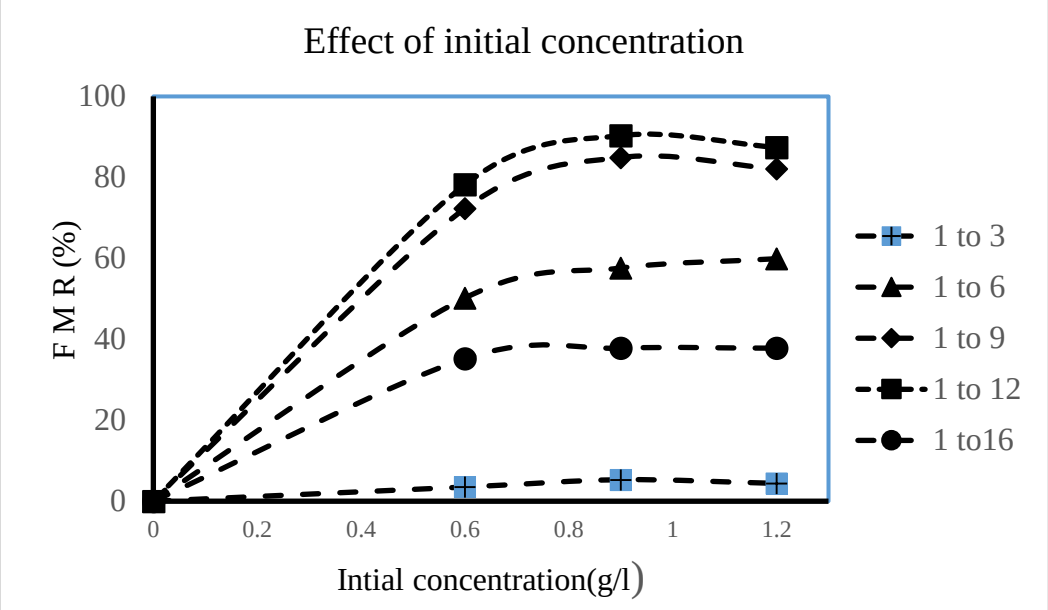
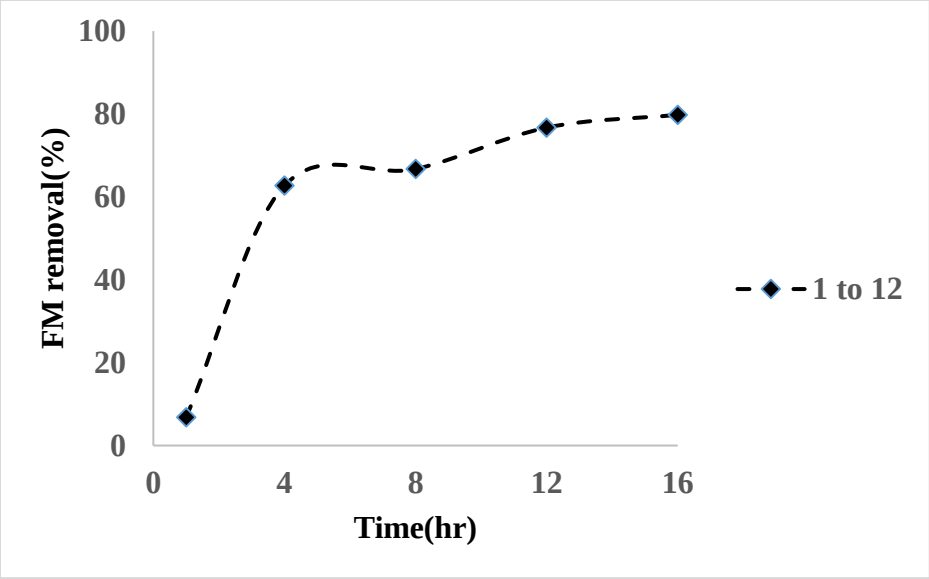


Figure 4. 6. Effect of initial soap concentration on TFMR

4.3.4. Fatty matter removal on the real wastewater

The batch experimental studies for real wastewater by selecting Adama Science and Technology University student laundry as a soap wastewater source. The result investigated showed that 81.8% of the fatty matter was removed from wastewater. The reduction in removal efficiency from that synthetic wastewater was due to a large number of competing pollutants within the waste.



Time(hr)	TFMR (%)
1	34.6
4	62.7
8	73.4
12	79.5
16	81.8

Figure 4. 7. Fatty matter removal on the real wastewater

4.4. Statistical Analysis of the Experimental Results for fatty matter removal using bio-based coagulant

The process consists of three main factors: time, coagulant dose, and initial concentration of soap with one response. After following the above series of procedures, the experimental outcomes of those particular results were measured for fatty matter removal. The result obtained from the Experiment was indicated in table 4.8

Table 4. 8. Results obtained from the experiment of coagulation

		Factor 1	**Factor 2**	**Factor 3**	**response**
		A: time	**B: initial concentration**	**C: dose**	**TFMR**
Std	**Run**	hr.	g/l	g/l	%
12	1	8.5	1.2	5	84.6
13	2	8.5	0.9	3	82.5
3	3	1	1.2	3	28.5
5	4	1	0.9	1	20.8
8	5	16	0.9	5	90.5
4	6	16	1.2	3	85.5

2	7	16	0.6	3	76.3
15	8	8.5	0.9	3	81.6
1	9	1	0.6	3	19.3
6	10	16	0.9	1	47.4
16	11	8.5	0.9	3	82.2
9	12	8.5	0.6	1	29.4
11	13	8.5	0.6	5	72.5
7	14	1	0.9	5	33.5
10	15	8.5	1.2	1	47.4
17	16	8.5	0.9	3	80.4
14	17	8.5	0.9	3	81.9

The removal was performed by using glycerol lactate as a coagulant at different factors: Time, coagulant dose, and initial soap Concentration with a constant mixing time of 30 min stirring Speed of 100 rpm, and 298k temperature.

Using Design Expert 11.1.2.0 software, the data shown in Table 4.8 were examined to determine the impact of the operating parameters of coagulant dose, time, and initial soap concentration. The percentage of removal was the dependent variable employed as a response parameter. To reduce the impact of unexpected variability in the observed response due to outside variables, all tests were conducted in random order. Summary of the design for the percentage removal with the three levels and three components. Using Design Expert 11 software, the experiments' design model is a quadratic polynomial with five centers for each block.

4.4.1. Effects of Experimental Variables for TFMR

Table 4. 9. Analysis of variance (ANOVA) for the TFMR

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	10868.04	9	1207.56	40.21	< 0.0001	significant
A	4880.72	1	4880.72	162.52	< 0.0001	
B	294.03	1	294.03	9.79	0.0166	
C	2315.40	1	2315.40	77.10	< 0.0001	
AB	0.0000	1	0.0000	0.0000	0.6070	

AC	231.04	1	231.04	7.69	0.0275	
BC	8.70	1	8.70	0.2898	< 0.0001	
A^2	1666.66	1	1666.66	55.50	0.0001	
B^2	375.25	1	375.25	12.50	0.0095	
C^2	800.75	1	800.75	26.66	0.0013	
Residual	99.13	7	14.16			
Lack of Fit	69.7	3	22.23	3.16	0.1479	not significant
Pure Error	29.43	4	7.36			
Cor Total	17603.98	16				

A = Time, B = Initial concentration of soap, C = Coagulant dose

From the ANOVA (Analysis of Variance) Table, 4.9 results the higher F-Value and the lower P-value (<0.0001) indicate significance for the regression model. As the result demonstrates the model, the F-value of 40.21 implies it is significant. There is only a 0.01% chance that an F-value this large could happen due to noise. A p-value less than 0.05 implies that the model is statistically significant. They are extremely indispensable to understanding the interaction among the independent variables. The result showed that time (Factor A), initial concentration of soap (Factor B), and coagulant dose (Factor C) are the major important variables in the coagulation process with P-values < 0.0001, < 0.0166, and 0.0001 respectively. Based on the value both the coagulant dose and time are the best important factor. From the second polynomial model, A^2, $B^{2,}$ and C^2 are significant models with P-values for both models < 0.0001. In the case of interaction effects, AC (time coagulant dose) and BC (initial soap concentration and coagulant dose) are significant model terms for fatty matter removal where the p-value is 0.0275. The Lack of Fit of the F-value 3.16 implies the Lack of Fit is not significant relative to the pure error. There is about a 14.79% chance that a Lack of Fit F-value large could occur due to noise.

