

**TREATMENT OF PALM OIL MILL EFFLUENT USING COAGULATION AND
ADSORPTION**

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BENIN CITY

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BY

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ENG2002040

**A PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMICAL ENGINEERING,
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**IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOT THE AWARD OF
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OCTOBER, 2025

CERTIFICATION

This is to certify that this project work was carried out and compiled by I, **IGBINWEKA DANIEL OMOZUHIOMWEN** with matriculation number **ENG2002040** of the Department of Chemical Engineering, Faculty of Engineering, University of Benin, Benin City, Edo State, Nigeria.

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DEDICATION

This project is dedicated to the Almighty God for His divine guidance, strength, and protection throughout this journey. It is also lovingly dedicated to my parents, Mr. Igbineveka (of blessed memory) whose memory remains a source of strength, and to my mother, Mrs. Igbineveka, for her endless love and support.

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ABSTRACT

Palm Oil Mill Effluent (POME) is a wastewater byproduct of palm oil production, characterized by its high organic content and potential pollutant to water bodies and capable of causing significant environmental damage. This study therefore seeks to evaluate the treatment methods by coagulation and adsorption processes to remove suspended solids and pollutants, thereby purifying the wastewater for safe discharge or reuse. These methods are essential for environmental protection, resource recovery, and economic sustainability.

The POME sample was collected, diluted, and analyzed to determine its physicochemical properties before treatment. Its pH was adjusted to both acidic and alkaline conditions using hydrochloric acid and sodium hydroxide, monitored with pH indicator paper. Processed periwinkle shell powder served as a natural coagulant and adsorbent. Standard laboratory instruments were used to assess parameters such as pH, turbidity, total dissolved solids, electrical conductivity, and salinity before and after treatment.

The study evaluated the effects of coagulant dosage, contact time, and pH on the treatment of Palm Oil Mill Effluent (POME) using a periwinkle shell–chitosan composite. Significant reductions in total dissolved solids (TDS) and salinity were achieved at moderate dosages (0.55–0.82 g/L), contact times of 105–150 minutes, and near-neutral pH (7–8.2), showing effective coagulation and adsorption. X-ray diffraction (XRD) analysis revealed crystalline peaks at 2θ values of 23.9°, 26.5°, 27.5°, 33.4°, 36.4°, 38.1°, 41.4°, 43.1°, 46.0°, 48.6°, 50.5°, and 53.1°, corresponding to aragonite, muscovite, quartz, and orthoclase phases. Crystallite sizes (111–702 Å) confirmed a fine heterogeneous structure with high surface activity, making the composite suitable for efficient and sustainable POME purification.

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NOMENCLATURE

POME – Palm Oil Mill Effluent

COD – Chemical Oxygen Demand

BOD – Biological Oxygen Demand

TSS – Total Suspended Solids

TDS – Total Dissolved Solids

EC – Electrical Conductivity

DO – Dissolved Oxygen

pH – Potential of Hydrogen

AOPs – Advanced Oxidation Processes

UF – Ultrafiltration

NF – Nanofiltration

RO – Reverse Osmosis

HRT – Hydraulic Retention Time

MLSS – Mixed Liquor Suspended Solids

ZLD – Zero Liquid Discharge

MBR – Membrane Bioreactor

PAC – Polyaluminium Chloride

VFA – Volatile Fatty Acids

PSO – Pseudo-Second-Order

PFO – Pseudo-First-Order

IPD – Intraparticle Diffusion

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND STUDY

In Nigeria, palm oil is an important agricultural product that plays a big role in the country's economy and is frequently used for frying and cooking. In Nigeria, it is a vital and significant commodity that greatly boosts the country's economy. Nigeria is currently among the top five producers of palm oil, which it exports to more than 150 nations worldwide. This significance has led to the conversion of a sizable portion of land into an oil palm plantation estate. In order to convert the growing quantity of oil palm fresh fruit bunch (FFB) into crude palm oil, a rising number of palm oil mills have been constructed concurrently (Liew et al., 2014). The industry's expansion coincides with an increase in wastewater, or palm oil mill effluent (POME), produced and discharged into waterways, endangering the ecosystem. A significant amount of wastewater is produced during the water-intensive process of obtaining crude palm oil from the fresh fruit bunch.

POME is a thick, viscous, brownish liquid waste that is non-toxic but smells bad. It contains soluble components that could have a big environmental impact. Water, oil, suspended solids, dissolved solids, and sand make up the majority of POME. (2012) Ali et al. In Nigeria, where the palm oil industry is a substantial economic activity, palm oil mill effluent (POME) is a serious environmental concern. In addition to macromolecules like polysaccharides, lipids, proteins, and several monocyclic and polymeric aromatic molecules, POME is a highly polluting wastewater with a typical biochemical oxygen demand of 25 g/l, a chemical oxygen demand of 50 g/l, oil and grease of 8 g/l, suspended solids of 20 g/l, and total solids of 40 g/l (Chantaraporn et al., 2010).

POME is thought to be responsible for around 30% of the overall biochemical oxygen demand (BOD) burden that the aquatic environment is subjected to (Liew et al., 2014). Because POME will negatively impact the soil and vegetation system, it cannot be released directly into the land. Additionally, it cannot be dumped straight into watercourses without treatment since doing so would deplete and distract aquatic life. Additionally, according to Parthasarathy et al. (2016), POME is 100 times more harmful than household sewage.

When untreated palm oil mill wastewater is dumped into nearby rivers and lakes, it becomes a major source of water pollution. In order for the organisms in the receiving system to accept the effluent, it must be adequately treated (Oyakhilome et al., 2014). In order to reduce environmental pollution and advance sustainability in the palm oil sector, POME treatment is essential. POME, a wastewater byproduct of the manufacturing of

palm oil, has significant concentrations of organic materials, oil, grease, and other contaminants. In addition to preventing water contamination and perhaps recovering valuable materials, proper treatment lessens the environmental impact of releasing this effluent into water bodies.

Aerobic, anaerobic, and facultative ponds zero discharge technology, land application, adsorption, biological treatments, coagulation, flocculation, ultrafiltration, and membrane technology were among the conventional treatment techniques reportedly employed to treat POME. Nonetheless, the fundamental processes in the majority of water and wastewater treatment facilities are coagulation and adsorption.

The process of coagulation neutralizes charges and creates a gelatinous substance that bridges or traps particles, creating a mass big enough to settle or become stuck in the filter. Charge neutralization and sweep flocculation are part of its mechanism. When negatively charged colloidal particles in POME are destabilized by the addition of a positive charge coagulant, which causes them to group together. When a lot of coagulant is applied, a bulky precipitate called floc is created that captures and eliminates suspended particulates. This process is known as sweep flocculation.

The Environmental Protection Agency (2002) defines coagulation as the result of adding specific chemicals to raw water that contains non-settleable or slowly settling particles. The chemicals hydrolyze and neutralize the electrical charges on the colloidal particles, causing them to form floc agglomerations that will be eliminated through filtration and clarifying. The suspended particles can be changed via the coagulation process to make them stick to one another. A positive metal ion is supplied to water during coagulation in order to lower the surface charge to the point where particles are not repelled from one another. To combine the non-settling particles into larger, heavier masses of solids known as floc, chemicals (coagulant) are introduced to the water.

Because adsorption may eliminate impurities including oil and grease, color, and other contaminants, it is a promising method for treating POME. For a variety of separations, adsorbents have been created. Although powder is rarely utilized, commercial materials are often employed as pellets, granules, or beads. The adsorbent can be used once and then discarded, or it can be used repeatedly in a regenerative manner, which is more popular.

The efficacy of a number of adsorbents in treating POME has been studied, including

chitosan, synthetic rubber, and activated carbon made from agricultural waste (Pandia et al., 2018). Mass transfer of organic and inorganic molecules from liquid bulk to active sites within the adsorbent causes adsorption. Physical adsorption and chemisorption are two forces that are involved in the mechanisms (Jahn et al., 2006). The steps of mechanisms are influenced by physical forces such as chemical bonding and van der Waals forces. In essence, the processes include adsorption on active sites, internal diffusion, and outward diffusion.

1.2 STATEMENT OF THE PROBLEM

When untreated palm oil mill wastewater is dumped into nearby rivers and lakes, it becomes a major source of water pollution. Untreated POME will eventually destroy marine life, particularly fish in the river, by going through a biodegradation process and consuming dissolved oxygen in the water. The watercourse will become acidic due to the untreated POME, which will have an impact on aquatic life. Additionally, the oil in untreated POME often forms a thick coating on the water's surface, preventing oxygen from being absorbed. The stream will become brownish and unfit for public consumption due to POME's dark brown hue and disagreeable odor (Bello et al., 2017).

The Department of Environment (DOE) states that treated POME can be released either onto land or into a water flow. The POME regulation contains seven pollutants for the discharge into the watercourse. BOD₅, suspended solids (SS), oil and grease (O&G), ammoniacal nitrogen (AN), total nitrogen (TN), pH, and temperature are all managed. BOD₅, which is fixed at 5000 mg/L, is the only criteria for the discharge onto the land.

1.3 PURPOSE AND GOALS

Coagulation and adsorption are used in POME treatment to remove contaminants and suspended solids, cleanse the wastewater, and make it suitable for reuse or disposal.

- The main goal of adsorption is to lessen POME's high organic content, color, and turbidity. This can be done by binding and removing these contaminants with adsorbents like activated carbon. Adsorption technology can help manage palm oil mill wastewater sustainably and provide a cleaner environment.
- The purpose of coagulation is to destabilize and aggregate these pollutants so that they can be more readily eliminated by

later processes, such as filter sedimentation. Coagulation essentially improves the overall effectiveness of the wastewater management process by acting as a pre-treatment stage in POME treatment.

1.4 STUDY SCOPE

In order to lower the pollutant load and enhance water quality, this study focuses on treating Palm Oil Mill Effluent (POME) using coagulation and adsorption techniques. POME is collected, diluted, and characterized in order to ascertain its physicochemical characteristics both before and after treatment. The effectiveness of natural coagulants and adsorbents—more especially, powdered periwinkle shells—in eliminating suspended particles, organic debris, and other contaminants from the effluent is also investigated. To assess treatment effectiveness, parameters such pH, turbidity, total dissolved solids (TDS), electrical conductivity (EC), salinity, and dissolved oxygen (DO) are examined. The scope is restricted to laboratory-scale studies intended to evaluate natural materials' potential in POME purification and their role in ecologically friendly waste management techniques.

1.5 THE STUDY'S IMPORTANCE

There are various reasons why this study is important.

1.5.1 Environmental Protection: By minimizing greenhouse gas emissions, preventing water contamination, and reducing environmental pollution, effective treatment of palm oil mill effluent (POME) promotes a safer and cleaner ecosystem (Rana et al., 2017).

1.5.2 Resource Recovery: Effective POME management permits the recovery of nutrients that can support agricultural use as well as valuable resources like biogas and biofuels (Saifullahi et al., 2024).

1.5.3 Economic Benefits: This approach helps produce palm oil more effectively and sustainably by lowering waste treatment costs, improving sustainability, and guaranteeing adherence to environmental rules.

CHAPTER TWO

LITERATURE REVIEW

2.1 PALM OIL PRODUCTION IN NIGERIA

The fruit of the oil palm tree (*Elaeis guineensis*) yields palm oil, an edible oil. "Red palm oil," sometimes known as crude palm oil, has a high beta-carotene content. Beta-carotene, vitamin E, and both saturated and unsaturated lipids are found in palm oil. Crude palm oil is bleached, deodorized, and neutralized to get refined palm oil. The fruit of the oil palm tree (*Elaeis guineensis*) is used to make this popular edible vegetable oil. Known for its durability at high temperatures and resistance to oxidation, this versatile component can be found in a wide range of items, including food, cosmetics, and even biofuels. The mesocarp (reddish pulp) of the fruit of the oil palm tree is the source of palm oil, a popular edible vegetable oil in Nigeria. It is a mainstay of Nigerian cookery, used in a variety of regional meals, soups, and sauces. In addition to cooking, it is a crucial component in the manufacturing of soap, candies, and biofuel. Nigeria is among the top producers of palm oil worldwide and one of the biggest in Africa. huge industrial mills as well as a huge number of smallholder and artisanal processors located throughout the country's central and southern areas produce palm oil.

Depending on the particular technique used, the yield or efficiency of oil extraction can vary greatly. The choice of extraction method, which affects the amount of oil that can be effectively extracted from the raw material, is one of the main causes of this variance. Chemical solvents are frequently a major factor in improving oil extraction. Because of their strong affinity for lipids, non-polar solvents including hexane, diethyl ether, and carbon tetrachloride are widely used among the different types of solvents. Because they dissolve fats and oils more easily than polar solvents, these solvents are very useful for separating oil from other components in the sample. Because increasing oil yield is a top concern in both commercial and laboratory settings, these non-polar chemicals are frequently chosen (Amaobi et al, 2017). Nigeria's palm oil industry, despite its economic importance, has difficulties managing the environment, especially when it comes to POME disposal. Carbon, oxygen, hydrogen, nitrogen, sulfur, phosphorus, potassium, calcium, magnesium, aluminum, iron, silicon, and chlorine are the main constituents of POME. The absence of dangerous heavy metals like lead, cadmium, mercury, manganese, or chromium is one of POME's benefits. It also includes roughly 42% lignin, 7% hemicellulose, and 11% cellulose (O-Thong et al., 2012). Untreated POME, which is composed of 95% water, 4% total solids, and 0.7% oil, is brown in color, full of colloidal suspension, and has an offensive and strong odor (Ahmad et al., 2010). POME must be treated before being safely released since improper treatment can pollute the environment, particularly water. POME is frequently dumped straight into streams or allowed to build up in adjacent bushlands without being properly treated in rural and semi-urban regions. Soil degradation, surface water contamination, and public health are all at danger. Nigerian palm oil producers, particularly small and medium-sized businesses, need affordable, efficient, and flexible wastewater treatment solutions due to their restricted

access to contemporary treatment facilities. In this situation, coagulation and adsorption work effectively, particularly when locally accessible materials like charcoal, plant leaves, and agricultural waste are utilized as treatment aids. Nigeria's abundance of agricultural waste, including sawdust, rice husks, cassava peels, coconut husks, snail shells, and palm kernel shells, presents a special chance to implement sustainable and circular economy-based solutions. These waste products can be used as natural coagulants or adsorbents, which would simultaneously lower costs and lessen their impact on the environment. Additionally, this strategy is in line with the current environmental engineering and palm oil sector drive for green technologies and local content development.

2.2 POME TREATMENT PROCEDURES

Although chemicals are not used in the process of extracting oil from fruit bunches, the resulting palm oil mill effluent (POME) still contains a significant quantity of organic waste and needs to be adequately treated before being released into the environment. POME is usually treated in a number of steps, from straightforward mechanical solid removal to intricate chemical and biological procedures meant to lower pollutant burdens. To guarantee that the final effluent satisfies discharge regulations, these treatment phases can be roughly divided into primary, secondary, and tertiary processes, each of which has distinct roles and objectives.

2.3 POME'S PRIMARY TREATMENT

Palm Oil Mill Effluent (POME) is mostly treated using physicochemical techniques like membrane technology, adsorption, and coagulation, as well as conventional biological techniques like activated sludge and ponding systems (both anaerobic and aerobic). To break down organic materials more effectively, advanced oxidation processes (AOPs) such as ozonation and photocatalysis are also being investigated.

2.3.1 Sludge that is activated

The organic debris in the wastewater is broken down by microorganisms in this system. By using microorganisms to break down organic contaminants in the wastewater, activated sludge treatment is a technique for purifying palm oil mill effluent (POME). A settling tank is used to separate the treated water from the activated sludge after air or oxygen is added in an aeration tank to promote microbial growth and organic matter consumption. This procedure is frequently used following anaerobic digestion to improve the effluent's overall quality and further remove contaminants. Activated sludge treatment has been shown to enhance POME treatment in recent years (Chan et al., 2010 and Gobi et al., 2011). According to a different source, COD removal efficiency utilizing an activated sludge reactor following anaerobic digestion was found to be 83% and 57% at 36 and 24 hours of hydraulic retention time (HRT), respectively. Additionally, a group of scientists demonstrated that by keeping the MLSS at 2,500–4,000 mg/L at HRT of three days, the Sequencing Batch Reactor (SBR) could reach 82% COD removal efficiency (Fun et al., 2007).

