APPLICATION OF OKRA (*ABELMOSCHUS ESCULENTUS*) MUCILAGE-BASED AS NATURAL COAGULANT IN DOMESTIC WASTEWATER TREATMENT

ONG KAI QI

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By

ONG KAI QI

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Name of Student: Ong Kai Qi

I hereby declare that all corrections and comments made by the supervisor(s)and examiner have been taken into consideration and rectified accordingly.

Signature:

Date : 3.8.2022

Approved by:

(Signature of Supervisor)

Name of Supervisor : Dr. Nik Azimatolakma Awang

Date : 8.8.2022

Approved by:

(Signature of Examiner)

Name of Examiner : Assoc. Prof. Dr. Puganeshwary Palaniandy

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ABSTRAK

Kajian ini menyelidik ekstrak bahan penggumpal karbohidrat menggunakan air bagi koagulan karbohidrat daripada bendi (AEaqs) dan mengkaji ciri kimia ekstraknya (iaitu titik isoelektrik dan Fourier Transform Infra). AEaqs bertindak sebagai bahan penggumpal anionik untuk menggalakkan mekanisme penyambungan melalui kumpulan berfungsi utama, karboksil (C=O) dan hidroksil (OH). Keupayaan ekstrak AEaqs untuk bertindak sebagai bahan penggumpal primer dan bantuan bahan penggumpal kepada alum dinilai menggunakan air sisa domestik yang dikumpul daripada kolam pengoksidaan Kampus Kejuruteraan Universiti Sains Malaysia. Parameter yang diuji dalam air sisa domestik berada di bawah had Piawaian B, Peraturan Kualiti Alam Sekitar (kumbahan) 2009, Malaysia. Analisis ujian balang adalah berdasarkan peratusan penyingkiran kekeruhan, pepejal terampai (SS), COD dan zink (Zn^{2+}). Selain itu, keadaan optimum (pH dan dos), mekanisme pengumpalan dan saiz flok turut dikaji. Keputusan telah menunjukkan bahawa 20 mg/L AEaqs pada pH 12 mencapai 54%, 59%, 53% dan 21% penyingkiran kekeruhan, SS, COD dan Zn^{2+} daripada nilai awalnya 23.14 NTU, 34 mg/L, 187 mg/L dan 0.14 mg/L. Sebagai perbandingan, 30 mg/L alum pada pH 6 dapat mengeluarkan 99%, 100%, 44% dan 36% kekeruhan, SS, COD dan Zn^{2+}. Sebagai bantuan bahan penggumpal, 30 mg/L alum + 2 mg/L AEaqs pada pH 6 mencapai penyingkiran tertinggi dalam kekeruhan, SS, COD dan Zn^{2+} dengan 32%, 18%, 64%, 14%. Pengurangan 50% isipadu alum daripada dos optimumnya bersama dos AEaqs (15 mg/L alum + 5 mg/L AEaqs) berjaya menghilangkan -51%, -76%, 39%, 21% kekeruhan, SS, COD dan Zn^{2+}. Kadar penyingkiran keseluruhan untuk AEaqs sebagai bahan penggumpal primer dan bantuan bahan penggumpal adalah lebih rendah berbanding ketika alum digunakan secara bersendirian.