As shown from the ANOVA result, initial soap concentration indicates insignificant when compared with the other factors. This result confirmed that its contribution to fatty matter removal was the lowest. In addition, the interaction effects of time and initial concentration of soap (AB) and initial concentration of soap and coagulant Dose (BC) did not influence the coagulation

process due to their highest P-value. Their P- values were 1 and 0.607 respectively which is greater than 0.1, which indicates the model terms are not significant. In the same way.

The model equations from the ANOVA result can be used to relatively compare the removal efficiency of fatty matter within factors and levels. The equation in terms of coded factors can be used to make predictions about the fatty matter removal efficiency for the given levels of each factor. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. The highest coefficients from the equation indicate the most influential factor that affects the coagulation process of fatty matter removal. On the other side, the equation in terms of actual factors can be used to make predictions about the Removal efficiency of fatty matter removal for given levels of each factor. The levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

The Final Coded Equations for fatty matter removal from ANOVA result:

Fatty Matter Removal Efficiency (FMR) = +81.74 + 24.7* A + 6.06*B + 17.01*C + 7.6*AC - 1.47*BC – 19.9*A^2 + 9.44*B^2 – 13.79*C^2

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

Final Equation of Actual Factors

Fatty Matter Removal Efficiency (FMR) = -125.223 + 7.786*time + 216.393 initial concentration of soap + 27.098 coagulant dose + 1.97*time*initial concentration of soap + 0.507* time* coagulant dose – 2.458 initial concentration*coagulant dose – 0.354*$time^2$ -104.89*initial concentration of $soap^2$ * - 3.448*$dose^2$

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

4.4.2. Model Adequacy

The regression model was found to be significant with the correlation coefficients of determination of R-Squared, adjusted R-Squared, and predicted R-Squared having a value of 0.9944, 0.9871, and 0.9340 respectively as shown in table 4.10. The goodness fitness of the model was measured by a coefficient of determination R^2, which measured how well the real experimental data points have been approximated together with the regression model. As the result in a table, 4.10 expressed the value of $R^2 = 0.9944$ indicates the better fit of experimental data to that of the predicted one. The value of the larger Regression coefficient (R^2) approach to 1 illustrates, the validity of the model in which it implies an acceptable agreement among the actual and predicted data. It implies that 99.44% of the experimental variable studied attributed to the removal of fatty matter. The Predicted R^2 of 0.9340 is in reasonable agreement with the Adjusted R^2 of 0.9871; i.e. the difference is less than 0.2. Therefore, this demonstrates that the measured data is very well fitted with a quadratic model of the predicted value. Model Precision measures the signal-to-noise ratio. A ratio greater than 4 is desirable. Your ratio of 19.849 indicates an adequate signal. This model can be used to navigate the design space.

Table 4. 10. Model adequacy measures for TFMR

Std. Dev.	3.76	**R^2**	0.9944
Mean	49.86	**Adjusted R^2**	0.9871
C.V. %	7.55	**Predicted R^2**	0.9340
Press	89.8	**Model Precision**	30.5940

Generally, the smaller the value of the Predicted residual sum of squares (PRESS) static obtained 89.8 indicates, the better the experimental data points fit the model. Moreover, the high R^2 value and p-value as well as not significant fit indicate the best prediction of the experiment solution. Therefore, the result in both tables 4.8 and 4.9 proved that, the best performance of the fitness of the model equations.

Figure 4.8 indicates the relationship between Actual and predicted values, which can be introduced from the regression model equations of a Design Expert chart based on predicted and actual percent fatty matter removal. The straight line shows the predicted values and the small rectangles represent the actual experimental values. The straight line is very close to the actual values and

has a correlation coefficient R^2 equal to 0.9944, which confirms the accuracy of the model. The result confirmed that all the actual and predicted values are close to the line of a perfect fit. Both of them showed good agreement between them.

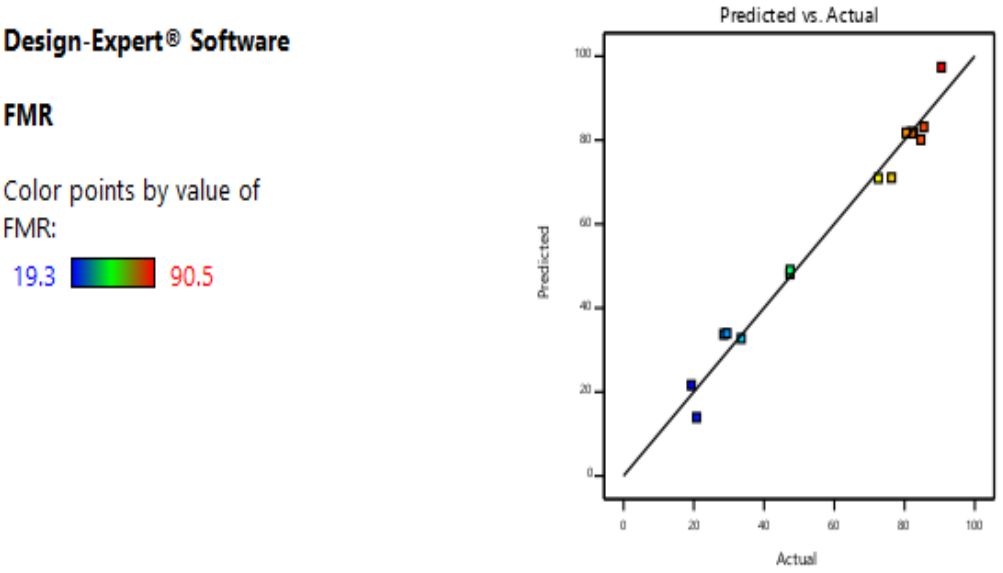


Figure 4. 8. Predicted vs Actual value fatty matter removal plot

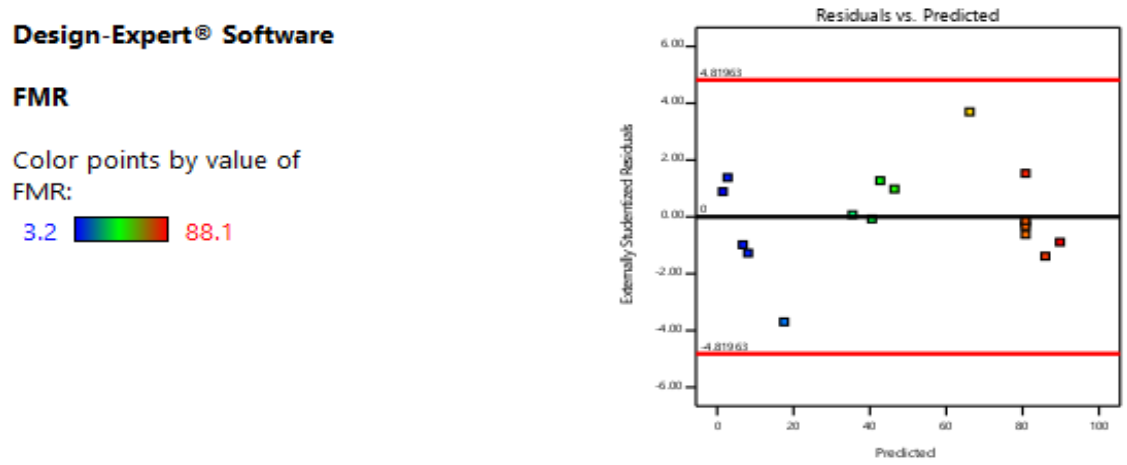


Figure 4. 9. Residual versus predicted values for fatty matter removal

If the model is correct and the assumptions are satisfied, the residuals should be structured less; in particular, they should be unrelated to any other variable including the predicted response. A

simple check is to plot the residuals versus the fitted (predicted) values. A plot of the residuals versus the rising predicted response values tests the assumption of constant variance. The plot shows random scatter which justifies no need for an alteration to minimize personal error. The effects of the parameters for the percentage of fatty matter removal are to generate one-factor response and surface plots of the equation.

4.4.3. Optimization of the Response

To find the optimum value of each independent variable, four statistical metrics such as the larger regression coefficient (R^2), smaller lack of fit F-value, lower p-value, and higher sum squares have been used. The response optimization was used to identify the combination of independent variables particularly, time, initial soap concentration, and coagulant dose within the response of fatty matter removal efficiency using glycerol lactate coagulant (FMR). The goal of the process was to obtain high fatty matter removal percentage. Therefore it has been set as a maximum while the process parameters such as initial soap concentration and coagulant dose have been set as "within the range", while the time target to minimize the response surface method was used to optimize the process versus process parameters. According to the result obtained from optimizer Design-Expert version 11 software the maximum optimum removal efficiency of the fatty matter was achieved at 87.75% % by using 11.54 hr time, 1.104 g/l of initial concentration of soap, and 4.221 g/l of coagulant dose.