2.3.2 ACTIVATED SLUDGE MECHANISM

POME is added to an aeration tank, where it is combined with a microbial community known as activated sludge and allowed to aerate. The oxygen required for the microorganisms to flourish and break down the organic contaminants is supplied by the aeration. By using the organic materials in the POME as food, the microorganisms in the activated sludge efficiently eliminate contaminants. The effluent is sent to a settling tank following the aeration process. Here, the treated water is separated and released while the activated sludge (biomass) sinks to the bottom and forms a sludge blanket. The method used to prepare the sludge-adsorbents is often the same as that used to prepare the activated carbon (C. Liu et al., 2010). Sludge-adsorbent is frequently prepared using activating agents like CO₂, H₂SO₄, KOH, ZnCl₂, etc.

2.3.3 Advantages of Activated Sludge

High levels of pollution removal are possible with activated sludge systems, especially for organic materials.

Activated sludge systems can be more compact than certain other treatment techniques, which makes them appropriate for a range of treatment plant sizes. Systems for activated sludge can be modified to handle various POME kinds and produce the required effluent quality. (Daphtary Neel, 2020)

2.3.4 Activated Sludge Difficulties

Because aeration uses a lot of energy, it can be very expensive to operate. The management of the activated sludge itself, including dewatering and disposal, can be costly and environmentally problematic. Activated sludge systems are good at eliminating carbonaceous pollutants, but they might not always be as good at eliminating nutrients like phosphorus and nitrogen. Filamentous bacteria can occasionally take over the activated sludge, which can have a detrimental effect on the treatment process by causing poor settling and sludge thickening. Temperature can have an impact on the effectiveness of activated sludge treatment; lower temperatures may lessen microbial activity. (Daphtary Neel, 2020)

2.4 THE PONDING SYSTEM

Pollutants can be naturally assimilated by a combination of anaerobic and aerobic ponds. While aerobic ponds further reduce pollutants, anaerobic ponds manage the initial breakdown of organic materials. A popular, albeit somewhat antiquated, technique for handling palm oil mill effluent (POME) is ponding systems. POME conducts both anaerobic and aerobic biological treatment processes in these systems, which usually consist of a number of ponds. Ponding systems are criticized for their vast land footprint, extended hydraulic retention durations, and potential for methane emission, a greenhouse gas, despite being inexpensive and requiring little technical expertise. POME is directed into cooling, anaerobic, facultative, and algae ponds within treatment ponds. The cooling pond is required to cool the high-temperature fresh POME that is released during the

extraction process. An anaerobic pond was first used to treat POME, providing an economical way to break down a lot of sediments without aeration. Anaerobic effluents have been documented in the literature at low sludge concentrations of 5–10% (Khadaroo et al., 2020).

2.4.1 Ponding System Mechanism

After being released from the extraction of palm oil, fresh POME is first chilled in a special pond. Methane is produced as a byproduct of the anaerobic bacteria that break down organic materials in these ponds. Both aerobic and anaerobic decomposition are possible in these ponds, which further reduces contaminants. In these ponds, algae develop, eating nitrogen and phosphorus as well as aiding in the removal of carbonaceous pollutants. Complex polymers including proteins, lipids, and carbohydrates were broken down into their corresponding monomers during the process. For instance, the thermotolerant bacteria boosted the synthesis of sugar, amino acids, and fatty acids. Acetic acid (HAc), propionic acid (HPr), butyric acid (HBu), valeric acid (HVa) with trace-non VFA, lactic acid, and other volatile fatty acids (VFA) are produced by the fermentation of carbon-containing monomers during the acidogenesis process (Lam et al., 2011). The long chain VFA (HPr, HBu, and HVa) from acidogenesis was converted by acetogenesis into hydrogen and carbon dioxide (CO₂). Acetoclastic methanogens used HAc and CO₂ to create methane gas, whereas hydrogenotropic methanogens consumed hydrogen and CO₂. Methane gas is regarded as the biogas production's ultimate value-added product. Anaerobically treated POME turned alkaline and turned blackish brown when the long chain fatty acids (LCFA) and VFA were broken down. This was because the lignin partially broke down into phenolic (Bello et al., 2013).

2.4.2 Ponding System Advantages

When compared to alternative treatment techniques, ponding systems often have cheaper initial and ongoing expenditures. They are reasonably easy to use and require little technological knowledge. Ponding systems rely on natural processes for aeration rather than external energy input. (Hayawin and others, 2018).

2.4.3 Ponding System Difficulties

One disadvantage of ponding systems is their large land area requirements. For proper treatment, POME must remain in the ponds for long stretches of time (often 100–120 days).

Methane, a strong greenhouse gas, is released into the atmosphere by anaerobic ponds. Ponding system treated effluent may not always satisfy strict discharge requirements for color and Biochemical Oxygen Demand (BOD). (Hayawin and others, 2018).

2.5 MEMBRANE TECHNOLOGY

Membranes are essential for gas separation and water reclamation operations, such as CO₂ collection, hydrogen purification, and biogas upgrading (Merkel et al., 2010, Devaisy et al., 2023). They are acknowledged for their reduced energy consumption, low carbon footprint,

and effective separation (Rahimalimamaghani et al., 2023). Bacteria, suspended particles, and other contaminants can be removed via membranes. One very successful technique for handling palm oil mill effluent (POME) is membrane technology.

2.5.1 Membrane Technology Mechanism

These membranes (UF, NF, and RO) use pressure to separate contaminants according to size exclusion. MBRs efficiently break down organic materials and separate treated effluent by combining membrane filtration with biological treatment. PMRs incorporate photocatalysts within the membrane system, allowing for the photocatalytic elimination of organic pollutants as well as size-based separation. Treatment efficiency can be increased by combining various membrane technologies (such as UF, NF, and RO).

2.5.2 Advantages of Membrane Technology

By recovering clean water from POME, membrane technology lessens the need for fresh water sources.

Membrane systems efficiently eliminate dissolved salts, organic materials, and suspended solids from POME.

Membrane techniques can help recover important byproducts from POME, like methane and biogas.

Membrane technology reduces the environmental impact of palm oil production by recovering resources and treating POME. (Devaisy and others, 2023).

2.5.3 Membrane Technology's Challenge

POME is a complicated effluent that can cause membrane surface fouling due to its high organic content and suspended particles. Membrane permeability is reduced by fouling, which lowers efficiency and need regular cleaning or replacement. The sticky nature of the organic substance and the presence of colloidal particles are two possible causes of this fouling.

Membrane technology may require a substantial upfront equipment and membrane investment. Because of the energy required for cleaning and pumping, as well as the requirement for regular membrane replacement, operating expenses might also be high. To guarantee peak performance and avoid fouling, membrane systems need close observation and upkeep. This may entail hiring specialists and maybe requiring downtime for repairs or cleaning.

It can be difficult to scale up membrane technology to manage the massive amounts of POME generated by palm oil mills. It can also be challenging to adapt the system to different designs of palm oil mills and different effluent characteristics. Although membrane technology might lessen pollution, improper management of the disposal of fouled membranes and cleaning treatments can create additional environmental problems.

2.6 ADVANCED OXIDATION PROCESS

Advanced oxidation process (AOP) mechanisms in the industries mainly utilize hydroxyl radical to destruct the structure of organic compounds (Bashir et al., 2016). Sulfate radical-based AOPs have gained attention in recent years to replace hydroxyl radical due to: higher redox potential, reaction in wider pH range and higher efficiency in oxidizing persistent organic pollutants with unsaturated bonds or aromatic rings that are difficult to be treated during microbial degradation (Onn et al., 2020). The process of AOPs takes place in two steps: on-site generation of radicals, then proceeds with the reaction of radicals with organic or biological pollutants. Persulfate activation to form sulphate radicals can be achieved through several methods such as thermal, ultraviolet, ultrasound or heterogeneous and homogeneous catalysis (Guerra-Rodríguez et al., 2018). Although sulfate radical-based research has been increasing, there is limited information on the thermally activated persulfate and its performance in treating treatment POME. Typically, thermal activation is one of the most effective methods for persulfate activation to generate reactive species. Since the bond energy of peroxide (O–O) in persulfate is 140–213.3 kJ mol⁻¹, a thermal activation at >30 °C is enough to break the O–O bond for the generation of sulfate anion radicals (SO₄•⁻). These SO₄•⁻ radicals can be further converted to hydroxyl radicals (•OH), which are also an effective species in degrading organic contaminants.

2.6.1 Benefits of Advanced Oxidation Process

AOPs can degrade pollutants that are difficult to remove with conventional methods.

They can degrade a wide range of pollutants, not just specific types.

AOPs can potentially mineralize pollutants, breaking them down into simpler, less harmful substances.

Compared to some other methods, AOPs can produce less sludge.

Treated POME can be reused for various purposes, including fertilizer production. (Pérez-Rodríguez et al. 2019)

2.6.2 Challenges of Advanced Oxidation Process

AOPs can be more expensive than some conventional methods. Some AOPs, like Fenton processes, can generate sludge that requires further treatment. (Pérez-Rodríguez et al. 2019)

2.7 SECONDARY TREATMENT OF POME

Secondary treatment of Palm Oil Mill Effluent (POME) focuses on further purifying the effluent after it has undergone primary (biological) treatment. This stage aims to remove residual color, chemical oxygen demand (COD), and other pollutants to meet stringent discharge standards or enable effluent reuse. Common tertiary treatment methods include advanced oxidation processes (AOPs), adsorption using resins, and membrane filtration.

2.8 SECONDARY POME TREATMENT METHODS

2.8.1 Adsorption

Because adsorption may eliminate impurities including oil and grease, color, and other contaminants, it is a promising method for treating POME. For a variety of separations, adsorbents have been created. Although powder is rarely utilized, commercial materials are often employed as pellets, granules, or beads. The adsorbent can be used once and then discarded, or it can be used repeatedly in a regenerative manner, which is more popular. The efficacy of several adsorbents in treating POME has been studied, including chitosan, synthetic rubber, and activated carbon made from agricultural waste. (Pandia and others, 2018). Mass transfer of organic and inorganic molecules from liquid bulk to active sites within the adsorbent causes adsorption.

2.8.2 Adsorption Mechanism

incorporate two forces, like chemisorption and physical adsorption (Jahn et al., 2006). The steps of mechanisms are influenced by physical forces such as chemical bonding and van der Waals forces. In essence, the processes include adsorption on active sites, internal diffusion, and outward diffusion. First, mass transfer of molecules on the boundary layer surrounding the adsorbent particles occurs due to external diffusion, which occurs when the concentrations of the liquid bulk and the adsorbent surface differ. While some stick molecules on the adsorbent surface return to the liquid bulk, others get adsorbed into the pores.

When the concentrations of the latter are identical, this process occurs at equilibrium. Isotherms are used to give the equilibrium relations since they are crucial for data interpretation and prediction (Oyekanmi et al., 2019). Second, as the adsorbed molecules move through the pores, internal diffusion occurs. Internal diffusion models, such as Boyd's model and Weber Morris Intra Particle Diffusion (IPD), can be used to study the transport mechanism. Third, the process is quite quick once the molecules get to the active sites in the adsorbent particles, making the investigation more challenging. Both physical adsorption and chemisorption are involved in the molecular binding process. As chemical bonding agents toward the molecules, the active functional groups are crucial for chemisorption (Mohammed et al., 2014). As a result, the adsorption mechanisms rely on both adsorbent and adsorbate properties (Saleem et al., 2019). Common kinetic models (Pseudo-First-Order, Pseudo-Second-Order, Elovich models) and mass transfer models (Weber-Morris IPD, Boyd's model) can be used to calculate the rate of adsorption in the aforementioned steps. Rate constant steps are determined using the Pseudo-First-Order (PFO), Pseudo-Second-Order (PSO), and Elovich models, whereas rate regulating steps are determined using the IPD model (Kumar et al., 2012). Nevertheless, PFO and PSO lack sufficient particular physical meanings and are empirical models (Wang and Guo 2020). As a result, the IPD model can be used to examine the diffusion mechanism, however PFO and PSO models are not appropriate (Gao et al., 2013).

2.9 COD (chemical oxygen demand)

This is one of the criteria used to evaluate the water or wastewater's quality. The total quantity of all chemicals, including organic and inorganic materials, found in water bodies is measured by COD analysis. Because there are more substances that can be chemically oxidized than those that can be biologically broken down, COD often has greater values than BOD (Abu Shmeis 2018). Strong oxidizing agents like potassium dichromate ($K_2Cr_2O_7$) in contaminated water bodies (wastewater) and potassium permanganate ($KMnO_4$) in clean water bodies oxidize materials (Dhanjai et al., 2019). The environment and living things may be harmed by the elevated COD levels. The materials found in oil palms and the procedures used to make the finished products of palm oil are the sources of the COD level in POME. COD levels in raw POME are approximately 50,000 mg/L, while those in bio-treated POME are 1730 mg/L (Amosa 2016). The Malaysian Department of Environment (DOE) 1986 states that the COD discharge limit is 1000 mg/L (Bello et al., 2013). Since many oil palm enterprises have utilized biological treatment, such as ponding systems, it is required to move on to a more effective POME treatment.

2.10 ACTIVATED CARBON ADSORPTION

This approach to treating POME is really promising. Studies on POME adsorption utilizing activated carbon made from agricultural biomass wastes are displayed in Table 1. (Mohammed and Chong 2014) used banana peel as an adsorbent in an experiment to remove COD from POME. Because of the hydrogen bonds and electrostatic attractions between POME and banana peel AC, the lower pH resulted in increased uptakes in COD, TSS, and color removals. Additionally, Wafti et al. (2017) assessed the use of empty fruit bunches as AC precursors in POME treatment. As a result, the adsorption capabilities of AC and CAC were similar. 0.5, 1, 2, 3, 5, 6, 7, 8, and 9 w/v% were the dosages utilized. With dosages up to 2 w/v%, the adsorption capabilities grew dramatically; with higher dosages, they increased more gradually until the maximum COD reduction took place. The availability of sites and greater surface area in AC contributed to the higher elimination of COD (Garg et al., 2003).

2.11 BOD, or biological oxygen demand

is an indicator of organic matter in water bodies. The BOD level indicates how much oxygen microbes require to break down organic materials. In contrast to COD analysis, which takes less time and adequately identifies both biodegradable and non-biodegradable materials, BOD analysis can take up to five days to measure the biodegradable chemicals (Dhanjai et al., 2019). The process of measuring BOD involves comparing the dissolved oxygen (DO) concentrations before and after samples are incubated at a specific temperature of 20°C in a dark environment (Walker et al., 2019). The BOD content of raw POME is over 25,000 mg/L, which is significantly more than the 50 mg/L discharge limit established by Malaysian DOE 1986 (Bello et al., 2013). After being removed from mill processes, it includes the complex form of organic acids that causes the low pH (Lee et al., 2019). POME's high organic matter content used up a lot of DO, which is necessary for biodegradation. Because it depends on the availability and sufficiency of DO, aquatic life will be negatively impacted. Effective techniques like adsorption have been used to remove organic contaminants (Bello and Abdul 2017).

2.12 SOLIDS TOTALLY SUSPENDED (TSS)

This is the deposition of particles that cause water bodies to appear murky and have poor water quality. TSS analysis uses a glass fiber filter to determine the weight of suspended particles larger than 2µm (Walker et al., 2019). The color of wastewater, notably the black appearance of POME, is influenced by suspended solids. Aquatic ecosystems are negatively impacted by the 40,500 mg/L of TSS found in raw POME. By obstructing sunlight and decreasing photosynthesis, TSS has an impact on the ecosystem (Patel et al., 2020).

2.13 REMOVAL OF COLOUR

Water quality can be easily determined by looking at its color. The high levels of BOD, COD, and TSS that need to be lowered are indicated by the wastewater's dark color. The sterilization of fresh fruit bunches is what gives POME its color. The diverse organic components in POME are responsible for the brownish color and disagreeable odor. Lignocellulosic components are broken down during the sterilization of fresh fruit bunches, resulting in the formation of organic materials like anthocyanin, lignin, tannin, carotene pigment, polyphenol compounds, polyalcohol, and melanoidin, which give POME its oily, viscous texture and brownish color (Amat et al., 2015). The DOE of Malaysia states that there is no set restriction for POME color. However, the public's concerns about the poor water quality are heightened by the turbid and dark color of wastewater, which encourages researchers to continue their work on wastewater color removal.

2.14 COAGULATION

In order to facilitate their removal through sedimentation or other separation techniques, suspended particles in the POME are destabilized and clumped together using coagulants. Both chemical coagulants like alum and PAC and natural coagulants like *Moringa oleifera* seeds, peanut-okra, and wheat germ-okra are frequently utilized. To make it easier to remove contaminants, chemicals are added to destabilize and clump them together. The impact that results from adding specific chemicals to raw water that contains slowly settling or non-settleable particles is referred to as coagulation (Ireland Environmental Protection Agency, 2002).