ABSTRACT

This research investigates water treated extraction (AEaqs) of carbohydrate coagulants from okra (*Abelmoschus esculentus*) and inspects its chemical characteristics (i.e. isoelectric point (IEP) and Fourier Transform Infrared). AEaqs serves as anionic coagulants to boost bridging mechanisms by carboxyl (C=O) and hydroxyl functional groups (OH). The ability of AEaqs extract to behave as primary coagulant and coagulants aid together with alum was evaluated using the domestic wastewater collected from oxidation pond of treatment plant in the Engineering Campus of Universiti Sains Malaysia. The parameters analysed in domestic wastewater was below the limits set in Standard B, Environmental Quality (sewage) Regulation 2009, Malaysia. Jar test results were analysed according to the removal percentage of turbidity, suspended solids (SS), COD and zinc (Zn^{2+}). In addition, the optimum conditions (pH and dosage), coagulation mechanism and floc structure were also explored. Results had shown that 20 mg/L AEaqs at pH 12 achieved 54%, 59%, 53% and 21% of turbidity, SS, COD and Zn^{2+} removals from its initial values of 23.14 NTU, 34 mg/L, 187 mg/L and 0.14 mg/L respectively. On the other hand, 30 mg/L alum at pH 6 removed 99%, 100%, 44% and 36% of turbidity, SS, COD and Zn^{2+} removals respectively. As coagulant aids, 30 mg/L alum + 2 mg/L AEaqs at pH 6 gained the highest removal in domestic wastewater sample of turbidity, SS, COD and Zn^{2+} with 32%, 18%, 64%, 14% respectively. A 50% reduction in the concentration of alum from its optimal dosage with doses of AEaqs (15 mg/L alum + 5 mg/L AEaqs) managed to remove -51%, -76%, 39%, 21% of turbidity, SS, COD and Zn^{2+}. The overall removal rates for AEaqs as primary coagulant and coagulant aid were lower as compared to when alum was used alone.

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LIST OF ABBREVIATIONS

AEaqs	Extract of *Abelmoschus esculentus* with water
Alum	Aluminium sulphate
COD	Chemical Oxygen Demand
EDX	Energy Dispersive X-Ray Spectroscopy
FTIR	Fourier Transform Infrared
IEP	Isoelectric point
NTU	Nephelometric Turbidity Unit
PZC	Point of zero charge
SS	Suspended solids

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Water is the most precious natural resource; no living species, including humans, can thrive without it. Rapid population growth, rising living standards, climate change, industrialization, agriculture and urbanization are all contributing to global water shortage (Choy et al., 2014). In order to preserve our global water supply, many major cities around the world considered treated municipal wastewater as a supplementary water source for a range of uses such as agricultural irrigation, landscaping, industrial activities (cooling and process demands), groundwater replenishment, recreational, and other uses (Bukhari, 2008).

Adequate water treatment and sanitation are required to remove turbidity, pollutants and other infectious bacteria. To remove particles from water, several technologies have been utilised, including coagulation, flotation, ion exchange, and electrolytic techniques (Shahimi et al., 2021). The application of a coagulation flocculation system to treat wastewater is still a popular practise on both large and small scales wastewater treatment. The idea of coagulation-flocculation is to agglomerate small particles and colloidal particles into bigger particles in order to eliminate turbidity, natural organic matter, and other soluble organic and inorganic contaminants in wastewater (Teh et al., 2016).

The majority of the coagulants utilized in the treatment are conventional aluminium, ferric-based salts, or synthetic organic coagulants. Though chemical coagulation is a prominent method for treating wastewater before it is transported into a biological treatment unit, it has some downsides (e.g. high levels of coagulant and flocculant residue in water bodies that can harm human health and pollute the environment) (Zaman, 2018). To mitigate these disadvantages, natural flocculants can be employed in coagulation-flocculation treatment.

Natural coagulants can be grouped into two types: plant-based coagulants and non-plant-based coagulants. Since ancient times, plant materials have been used as natural coagulants and flocculants to reduce turbidity in wastewaters (Patale and Pandya, 2012). Plant-derived flocculants are polysaccharides in their natural condition, with a mucilaginous texture and a neutral pH (Das et al., 2021). Mucilage, a viscous and complex carbohydrate with a high water retention capacity, is primarily accountable for the robust coagulation capability of plant-based coagulants (Vijayaraghavan et al., 2011).

Natural coagulants are shown to have a substantial positive impact on the environment and ecology. Natural coagulants are biodegradable and environmentally friendly. These coagulants are more sustainable than chemical coagulants since they pose less health hazards, and the costs of distributing them are less expensive because they are available locally (Shahimi et al., 2021). Encouraged by these facts, okra (*Abelmoschus esculentus*) is selected as natural coagulant in the coagulation-flocculation process in treating domestic wastewater.