4.4.4. Response Surface plots Interaction Effects

The effect of the interactions between the three independent input and output variables and their interactions on the dependent variables can be seen using three-dimensional (3D) response surface plots. As observed from Figure 4.10 - 4.12 indicates the Plots of three-dimensional response surfaces of the function of two variables with the response and the remaining variables at the central level of the other variables.

Figure 4.10 indicates the interaction effects of time and initial soap concentration on fatty matter removal at a fixed coagulant dose and mixing rate and time. Initially, as initial concentration increases, the removal efficiency of fatty matter also increases. The rise of initial soap concentration leads to increments in the removal of fatty matter from the solution due to the occurrences of large vacant places in the glycerol lactate coagulant. As the initial concentration beyond 0.9 g/l increases, the removal efficiency decreases because the vacant sites of the coagulant

are occupied by the pollutant. In the other case as the time increases leads to an increase in the removal efficiency of fatty matter and reached an optimum of 87.75 at 11.54 hr. The rise of time leads to increments in the removal of fatty matter from the solution due to the high surface contact of the pollutant and the coagulant.

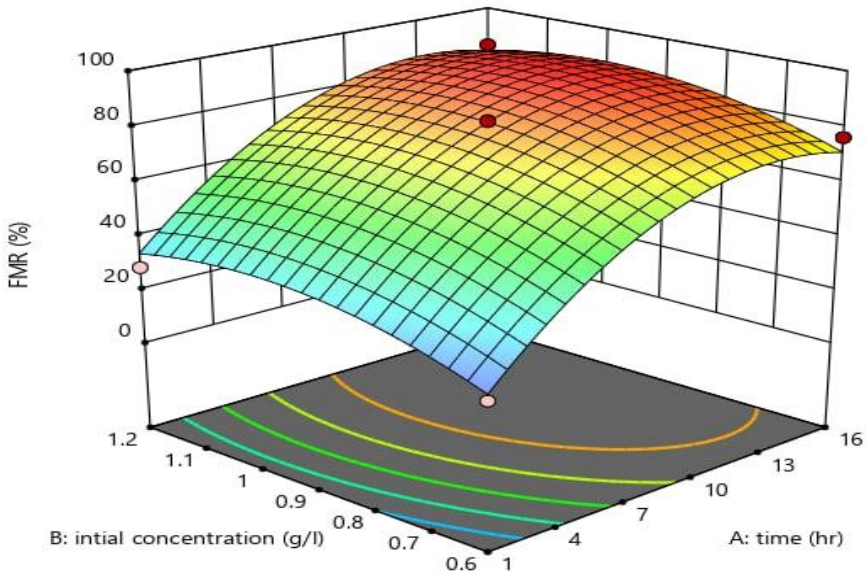


Figure 4. 10. 3D interaction effect of time and initial soap concentration

Figure 4.11 showed the interaction between initial soap concentration and coagulant dose. As demonstrated by increasing the initial soap concentration, the percentage removal of fatty matter removal increased proportionally. The process happened due to a higher concentration gradient that was performed as a driving force to conquer the mass transfer between the bulk solution of soap ion and coagulant surfaces of glycerol lactate. The coagulant dose has no more effect on fatty matter removal at a lower value of initial soap concentration. As the initial soap concentration increased, the percentage of fatty matter removal also increased as a result of the coagulant dose increased. Therefore, the coagulant dose was the main independent variable that affects the removal of fatty matter from the solution.

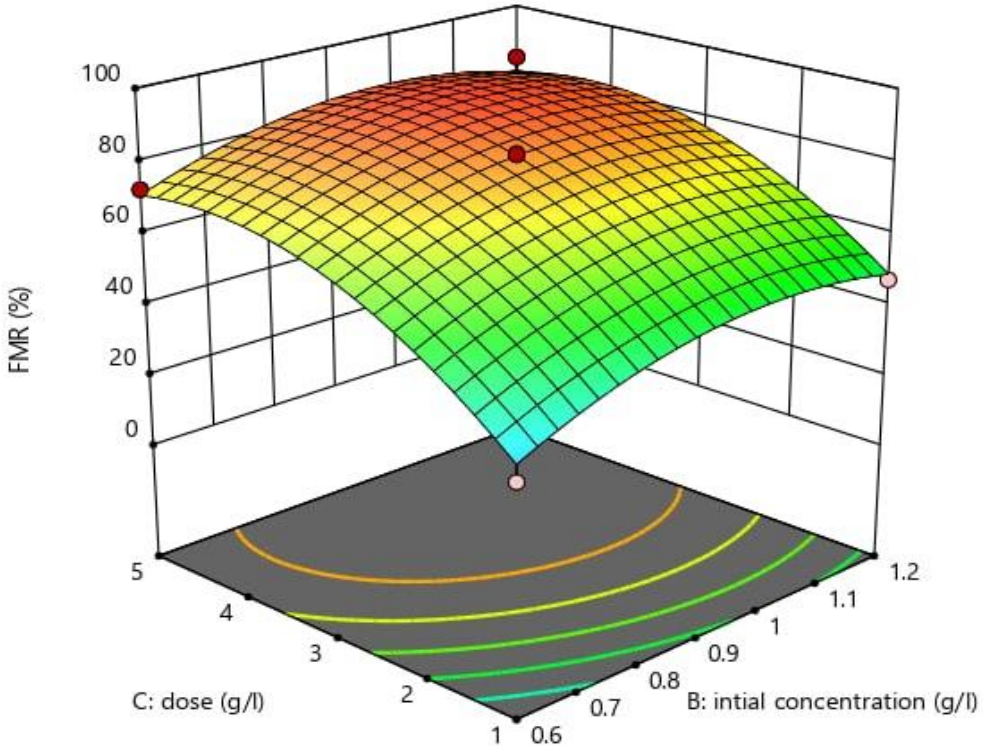


Figure 4. 11. 3D interaction effect of coagulant dose and initial soap concentration

Figure 4.12 illustrates the 3D interaction effect of coagulant dose and time on fatty matter removal efficiency at constant initial soap concentration and mixing rate and time. As the coagulant dose increased, there was an increment in fatty matter removal efficiency. However, the percentage of fatty matter removal did not increase with increasing time at any coagulant dosage. The coagulant has reached the maximum removal capacity within or before 12 hours. In the other words, the surface coverage of the coagulant was already saturated with the fatty matter in less than 12 hours. Therefore, it can be concluded that the percentage of fatty matter removal was highly dependent on the coagulant dose and time but fatty matter removal did not more depend on the contact initial concentration of soap.

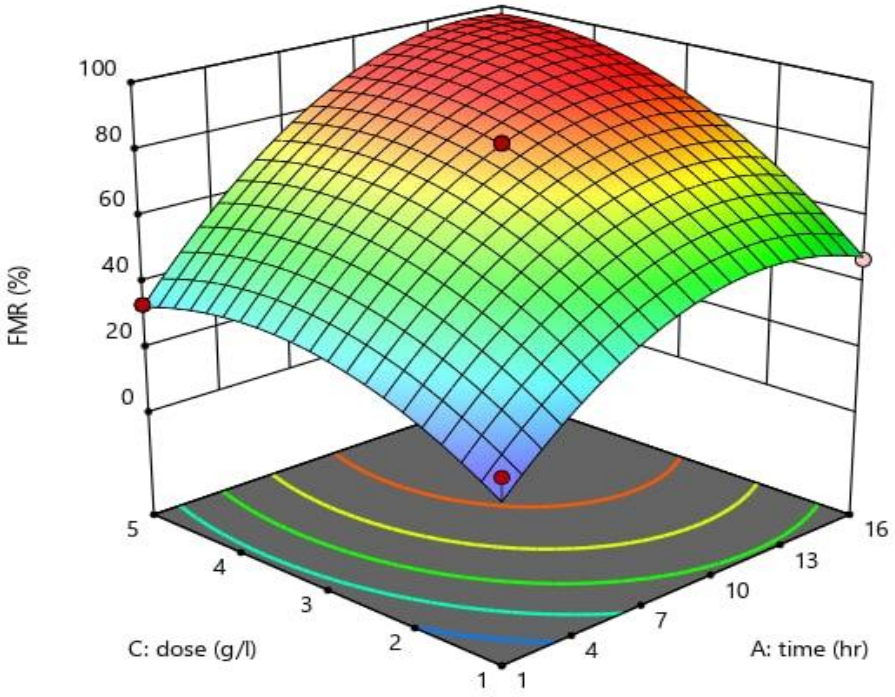


Figure 4. 12. 3D interaction effect of time and coagulant dose

4.5. FTIR analysis of glycerol lactate after using as a coagulant

Figure 4-13 below shows the Fourier transform infrared (FTIR) result for the purified coagulant, flocs (solid particles that are formed after we use the coagulant), and soap. From the FTIR results as shown in the figure below there is an O-H stretching, C=O (ester bond), and, R-COONa within the range of (3600- 3100 cm $^{-1}$ peaks), (1770 - 1720 cm^{-1}) and, (1616-1550 cm^{-1}) respectively. The graph shows that after the synthesized product was used as a coagulant for soap wastewater treatment the intensity of O-H stretching of the floc (coagulant after use for the soap wastewater treatment) was decreased. O-H stretching was decreased because the H^+ leaves the coagulant anion, and at the same time, the Na^+ joins with the coagulant anion, making a molecule with a COONa group on one end of the coagulant. The peak shown in the wave number 1570 cm-1 indicates the presence COONa. The peak around 1746 cm-1 wave number also decreased because the conjugate is grow over the O-H end and the O-H stretching decreased after being used for treatment purposes.