2.15 COAGULATION MECHANISM

The colloidal particles' electrical charges are hydrolyzed and neutralized by the chemicals, causing them to aggregate into floc, which will be eliminated by filtration and clarifying. The suspended particles can be changed via the coagulation process to make them stick to one another. A positive metal ion is supplied to water during coagulation in order to lower the surface charge to the point where particles are not repelled from one another. To combine the non-settling particles into larger, heavier masses of solids known as floc, chemicals known as coagulants are added to the water (Ahmad et al., 2002).

2.16 COAGULANTS IN NATURE

Natural coagulants derived from plant parts like seeds, roots, and leaves can be used to treat the effluent from palm oil mills. These naturally occurring organic polymer components are significant because they include human-safe acrylamide monomers. Due to their abundance and other beneficial, environmentally friendly qualities, they have the potential to be commercialized in the future (Hegazy et al., 2011). The primary benefits of employing natural plant-based coagulants for water treatment are evident: they are highly biodegradable, inexpensive, and unlikely to result in treated water with an extreme pH (Jahn and Dirar 2006). A new POME treatment option could be the application of natural coagulant in POME wastewater by physical treatment from the coagulation-flocculation process. When treating POME, chemical coagulants or flocculants like alum (aluminum sulfate) or polyaluminum chloride (PAC) are not a suitable option. Alum and PAC include aluminum, which has some drawbacks. For example, a high aluminum concentration in water may be linked to health issues for people (Ndabigengesere and Narasiah 1998).

Inorganic coagulants create a lot of sludge and may be harmful to aquatic life. In addition to the high expense of these chemicals, sludge with a high residual metal concentration may be challenging to dispose of. Therefore, the best options for treating POME are natural, eco-friendly coagulants and biodegradable flocculants (Bhatia and Ahmad 2008). A great quantity and variety of natural compounds have been investigated for their coagulation capabilities due to the possibility of natural coagulants as a suitable option to create potable water. Thus far, research has demonstrated the efficacy of natural chemicals derived from *Prosopis juliflora*, *Cactus latiflora*, and *Moringa oleifera* as coagulants in wastewater treatment. Additionally, significant chemical savings will be attained by using molecules produced from natural sources. In addition, natural products are safe for the environment and easily biodegradable (Ghebremichael 2004).

Due to its nutritional and therapeutic benefits, *Moringa oleifera* is one of the most prized and widely grown plants in the world. The species is a member of the Moringaceae, a single-generic family. There are fourteen species in the genus *Moringa*, which includes both trees and shrubs. *Moringa oleifera* (*moringa oleifera*Lam) is the species' true botanical name (Nand et al., 2012).

Moringa oleifera seeds have a number of qualities that are helpful in the medical area, such as buffering ability and antibacterial qualities. By eliminating microorganisms, suspended particles, and excessive water turbidity, these elements are helpful in the cleanup of wastewater. Khairuldin and Chaudhuri (2009). According to reports, the coagulation activity of *moringa oleifera* seeds has been tested on an industrial scale for the treatment of industrial wastewater, including palm oil mill effluent (POME), and the benefits of the seeds for high effluent removal were emphasized. Seeds extract of *moringa oleifera* was found as an effective purifier for removing suspended materials such as solids, turbidity and other waste products (Vieira et al., 2010).

There have been reports of several POME treatment approaches. However, since moringa oleifera seed works well for treating water and is an inexpensive resource with a wide range of uses, it will be advantageous to employ this species wisely to enhance the treatment of industrial waste. It would be a sustainable alternative to the traditional coagulant system and aid in raising the economic standing of a nation with a large population of the species (Vieira et al., 2010). Zeolite and chitosan have been used as coagulants in water treatment in recent years (Folkard et al., 2000).

A cellulose-like polyelectrolyte biopolymer, chitosan (N-acetyl-d-glucosamine) is produced by deacetylating chitin. Because of its exceptional qualities, including biodegradability, biocompatibility, adsorption property, flocculating ability, polyelectrolyticity, and potential for regeneration in a variety of applications, chitosan has been suggested as a good coagulant resource material (Ravi 2000). It is a linear cationic polymer that is non-toxic, has a high molecular weight and charge density, and dissolves easily in acidic solutions (An et al., 2001). Chitosan was employed as a coagulation agent to eliminate suspended particles and leftover oil. According to Ahmad et al. (2006), the chitosan-produced flocs develop quickly and increase to a bigger size that is easily sedimented.

(Sethupathi 2004) compared the adsorption equilibrium, isotherm, thermodynamic, and kinetic effects of using chitosan powder and chitosan flake on residue oil removal efficiency from POME. A number of experiments were conducted with various parameters and situations. Weight dosage, beginning concentration, sedimentation duration, mixing time, mixing rate, pH, and temperature were the control variables. Using 0.5g/l of powdered chitosan, sedimentation duration of 30 minutes, mixing time of 30 minutes, mixing rate of 100 rpm, pH of 4.0 to 5.0, and temperature between 50 and 70°C, the best removal of leftover oil was achieved. The elimination percentages of 99% were rather excellent. To demonstrate that the leftover oil had been absorbed by chitosan, FT-IR and SEM micrographs of chitosan powder and flake before and after adsorption were shown. When compared to chitosan flake, chitosan powder shown a greater ability to remove residual oil. The study confirmed that chitosan, a biopolymer, is a viable substitute for coagulating and adsorbing POME residue oil.

(Ahmad et al., 2005) selected bentonite clay, chitosan, and activated carbon as adsorbents to determine the most effective residual oil remover from POME. For every parameter examined, chitosan demonstrated the best removal when compared to the other adsorbents. Using a dosage of 0.5 g, a mixing period of 30 minutes, a mixing rate of 100 rpm, sedimentation for 30 minutes, and a pH range of 4.0 to 5.0, chitosan was able to effectively remove 99% of residual oil and reduce the suspended solid content to a value of 25 mg/l from POME. To achieve the same percentage of removal as chitosan, the ideal dosages for activated carbon and bentonite clay were 8.0 g and 10.0 g/l, respectively, 30 minutes of mixing at 150 rpm, 80 and 60 minutes of settling time, and a pH of 4.0–5.0. In contrast to chitosan, (Saifuddin and Dinara 2011) investigated the possibility and efficacy of using chitosan-magnetite nanocomposite particles as a principal coagulant and flocculent for pre-treatment of palm oil mill effluent (POME). To find their ideal circumstances, a number of batch coagulation procedures using chitosan-magnetite nanocomposite particles and chitosan under various dose and pH conditions were carried out. Even at POME's initial pH of 4.5, chitosan-magnetite particles demonstrated superior parameter reductions with significantly lower dose consumption than chitosan. Turbidity, TSS, and COD levels were reduced by 98.8%, 97.6%, and 62.5%, respectively, at pH 6 with the ideal chitosan-magnetite dosage of 250 mg/L. Charge neutralization and the polymer bridging mechanism worked together to bring about the coagulation of POME by chitosan-magnetite at this pH. However, in order to achieve the highest turbidity, TSS, and COD reductions (97.7%, 91.7%, and 42.70%, respectively), chitosan appears to require a significantly larger dosage (370 mg/L). Interestingly, chitosan and the chitosan-magnetite nanocomposite both did a very good job of coagulating and removing the suspended solid and residual oil from POME. For palm oil mill effluent, coagulation with either chitosan-magnetite or chitosan might be considered an efficient and eco-friendly pre-treatment method with little to no harmful residual waste.

Zeolites are crystalline aluminosilicates with micropores. According to Chester and Derouane (2001), aluminum, silicon, and oxygen are arranged in a regular structure of $[\text{SiO}_4]$ - and $[\text{AlO}_4]$ -tetrahedral units that form a framework with tiny pores (also known as tunnels, channels, or cavities) of roughly 0.1–2 nm diameter running through the material. Zeolites are hydrated aluminosilicate minerals that are found in nature. They are part of the class of minerals called "tectosilicates." The majority of naturally occurring zeolites are created when fresh water from playa lakes or seawater alters glass-rich volcanic rocks called tuff (Badillo-Almaraz et al., 2003). Three-dimensional SiO_4 and AlO_4 tetrahedra frameworks make up zeolite formations. The isomorphous substitution of Si^{4+} by Al^{3+} results in a negative charge in the lattice, and the aluminum ion is tiny enough to take the location in the center of the tetrahedron of four oxygen atoms. The exchangeable cation (calcium, potassium, or sodium) balances the net negative charge. Certain cations in solutions, including lead, cadmium, zinc, and manganese, can be exchanged for these cations (Barer 2002).

Zeolite is a microporous mineral that contains cationic elements (K, Na, Ca, and Mg) and H₂O because it may release water and exchange ions. It has a negative charge neutralization mineral that can be neutralized by alkali metals. As an adsorbent of environmentally contaminating cations (Pb, Al, Fe, Mn, Zn, and Cu), it offers additional advantages. The environmental contamination was lessened by the zeolite (Oste et al., 2002). Because of their large specific surface areas and net negative charge, which can be electrically compensated for by inorganic and organic cations from the environment, clay minerals like bentonite and zeolite are among the possible substitutes (Konig et al., 2012). Shavandi et al. (2012) looked into the ability of natural zeolite to remove heavy metal ions from palm oil mill effluent, specifically zinc Zn(II), manganese Mn(II), and iron Fe(III). Heavy metal sorption was assessed in relation to contact time, agitation speed, pH, and sorbent dosage. Zeolite's capacity for desorption was also examined. The sorption was quick, reaching equilibrium in 180 minutes. The rate of adsorption thereafter gradually decreased. Natural zeolite was shown to have highest sorption capacities of 64.601, 53.644, and 52.446% for Fe, Zn, and Mn, respectively. At pH 7, the best adsorption took place. Based on the findings, 25.0 g of zeolite was determined to be the ideal dosage. It was found that the adsorption capacity was correlated with the mass of sorbent. While the pseudo-second-order model adequately represented the kinetic data, the equilibrium data were consistent with the Langmuir isotherm model. Maximum

For Fe, Zn, and Mn, HCl achieved desorption with 69.638, 58.575, and 61.516% of the initial adsorbed quantity, respectively. In the studies, over 50% of Zn (II), Mn (II), and over 60% of Fe (III) could be eliminated. It is proposed that natural zeolite is a reasonably inexpensive naturally occurring material for the removal of metal ions from POME because of its quick adsorption–desorption characteristics and reusability.

2.17 COAGULANTS IN CHEMICS

Chemical coagulation is the process by which colloidal particles combine to form larger particles that eventually settle as a precipitate or float on the surface. Turbidity reduction is a crucial unit process in water treatment that is typically caused by the addition of electrolytes. When an electrolyte is added to a colloidal solution, the oppositely charged ions are absorbed by the sol particles, neutralizing the solution. After that, the neutral particles begin to build up and eventually settle down as bigger particles. Filtration and sedimentation come after its use in water treatment (Karim and Lau 1987).

Most suspended particles can be effectively removed by inorganic coagulants, which are often the least expensive. They create a lot of floc, which can trap bacteria as they settle (Lee et al., 2012). The degree of instability of the colloid particles is influenced differently by each coagulant. They are efficient at eliminating a variety of contaminants from water, such as dissolved organic materials and colloidal particles. The destabilizing action of the counter-ion increases with its valence, and a lower dose is required for coagulation. The metal salt coagulants quickly produced hydrolytic products, which are easily absorbed by the colloid particles and destabilize their electrical charge. In addition, the improvement of the hydrolytic reaction depends on the wastewater's consistent pH throughout the coagulation process (Duan and Gregory 2003).

The most popular coagulant in wastewater treatment is aluminum sulphate (alum), an inorganic salt, because it works better at lower temperatures, has a wider pH range, and creates positively charged Al species that adsorb to negatively charged natural particles to neutralize charges. It has been demonstrated that polyelectrolytes work well as a pre-treatment coagulant to separate oil and grease by destabilizing oil droplets and destroying emulsions by adding alum and PAC (Zhao 2003). Using iron coagulants instead of aluminum has the advantage of producing denser and more durable flocs. Additionally, they are less susceptible to overdosing and can function across a wider pH range. Ferric chloride was added, producing fine, grey-brown flocs that have a tendency to form huge, amorphous aggregates. The translucent supernatant exhibited a very dark brown tint above the difficulty settling floc.

Six different doses of aluminum sulphate (alum): 90, 180, 270, 360, 450, and 540 mg combined with four different mixing ratios of (water: POME): 0.5:1.0, 0.75:1.0, 1.0:1.0, and 1.25:1.0 were used to evaluate the performance of the coagulation process by the percentage reduction efficiency of COD, BOD, and TSS of POME. According to (Reem 2011), the coagulant exhibited the greatest increase in TSS percentage elimination from 89.7% to 92.3% at a mixing ratio of 0.5:1.0. However, adding more coagulant than the recommended dosage (275.5 mg) at a mixing ratio of 1.25:1.0 caused the TSS elimination effectiveness to steadily decline from 90.6 to 82.9%. The decline most likely resulted from restabilization brought on by an excessive amount of coagulant. (Mohd and others, 2001). By tracking the decrease in the level of the cleared liquid interface over time, the settling rate of POME with 0–20 g/L alum was examined. He saw that the majority of the settling took place in the first five to seven hours. The rates were steady and settling was at its fastest over the first three to four hours. At 11 g/L of alum, the best settling was achieved, resulting in the lowest solids volume of 30% of the initial volume. The clarified liquor had the lowest suspended particles and COD, and the settling rate at 11 g/L alum was almost three times faster than raw POME. Alum clearly has the ability to significantly lower the suspended particles in POME, but not its COD or BOD. Using a modified jar test method, the efficacy of alum, polyaluminum chloride (PAC), FeCl₃, and FeSO₄ in addition to an anionic polymer was evaluated based on the removal of BOD, COD, and SS. The findings demonstrated that coagulation-flocculation processes on settleable solid-free POME were typically optimized at 150–200 ppm of FeSO₄ and FeCl₃ and 300–350 ppm of alum and PAC. It was discovered that the strength of the wastewater affected the ideal coagulant dosages. At a dosage of 200 ppm, the FeCl₃ coagulant was shown to have a 70% suspended solid removal effectiveness. Tan and associates (2006). examined four distinct coagulant combinations (Type A, B, C, and D) that included commercial polymer SR316 as a flocculent for use in the pre-treatment of POME. These combinations included aluminum sulfate, ferric chloride, ferric sulfate, and ammonium sulfate. When compared to Type C and Type D coagulants, which use ferric salts as the primary coagulant, the combination of Type A and Type B coagulants (aluminum sulphate as the primary coagulant) with SR316 does not demonstrate superior COD elimination. Using coagulant Type C1 with aluminum sulphate (1% w/w), ferric chloride (5% w/w), and ammonium sulphate (1% w/w), a 97% turbidity removal efficiency was attained. As an alternative, 62% of the COD was removed using coagulant Type C2, which contained 10% w/w ferric chloride, 1% w/w aluminum sulfate, and 1% w/w ammonium sulfate.

2.18 MEMBRANE SEPARATION

Membrane separation processes are widely regarded as among the most promising and effective technologies for the advanced treatment of industrial wastewater, including palm oil mill effluent (POME). Membrane separation operates on the principle of selective permeation, where certain

components of a fluid mixture are permitted to pass through a semi-permeable barrier while others are retained. This selectivity depends on the physical and chemical characteristics of the membrane material as well as the applied pressure and feed composition (Ahmad & Ooi, 2019).

In wastewater treatment, membrane processes can be classified into several categories which are microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO)—depending on pore size and operational pressure. MF membranes, with pore diameters ranging from 0.1–1.0 μm , are effective in removing suspended solids and emulsified oil. UF membranes (0.01–0.1 μm) remove macromolecules such as proteins, polysaccharides, and colloidal organics. NF membranes (0.001–0.01 μm) can reject multivalent ions and organic compounds, while RO membranes (<0.001 μm) remove dissolved salts and low-molecular-weight organics (Rahman et al., 2020).

2.18.1 Types of Membrane Configurations

Membrane modules are fabricated in several configurations including flat-sheet, spiral-wound, tubular, and hollow-fiber modules. Flat-sheet membranes are commonly used in laboratory studies due to their simplicity, while spiral-wound configurations are favored in industrial installations for compactness and high surface-area-to-volume ratio. Tubular membranes are used when treating viscous or high-solid wastewater such as POME because they tolerate higher fouling loads and are easier to clean mechanically (Zawawi & Lee, 2022). Hollow-fiber membranes, often used in ultrafiltration and microfiltration, are advantageous due to their large surface area and low energy consumption.