1.2 PROBLEM STATEMENT

Chemically enhanced primary treatment (CEPT) removes suspended particles, organic carbon, and nutrients from raw wastewater via coagulant chemicals (Shewa and Dagnew, 2020). It has gained a lot of interest in recent years because to its improved treatment capacity and related advantages over the conventional main treatment technique. Coagulation-flocculation has been widely used as a pre-treatment prior to membrane filtration in order to reduce membrane fouling (Ang and Mohammad, 2020). With its simplified operation, uncomplicated design, and low energy demand, it can enhance the quality of treated effluent while limiting the size, complexity, and cost of secondary treatment.

Even though classic chemical-based coagulants such as aluminium and iron (III) salts are useful in wastewater purification, they have a number of limitations such as inefficiency at low temperatures, severe health consequences, huge sludge densities, and a change in the pH of the purified water (Shamsnejati et al., 2015). Aluminium is regarded as a significant factor contributing to poisoning in dialysis encephalopathy (Thakur and Choubey, 2014). Likewise, many studies have associated Alzheimer's disease to residual aluminium ions in treated water (Anastasakis et al., 2009). As a result, alternative natural coagulants that are less costly and more environmentally friendly should be developed to supplement, if not totally substitute, alum, ferric salts, and synthetic polymers in order to accelerate the transition to a clean technology era.

Recently, natural coagulants have sparked attention in underdeveloped nations since they are harmless to humans, cost effective, widely available from renewable resources and biodegradable (Anastasakis et al., 2009). Moreover, they generate less voluminous sludge and without impact on the pH of the purified water (Shamsnejati et al., 2015). A few studies had conducted by Matilainen et al. (2010), Al-Aubadi and Hashim (2015) and Kumar et al. (2020) using natural coagulant as coagulant aid. However, there are very few studies that have been reported utilising natural coagulants as coagulants aids. As a result, comparing the efficiency of okra as a coagulant and instantly comparing the data obtained from other established literatures is difficult.

Numerous researchers such as Thakur and Choubey (2014), Effendi et al. (2015), Freitas et al. (2015), Shamsnejati et al. (2015) and Fard et al. (2021) focused on coagulation-flocculation process using mucilage natural coagulant. These reported studies had proven the effectiveness of natural coagulant extracts in removing pollutants in industrial wastewater and synthetic wastewater that consists of kaolin, humic acid, and dyes. Unfortunately, the implementation of natural coagulants in water industry is not widely accepted or applied. This is because natural coagulants have a lower coagulation capability, a booster dose of natural coagulant is necessary to achieve the same results as chemical coagulants. Additional expenditures related with additional dose consumption, such as natural coagulant extraction, storage, shipping, and handling, cause doubt over the use of natural coagulant as a green material (Ang and Mohammad, 2020).

Moringa oleifera, *strychnos potatorum*, nirmali seeds, and cactus are examples of common and established natural coagulants with high removal efficiency (Shahimi et al., 2021). Application of mucilage natural coagulant have a promising result in removal of turbidity in wastewater treatment. However, further studies need to be done on the efficiency on removing other parameters such as SS, COD and heavy metals.

Importing chemicals such as polyaluminium chloride and alum is expensive for developing countries due to the difficulty in assessing the chemical coagulants. The importance of the research is to improve the efficacy of coagulation in wastewater treatment by utilising locally accessible natural coagulant to lower the expense of treatment. Reducing the chemical coagulant usage helps to reduce less toxic sludge and less impact to human health.

The present study concentrates on domestic wastewater sampled from the oxidation pond in the Engineering Campus of Universiti Sains Malaysia. The study also focuses on the use of natural polymer extracts from okra as primary coagulant and coagulant aid with alum as primary coagulant.

1.3 RESERCH OBJECTIVES

The objectives of the research are:

i. To investigate the characteristic of okra (*Abelmoschus esculentus*) mucilage based on isoelectric point (IEP) and functional group properties.
ii. To determine the optimum condition (pH and dosage) of okra mucilage as primary and coagulant aid in removing turbidity, SS, COD and Zn^{2+} in domestic wastewater.
iii. To study the mechanism of coagulation process using okra mucilage as primary and coagulant aid based on removal efficiency and floc analysis.