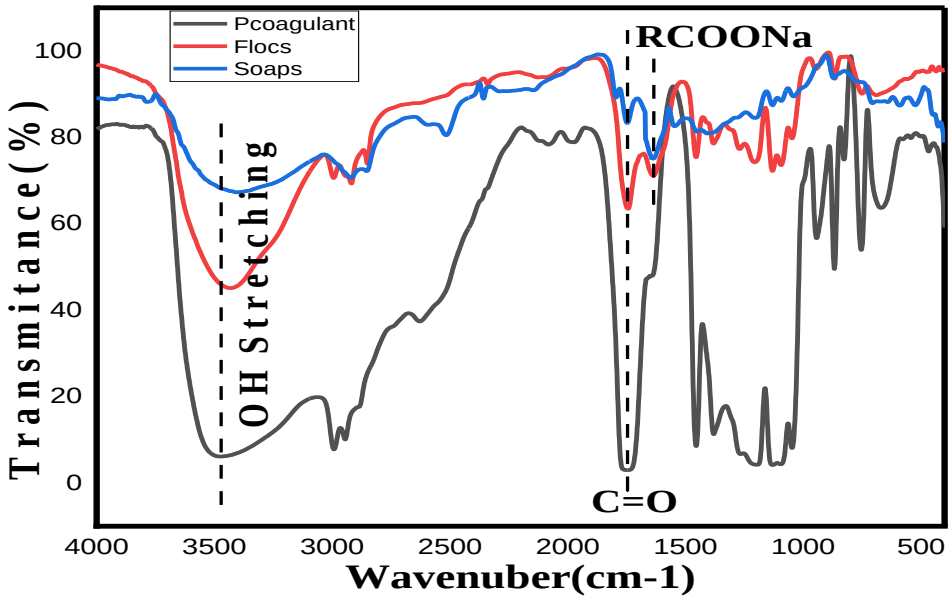


Figure 4. 13. FTIR graph of the synthesized coagulant after the coagulation process

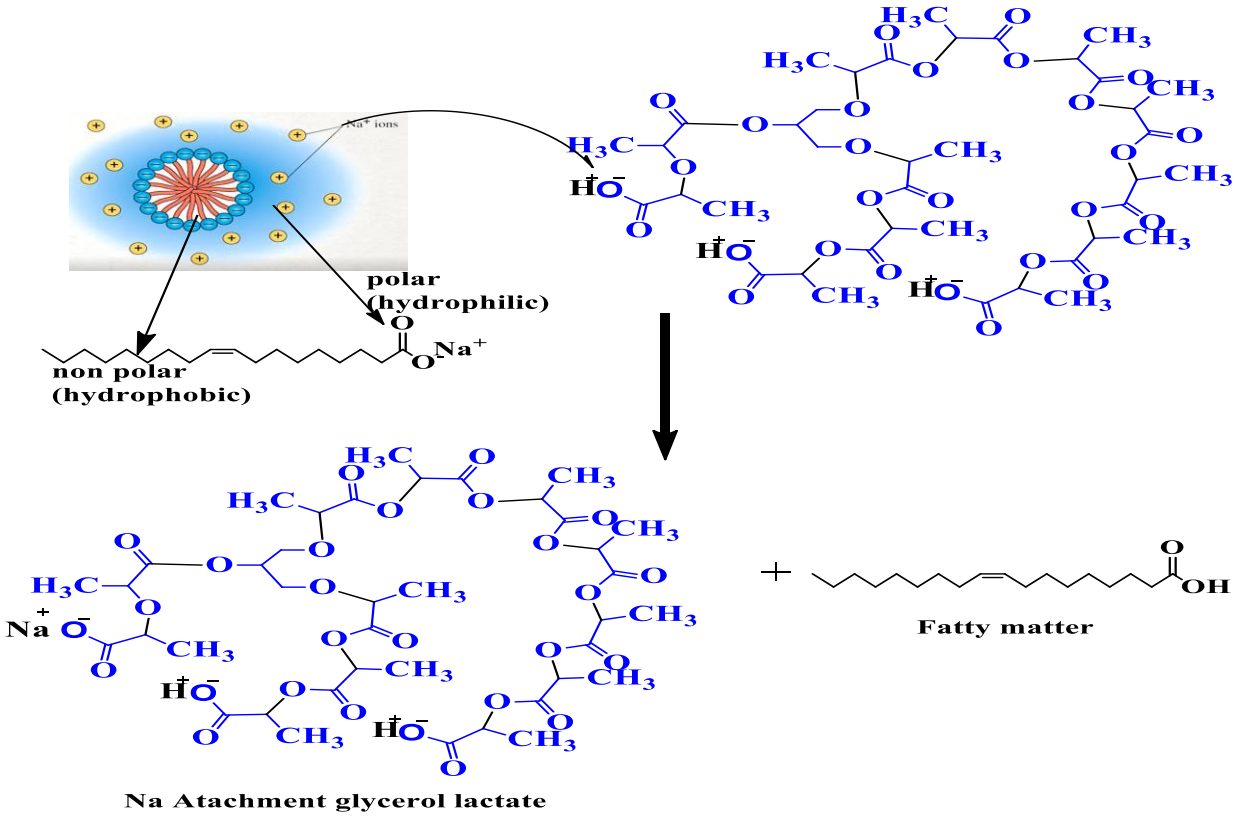


Figure 4. 14. Interaction of soap and glycerol lactate

The process to get fatty matter from soap is the Na^+ and H^+ trade places. The Na^+ leaves the fatty anion, so it can go be friends with glycerol lactate in solution, and at the same time, the H^+ joins with the fatty anion, making a molecule with a COOH group on one end, a fatty matter. The fatty matter is not soluble in water, so it "crashes out". It gathers together into droplets and floats on top of the heavier aqueous (water) solution.

Finally, it can be concluded that the product is made of a glycerol core, and lactic acid conjugate act as a coagulant by trapping sodium ion (Na^+) by the O-H end of the conjugate and forming fatty matter.

4.6. Effect of electro-coagulation parameters on TFMR

When bio-based coagulation which is produced from glycerol was used it needs 11.54hr to reach 87.75% fatty matter removal so, to improve the fatty matter in a short time electrocoagulation was used in this experiment.

4.6.1. Effect of voltage

Regarding the effect of voltage on the percentage removal of fatty matter in soap wastewater, the laboratory experimental result is shown in table 4.11 and Figure 4.15. According to the result, it is observed that when voltage increases the percentage removal of fatty matter increases. The fatty matter reduction percentage increases somewhat proportionally to voltage for 10v, 20v, and 30v. That is with higher voltage showing proportionally higher reductions. The reason for the increment in the removal of the fatty matter is when voltage increases the number of bubbles and flocs formed also increases. The bubbles formed during the reaction results in the floating of lighter pollutants. This again results in the formation of more flocs and increases the rate of coagulation. The increase in the rate of coagulation again increases the removal of pollutants.

The explanation for the increment in the percentage removal of the fatty matter of soap wastewater is as follows. Operating voltage and electric current are critical in batch electrocoagulation. The voltage directly determines both the coagulant dose and bubble generation rate, as well as strong influences on both mixings of solution and mass transfer at the electrodes(Lumina, Pavithra, & Harinath, 2010). The metal ion can react with the OH- ions produced at the cathode during gaseous H_2 evolution, to yield insoluble hydroxide which traps pollutants out of the solution. It also contributes to coagulation by neutralizing the negatively charged colloidal particles. Metal

hydroxide flocs formed during electro-coagulation have a large surface area beneficial for rapid adsorption of soluble organic compounds and ensnaring of colloidal particles.