2.18.2 Mechanism of Separation

The mechanism by which membranes separate components is governed by molecular size exclusion and electrostatic interactions. During filtration, the applied trans-membrane pressure (TMP) acts as the driving force for solvent permeation. Solute molecules larger than the membrane pores are retained (size exclusion), whereas smaller molecules pass through. Electrostatic repulsion also influences rejection efficiency when charged membranes interact with ionic solutes. For POME, membranes are designed to retain oil globules, suspended solids, proteins, and color pigments while allowing clean water to pass through (Lim et al., 2022).

2.18.3 Application in POME Treatment

Membrane technology has been widely applied as a polishing stage following biological or physicochemical treatment of POME. A study by Chin et al. (2020) employed an anaerobic-UF hybrid system that achieved 98 % chemical oxygen demand (COD) reduction, producing permeate suitable for irrigation. Similarly, Tay et al. (2021) used a two-stage UF/NF process which reduced total suspended solids (TSS) to less than 50 mg/L and achieved 96 % color removal. Reverse osmosis membranes have been used to produce high-quality water for reuse within palm oil mills, with COD values as low as 100 mg/L (Abdullah et al., 2022).

Membrane bioreactors (MBRs), which integrate biological degradation and membrane filtration in a single unit, have also shown excellent results. Habib et al. (2021) reported that an MBR treating aerobically digested POME achieved 99 % BOD removal and 97 % color reduction at a hydraulic retention time of 10 h. The MBR process effectively retains biomass and organic particulates, allowing for extended microbial contact and improved degradation efficiency.

2.18.4 Membrane Materials and Surface Modification

Polymeric materials such as polyethersulfone (PES), polyvinylidene fluoride (PVDF), and polysulfone (PSf) are frequently used due to their good mechanical strength and chemical resistance. However, membrane fouling remains a significant challenge caused by the deposition of organic macromolecules, colloids, and microorganisms on the membrane surface. To mitigate fouling, membranes are modified using hydrophilic coatings or embedded nanoparticles such as TiO₂, ZnO, or graphene oxide. Modified PVDF membranes incorporating TiO₂ nanoparticles demonstrated 40 % higher flux and superior antifouling performance compared to unmodified membranes (Sofiah et al., 2023).

2.18.5 Advantages of Membrane Technology

Membrane treatment provides several advantages including compact design, high separation efficiency, and the potential for water recycling. The technology also eliminates chemical additives, resulting in minimal secondary pollution. Energy consumption is comparatively lower than in thermal separation methods, and membranes can be integrated with other systems such as adsorption and electrocoagulation to enhance overall performance (Devaisy et al., 2023).

2.19 ELECTROCOAGULATION OF POME

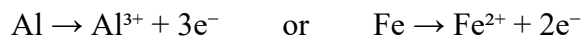
Electrocoagulation (EC) is a modern treatment method that combines coagulation, flotation, and electrochemical oxidation in a single process. It is particularly effective for high-strength wastewater such as POME, which contains emulsified oil, colloids, and high organic load (Yusoff et al., 2021).

2.19.1 Fundamental Principles

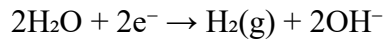
Electrocoagulation utilizes sacrificial metal electrodes, typically aluminium or iron, that dissolve under an applied electrical potential to release coagulant ions in situ. The generated ions hydrolyze to form hydroxides, which destabilize suspended and dissolved contaminants through charge neutralization and adsorption. The evolution of hydrogen gas at the cathode assists in the flotation of flocculated particles to the surface, forming a sludge layer that can be easily removed (Mansor et al., 2020).

The main electrochemical reactions occurring during EC are:

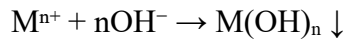
- **At the anode:**



- **At the cathode:**



- **In solution:**



The precipitated metal hydroxides act as coagulants and adsorb pollutants effectively.

2.19.2 Factors Influencing Electrocoagulation

Several operational parameters influence the EC efficiency: current density, pH, electrode material, inter-electrode distance, and treatment time. Optimal current densities (10–25 mA cm⁻²) enhance ion generation without excessive energy consumption. The solution pH affects metal hydroxide solubility; for aluminium electrodes, the optimum range is pH 6–8, whereas for iron electrodes, it is pH 5–7 (Saranya et al., 2021). Increasing treatment time generally improves pollutant removal until a plateau is reached.

Electrode material also plays an essential role. Aluminium electrodes tend to produce amorphous flocs with high adsorption capacity, while iron electrodes generate denser flocs that settle faster. Combined Al–Fe configurations often yield higher removal efficiencies due to synergistic effects (Yap et al., 2022).

2.19.3 Performance in POME Treatment

In treating POME, EC has achieved remarkable pollutant removal efficiencies. A study by Mansor et al. (2020) reported 93 % COD removal, 97 % TSS removal, and 90 % color reduction at 25 mA cm⁻² for 45 minutes using iron electrodes. Another investigation by Abdullah et al. (2023) found that aluminium electrodes achieved 98 % turbidity and 95 % color reduction under optimized pH and current density. EC also reduces oil and grease concentrations by over 90 %, making it a potential pre-treatment before membrane filtration.

2.19.4 Advantages of Electrocoagulation

- **Chemical-free operation:** No need for external coagulants, reducing chemical handling and sludge volume.
- **Compact and rapid process:** Requires smaller reactor footprints than chemical coagulation.
- **Simultaneous pollutant removal:** Removes color, suspended solids, and oil simultaneously.
- **Disinfection effect:** The generation of reactive oxygen species helps destroy microorganisms (Ong et al., 2020).

2.19.5 Hybrid EC Systems

To enhance performance, EC is often coupled with adsorption or membrane filtration. The EC step destabilizes particles and removes oils, reducing fouling in downstream membranes. Hybrid EC–adsorption systems using activated carbon or biochar achieved over 99 % turbidity and 95 % COD removal at lower energy input (Hamzah et al., 2022). These systems are attractive for sustainable industrial wastewater treatment.

2.20 HYBRID COAGULATION–ADSORPTION SYSTEMS

The integration of coagulation and adsorption has been widely explored to achieve complete pollutant removal from complex wastewater such as POME. The process exploits the complementary strengths of each technique: coagulation efficiently removes suspended solids and colloids, while adsorption targets dissolved organic compounds, heavy metals, and color-forming substances (Sharma & Bhatia, 2020).

2.20.1 Process Mechanism

Coagulation involves the addition of chemical or natural coagulants that neutralize the negative charges on colloidal particles, leading to floc formation. Adsorption then follows, whereby dissolved molecules attach to the surface of porous adsorbents through physical or chemical interactions. The combined approach produces synergistic effects: the coagulant pre-treatment reduces turbidity and enhances the accessibility of pollutants to adsorbent surfaces. In some cases, adsorbent particles are added during the coagulation stage to form composite flocs that settle faster and exhibit enhanced pollutant uptake (Hameed et al., 2021).

2.20.2 Adsorbent Materials Used

A variety of adsorbents have been investigated for hybrid treatment of POME. Activated carbon, derived from agricultural residues such as coconut shells and rice husks, is the most common due to its large surface area and pore volume. Biochar, produced through pyrolysis of biomass, offers a cost-effective alternative with similar adsorption capabilities. Zeolite, a microporous aluminosilicate mineral, is effective for removing heavy metals and ammonium ions. Chitosan, a natural polymer derived from crustacean shells, acts as both coagulant and adsorbent due to its cationic amine groups.

According to Lee et al. (2020), the use of chitosan-modified activated carbon increased COD removal from 85 % (using chitosan alone) to 98 %. Biochar impregnated with iron oxide achieved simultaneous color and COD reductions exceeding 90 % (Pasong et al., 2022).

2.20.3 Operational Parameters

The performance of hybrid coagulation-adsorption systems depends on several parameters:

- **pH:** Influences coagulant ionization and adsorbate–adsorbent interaction. Optimal performance is generally observed at pH 5–7.
- **Coagulant and adsorbent dosage:** Insufficient dosage results in incomplete pollutant removal, while excessive dosage causes re-stabilization of colloids.
- **Mixing and settling time:** Proper mixing ensures effective particle collisions, whereas sufficient settling allows floc separation.
- **Temperature:** Higher temperature can enhance diffusion but may also affect coagulant hydrolysis stability (Abubakar et al., 2022).

2.20.4 Modeling and Kinetics

Adsorption kinetics in hybrid systems are commonly interpreted using pseudo-first-order (PFO) and pseudo-second-order (PSO) models. Most studies show better fitting with PSO, indicating that chemisorption is the dominant mechanism. Equilibrium isotherms such as Langmuir and Freundlich are applied to determine maximum adsorption capacity and surface heterogeneity. For instance, a Langmuir maximum capacity of 125 mg COD g⁻¹ was reported for rice-husk biochar after coagulation pre-treatment (Hassan et al., 2021).

2.20.5 Performance of Hybrid Systems

Numerous studies have demonstrated the superior performance of hybrid systems. Abubakar et al. (2022) achieved 97 % COD and 99 % color removal using chitosan–biochar at 400 mg L⁻¹ coagulant and 2 g L⁻¹ adsorbent dosage. Zainudin et al. (2021) found that a chitosan–bentonite composite removed up to 99 % turbidity and 92 % COD at pH 6. Biochar derived from palm kernel shells also displayed excellent adsorption capacity (120 mg g⁻¹) and reusability for up to five cycles (Rahmad et al., 2020).

2.20.6 Advantages of Coagulation–Adsorption Systems

- **Enhanced removal efficiency:** Combination addresses both particulate and dissolved pollutants.

- **Reduced chemical consumption:** Natural coagulants minimize chemical sludge generation.
- **Operational simplicity:** Can be implemented using locally available materials.
- **Compatibility with other processes:** Serves effectively as a pre-treatment before membrane filtration or biological polishing (Hamzah et al., 2022).

2.21 BIOSORPTION AND BIOCOAGULANTS

Biosorption is an environmentally friendly process that uses biological materials to remove organic and inorganic pollutants from wastewater. Unlike conventional chemical treatments, biosorption utilizes renewable, low-cost biomass or its derivatives to adsorb contaminants. For palm oil mill effluent (POME), biosorption has emerged as an attractive solution due to the abundance of agro-waste materials in palm oil-producing regions such as Nigeria, Malaysia, and Indonesia (Akinbile et al., 2020).

2.21.1 Principles of Biosorption

Biosorption involves the passive binding of pollutants onto biological materials through mechanisms such as ion exchange, complexation, adsorption, and micro-precipitation. The process is independent of metabolic activity, allowing dead biomass to be used effectively. Functional groups present on biosorbent surfaces such as carboxyl, hydroxyl, amino, phosphate, and sulfate groups are responsible for metal and organic pollutant binding (Sulaiman et al., 2021).

The biosorption capacity depends on various parameters including pH, initial concentration of pollutants, biosorbent dosage, and temperature. At lower pH, competition between hydrogen ions and metal ions for active sites occurs, while higher pH enhances adsorption by deprotonating active functional groups.

2.21.2 Types of Biosorbents Used for POME

Various biosorbents have been utilized for treating POME and similar effluents. Common examples include:

- **Chitosan:** Derived from chitin deacetylation, chitosan is a natural polysaccharide with amino and hydroxyl functional groups capable of binding pollutants. Studies indicate that chitosan exhibits excellent flocculation, chelation, and film-forming abilities (Anuar et al., 2020).
- **Peanut shell and coconut husk:** These agricultural wastes are lignocellulosic and contain cellulose, hemicellulose, and lignin, which provide hydroxyl and carboxyl groups for adsorption.
- **Banana peel:** When carbonized, it exhibits significant COD and color removal capacities (Rahmad et al., 2020).
- **Microalgae biomass:** Dried algal biomass (such as *Chlorella vulgaris*) has been used to remove nutrients and color pigments from POME (Yahya et al., 2021).

The wide availability of these materials makes biosorption a cost-effective option, especially in developing countries.

2.21.3 Biocoagulants from Plant Extracts

In recent years, biocoagulants derived from plants such as *Moringa oleifera*, *Cactus opuntia*, *Tamarindus indica*, and *Plantago ovata* have been studied as sustainable alternatives to chemical coagulants. These natural materials contain cationic proteins or polysaccharides that neutralize negatively charged particles in POME. For example, *Moringa oleifera* seed extract contains positively charged water-soluble proteins (6–16 kDa) that act as natural polyelectrolytes (Jahn & Dirar, 2006).

In an experiment conducted by Ahmad et al. (2019), *Moringa oleifera* extract removed 90% turbidity and 87% COD from POME at a dosage of 1 g/L. Similarly, cactus mucilage demonstrated up to 85% turbidity and 80% color reduction due to the presence of galacturonic acid and polysaccharide chains (Anwar et al., 2021).

2.21.4 Advantages of Biosorption and Biocoagulation

- Utilization of renewable resources and agricultural wastes.
- Lower operational costs compared to chemical methods.
- High efficiency in removing color, COD, and heavy metals.
- Production of biodegradable sludge, which can be used as fertilizer.

2.22 ADVANCED MEMBRANE–ADSORPTION COMBINATIONS

Membrane–adsorption hybrid systems combine the molecular selectivity of membranes with the sorptive capacity of adsorbents. The concept aims to overcome the fouling problems of standalone membranes and the regeneration challenges of adsorbents. In such systems,

adsorption can occur either before membrane filtration (pre-adsorption), simultaneously (integrated adsorption–membrane), or after filtration (post-adsorption polishing) (Tay et al., 2022).

2.22.1 Mechanism of Coupling

Pre-adsorption reduces pollutant load and colloid concentration, minimizing fouling. Integrated systems use an adsorbent bed or suspension near the membrane surface to capture foulants before they deposit. Post-adsorption polishing removes residual organics that permeate through the membrane. The adsorption step may use activated carbon, zeolite, or biochar, depending on pollutant characteristics (Devaisy et al., 2023).

2.22.2 Application in POME Treatment

Integrated UF–adsorption processes have been applied to POME with remarkable success. Wong et al. (2020) demonstrated that coupling UF with granular activated carbon reduced COD by 98% and color by 95%, compared to 85% COD removal by UF alone. The hybrid system also extended membrane lifespan by reducing fouling by 60%. Similarly, an NF–adsorption hybrid achieved near-zero discharge when biochar was added as the adsorbent (Rahmad et al., 2021).

Another promising approach involves embedding adsorbent nanoparticles within membrane matrices (mixed-matrix membranes). For example, PVDF membranes embedded with zeolite and graphene oxide nanoparticles improved permeability and anti-fouling capacity by up to 40% while removing 97% COD and 99% color (Abdullah et al., 2022).

2.22.3 Advantages and Sustainability

- Combines physical separation and adsorption, achieving near-complete removal of pollutants.
- Reduces fouling frequency, extending membrane lifespan.
- Enables recovery and reuse of permeate water.
- Compatible with renewable adsorbents and circular economy concepts.

2.23 ZERO LIQUID DISCHARGE AND RESOURCE RECOVERY FROM POME

The concept of Zero Liquid Discharge (ZLD) represents the highest level of wastewater management, where no liquid effluent is released into the environment. Instead, all water is recovered, and residual solids are processed for reuse or safe disposal (Chowdhury et al., 2020). ZLD systems integrate multiple unit operations, including coagulation, adsorption, membrane filtration, and evaporation.

2.23.1 Importance of ZLD in Palm Oil Mills

Palm oil mills produce vast quantities of effluent approximately 2.5 tons of POME per ton of crude palm oil (CPO) produced. Without adequate treatment, this effluent poses serious threats to water bodies and ecosystems. Implementing ZLD not only prevents pollution but also recovers resources such as water, nutrients, and biogas (Ahmad et al., 2019).

2.23.2 Recovery of Valuable Products

- **Water recovery:** Using RO and NF membranes, more than 80% of water can be recovered for reuse in washing and steam generation (Zawawi & Lee, 2022).
- **Biogas generation:** Anaerobic digestion of POME produces methane-rich biogas that can substitute fossil fuels, reducing greenhouse gas emissions (O-Thong et al., 2012).
- **Nutrient recovery:** Solids and sludge from treatment contain nitrogen, phosphorus, and potassium and can be processed into organic fertilizers (Akinbile et al., 2020).

2.23.3 Integration with Coagulation–Adsorption Processes

Coagulation and adsorption play crucial roles as pre-treatment units in ZLD systems. They significantly reduce COD and suspended solids, improving downstream membrane performance. Bio-based coagulants and adsorbents reduce operational costs and environmental impact.

2.23.4 Environmental and Economic Benefits

ZLD contributes to cleaner production, compliance with discharge regulations, and sustainable palm oil certification. Although initial capital costs are high, the long-term economic benefits from water reuse and energy recovery are substantial (Rahmad et al., 2021).