1.4 SCOPE OF STUDY

The study is focus mainly on:

i. The removal efficiency of okra mucilage as primary coagulating agent and coagulant aid in domestic wastewater treatment based on parameters such as turbidity, SS, COD and heavy metal (Zn^{2+}).
ii. Mechanism of coagulation and flocculation process by okra mucilage.
iii. Determination of optimum conditions such as dosage of coagulant and pH to improve the coagulation efficacy.

CHAPTER 2

LITERATURE REVIEW

2.1 DOMESTIC WASTEWATER

Organic molecules from all human activities, including domestic, industrial, and agricultural sources, pollute the wastewater, posing an ecological risk to animals and humans (Aboussabiq et al., 2014). Before being discharged into the environment or before biological treatment, it requires advanced treatment. Domestic wastewater or sewage is mainly composed of effluent from domestic activities such as showering, dishwashing, laundry, and toilet flushing (Shahimi et al., 2021). Every day, a person produces 225 litres of sewage in Malaysia.

Domestic wastewaters are collected in “sewers” which are underground conduits. Normally, sewer flow is gravity-fed into a public sewer system and treated at a public sewage treatment facility to fulfil sewage effluent discharge limits to public water bodies. Many villages and cities in Malaysia have a separate sewer system. Separate pipes transmit wastewater from storm sewers, industrial sewers, and sanitary sewers. This system will not experience CSOs (Combined Sewer Overflows), which are common in combined sewer systems (IWK, 2019).

In Malaysia, there are three types of sewerage systems. These are classified as public sewage treatment plants, private sewage treatment plants, and individual septic tanks. Individual septic tanks are designed for a single house or individual buildings with a population equivalent (PE) of up to 150 people, and these systems only partially treat sewage (Nasir, 2014). Public sewage treatment facilities, on the other hand, include

Imhoff tanks, oxidation ponds, mechanical plants, network pump stations, and communal septic tanks.

2.2 CHARACTEERISTIC OF DOMESTIC WASTEWATER

The instantaneous change in the volume and physical, chemical, and biological properties of wastewater had become a challenge in wastewater treatment. Batch discharge into the system, a sudden decrease or increase in input flow and concentration (process start-up or shut-down and heavy rainfalls), diurnal, weekly, and seasonal input load oscillations all modify the characteristics of wastewater) (Niku and Schroeder, 1981). Physical, chemical, and/or biological treatment will be used depending on the level of contaminants and local requirements. As a result, determining the characteristics of domestic wastewater is critical for the design and operation of collection, treatment, and disposal facilities, as well as for environmental quality engineering management.

2.2.1 Turbidity

Turbidity is an optical property of water that measures the amount of light dispersed by suspended and undissolved particles when light is transmitted through the sample. It is measured using turbidimetry or nephelometry techniques and is represented in arbitrary units (Nephelometric Turbidity Unit, NTU). Turbidity quantifies clearness of water that often shows the presence of dispersed, suspended solids; particles that are not in true solution such as silt, clay, algae, and other microorganisms; organic debris, and other minute particles (Lambrou et al., 2010). Turbidity decreases the clarity of water to transmitted light as it increases by scattering and adsorbing light. Turbid particles also provide shelter to pathogen from disinfection resulting in an outbreak of waterborne disease.

Many parameters, including particle size distribution, particle shape and surface condition, scattering particle refractive index, and light wavelength, affect the direct relationship between turbidity data and suspended solids concentration (Tan et al., 2017). When the concentration of suspended particles is identical, but the material composition is different, it is unfeasible to scatter the same quantity of light (Du et al., 2021). As a result, turbidity is correlated to suspended matter, however the relationship is difficult to quantify.

2.2.2 Chemical oxygen demand

Chemical oxygen demand (COD) is a water quality metric that oxidises wastewater with a boiling acid dichromate solution to evaluate the amount of biologically active and biologically inactive organic compounds in water (Khan and Ali, 2018). During COD tests, silver sulphate is added to aid in complete oxidation. The chloride ion combines with silver ions to precipitate silver chloride, which removes silver's catalytic activity and results in a negative interference. In order to minimize reaction interference, mercury sulphate is added. All organic matter is transformed to carbon dioxide during a COD test. Dichromate (Cr^{6+}, orange colour) is reduced to chromate (Cr^{3+}, green colour) via Equation 2.1 (Hu and Grasso, 2005):