Table 4. 11. Effect of voltage on TFMR

Rxn time(min)	**Voltage(V)**		
	10V	20V	30V
30	33.5	54.3	60.2
60	44.7	65.4	76.8
90	51.3	73.1	79.4
120	57.4	74.8	80.6

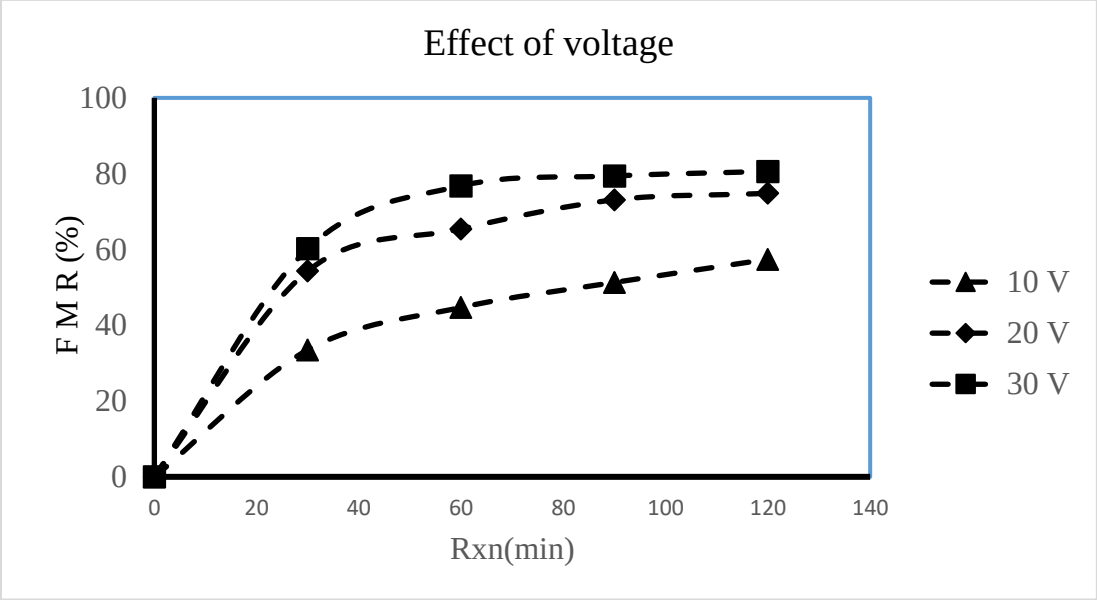


Figure 4. 15. Effect of voltage on TFMR

These flocs are finally easily removed from the aqueous medium by sedimentation or flotation(Janpoor, et al., 2011). As reported by several authors, they concluded that the percentage of pollutant removal is increased due to an increase in voltage(Sharma & Simsek, 2019).

4.6.2. Effect of inter-electrodes distance on TFMR

In the EC process, inter-electrode distance plays an important role in EC strength because the electrostatic field depends on the distance between the anode and the cathode. An optimum distance between electrodes provides maximum pollutant removal efficiency. Minimum inter-electrode distance provides low pollutant removal efficiency. The more the inter-electrode distance the slower the movement of the generated ions. Due to the slower movement ions gets extra time to form the floc required for the coagulation of pollutants. Whereas an additional increase in the inter-electrode distance above optimal value decreases anodic dissolution and will increase the distance that ions essential to travel for floc formation, which results in a decrease in the electrocoagulation efficiency. Electrocoagulation efficiency depends upon the conductivity of the solution. As shown in the equation below the electrical conductivity is directly proportional to the inter-electrode distance. An increase in distance between the anode and the cathode (D), increases resistance (R) offered by the cell.

$$R = \frac{D}{KA}$$

Where K is the cell-specific conductance and A is the electrode surface area. According to Ohm

Table 4. 12. Effect of inter-electrode distance on TFMR

Rxn time(min)	**Inter-electrode distance(c.m)**	
	2c.m	**4c.m**
30	60.2	51.7
60	76.8	66.3
90	79.4	70.8
120	80.6	75.7

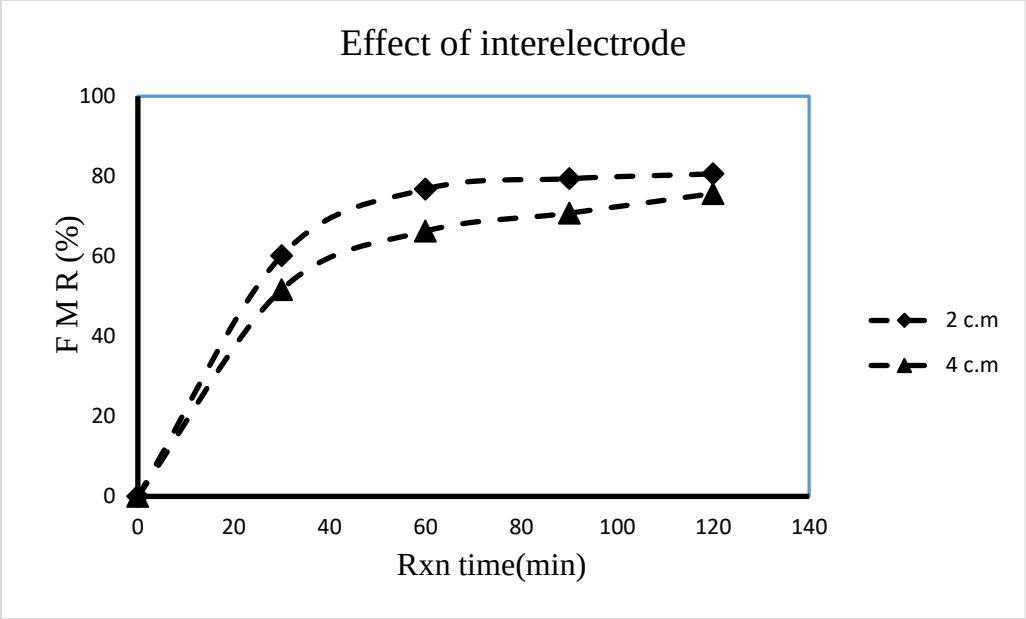


Figure 4. 16 . Effect of inter-electrode distance on TFMR

When we compare the percentage removal of 2cm and 4cm at a variable reaction time of 30min, 60min, 90min, and 120min at a constant voltage of 30V. The maximum percentage removal of the fatty matter is obtained at a distance of 2cm and 120min. This can be observed in figure 4.16, and table 4.12. The main reason for this is that when the resistance between electrodes at constant voltage is decreased the current increases thus increasing the concentration of coagulants and bubbles. When coagulation increases the removal efficiency of pollutant also increase (Janpoor, et al., 2011).

4.6.3. Effect of Reaction time and settling time on TFMR

Reaction time and settling time differences also affect the removal of fatty matter during the electro-coagulation process. In this study, its effect is studied in the laboratory and the result is shown in figure 4.17 and table 4.13 below.

Table 4. 13. Effect of time on TFMR

Rxn time(min)	**Settling time(min)**				
	30	60	90	120	180
30	60.2	65.3	69.2	73.9	76.3
60	76.8	80.1	83.6	84.4	87.5
90	79.4	87.1	88.3	95.9	95.2
120	80.6	90.8	93.4	95	95.6

In this study the effect of time on the percentage removal of the fatty matter was studied with settling times of 30min, 60min, 90 min, 120min, and 180min separately and the result is shown in graph 4.16 below. When we see the effect of time on fatty matter removal at 30min settling time, it is observed that the percentage of removal increases as the reaction time increase. In the same way, the percentage removal also increased 60min and 90min times as the reaction time increased but in 120min and 180 min settling times the percentage removal was slightly changed after 90 min reaction time. The maximum percentage removal of the fatty matter is observed at the 90th minute reaction time of the 120 min settling time. An increase in electrolysis time up to the optimum level increases the pollutant removal efficiency but it does not increase beyond the optimum level. The actual fact is that at constant current density coagulant formation increases with an increase in electrolysis time which leads to increased removal efficiency. Whereas the above optimum electrolysis time and increase in coagulant dose do not increase the pollutant removal due to the presence of a sufficient number of flocs(Mallesh, 2018).