2.24 OPTIMIZATION TECHNIQUES FOR COAGULATION AND ADSORPTION OF POME

Optimization is vital for identifying the most effective combination of process variables—such as coagulant dosage, pH, mixing rate, and contact time—to achieve maximum pollutant removal with minimal resource use. Traditional one-factor-at-a-time methods are inefficient, whereas

statistical tools like Response Surface Methodology (RSM) and Central Composite Design (CCD) provide comprehensive models of parameter interactions (Abubakar et al., 2022).

2.24.1 Response Surface Methodology (RSM)

RSM is a mathematical and statistical tool used for modeling and analyzing problems in which a response of interest is influenced by several variables. The relationship between the response (e.g., COD removal) and variables (e.g., dosage, pH) is represented by a second-order polynomial equation. The significance of each parameter is tested using analysis of variance (ANOVA).

For instance, in a study using chitosan as coagulant, the RSM model achieved 97.8% COD removal at an optimal dosage of 0.55 g/L, pH 7, and time of 105 minutes (Ahmad et al., 2019). The regression model showed high accuracy with $R^2 = 0.9823$ and Adeq Precision = 25.44, confirming model reliability.

2.24.2 Artificial Neural Networks (ANN)

ANN models are increasingly being applied to predict and optimize wastewater treatment parameters. Unlike RSM, ANN does not require a predefined equation form and can capture complex nonlinear relationships between inputs and outputs. For coagulation–adsorption of POME, ANN models have achieved prediction accuracies above 95%, outperforming traditional regression approaches (Ismail et al., 2021).

2.24.3 Genetic Algorithms (GA) and Machine Learning (ML) Tools

Genetic Algorithms can complement RSM and ANN by searching for global optima in large parameter spaces. ML algorithms such as random forests, support vector machines (SVM), and gradient boosting have been tested to predict pollutant removal efficiency using experimental datasets (Chin et al., 2020). These advanced computational tools offer rapid, reliable, and reproducible optimization frameworks.

2.24.4 Statistical Parameters and Validation

Key performance metrics include coefficient of determination (R^2), adjusted R^2 , predicted R^2 , F-value, and p-value. High R^2 (>0.95) indicates a strong fit between predicted and experimental data. Model adequacy is validated through residual analysis and lack-of-fit tests (Abubakar et al., 2022).

2.24.5 Importance of Optimization

Optimization ensures cost-effectiveness, enhances reproducibility, and minimizes environmental footprint. It also facilitates scale-up from laboratory to industrial level, ensuring consistent performance under variable operational conditions.

2.25 ENVIRONMENTAL AND ECONOMIC ASSESSMENT

Environmental and economic assessments are critical for evaluating the sustainability of treatment systems for POME. The assessment typically considers material use, energy input, greenhouse gas emissions, and cost-benefit analysis (Hassan et al., 2021).

2.25.1 Life Cycle Assessment (LCA)

LCA evaluates the environmental impact of each stage of the treatment process—from raw material extraction to waste disposal. Studies have shown that integrating natural coagulants and adsorbents reduces carbon emissions by 40% compared to synthetic chemicals (Rahmad et al., 2021). Membrane-based ZLD systems powered by biogas energy can achieve net-zero carbon emissions (Zawawi & Lee, 2022).

2.25.2 Cost-Benefit Analysis (CBA)

Economic feasibility is determined by comparing operational and capital costs with recovered resource value. Hybrid coagulation–adsorption systems exhibit lower cost per cubic meter treated (USD 0.50–0.80) compared to membrane-only systems (USD 1.2–1.8) (Ahmad et al., 2019).

2.25.3 Social and Regulatory Implications

Compliance with environmental regulations, such as Nigeria’s National Environmental Standards and Regulations Enforcement Agency (NESREA) discharge limits, is essential. Sustainable treatment technologies contribute to corporate social responsibility and enhance the reputation of palm oil producers (Akinbile et al., 2020).

2.26 RECENT TRENDS AND EMERGING TECHNOLOGIES IN POME

TREATMENT

Over the past decade, innovations in material science, process design, and biotechnology have driven a new generation of wastewater treatment technologies. The treatment of palm oil mill

effluent (POME) is gradually evolving from conventional chemical and biological systems to highly integrated, energy-efficient, and sustainable technologies that emphasize resource recovery and circular economy principles (Yahya et al., 2022).

2.26.1 Nanotechnology in Coagulation and Adsorption

Nanomaterials offer unique surface and catalytic properties that enhance pollutant removal. Nanoparticles such as titanium dioxide (TiO₂), zinc oxide (ZnO), iron oxide (Fe₃O₄), and graphene oxide (GO) have been incorporated into coagulants and adsorbents to improve adsorption efficiency and reusability (Abdullah et al., 2023).

Magnetic nanocomposites, for example, combine magnetic iron oxide cores with adsorptive shells of activated carbon or chitosan. These materials exhibit high surface area and can be recovered easily using external magnets, reducing sludge generation (Bhatia & Singh, 2021). TiO₂-modified chitosan nanocomposites achieved over 99 % color and 95 % COD removal in POME treatment at ambient temperature (Lim et al., 2023).

2.26.2 Photocatalytic Oxidation

Photocatalysis utilizes light-activated catalysts to degrade organic compounds into harmless end products such as CO₂ and H₂O. When TiO₂ nanoparticles are irradiated with UV or visible light, they produce hydroxyl radicals (OH) capable of oxidizing complex organics. Integration of photocatalysis with coagulation or adsorption enhances overall performance by mineralizing non-biodegradable fractions (Rahmad et al., 2022).

Hybrid systems such as coagulation-photocatalysis and adsorption-photocatalysis have achieved significant results. In one study, TiO₂-assisted solar photocatalysis removed up to 98 % of residual color after coagulation with alum, demonstrating the potential of light-driven systems for tropical regions (Anwar et al., 2021).

2.26.3 Electrochemical Advanced Oxidation Processes (EAOPs)

EAOPs, including electro-Fenton, anodic oxidation, and electro-peroxone processes, generate reactive radicals directly from water electrolysis. These radicals degrade refractory organics that persist after primary treatment. EAOPs are particularly effective for polishing POME effluent prior to discharge (Ong et al., 2020).

Combining electrocoagulation with electro-oxidation in a dual-chamber reactor has shown over 97 % color and COD removal, while simultaneously reducing total dissolved solids (Yusoff et al., 2021).

2.26.4 Enzymatic and Microbial Biosystems

Recent studies explore enzymes such as laccases, peroxidases, and lipases for selective degradation of color and oil residues in POME. These enzymes catalyze oxidation reactions under mild conditions and generate minimal secondary waste. Enzyme-immobilized beads and biocatalytic membranes provide reusable systems with stable activity over several cycles (Akinbile et al., 2020).

Microbial consortia of *Aspergillus niger*, *Pseudomonas aeruginosa*, and *Bacillus subtilis* have demonstrated synergistic COD removal exceeding 90 %, converting organic acids and lipids to methane precursors (O-Thong et al., 2012).

2.26.5 Membrane Bioreactors (MBRs) and Anaerobic Membrane Systems

The integration of biological degradation with membrane separation such as Anaerobic Membrane Bioreactors (AnMBRs) represents a major technological advancement. AnMBRs retain active biomass while producing a clear permeate, enabling high-rate digestion and biogas recovery (Habib et al., 2021).

Studies report methane yields up to $0.35 \text{ m}^3 \text{ CH}_4 \text{ kg}^{-1} \text{ COD removed}$ and effluent $\text{COD} < 200 \text{ mg L}^{-1}$ (Rahmad et al., 2021). The combination of AnMBR and nanofiltration allows total water recovery in ZLD systems.

2.26.6 Integration of Renewable Energy Sources

To enhance sustainability, many palm oil mills are shifting toward renewable-energy-powered treatment systems. Biogas generated from anaerobic digestion is used to power pumps and blowers, offsetting electricity costs. Solar photovoltaic (PV) panels coupled with electrocoagulation units have proven effective in decentralized rural applications (Chowdhury et al., 2020).

2.26.7 Digitalization and Smart Monitoring

Industry 4.0 concepts such as real-time sensors, Internet-of-Things (IoT) monitoring, and predictive analytics are increasingly being applied. Smart sensors measure pH, turbidity, COD,

and conductivity, feeding data into machine-learning models that predict performance and suggest control adjustments (Ismail et al., 2021).

Such automation minimizes manual intervention and ensures continuous compliance with environmental regulations.

2.27 RESEARCH GAPS AND FUTURE DIRECTIONS

Although substantial progress has been achieved in POME treatment technologies, several research and implementation gaps remain.

2.27.1 Limited Industrial-Scale Implementation

Most studies on coagulation, adsorption, and hybrid systems are conducted at laboratory or pilot scale. Scaling up to full industrial operation requires addressing challenges such as material supply, reactor design, and cost optimization. Only a small percentage of palm oil mills in sub-Saharan Africa currently utilize advanced treatment systems (Akinbile et al., 2020).

2.27.2 Inconsistent Performance of Natural Coagulants

Biocoagulants like *Moringa oleifera* and chitosan exhibit variability due to seasonal changes in composition, storage stability, and extraction techniques. Standardization of preparation protocols and quality assurance methods is needed to ensure consistent performance (Anuar et al., 2020).

2.27.3 Regeneration and Disposal of Adsorbents

While biochar and activated carbon are effective adsorbents, regeneration remains a challenge. Thermal regeneration consumes energy, whereas chemical methods may cause pore blockage or structural collapse. Research on low-temperature or microwave-assisted regeneration is ongoing (Hamzah et al., 2022).

2.27.4 Sludge Management

Coagulation and biosorption processes generate sludge containing organic matter and metals. Improper disposal can lead to secondary pollution. Converting sludge into bio-fertilizers or construction materials (e.g., bricks) has been proposed, but large-scale validation is required (Yahya et al., 2021).

2.27.5 Integration with Resource Recovery

Future POME management should integrate treatment with recovery of water, nutrients, and energy. Circular economy strategies that valorize by-products such as producing bio-hydrogen or single-cell protein could transform waste into revenue streams (Rahmad et al., 2021).

2.27.6 Development of Cost-Effective Nanocomposites

Although nanomaterials improve performance, their high production cost and potential ecotoxicity limit adoption. Research into green synthesis of nanoparticles using plant extracts or waste biomass could lower cost and environmental risk (Bhatia & Singh, 2021).

2.27.7 Policy and Regulatory Frameworks

Lack of stringent enforcement and incentives discourages mills from investing in advanced treatment. Governments must develop policies that promote wastewater valorization, tax incentives, and funding for small-scale technology adoption (Akinbile et al., 2020).

2.28 KINETICS AND ISOTHERM MODELS FOR COAGULATION AND ADSORPTION IN POME TREATMENT

2.28.1 Fundamental Principles

Understanding the kinetics and equilibrium behavior of coagulation and adsorption processes is essential for designing and optimizing the treatment of Palm Oil Mill Effluent (POME). These models provide insights into reaction mechanisms, rate-controlling steps, and the capacity of coagulants and adsorbents to remove pollutants.

Kinetic and isotherm studies help determine the optimum contact time, coagulant dosage, and adsorbent loading required to achieve maximum pollutant removal while minimizing energy and material costs (Ahmad et al., 2019). For high-strength effluents such as POME which contain complex mixtures of suspended solids, dissolved organics, oils, and color pigments—understanding these models ensures accurate process design and scalability.

2.28.2 Kinetics of Coagulation in POME Treatment

The coagulation process involves two main stages:

1. Rapid destabilization of colloidal particles by the addition of coagulants, and
2. Slow aggregation or flocculation of the destabilized particles into settleable flocs.

Kinetic modeling of coagulation is based on particle collision theory and rate of floc formation, which are influenced by mixing intensity, coagulant concentration, and particle size distribution (Hameed et al., 2021).

2.28.2.1 Coagulation Reaction Rate

The coagulation process is often described by a second-order kinetic model, which assumes that the rate of floc formation is proportional to the product of the concentrations of two colliding particles:

$$\frac{dC}{dt} = -KC^2 \dots\dots\dots\{2.1\}$$

Where:

- C = concentration of suspended particles (mg/L)
- k = coagulation rate constant (L/mg·min)
- t = time (min)

Integrating this expression gives:

$$\frac{1}{C_t} - \frac{1}{C_o} = kt \dots \dots \dots \{2.2\}$$

A plot of $1/C_t$ versus t yields a straight line, and the slope corresponds to the coagulation rate constant k (Rahmad et al., 2021).

2.28.2.2 Factors Influencing Coagulation Kinetics

- **pH:** Affects the hydrolysis and polymerization of coagulants such as alum or ferric chloride.
- **Dosage:** Higher dosages increase particle destabilization but can lead to restabilization if overdosed.
- **Mixing Speed and Time:** High-speed mixing promotes particle collision, while gentle flocculation allows aggregation.
- **Temperature:** Influences viscosity and collision frequency (Anuar et al., 2020).

2.28.2.3 Kinetic Observations in POME Studies

Abubakar et al. (2022) reported that coagulation of POME using chitosan followed pseudo-second-order kinetics, suggesting that the rate-limiting step was particle–polymer interaction rather than simple diffusion. The apparent rate constant increased with increasing coagulant dosage and mixing intensity.

Similarly, Mansor et al. (2020) found that electrocoagulation followed second-order kinetics, indicating that metal hydroxide species generated at the electrodes play a dominant role in pollutant destabilization and floc formation.

2.28.3 Kinetics of Adsorption in POME Treatment

Adsorption kinetics describe the rate at which pollutants are transferred from the liquid phase onto the surface of adsorbents. In POME treatment, adsorption is mainly applied after coagulation to remove dissolved organics, color, and residual COD. Kinetic studies are crucial for understanding adsorbate–adsorbent interactions, determining equilibrium time, and identifying rate-controlling mechanisms such as film diffusion, pore diffusion, or chemisorption (Lee et al., 2020).

2.28.3.1 Pseudo-First-Order (PFO) Model

The Lagergren pseudo-first-order (PFO) model assumes that the rate of adsorption is proportional to the difference between the equilibrium adsorption capacity and the amount adsorbed at time t (Ho & McKay, 1999):

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \dots\dots\dots\{2.3\}$$

Where:

- q_e = amount of solute adsorbed at equilibrium (mg/g)
- q_t = amount adsorbed at time t (mg/g)
- k_1 = pseudo-first-order rate constant (1/min)

If the plot of $\log(q_e - q_t)$ versus t gives a straight line, the process follows first-order kinetics.

However, several studies have shown that PFO poorly fits adsorption data for POME because adsorption often involves chemisorption rather than physisorption (Rahmad et al., 2021).

2.28.3.2 Pseudo-Second-Order (PSO) Model

The Ho and McKay pseudo-second-order (PSO) model is widely used for adsorption of organics from POME. It assumes that chemisorption involving valence forces through electron sharing or exchange dominates the process (Hameed et al., 2021):

$$\frac{t}{qt} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \dots\dots\dots \{2.4\}$$

Where k_2 is the rate constant of pseudo-second-order adsorption (g/mg·min). A plot of t/qt versus t yields a straight line if the process follows PSO kinetics.

In a study by Lee et al. (2020), adsorption of COD and color on chitosan–biochar composites followed the PSO model with correlation coefficients (R^2) above 0.995, confirming the chemisorption nature of the interaction.

2.28.3.3 Intraparticle Diffusion Model

The Weber–Morris intraparticle diffusion model is used to determine whether diffusion inside the adsorbent pores controls the adsorption rate:

$$C_{qt} = k_i t^{\frac{1}{2}} + C \dots\dots\dots \{2.5\}$$

Where k_i is the intraparticle diffusion rate constant (mg/g·min^{0.5}), and C represents the boundary layer thickness.

If the plot of qt versus $t^{\frac{1}{2}}$ is linear and passes through the origin, intraparticle diffusion is the sole rate-limiting step (Hamzah et al., 2022).

Most POME adsorption systems show multi-linear plots, indicating that adsorption occurs in two or more stages:

1. External film diffusion
2. Intraparticle diffusion
3. Equilibrium adsorption on active sites.

2.28.4 Adsorption Isotherm Models

Isotherm models describe the equilibrium relationship between the amount of solute adsorbed on the adsorbent and its concentration in solution at constant temperature. These models help evaluate surface properties, adsorption capacity, and mechanisms.