$$Cr_2O_7{}^{2-} + 6e^- + 14H^+ \rightarrow 2Cr^{3+} + 7H_2O \qquad (2.1)$$

According to Prajapati et al. (2015), a maximum of 85% COD was reduced at pH 5 when using optimum alum dose of 60 mM coagulant mass loading to treat rice grain–based biodigester distillery effluent. Any deviation from pH 5 lowers COD removal effectiveness. This might be due to the sweep coagulation of alum that functioned best at optimum pH 5. Furthermore, Freitas et al. (2015) reported that 85.69% of COD was removed using a low amount of the okra mucilage (3.20 mg/L) at pH 6.

2.2.3 Heavy metals

Heavy metals are metals having high atomic weights in the range of 63.5–200.6 g mol^{-1} and the densities more than 5 gr cm^{-3} (Shadman et al., 2019). Numerous essential heavy metals such as copper, iron, manganese, cobalt, zinc, and nickel are required by plants to produce cofactors that are structurally and functionally important for enzymes and other proteins. Cadmium, lead, mercury, chromium, and aluminium are nonessential heavy metals that are not needed by plants for any metabolic processes (Raychaudhuri et al., 2021). These metals are released into the environment through both natural and anthropogenic sources, including industrial discharge, automotive exhaust, and mining, polluting the water.

Zinc is a highly reactive metal that combines with oxygen and other nonmetals and reacts with dilute acids to produce hydrogen. Zinc is an essential mineral for humans. However, overdose of zinc may lead to a series of health issues, including stomach pains, skin irritations, nausea, and anaemia (Bakar et al., 2021). According to Marcussen et al. (2010), water with a high zinc concentration has a milky appearance and astringent flavour. When the water is heated, high levels of zinc may produce a greasy film on top of the water.

Heredia and Martín (2009) investigated the effectiveness of tannin-based coagulant to remove Zn^{2+}, Ni^{2+} and Cu^{2+} by coagulation flocculation process. Cu^{2+} concentrations were found to be decreased up to 90% at optimum pH 6 while Zn^{2+} and

Ni^{2+} concentrations were diminished up to a 75% and 70% at optimal pH 7 and 8 respectively. According to Tang et al. (2016), heavy metal precipitation occurs in alkaline environments, with negatively charged hydrolysed species of typical inorganic coagulants at high pH values (pH 9-10).

2.2.4 Solids

Solids in a water sample are classified into numerous types, including total, dissolved, suspended, organic, inorganic, and floatable solids. Rocks, minerals, and metals are examples of inorganic matter. Some of the most prevalent and significant inorganic dissolved solids are chlorides, phosphates, nitrates, and certain metals. The inorganic portion can be extremely harmful and also unappealing in environmental receiving waters. Organic matter (OM) in water is divided into two categories: dissolved and non-dissolved. Carbohydrates/polysaccharides, amino acids/peptides/proteins, lipids, humic substances, and anthropogenic organic pollutants are all components of dissolved organic matter (Shi et al., 2021). Particulate organic matter includes plant debris, algae, phytoplankton cell and bacteria (Mostofa et al., 2013).

Suspended matter in water is the solid matter trapped from water in a 0.45 μm filter membrane and dried to a consistent weight at 103°C-105°C (Du et al., 2021). The impacts of suspended particles on chlorine disinfection are generally proportionate to the number and size (Liang et al., 2013). The sponge-like matrix of suspended particles captured bacteria and protected them from disinfection. In addition, organic debris may further restrict chlorine disinfection effectiveness through stabilising bacteria cell membranes (Friedler et al., 2021), resulting in carcinogenic unwanted disinfection by-products such as trihalomethanes (THMs) and haloacetic acids (HAAs) (Mazhar et al., 2020).

2.3 TYPE OF COAGULANTS

During the process of coagulant-flocculation, chemicals, such as coagulants or flocculants, are introduced into wastewater to facilitate in the elimination of contaminants. Coagulants are surface-charged compounds or substances that enhance the instability of suspended particles and the production of floc (Chum, 2020). Coagulants are categorized into three types based on their chemical composition: inorganic-based coagulants, organic-based flocculants, and hybrid materials (Tang et al., 2016).