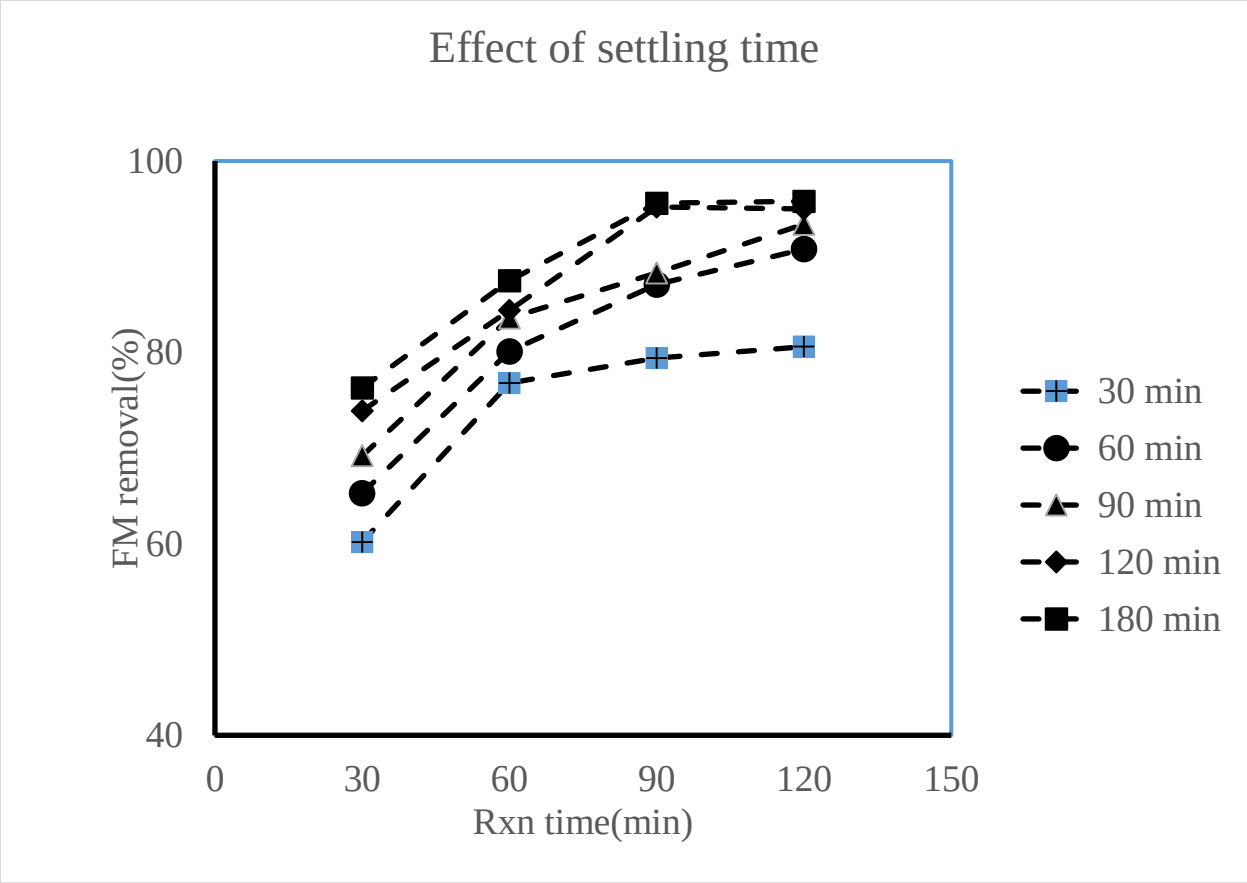


Figure 4. 17. Effect of reaction time and settling time on TFMR

The specific removals in the fatty matter are very dependent on the time necessary to perform the electrocoagulation process(Zetriuslita & Athar, 2014). The reason for that during the electrolysis process, the anode reaction occurs on the positive electrode and the cathode reaction occurs on the negative electrode. The ion released during the reaction neutralizes the electrical charge of the pollutant particles forming coagulation. Removal efficiency is directly dependent on the concentration of ions that are produced on the electrodes. With increasing the time of electrolysis, the concentration of ions that are produced increases, thereby the hydroxide clots increase(Mohammadi, et al., 2017).

4.7. Statistical Analysis of the Experimental Results for TFMR using bio-based coagulant and electrocoagulation

The process consists of four main factors: reaction time, voltage, settling time, and distance between electrodes with one response. After following the above series of procedures, the

experimental outcomes of those particular results were measured for fatty matter removal. The result obtained from the Experiment was indicated in table 4.14

Table 4. 14. Results obtained from the electrocoagulation experiment

Std		**Factor 1**	**Factor 2**	**Factor 3**	**Factor 4**	**Response 1**
	Run	**A: Voltage**	**B: reaction time**	**C: distance**	**D: settling time**	**TFMR**
		V	min	cm	Min	%
24	1	20	120	3	180	87.5
25	2	20	75	3	105	76.6
3	3	10	120	3	105	68.5
14	4	20	120	2	105	88.4
23	5	20	30	3	180	66.2
17	6	10	75	2	105	56.5
22	7	20	120	3	30	72.4
26	8	20	75	3	105	74.2
5	9	20	75	2	30	69.3
21	10	20	30	3	30	50.1
28	11	20	75	3	105	75.4
20	12	30	75	4	105	77.0
12	13	30	75	3	180	85.5
1	14	10	30	3	105	40.6
29	15	20	75	3	105	74.3
18	16	30	75	2	105	85.4
7	17	20	75	2	180	81.4
4	18	30	120	3	105	91.8
15	19	20	30	4	105	57.2
11	20	10	75	3	180	56.7
9	21	10	75	3	30	43.2
16	22	20	120	4	105	83.5
27	23	20	75	3	105	73.45
19	24	10	75	4	105	48.3

8	25	20	75	4	180	73.2
6	26	20	75	4	30	59.7
13	27	20	30	2	105	65.7
2	28	30	30	3	105	67.3
10	29	30	75	3	30	72.1

The removal was performed by using glycerol lactate as coagulant and aluminum electrodes as electro-coagulant with different factors: reaction time, voltage, settling time, and distance between electrodes with a constant coagulant dose of 3g/l, initial concentration of soap 0.9g/l, mixing time 30 min, stirring Speed of 100 rpm and 298k temperature.

The resulting data obtained in Table 4.14, were analyzed using Design expert 11.1.2.0 software to decide the effects of operating parameters; reaction time, settling time, voltage, and distance between the electrodes. The dependent variable used as a response parameter was the percentage removal of fatty matter. All experiments were carried out in a randomized order to minimize the effect of unexpected variability in the observed response due to extraneous factors. Design Summary for percentage removal with three levels and four factors. The design model of the experiments is a quadratic polynomial and the center point per block is five using Design expert11 software.

4.7.1. Effects of Experimental Variables for TFMR

Table 4. 15. Analysis of variance (ANOVA) for TFMR

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	5180.66	14	370.05	131.71	< 0.0001	significant
A	2271.5	1	2271.5	808.47	< 0.0001	
B	1756.92	1	1756.92	625.32	< 0.0001	
C	189.61	1	189.61	67.49	< 0.0001	
D	585.2	1	585.2	208.29	< 0.0001	
AB	3.06	1	3.06	1.09	0.03142	
AC	2.46	1	2.46	1.25	0.2475	
AD	12.73	1	12.73	7.14	0.0121	

BC	3.24	1	3.24	1.15	0.0301	
BD	0.2025	1	0.2025	0.0721	0.007923	
CD	0.4225	1	0.4225	0.1504	0.00704	
A^2	312.04	1	312.04	111.06	< 0.0001	
B^2	8.37	1	8.37	2.98	0.1064	
C^2	0.8445	1	0.8445	0.3006	0.5921	
D^2	97.94	1	97.94	34.86	< 0.0001	
Residual	39.33	14	2.81			
Lack of Fit	33.01	10	3.3	2.09	0.2493	not significant
Pure Error	6.33	4	1.58			
Cor Total	5219.99	28				

A = voltage, B = reaction time, C = distance between electrode and D = settling time

From the ANOVA (Analysis of Variance) Table, 4.15 results the higher F-Value and the lower P-value (<0.0001) indicate significance for the regression model. As the result demonstrates the model, the F-value of 131.71 implies it is significant. There is only a 0.01% chance that an F-value this large could happen due to noise. A p-value less than 0.05 implies that the model is statistically significant. They are extremely indispensable to understanding the interaction among the independent variables. The result showed that voltage (Factor A), reaction time (Factor B), inter-electrode distance (Factor C), and settling time (Factor D) are the major important variables in the electrocoagulation coagulation process with P-value < 0.0001. Based on the value both parameters are the best important factor. From the second polynomial model, A^2 and D^2 are significant models with P-values for both models < 0.0001. In the case of interaction effects AB (voltage and reaction time), BC (reaction time and distance between electrodes), BD (reaction time and settling time), and CD (distance between the electrodes) are significant model terms for fatty matter removal where their p-values are 0.03142, 0.0301, 0.007923 and 0.00704. The Lack of Fit of the F-value of 2.09 implies the Lack of Fit is not significant relative to the pure error. There is about a 24.93% chance that a Lack of Fit F-value large could occur due to noise.