2.28.4.1 Langmuir Isotherm

The Langmuir model assumes monolayer adsorption on a homogeneous surface with identical sites and no interaction between adsorbed molecules (Langmuir, 1918):

$$q_e = \frac{q_{max} b C_e}{1 + b C_e} \dots\dots\dots \{2.6\}$$

Where:

- q_e = adsorption capacity (mg/g)
- q_{max} = maximum monolayer adsorption capacity (mg/g)
- b = Langmuir constant related to adsorption energy (L/mg)
- C_e = equilibrium concentration of solute (mg/L)

The separation factor $RL = \frac{1}{1+bC_0}$ indicates adsorption favorability ($0 < R_L < 1$).

Lee et al. (2020) found that adsorption of organics from POME on chitosan-based adsorbents fitted the Langmuir model, indicating monolayer coverage and chemisorption.

2.28.4.2 Freundlich Isotherm

The Freundlich model applies to heterogeneous surfaces with non-uniform adsorption energies (Freundlich, 1906):

$$qe = KfCe^{\frac{1}{n}} \dots \dots \dots \{2.7\}$$

Where:

- K_f = Freundlich constant (adsorption capacity)
- n = heterogeneity factor ($1 < n < 10$ indicates favorable adsorption)

Biochar and activated carbon adsorbents often follow the Freundlich model because their heterogeneous surfaces provide multiple adsorption sites (Rahmad et al., 2021).

2.28.4.3 Temkin and Dubinin–Radushkevich (D–R) Isotherms

The Temkin model considers adsorbate–adsorbent interactions, while the D–R isotherm helps distinguish physical from chemical adsorption based on adsorption energy. These models, though less common, provide additional validation for adsorption mechanisms (Lim et al., 2023).

Model	Equation Type	Application	Observation in POME Studies
Pseudo-First-Order	Linear logarithmic	COD/Color adsorption	Weak correlation ($R^2 < 0.9$)
Pseudo-Second-Order	Linear t/q vs t	COD/Color adsorption	Strong correlation ($R^2 > 0.98$), chemisorption
Intraparticle Diffusion	Multi-linear	COD removal	Multi-stage diffusion
Langmuir	Monolayer	Chitosan and activated carbon	Best fit for uniform sites
Freundlich	Multilayer	Biochar and natural adsorbents	Fits heterogeneous adsorption
Temkin & D-R	Energy-based	Nano-adsorbents	Useful for mechanism confirmation

Table 2.1: Summary of Kinetic and Isotherm Behaviour in POME Systems

Coagulation kinetics in POME treatment generally follow second-order reaction models, while adsorption processes align more closely with pseudo-second-order and Langmuir or Freundlich

isotherms, depending on the adsorbent surface properties.

These models collectively describe the rate and capacity of pollutant removal, guiding the design of efficient physicochemical treatment systems (Abubakar et al., 2022; Hameed et al., 2021).

CHAPTER THREE

MATERIALS AND METHODS

3.1 MATERIALS

The Palm Oil Mill Effluent (POME) used for this study was collected directly from the discharge point of a local palm oil mill. The sample was stored in clean plastic containers and allowed to be opened down and cool to room temperature before analysis. The sample was diluted and homogenized by stirring to ensure uniformity prior to treatment. Some parameters such as pH, salinity and turbidity were measured immediately after collection, while others such as salinity and turbidity were determined later under standard laboratory conditions

Palm Oil Mill Effluent (POME) sample was obtained from a local palm oil mill at Ikpoba Okha Local Government Area, Edo State. Perewinkle shells were gotten from Yanga market at Oredo Local Government Area, Edo State.

3.2 EQUIPMENT AND APPARATUS

1. **Beakers (250 mL and 500 mL):** Used for mixing, measuring, and holding the POME samples during treatment. Measuring cylinders (100 mL): Utilized for accurate measurement of liquids such as POME samples and coagulant solutions.
2. **Orbital shaker:** To provide uniform mixing and sufficient contact between the coagulant particles and the POME sample, ensuring efficient collision, floc formation, and pollutant removal.

3. **pH meter:** Used to determine the acidity or alkalinity of the POME sample before and after treatment
4. **EC meter:** Used to determine the Electrical Conductivity (EC) of the sample, which reflects the ionic content and overall water quality
5. **Salinity meter:** Measures the salt concentration in the POME, providing insight into its ionic strength and potential environmental impact.
6. **Turbidity meter:** Used to measure the cloudiness or haziness of the POME sample, which is directly related to the amount of suspended solids.
7. **Dissolved oxygen meter:** Used to measure the amount of dissolved oxygen in the pome sample, indicating the level of organic pollution and biological activity

Apparatus	Manufacturer	Model
Tds Meter	HM Digital	COM-100
EC Meter	Hanna Instruments	HI99301
Salinity Meter	Hanna Instruments	HI9829
Turbidity Meter	Lovibond	TB350 IR
pH Meter	Apera Instruments	PH700
Orbital Shaker	Fison	FM-OSH-B103
D.O Meter	AZ Instrument	8402 AZ

Table 3.1: Equipment and Apparatus

3.3 pH ADJUSTMENT OF DILUTED PALM OIL MILL EFFLUENT (POME)

A total of 200 mL of palm oil mill effluent (POME) was diluted with 400 mL of distilled water to obtain a diluted POME sample.

To adjust the acidity, 400 mL of the diluted POME was treated with 0.5 M hydrochloric acid (HCl) to achieve a pH of 5.8, while 100 mL of the same sample was adjusted to a pH of 5.0 using the same acid concentration.

For pH adjustment to the alkaline range, 400 mL of the diluted POME was titrated to a pH of 8.2 using 1 M sodium hydroxide (NaOH) solution. The base solution was prepared by dissolving 1 gram of NaOH in 40 mL of distilled water.

During all pH adjustments, pH indicator paper was used to monitor and confirm the pH levels of the solutions.

All materials and reagents used were of analytical grade and handled according to standard laboratory procedures.

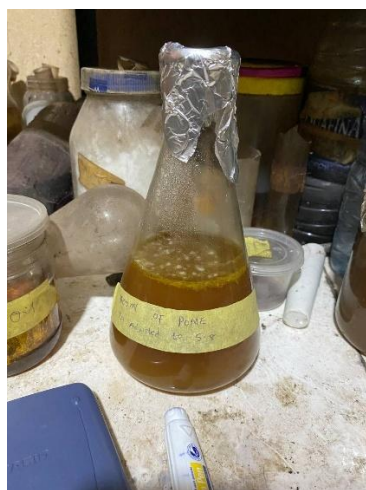


plate 3.1 400 ml POME (5.8)

plate 3.2 400 ml POME (8.2)

3.4 PREPARATION OF COAGULANT (PERWINKLE SHELL)

The shells were thoroughly washed with water to remove adhering dirt and organic matter. They were then oven-dried until all moisture was removed. The dried shells were crushed into fine powder using a mechanical grinder and sieved to obtain a uniform particle size. The powdered material was stored in an airtight container for use as a natural coagulant and adsorbent.

3.5 CHARACTERIZATION OF POME

The Palm Oil Mill Effluent (POME) sample was characterized to determine its physical and chemical properties before treatment. This provided the baseline data necessary to assess the efficiency of the coagulation and adsorption processes carried out using perewinkle shell as coagulant and adsorbent.

3.6 PHYSICAL PROPERTIES OF POME

1. Turbidity

Turbidity was determined using a turbidimeter. The value obtained was 111.36 NTU, showing high levels of suspended solids and colloidal particles.

2. Electrical conductivity (EC)

The electrical conductivity was recorded as 1957 μ S/cm suggesting a high ionic concentration in the effluent.

3. Salinity

The salinity level was 2.22ppt which could affect microbial activity in biological treatment systems.

4. Total dissolved solids (TDS)

The TDS value was 971ppm, representing the amount of dissolved ions and salts in the effluent

3.7 CHEMICAL PROPERTIES OF POME

The following chemical parameters were analyzed to understand the pollution strength of the POME

1. pH

The measured pH value was 7.25, indicating that the effluent was slightly alkaline, possibly due to microbial activity and the breakdown of organic matter during fermentation.

2. Dissolved Oxygen

The dissolved oxygen (DO) level of the sample was 0.0 mg/L, indicating a complete absence of oxygen in the effluent, which suggests severe oxygen depletion and poor aeration conditions.

3.8 OPTIMIZATION AND MODEL ANALYSIS

The optimization of the coagulation-adsorption process for palm oil mill effluent (POME) was conducted using Response Surface Methodology (RSM) with a Central Composite Design (CCD) approach. The independent variables considered were A-Coagulant

Dosage (g/L), B- Reaction Time (min), and C – PH, while the response variable was Total Dissolved Solids (TDS) Reduction (%).

A total of 20 experimental runs were carried out, and the data were fitted to a quadratic model to predict the interaction effects of the variables.

Design	Info
File Version	13.0.1.0
Study Type	Response Surface
Design Type	Central Composite
Design Model	Quadratic
Build Time (ms)	1.0000
Subtype	Randomized
Runs	20.00
Blocks	No Blocks

Table 3.2 Build Information

Name	Minimum	Maximum	Coded Low	Coded High	Mean
A: Coagulant dosage (g/L)	1.0000	10.00	-1 ↔ 2.82	+1 ↔ 8.18	5.50
B: Time (minute)	30.00	180.00	-1 ↔ 60.40	+1 ↔ 149.60	105.00
C: pH	5.00	9.00	-1 ↔ 5.81	+1 ↔ 8.19	7.00

Table 3.3 Design Factors

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 EXPERIMENTAL DESIGN AND ANALYSIS OF COAGULATION- ADSORPTION PROCESS

The optimization of the treatment of Palm Oil Mill Effluent (POME) using the Periwinkle Shell–Chitosan composite was conducted through a Design of Experiment (DOE) approach. The study investigated the effects of three key independent variables- coagulant dosage (A), contact time (B), and pH (C) on two major response parameters: Total Dissolved Solids (TDS) and Salinity.

The experimental runs were arranged systematically according to the design matrix, with each run representing a unique combination of the selected factors and their respective levels. The results obtained from the laboratory analyses are summarized below in Table 4.1

Std	Run	Factor 1 A:Coagulant do... g/L	Factor 2 B:Time minute	Factor 3 C:pH	Response 1 TDS ppm	Response 2 Salinity ppt
12	1	0.55	180	7	2972	3.54
5	2	0.82	60	5.8	2095	2.35
16	3	0.55	105	7	2928	3.65
7	5	0.82	150	8.2	2050	2.17
8	8	0.82	150	8.2	1984	2.30
2	9	0.82	60	5.8	1931	2.03
3	10	0.28	150	5.8	1984	2.12
9	11	0.1	105	7	3021	3.71
10	13	1	105	7	3082	2.29
14	15	0.55	105	9	3062	3.77
1	16	0.28	60	5.8	1779	1.95
13	17	0.55	105	5	2046	2.08
6	18	0.82	60	8.2	2013	2.11
11	19	0.55	30	7	2985	3.62
4	20	0.82	150	5.8	1961	2.17

v

Table 4.1: design experimental table

4.2 DISCUSSION OF EXPERIMENTAL RESULTS

From the results presented in Table 4.1, the Total Dissolved Solids (TDS) and Salinity of the effluent were influenced by variations in coagulant dosage, contact time, and pH.

At low coagulant dosages (0.10–0.28 g/L), the removal efficiency was relatively poor, with higher TDS and salinity values (above 2900 ppm and 3.6 ppt), indicating insufficient coagulant particles to destabilize suspended solids and colloidal impurities.

As the dosage increased to moderate levels (0.55–0.82 g/L), a significant decrease in both TDS and salinity was observed. The lowest TDS value (1779 ppm) and salinity (1.95 ppt) occurred at a dosage of 0.28 g/L, contact time of 60 minutes, and pH 5.8, indicating optimal particle aggregation and pollutant removal under these conditions. However, excessively high dosages (≥ 1.0 g/L) resulted in a rebound in TDS (3082 ppm), likely due to restabilization of colloids and oversaturation of active sites, a phenomenon known as charge reversal.

The effect of contact time was also significant. Runs with prolonged contact times (105–150 minutes) exhibited improved pollutant removal compared to shorter times (30–60 minutes), as longer agitation periods promoted better floc formation and sedimentation. However, after 150 minutes, little improvement was observed, suggesting that equilibrium adsorption had been reached.

The influence of pH was most favorable in near-neutral to slightly alkaline conditions (pH 7–8.2). Under these conditions, both coagulation and adsorption mechanisms were enhanced, as the surface charge of the composite and the ionization state of pollutants favored electrostatic attraction. At lower pH values (5.0–5.8), protonation of chitosan's amino groups reduced active sites, slightly lowering efficiency.

Overall, the best treatment efficiency was obtained at moderate coagulant dosage (0.55–0.82 g/L), contact times between 105–150 minutes, and pH 7–8.2, yielding TDS values as low as 1779–2050 ppm and salinity between 1.95–2.17 ppt. These results demonstrate that the

periwinkle shell–chitosan composite effectively reduces pollutant load in POME through synergistic coagulation and adsorption processes.

The FTIR spectrum confirms characteristic chitosan functional groups, including O–H/N–H stretching around $\sim 3448\text{ cm}^{-1}$ and amide/amine peaks near $\sim 1650\text{--}1470\text{ cm}^{-1}$. This verifies successful formation of periwinkle shell chitosan.

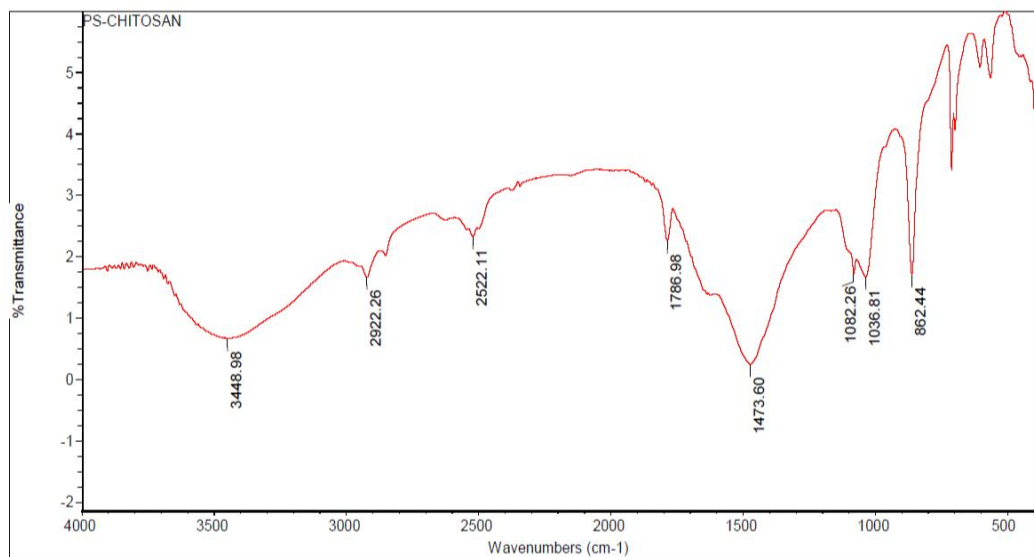


Figure 4.1: FTIR spectrum of the functional group present in periwinkle shell chitosan

Wave number (cm-1)	Intensity	Functional group	Compound	Infor
1036.81	1.672	S = O	sulfoxide	Strong, stretching
1082.26	1.723	C – O	Primary alcohol	Strong, stretching
1473.6	0.25	C – H	Methylene	Bending

1786.98	2.26	C = O	conjugated acid halide	Strong, stretching
2522.11	2.332	O – H	carboxylic acid	Strong, broad, stretching
2922.26	1.67	C – H	Alkane	Medium, stretching
3448.98	0.667	N – H	Primary, amine	Medium, stretching

Table 4.2: FTIR wave number interpretation for periwinkle shell chitosan

EDXRF Analysis of Chitosan

The Energy Dispersive X-Ray Fluorescence (EDXRF) spectrum shown below in Figure 4.2 presents the elemental composition of the prepared chitosan used for coagulation and adsorption of Palm Oil Mill Effluent (POME). The analysis reveals distinct peaks corresponding mainly to calcium (Ca) and strontium (Sr), with trace signals of iron (Fe) and tin (Sn), indicating the presence of naturally occurring mineral elements within the biopolymer structure. The high intensity Ca and Sr peaks suggest their potential role in enhancing the flocculating and adsorptive properties of chitosan through charge neutralization and bridging mechanisms. The overall spectrum confirms that the chitosan possesses essential inorganic components beneficial for effective pollutant removal in wastewater treatment.