2.3.1 Inorganic coagulants

Aluminum and iron are two commonly utilized coagulant hydrolyzing metal salts. Pliny (about 77 AD) was the first to cite 'alum,' or aluminum sulphate, as a water purifier (Duan and Gregory, 2003). Aluminum sulphate, aluminum chloride, sodium aluminate, aluminum chlorohydrate, polyaluminum chloride, polyaluminum sulphate chloride, polyaluminum silicate chloride, and forms of polyaluminum chloride containing organic polymers are examples of common aluminum-based coagulants. Iron coagulants include ferric sulphate, ferrous sulphate, ferric chloride, ferric chloride sulphate, polyferric sulphate, and ferric salts with organic polymers. Other compounds, such as hydrated lime and magnesium carbonate, are also utilized as coagulants.

Aluminum and iron coagulants' efficacy is determined by their capacity to generate multicharged polynuclear complexes with improved adsorption properties. When aluminum and ferric coagulants are mixed with water, they dissociate very instantly, forming hydrated reaction products. Which hydrolysis species is efficient for treatment is determined by rapid and slow mixing, pH, and coagulant dosage (Kurniawan et al., 2020).

Iron-based coagulants were found to be more effective than aluminum-based coagulants in several investigations. According to Baghvand et al. (2010), the maximum turbidity removal efficacy over the applied turbidity range was between 82.9-99.0 % for alum (pH 6-7) and 92.9-99.4% for ferric chloride (pH 5-6) with the same dose 10-20 mg/L. Furthermore, according to Bakar and Halim (2013), COD removal efficiency was 64% and TSS removal efficiency was 91% using 200 mg/L ferric chloride (pH 6), while COD removal was 54% and TSS removal was 94% using 100 mg/L alum (pH 7). Zinc removal was 68% at the optimum dose of 1 mg/L ferric chloride (pH 6), compared to 44 percent at 4 mg/L alum (pH 7).

2.3.1.1 Aluminum sulphate

Aluminum is the vastest metallic element in the earth's crust, accounting for around 8.1% of all metallic elements. Aluminum sulphate (alum) is utilized as a coagulant for suspended solids in water treatment. It's made by combining sulfuric acid and bauxite ores to make it. The dry product has the approximate formula $Al_2(SO_4)_3 14H_2O$ with an aluminum content ranging from 7.4% to 9.5% (typically near to 9% as Al) by mass due to the evaporation of water (Bratby, 2016).

The pH of aluminum sulphate solutions is comparable to equimolar acetic acid solutions. Solubility, complexation, and polymerization of these aluminum compounds are influenced by ambient pH and the presence of appropriate ligands, as well as, to a lesser extent, temperature, and duration in solution.

2.3.1.2 Hydrolysis of aluminum

Metal coagulants go through a sequence of hydrolytic processes as they convert from the free aquo metal ion to the insoluble metal hydroxide precipitate. Al^{3+} has a main hydration shell composed of six octahedrally coordinated water molecules, for example, $Al(H_2O)_6^{3+}$. Water molecules in the primary hydration shell are polarized due to the high charge on the metal ion, resulting in the removal of protons based on the pH of the solution. According to Equation 2.2, water molecules in the primary hydration shell are deprotonated by replacing them with hydroxyl ions, resulting in a lower positive charge.

$$Al^{3+} \rightarrow Al(OH)^{2+} \rightarrow Al(OH)_2{}^{+} \rightarrow Al(OH)_3 \rightarrow Al(OH)_4{}^{-} \qquad (2.2)$$

This is an oversimplified method as Al hydrolysis products formed can be dimeric, trimeric, or polynuclear. Increased pH causes the equilibria to shift right since each step entails the removal of a proton. Aluminum hydroxide is poorly soluble in water and precipitates at intermediate pH levels. The soluble aluminate ion is generated when the pH rises, making the hydroxide significantly less soluble than aluminum hydroxide. (Gregory and Duan, 2001; Duan and Gregory, 2003; Sahu and Chaudhari, 2013; Bratby, 2016).