As shown from the ANOVA, the interaction effects of voltage and distance between the electrodes (AC) and voltage and settling time (AD) did not influence the electrocoagulation process due to

their highest P-value. Their P- values were 1 which is greater than 0.1, which indicates the model terms are not significant. In the same way.

The model equations from the ANOVA result can be used to relatively compare the removal efficiency of fatty matter within factors and levels. The equation in terms of coded factors can be used to make predictions about the fatty matter removal efficiency for the given levels of each factor. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. The highest coefficients from the equation indicate the most influential factor that affects the coagulation process of fatty matter removal. On the other side, the equation in terms of actual factors can be used to make predictions about the Removal efficiency of fatty matter removal for given levels of each factor. The levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

The Final Coded Equations:

Fatty Matter Removal Efficiency (FMR) = +74.78 + 13.76*A + 12.1*B – 3.98*C + 6.98*D – 0.875*AB +0.5*BC – 0.225*BD + 0.325*CD – 6.94*A^2 – 1.14*B^2 -0.3608*C^2 -3.89*D^2

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

Final Equation of Actual Factors

Fatty Matter Removal Efficiency (FMR) = -10.9 + 4.296*voltage + 0.339*reaction time – 3.765*distance between electrodes + 0.23*settling time – 0.002*voltage*reaction time – 1.358*voltage*distance between electrodes - 5.47*voltage*settling time + 0.02*reaction time*distance between electrodes – 0.000067*reaction time*settling time + 0.0043*distance*settling time – 0.069*$voltage^2$ – 0.00056*reaction $time^2$ – 0.36*distance between $electrodes^2$ – 0.00069*$settling\ time^2$

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the

coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

4.7.2. Model Adequacy

The regression model was found to be significant with the correlation coefficients of determination of R-Squared, adjusted R-Squared, and predicted R-Squared having a value of 0.9925, 0.9849, and 0.9617 respectively as shown in table 4.16. The goodness fitness of the model was measured by a coefficient of determination R^2, which measured how well the real experimental data points have been approximated together with the regression model. As the result in a table, 4.16 expressed the value of $R^2 = 0.9925$ indicates the better fit of experimental data to that of the predicted one. The value of the larger Regression coefficient (R^2) approach to 1 illustrates, the validity of the model in which it implies an acceptable agreement among the actual and predicted data. It implies that 99.25% of the experimental variable studied attributed to the removal of fatty matter. The Predicted R^2 of 0.9617 is in reasonable agreement with the Adjusted R^2 of 0.9849; i.e. the difference is less than 0.2. Therefore, this demonstrates that the measured data is very well fitted with a quadratic model of the predicted value. Adeq Precision measures the signal-to-noise ratio. A ratio greater than 4 is desirable. Your ratio of 42.9 indicates an adequate signal. This model can be used to navigate the design space.

Table 4. 16. Model adequacy measures of electrocoagulation for TFMR

Std. Dev.	1.68	**R^2**	0.9925
Mean	69.68	**Adjusted R^2**	0.9849
C.V. %	2.41	**Predicted R^2**	0.9617
press	86.8	**Adeq Precision**	42.9003

Generally, the smaller the value of the Predicted residual sum of squares (PRESS) static obtained 86.8 indicates, the better the experimental data points fit the model. Moreover, the high R^2 value and p-value as well as not significant fit indicate the best prediction of the experiment solution. Therefore, the result in both tables 4.15 and 4.16 proved that, the best performance of the fitness of the model equations.

Figure 4.18 indicates the relationship between Actual and predicted values, which can be introduced from the regression model equations of a Design Expert chart based on predicted and actual percent fatty matter removal. The straight line shows the predicted values and the small rectangles represent the actual experimental values. The straight line is very close to the actual values and has a correlation coefficient R^2 equal to 0.9925, which confirms the accuracy of the model. The result confirmed that all the actual and predicted values are close to the line of a perfect fit. Both of them showed good agreement between them.

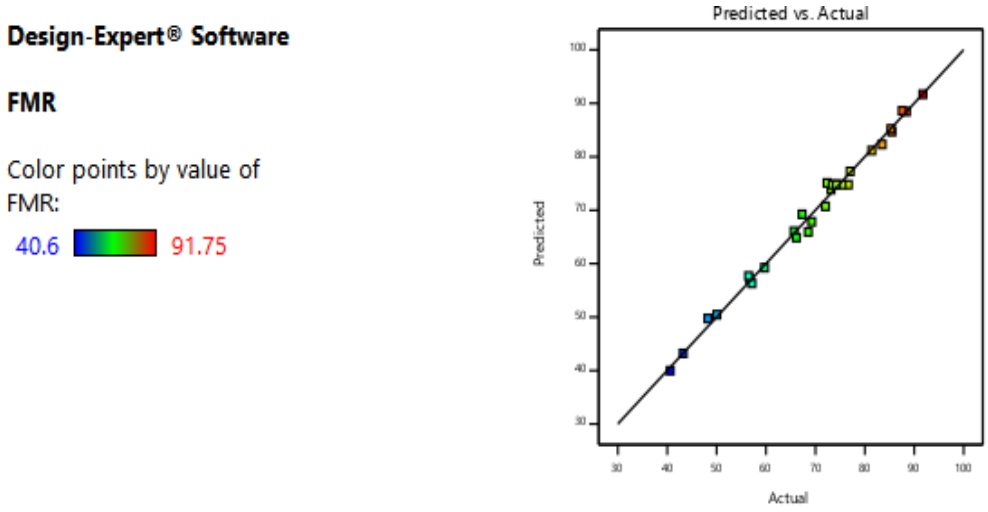


Figure 4. 18. Predicted vs Actual value fatty matter removal plot

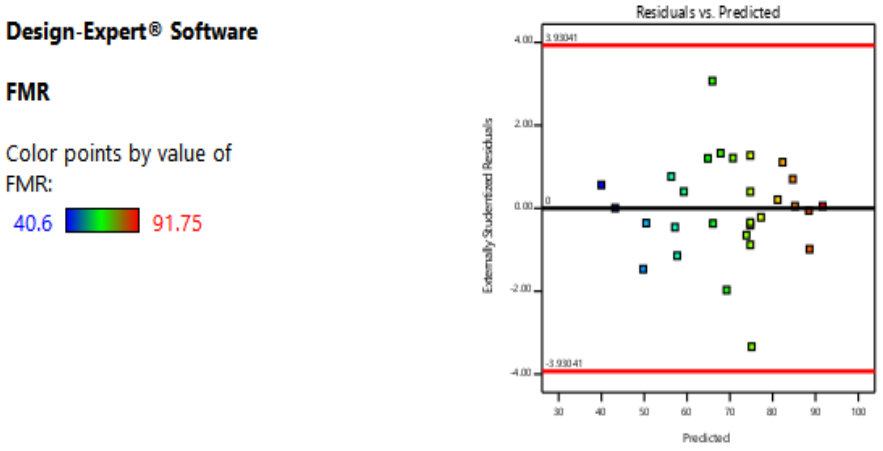


Figure 4. 19. Residual versus predicted values for fatty matter removal

If the model is correct and the assumptions are satisfied, the residuals should be structured less; in particular, they should be unrelated to any other variable including the predicted response. A simple check is to plot the residuals versus the fitted (predicted) values. A plot of the residuals versus the rising predicted response values tests the assumption of constant variance. The plot shows random scatter which justifies no need for an alteration to minimize personal error. The effects of the parameters for the percentage of fatty matter removal are to generate one-factor response and surface plots of the equation.

4.7.3. Optimization of the Response

To find the optimum value of each independent variable, four statistical metrics such as the larger regression coefficient (R^2), smaller lack of fit F-value, lower p-value, and higher sum squares have been used. The response optimization was used to identify the combination of independent variables particularly, reaction time, inter-electrode distance, voltage, and settling time within the response of fatty matter removal efficiency. The goal of the process was to obtain the optimum fatty matter removal percentage. Therefore it has been set as a maximum while the process parameters such as voltage, inter-electrode, and reaction time have been set as "within the range" while the settling time target to minimize the response surface method was used to optimize the process versus process parameters. According to the result obtained from optimizer Design-Expert version 11 software, the optimum removal efficiency of the fatty matter was achieved at 92.48% by using a 111.655min reaction time, and 24.076V voltage. 2.335c.m inter-electrode distance and 145.922min settling time.