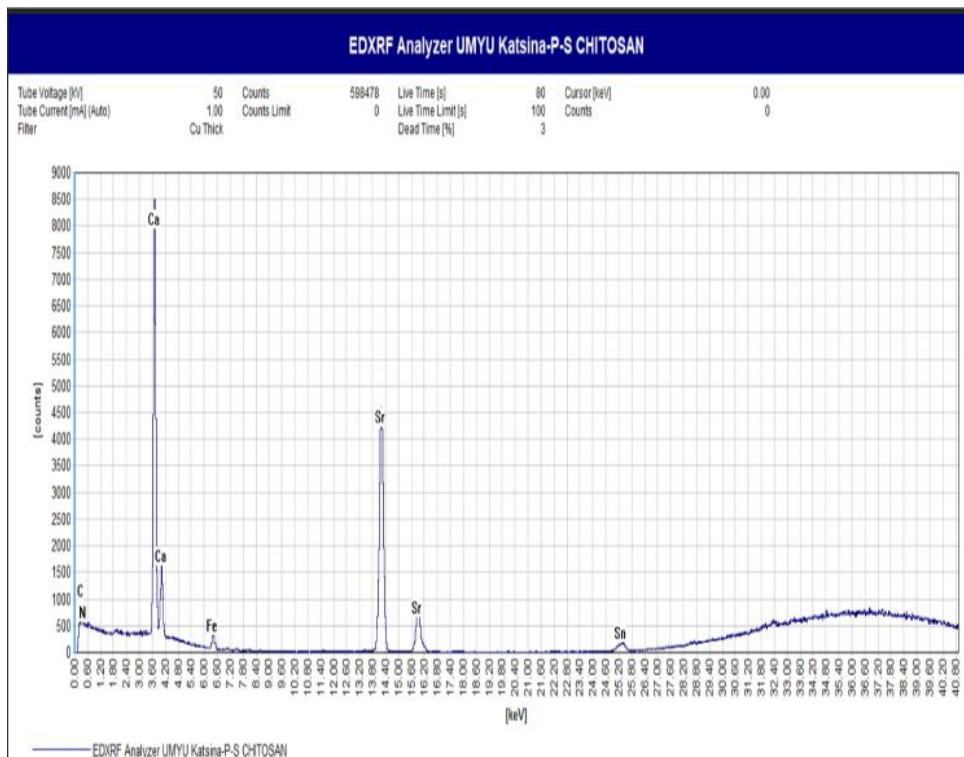


Figure 4.2: EDXRF Analyzer

Element	Conc. (wt%)
Fe2O3	0.059658
SiO2	0.658809
Al2O3	0.043764
P2O5	0.006084
SO3	0.010866
TiO2	0.013513
MnO	0.024753
CaO	88.0757
K2O	0.077494
CuO	0.000165
ZnO	0.000749
Cr2O3	0.001014
As2O3	0.000427
PbO	0.000491
NiO	9.93E-05
Cl	0.334101
Ta2O5	0.000971
WO3	0.096409
Nb2O5	0.474189
BaO	0.230914

Sb ₂ O ₃	0.15691
CeO ₂	1.878652
SrO	6.911519
Br	0.942742

Table 4.3: elemental composition of ps-chitosan

Run	Factor 1: catalyst dosage (g)	Factor 2: Time (minutes)	Factor 3: pH	Response 1: TDS Reduction (%)
1	0.55	180.0	7.0	83.24
2	0.82	60.0	5.8	38.66
3	0.55	105.0	7.0	87.64
5	0.82	150.0	8.2	87.64
8	0.82	150.0	8.2	44.05
9	0.82	60.0	5.8	87.64
10	0.28	150.0	5.8	87.64
11	0.1	105.0	7.0	73.52
13	1.0	105.0	7.0	63.02
15	0.55	105.0	9.0	50.11
16	0.28	60.0	5.8	25.73
17	0.55	105.0	5.0	87.64
18	0.82	60.0	8.2	41.58
19	0.55	30.0	7.0	87.64
20	0.82	150.0	5.8	53.86

Table 4.4: Experimental Design

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	7464.39	9	829.38	61.74	< 0.0001	significant
A-Coagulant dosage	669.16	1	669.16	49.81	< 0.0001	
B-Time	2.63	1	2.63	0.1959	0.6675	
C-pH	1.18	1	1.18	0.0881	0.7727	
AB	16.59	1	16.59	1.23	0.2925	
AC	307.02	1	307.02	22.86	0.0007	
BC	88.44	1	88.44	6.58	0.0281	
A²	5284.69	1	5284.69	393.41	< 0.0001	
B²	116.54	1	116.54	8.68	0.0146	
C²	1610.81	1	1610.81	119.91	< 0.0001	
Residual	134.33	10	13.43			
Lack of Fit	134.33	5	26.87			
Pure Error	0.0000	5	0.0000			
Cor Total	7598.72	19				

Table 4.5: TDS Reduction ANOVA for Quadratic model

Factor coding is coded

Sum of squares is **Type III - Partial**

The Model F-value of 61.74 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A, AC, BC, A², B², C² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

R²	0.9823
Adjusted R²	0.9664
Predicted R²	0.8626
Adequate Precision	25.4418
Std. Dev.	3.67
Mean	65.41
C.V. %	5.60

Table 4.6: Fit Statistics

The Predicted R² of 0.8626 is in reasonable agreement with the Adjusted R² of 0.9664; i.e. the difference is less than 0.2.

Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 25.442 indicates an adequate signal. This model can be used to navigate the design space.

Final Equation in Terms of Coded Factors

$$\text{TDS Reduction} = 87.65 + 7.00A + 0.4390B - 0.2943C - 1.44AB + 6.19AC + 3.33BC - 19.15A^2 - 2.84B^2 - 10.57C^2$$

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.

3D Surface Plot for TDS Reduction

Figure 4.3 shows the interactive effect of coagulant dosage (A) and treatment time (B) on the percentage reduction of Total Dissolved Solids (TDS) in POME at a fixed pH of 7. The surface plot illustrates that TDS removal efficiency increases progressively with higher coagulant dosage and longer contact time. The colour gradient from blue to red represents low to high TDS reduction, with the highest removal efficiency (up to ~87%) observed at higher dosage and extended treatment time. The smooth upward curvature of the plot indicates a strong positive relationship between both variables and TDS reduction, showing that optimum performance is achieved when both factors are maximized.

Factor Coding: Actual

TDS Reduction (%)

● Design Points
25.73 87.64

X1 = A

X2 = B

Actual Factor

C = 7

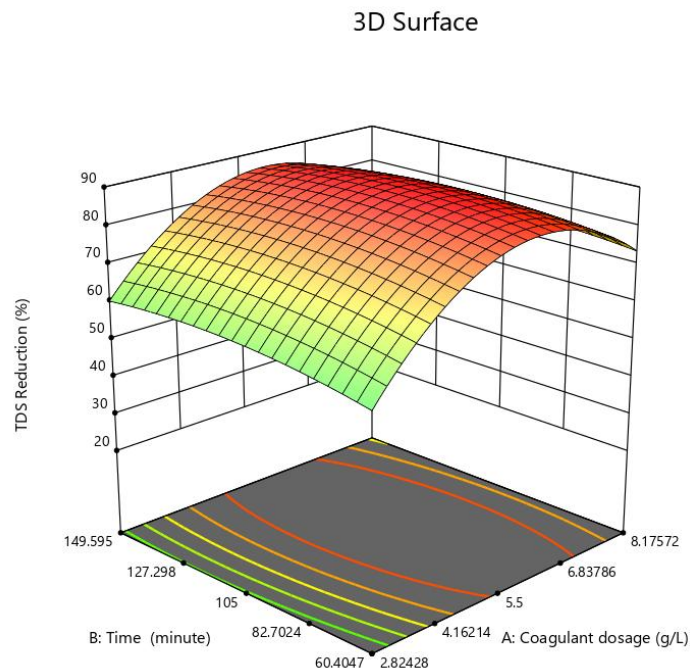


Figure 4.3: Colour points by value of TDS reduction

Figure 4.4 shows the combined effect of coagulant dosage (2.8–8.2 g/L) and pH (5.8–8.2) on TDS reduction at a fixed treatment time of 105 minutes. The plot indicates that TDS removal increases as both dosage and pH rise, with the highest reduction (about ~87%) achieved at higher dosage (~8 g/L) and slightly alkaline pH (~7–8). Lower dosage and acidic pH result in lower removal efficiency (~25%).

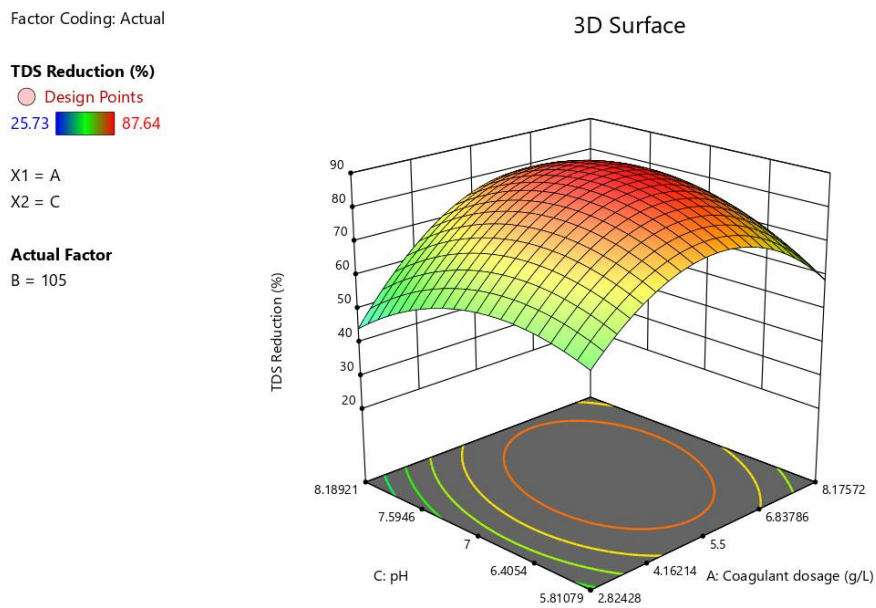


Figure 4.4: Colour points by value of TDS reduction

Figure 4.5 illustrates the effect of treatment time (60–150 min) and pH (5.8–8.2) on TDS reduction at a fixed coagulant dosage of 5.5 g/L. The TDS removal increases with longer treatment time and slightly alkaline pH, reaching a maximum of about ~87% at pH ~7–8 and ~150 min. Lower pH and short reaction time result in lower removal efficiency (~25–40%).

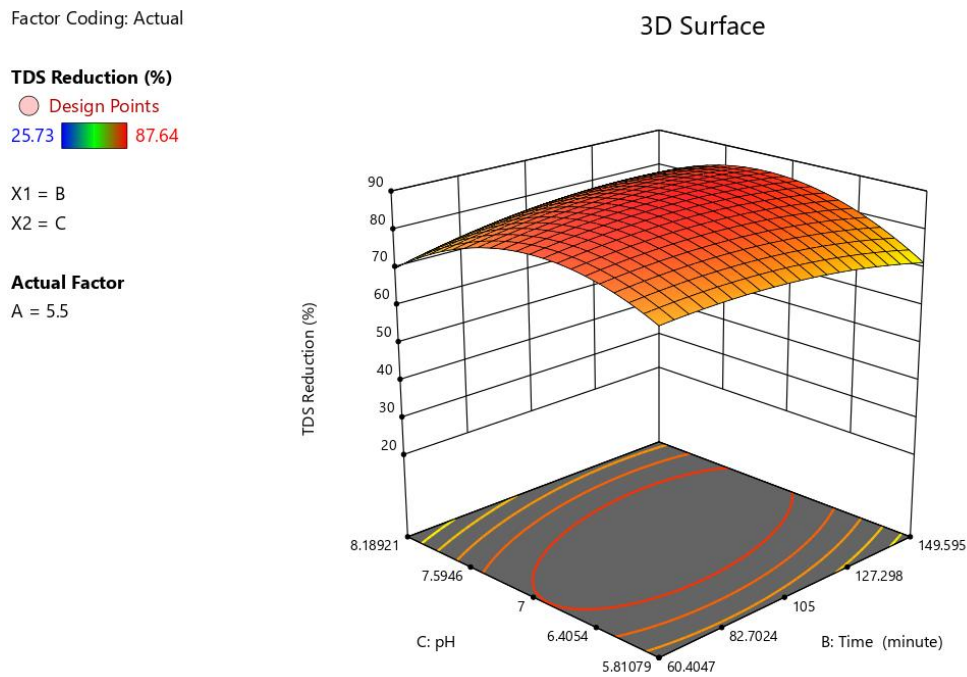


Figure 4.5: Colour points by value of TDS reduction

Figure 4.6 below presents the optimization results for TDS reduction using the desirability function. The model identified the optimal conditions as coagulant dosage = 5.5 g/L, time = 105 min, and pH = 7, achieving a predicted TDS removal of 87.65%. The desirability value of 1.000

indicates an ideal and highly satisfactory optimization outcome, meaning the selected conditions provide the best possible treatment efficiency.

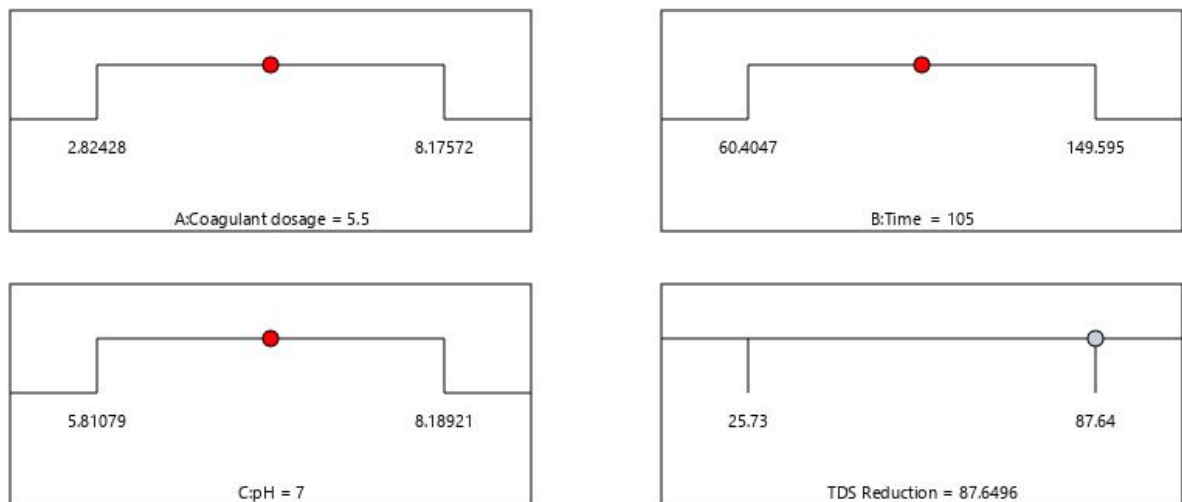


Figure 4.6:Desirability = 1.000

Solution 4 out of 100

4.3 CHARACTERIZATION OF PS-CHITOSAN COMPOSITE

The structural characterization of the Periwinkle Shell-Chitosan (PS-Chitosan) composite was performed using X-ray Diffraction (XRD) analysis to identify the crystalline phases and evaluate the degree of crystallinity. The XRD pattern of the composite exhibited sharp and intense diffraction peaks, confirming that the material is predominantly crystalline in nature.

The major diffraction peaks were observed at 2θ values of 23.9° , 26.5° , 27.5° , 33.4° , 36.4° , 38.1° , 41.4° , 43.1° , 46.0° , 48.6° , 50.5° , and 53.1° , each corresponding to specific mineral phases

present in the composite. The identified crystalline phases include Aragonite (CaCO_3), Muscovite [$\text{H}_2\text{KAl}_3(\text{SiO}_4)_3$], Quartz (SiO_2), and Orthoclase [$\text{Al}_2\text{O}_3 \cdot \text{K}_2\text{O} \cdot 6\text{SiO}_2$], as presented in Table 4.7.