Table 4. 17. Result from the optimum parameters

Parameters	**Raw wastewater**	**After treatment**			
		Using glycerol lactate	**Using Electrocoagulation**	**Using glycerol lactate and electrocoagulation**	**Using aluminum sulfate**
TDS removal (%)	1450 mg/l	57.91	71.27	72.43	61.52
Fatty matter removal (%)	74.26%	87.75	84.7	93.63	91.24
COD removal (%)	1500 mg/l	68.95	78.63	74.28	85.03
Turbidity removal (%)	165 NTU	96.52	97.49	97.503	97.89

CHAPTER FIVE

5. Conclusion and Recommendations

5.1. Conclusion

This research work focuses on the synthesis, purification, and characterization of bio-based coagulants and the preparation of electrocoagulation setup from aluminum electrodes for the application of soap wastewater treatment. The FTIR result analysis confirmed that the synthesis conjugate was purified and soap wastewater pollutant was successfully trapped by bio-based coagulant. The absorption peak at 1643 cm^{-1} indicates the carbonyl stretching of the remaining free lactic acid after the condensation reaction. In addition, the peak at 1570 cm^{-1} after the coagulation process was responsible for soap ion, which shows Na^{+} (COONa). Soap wastewater was used as a model pollutant. The sample of wastewater before and after treatment was characterized for its physicochemical properties. The total dissolved solids in the raw sample wastewater were found to be 1450 mg/l. The COD, turbidity, Ph, and total fatty matter values of the effluent were on average 1500 mg/l, 165 NTU, 9.07, and 74.26%, respectively. The Response Surface Method (RSM) was adopted to explore the model equation for fatty matter removal efficiency. The central composite design was selected to study the effect of variation in time, initial soap concentration, and coagulant dose of the coagulation process and study the effect of variation in voltage, reaction time, settling time, and inter-electrode distance of the electrocoagulation. The validation of the model using ANOVA analysis verified that the quadratic model selected is accurate, and statistically significant with a p-value less than 0.05. The optimum removal efficiency of 87.75 was obtained at t= 11.54hr, C_o = 1.104 g/L, dose = 4.221 g, and1:12 glycerol lactate ratio in coagulation process using glycerol lactate and 92.48 was obtained at reaction time = 111.655min, v = 24.076, d = 2.33c.m and settling time 145.922min in electrocoagulation process. The result shows the interaction effect of those parameters had a significant effect on the coagulation process. The batch experimental studies for real wastewater by selecting Adama Science and Technology University student laundry as a soap wastewater source. The result investigated showed that 81.8% of the fatty matter was removed from wastewater. The reduction in removal efficiency from that synthetic wastewater was due to a large number of competing pollutants within the waste.

5.2. Recommendation

For future work, the following recommendations are suggested based on the result obtained.

- ✓ It is better to study the removal efficiency of the bio-based coagulant on soap wastewater by varying the other operational variables like agitation speed, mixing time, and temperature in detail to improve the removal efficiency of the system.
- ✓ It is recommended to optimize the other responses like turbidity, BOD, TDS, COD, and TSS.
- ✓ Furthermore, glycerol lactate coagulant can be used for other types of wastewater.
- ✓ Future research should be done using this technology in combination with other cost-effective treatment methods to further treatment to improve the removal efficiency of the system.

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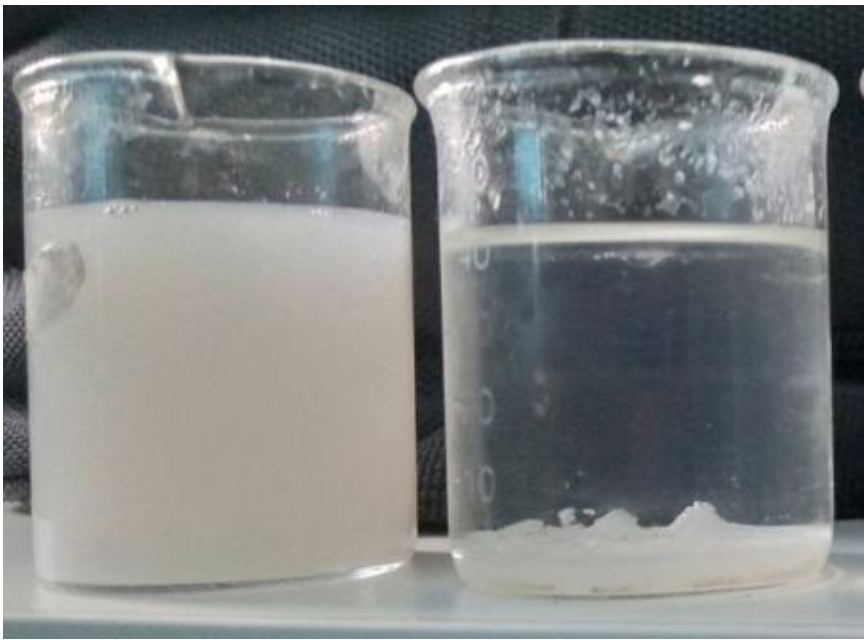
APPENDIXES

Appendix A: preliminary experiment

Fatty matter of real waste water

Experiment	FM(g/l)	Initial soap concentration(g/l)
Experiment 1	0.224	0.87
Experiment 2	0.162	0.63
Experiment 3	0.304	1.18
Experiment 4	0.196	0.76
Experiment 5	0.185	0.72



Before treatment and after treatment

Appendix B: final mass of fatty matter in the experiment

Time(hr.)	**Glycerol to lactic acid ratio**				
	1:3	1:6	1:9	1:12	1:16
1	0.654	0.504	0.420	0.458	0.582
4	0.451	0.486	0.401	0.202	0.511
8	0.513	0.414	0.251	0.125	0.452
12	0.623	0.288	0.108	0.059	0.410
16	0.637	281	0.106	0.77	0.355

Dose(g/l)	**Glycerol to lactic acid ratio**				
	1:3	1:6	1:9	1:12	1:16
1	0.641	0.489	0.405	0.337	0.442
3	0.632	0.284	0.102	0.063	0.413
5	0.582	0.253	0.084	0.049	0.383

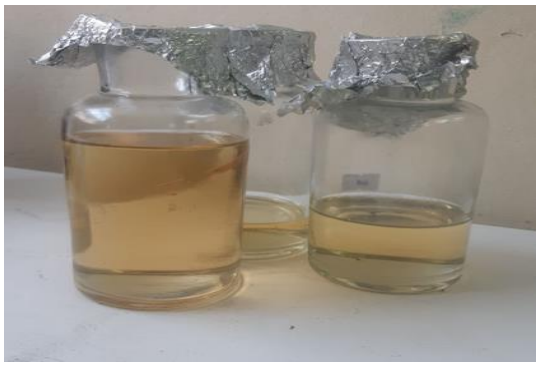
Initial soap concentration(g/l)	**Glycerol to lactic acid ratio**				
	1:3	1:6	1:9	1:12	1:16
0.6	0.430	0.222	0.124	0.097	0.290
0.9	0.633	0.283	0.101	0.065	0.415
1.2	0.852	0.537	0.159	0.112	0.378

Rxn time(min)	Voltage(V)		
	10V	20V	30V
30	0.444	0.305	0.266
60	0.369	0.231	0.155
90	0.325	0.180	0.138
120	0.284	0.168	0.130

Rxn time(min)	Inter-electrode distance(c.m)	
	2c.m	4c.m
30	0.266	0.323
60	0.155	0.225
90	0.138	0.195
120	0.129	0.162

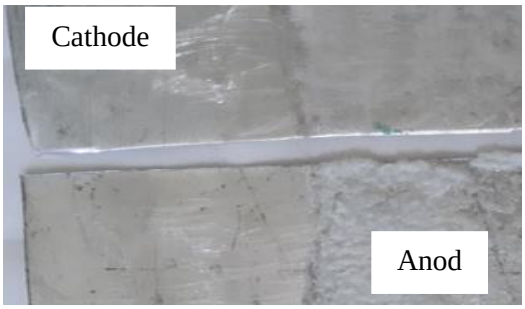
Rxn time(min)	Settling time(min)				
	30	60	90	120	180
30	0.267	0.232	0.206	0.174	0.158
60	0.154	0.133	0.110	0.104	0.084
90	0.153	0.086	0.078	0.027	0.32
120	0.132	0.061	0.044	0.033	0.294

Appendix C: Photographic Representation of laboratory work



Synthesized bio based coagulant

initial Ph



Al electrode

COD reactor

COD reagent

photometer

Turbidity

vacuum pampe

Appendix D: ANOVA analysis results