S/N	2 θ (°)	Phase Identified	Chemical Formula	Remarks
1	23.9	Muscovite	$\text{H}_2\text{KAl}_3(\text{SiO}_4)_3$	Layered silicate mineral contributing to ion exchange
2	26.5	Aragonite, Quartz	$\text{CaCO}_3, \text{SiO}_2$	Dominant CaCO_3 phase providing surface activity
3	27.5	Aragonite	CaCO_3	Indicates high calcium carbonate crystallinity
4	33.4	Aragonite	CaCO_3	Confirms calcite-like structural order
5	36.4	Aragonite, Quartz	$\text{CaCO}_3, \text{SiO}_2$	Suggests high mechanical stability
6	38.1	Aragonite, Muscovite	$\text{CaCO}_3, \text{H}_2\text{KAl}_3(\text{SiO}_4)_3$	Enhances structural strength and adsorption potential
7	41.4	Aragonite, Muscovite	$\text{CaCO}_3, \text{H}_2\text{KAl}_3(\text{SiO}_4)_3$	Reflects high crystallinity and stability
8	43.1	Aragonite, Muscovite	$\text{CaCO}_3, \text{H}_2\text{KAl}_3(\text{SiO}_4)_3$	Suggests well-ordered layered structure
9	46.0	Aragonite, Muscovite	$\text{CaCO}_3, \text{H}_2\text{KAl}_3(\text{SiO}_4)_3$	Indicates mixed mineral phase
10	48.6	Aragonite, Orthoclase	$\text{CaCO}_3, \text{Al}_2\text{O}_3 \cdot \text{K}_2\text{O} \cdot 6\text{SiO}_2$	Adds surface functional groups (Al–O, Si–O)
11	50.5	Aragonite	CaCO_3	Reflects fine crystallite structure
12	53.1	Aragonite	CaCO_3	Confirms CaCO_3 dominance

Table 4.7: Identified Crystalline Phases in PS–Chitosan Composite

4.4 PHASE COMPOSITION AND STRUCTURAL PROPERTIES

The XRD profile revealed that Aragonite (CaCO_3) was the most dominant phase, as indicated by its multiple strong peaks. Aragonite is a polymorph of calcium carbonate with a high specific surface area, making it suitable for adsorption and ion-exchange processes. The prominence of this mineral in the composite demonstrates that the periwinkle shell component retained its crystalline calcium carbonate structure after processing.

The presence of Muscovite, an aluminosilicate mineral, adds stability and enhances the composite's ability to bind heavy metal ions through electrostatic attraction. Quartz (SiO_2) contributes to mechanical hardness and chemical inertness, while Orthoclase introduces additional functional sites through Al–O–Si bonds, improving the composite's adsorption capacity.

The combination of these minerals results in a heterogeneous crystalline structure, which is beneficial for wastewater treatment as it provides multiple active binding sites for pollutant adsorption.

4.5 CRYSTALLITE SIZE AND SURFACE CHARACTERISTICS

The average crystallite sizes, calculated using the Scherrer equation, ranged from 111 Å to 702 Å, indicating the presence of fine, nano-scale particles. Smaller crystallite sizes increase the surface-to-volume ratio, leading to improved surface reactivity and higher adsorption efficiency.

The coexistence of sharp and broad peaks in the XRD pattern indicates a mixture of crystalline and amorphous regions. This semi-crystalline nature enhances the composite's surface flexibility, allowing easy penetration and interaction of pollutants during coagulation and adsorption.

Furthermore, the uniform distribution of aragonite and chitosan throughout the structure enhances ion-exchange, electrostatic attraction, and surface complexation, the main mechanisms governing pollutant removal.

4.6 INTERACTION BETWEEN ORGANIC AND INORGANIC PHASES

The PS–Chitosan composite demonstrates a strong interaction between the organic biopolymer (chitosan) and the inorganic mineral component (periwinkle shell). Slight shifts in diffraction peaks compared to standard aragonite patterns indicate partial interfacial bonding and integration between chitosan's amino ($-\text{NH}_2$) and hydroxyl ($-\text{OH}$) groups and the calcium carbonate lattice.

This interaction forms a hybrid matrix in which chitosan provides chemical reactivity and biocompatibility, while periwinkle shell derived aragonite contributes structural rigidity and porosity. Such integration results in a synergistic composite with improved adsorption sites, mechanical stability, and regeneration potential during repeated wastewater treatment cycles.

The structural hybridization also reduces particle agglomeration and enhances surface uniformity, which are key parameters for efficient coagulation and adsorption.

General information

Analysis date	2025-09-16 11:34:10	Measurement start time	2025-09-16 10:25:24
Analyst	Default	Operator	Default
Sample name	PS- CHITOSAN	Comment	
Measured data name	C:\WallPaper\16-09-2025\UMYU\PS- CHITOSAN_20250916_1...	Memo	

Peak list

No.	2 θ , °	d, Å	Height, cps	FWHM, °	Int. I., cps°	Int. W., °	Asymmetry	Decay(η L/mL)	Decay(η H/mH)	Size, Å
1	23.91(2)	3.719(4)	161(22)	0.25(10)	75(12)	0.46(14)	0.5(8)	0.6(9)	1.5(4)	337(137)
2	26.498(14)	3.3610(17)	1046(139)	0.21(4)	335(27)	0.32(7)	1.4(12)	0.5(3)	1.3(3)	406(84)
3	27.49(5)	3.242(5)	354(40)	0.26(4)	101(16)	0.29(8)	1.0(7)	0.2(9)	0.0(6)	331(46)
4	33.39(5)	2.681(4)	834(85)	0.22(4)	278(19)	0.33(6)	1.6(18)	1.0(5)	0.8(8)	398(81)
5	36.390(12)	2.4669(8)	493(54)	0.22(7)	174(12)	0.35(6)	0.8(9)	1.1(4)	1.1(4)	406(127)
6	38.17(3)	2.3558(19)	225(24)	0.79(10)	211(24)	0.9(2)	0.5(4)	0.8(4)	0.0(4)	111(15)
7	41.428(17)	2.1778(8)	380(66)	0.13(6)	76(12)	0.20(7)	0.7(13)	1.0(7)	1.0(6)	702(332)
8	43.18(5)	2.093(2)	351(56)	0.21(7)	100(14)	0.28(9)	1.0(10)	1.0(7)	0.2(7)	419(137)
9	46.072(17)	1.9685(7)	983(184)	0.21(4)	278(22)	0.28(8)	0.5(6)	1.1(3)	0.27(17)	428(72)
10	48.64(4)	1.8703(13)	347(46)	0.23(6)	111(13)	0.32(8)	0.7(6)	1.2(7)	0.2(6)	397(113)
11	50.53(2)	1.8050(7)	207(24)	0.29(6)	75(12)	0.36(10)	5(5)	0.0(6)	1.5(4)	318(62)
12	53.18(3)	1.7208(10)	221(17)	0.45(10)	163(21)	0.74(15)	1.2(11)	1.5(3)	0.0(7)	207(47)

Table 4.8: PS-Chitosan Analysis

No.	2 θ , °	Phase Name	Chemical Formula	Card No	Norm. I.	Profile Type	Distributi...	Degree of Orientation
1	23.91(2)	Muscovite: 0 2 3	H ₂ K Al ₃ (Si O ₄) ₃	00-001-1098	22.32	Split pseudo-Voigt	-	-
2	26.498(14)	aragonite: 1 1 1,Quartz: 1 0 1	Ca C O ₃ ,Si O ₂	01-085-6705,00-00...	100.00	Split pseudo-Voigt	-	-
3	27.49(5)	aragonite: 1 0 2	Ca C O ₃	01-085-6705	30.22	Split pseudo-Voigt	-	-
4	33.39(5)	aragonite: 2 0 1	Ca C O ₃	01-085-6705	82.97	Split pseudo-Voigt	-	-
5	36.390(12)	aragonite: 0 2 0,Quartz: 1 1 0,...	Ca C O ₃ ,Si O ₂ ,Al ₂ ...	01-085-6705,00-00...	51.96	Split pseudo-Voigt	-	-
6	38.17(3)	aragonite: 2 1 1,Muscovite: 1 3 3	Ca C O ₃ ,H ₂ K Al ₃ (...)	01-085-6705,00-00...	62.99	Split pseudo-Voigt	-	-
7	41.428(17)	aragonite: 1 2 1,Muscovite: 2 0...	Ca C O ₃ ,H ₂ K Al ₃ (...)	01-085-6705,00-00...	22.72	Split pseudo-Voigt	-	-
8	43.18(5)	aragonite: 0 2 2,Muscovite: 1 3 5	Ca C O ₃ ,H ₂ K Al ₃ (...)	01-085-6705,00-00...	29.69	Split pseudo-Voigt	-	-
9	46.072(17)	aragonite: 1 2 2,Muscovite: 0 2...	Ca C O ₃ ,H ₂ K Al ₃ (...)	01-085-6705,00-00...	82.82	Split pseudo-Voigt	-	-
10	48.64(4)	aragonite: 1 0 4,Orthoclase: 3...	Ca C O ₃ ,Al ₂ O ₃ · K...	01-085-6705,00-00...	32.98	Split pseudo-Voigt	-	-
11	50.53(2)	aragonite: 2 2 1	Ca C O ₃	01-085-6705	22.30	Split pseudo-Voigt	-	-
12	53.18(3)	aragonite: 1 2 3	Ca C O ₃	01-085-6705	48.62	Split pseudo-Voigt	-	-

No.	2 θ , °	Ring Factor	β Cluster
1	23.91(2)	-	-
2	26.498(14)	-	-
3	27.49(5)	-	-
4	33.39(5)	-	-
5	36.390(12)	-	-
6	38.17(3)	-	-
7	41.428(17)	-	-
8	43.18(5)	-	-
9	46.072(17)	-	-
10	48.64(4)	-	-
11	50.53(2)	-	-
12	53.18(3)	-	-

Table 4.9: Qualitative Analysis Results

Phase name	Formula	Figure of merit	Phase reg. detail	Space Group	DB Card Number
aragonite	Ca C O3	0.667	S/M(PDF-4 Minerals 2025)	62 : Pnma	01-085-6705
Muscovite	H2 K Al3 (Si O4)3	2.225	Import(PDF-4 Minerals 2025)	15 : C12/c1	00-001-1098
Quartz	Si O2	1.744	Import(PDF-4 Minerals 2025)	154 : P3221	00-001-0649
Orthoclase	Al2 O3 · K2 O · 6 Si O2	2.015	Import(PDF-4 Minerals 2025)	12 : C12/m1	00-002-0475

Phase Data View

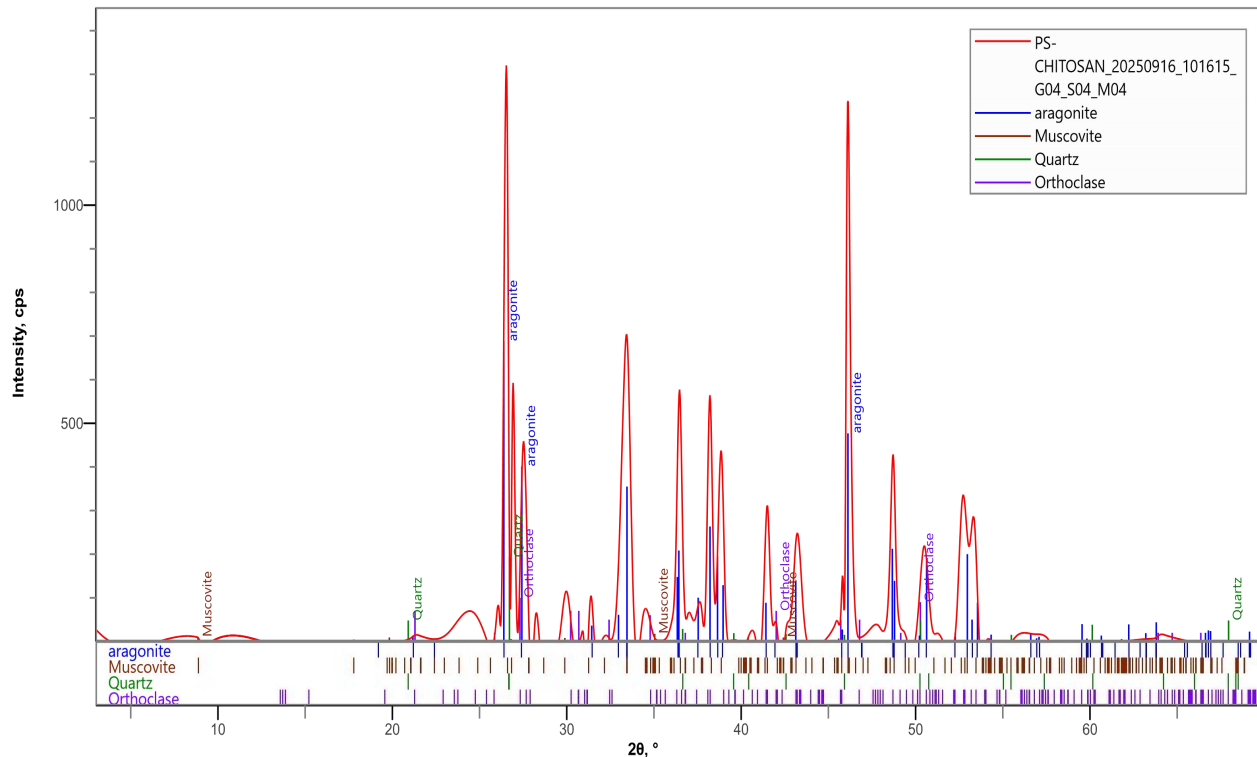


figure 4.7: Phase Data View

The XRD pattern confirms the crystalline phases present in the prepared periwinkle-shell chitosan. Major peaks correspond to aragonite, with minor phases of quartz, muscovite, and orthoclase, indicating the presence of calcium-based minerals and trace silicate components that support the structural integrity and adsorption capability of the chitosan.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

This study examined the treatment of Palm Oil Mill Effluent (POME) using coagulation and adsorption processes as environmentally sustainable and cost-effective methods for wastewater purification. Palm Oil Mill Effluent, a high-strength industrial wastewater, poses a serious threat to aquatic ecosystems and public health due to its high levels of organic matter, suspended solids, oil, and grease. Conventional treatment methods are often costly and energy-intensive, creating the need for alternative, nature-based solutions.

The research demonstrated that coagulation and adsorption using natural coagulants and adsorbents specifically periwinkle shell and chitosan composite can effectively reduce key pollutants such as turbidity, total dissolved solids (TDS), salinity, and electrical conductivity in POME. The results showed that process parameters such as coagulant dosage, pH, and contact time significantly affect treatment efficiency. Optimal performance was achieved at moderate dosages, near-neutral pH (7–8.2), and longer contact times (105–150 minutes), indicating favorable conditions for both charge neutralization and adsorptive removal of contaminants.

The characterization of the composite via X-ray diffraction (XRD) revealed crystalline peaks associated with aragonite, quartz, muscovite, and orthoclase, with crystallite sizes ranging between 111–702 Å, suggesting a fine heterogeneous surface with strong adsorption potential. These structural features enhanced pollutant binding and contributed to efficient purification performance.

In summary, the study concludes that the periwinkle shell–chitosan composite serves as a viable, eco-friendly, and sustainable material for POME treatment. The integration of coagulation and adsorption provides a synergistic mechanism capable of removing both colloidal and dissolved pollutants. This approach not only improves effluent quality for safe discharge or reuse but also aligns with global efforts toward green chemistry, resource recovery, and zero-liquid discharge (ZLD) in industrial wastewater management.

5.2 RECOMMENDATIONS

1. Adoption of Natural Coagulants and Adsorbents:

Industries involved in palm oil production should consider replacing or supplementing chemical coagulants with natural materials such as chitosan and periwinkle shells. These materials are biodegradable, locally available, and environmentally benign, making them suitable for sustainable wastewater management.

2. Optimization of Process Parameters:

Future treatment systems should maintain moderate coagulant dosage, contact times between 105–150 minutes, and neutral to slightly alkaline pH to maximize coagulation and adsorption efficiency.

3. Scale-Up Studies:

The process should be further evaluated at pilot or industrial scale to validate laboratory results under real operational conditions and determine the cost-benefit ratio for large-scale application.

4. Integration with Other Technologies:

The coagulation–adsorption method can be integrated with membrane filtration or biological treatment to achieve higher purification efficiency and meet stringent discharge standards. This hybrid approach could support the development of zero-liquid discharge systems for palm oil mills.

5. Monitoring and Compliance:

Regular monitoring of treated effluent parameters such as BOD, COD, TDS, EC, and salinity should be implemented to ensure compliance with environmental regulations and protect receiving water bodies.

6. Resource Recovery and Reuse:

The sludge generated after treatment can be explored for nutrient recovery or use as a soil conditioner, while the treated effluent may be reused for irrigation or washing processes, promoting circular economy practices within the palm oil sector.

7. Further Research:

Additional studies should investigate other local agro-waste materials (e.g., coconut husk, rice husk, snail shells) for potential use as coagulants or adsorbents. Also, exploring modification techniques (chemical activation or surface functionalization) could enhance adsorption capacity and reusability.

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APPENDIX

General information

Analysis date	2025-09-16 11:34:10	Measurement start time	2025-09-16 10:25:24
Analyst	Default	Operator	Default
Sample name	PS- CHITOSAN	Comment	
Measured data name	C:\WallPaper\16-09-2025\UMYU\PS- CHITOSAN_20250916_1...	Memo	

Multiple Profile