2.3.2 Organic/Natural coagulants

The growing necessity for eco-friendly and sustainable technologies has drawn researchers' attention to natural polyelectrolytes due to their biodegradability, natural flocculants, primarily polysaccharides, are regarded environmentally beneficial than inorganic and organic coagulants. Plant-derived polysaccharides can be utilised to replace organic flocculants at a lower cost. They're also abundant, stable, hydrophilic, and biodegradable, and they're made from renewable resources (Anastasakis et al., 2009).

Since ancient times, plant materials have been used as natural coagulants and flocculants to reduce turbidity in wastewaters (Patale and Pandya, 2012). Since the 16th century, *Strychnos potatorum* seeds were responsible for clarification of turbid Nile water (Jahn, 2001). Natural-based coagulants have been utilised in nations such as India and Myanmar 4000 years ago, with the use of nirmali (*Strychnos potatorum*), while almonds and soaked beans have been used in Sudan and Egypt since the 16th century as water purifying agents to improve water quality (Mohd-Salleh et al., 2019). However, natural coagulants are not widely incorporated in actual water and wastewater treatment plants due to a lack of industrial trust in utilizing them in treatment process (Ang and Mohammad, 2020).

In recent years, numerous plant materials have been described as a source of natural coagulants, but the most studied was the seed of *Moringa oleifera* which has been reported by numerous authors (Effendi et al., 2015; Hendrawati et al., 2016; Zaman, 2018; Chum, 2020). Dehghani and Alizadeh (2016) investigated the feasibility of using a plant extract, specifically *M. Oleifera*, to treat wastewater from an oil refinery. *M. Oleifera* removed approximately 38.60% of COD, 63.70% of turbidity, and 62.05% of total suspended solids (TSS) at 70 mg/L under optimal conditions (constant pH: 6, constant temperature: 20 C, and mixing speed: 300 rpm). In particular, Heredia and Martín (2009) reported that *M. Oleifera* can remove up to 80% of sodium lauryl sulphate (an anionic surfactant) via coagulation and flocculation.

2.3.2.1 Abelmoschus Esculentus

Okra is botanically known as *Abelmoschus esculentus*, formerly known as *Hibiscus esculentus*, and is a member of the Malvaceae family. It is an economical vegetable crop grown in tropical and subtropical climates around the world. Okra is a multifunctional crop due to the numerous applications of its fresh leaves, buds, flowers, pods, stems, and seeds. Fresh okra pod extracts are ubiquitous in nature, affordable, and harmless biopolymers, making okra an appealing material for industrial applications. When utilised as a plasma substitute or blood volume expander, okra mucilage has medical potential. Okra mucilage binds cholesterol and bile acid conveying toxins that the liver discharges into it (Gemede et al., 2015).

Furthermore, okra mucilage, as well as ethanolic and aqueous extracts of the pods, have been shown to reduce blood glucose levels in alloxan-induced diabetic models (Elkhalifa et al., 2021). Subsequently, it was discovered that other sections of okra, including as the blossoms, leaf, seed, and pods, also exhibited significant antioxidant efficacy (Liao et al., 2012). Furthermore, gold nanoparticles produced from okra pulp had significant microbicidal activity against *Bacillus cereus, Bacillus subtilis, E. coli, P. aeruginosa,* and *M. luteus* (de Carvalho et al., 2011). As a result, okra characteristics have become a key source in biological and pharmacological applications.

2.3.2.2 Mucilage of Abelmoschus Esculentus

Chemically, mucilage resembles gums and pectins but differ in their physical properties. Gums swell in water to produce sticky colloidal dispersions, while pectins gelatinize and mucilage forms slippery aqueous colloidal dispersions. The polysaccharides found in okra extracts are mostly pectins (Ghori et al., 2014). Okra gum is a natural polysaccharide that is comprised of D-galactose, L-rhamnose, and L-galacturonic acid (Freitas et al., 2015). At 20°C, a 0.5 percent (w/v) dispersion of okra mucilage had a specific gravity of 0.9975 gm/ml and a pH of 4.0 (Sangwan et al., 2011).

According to Das et al. (2021), mucilage is a plant-derived mixture of monosaccharides with viscous colloidal dispersion qualities in water, containing arabinose, glucose, galactose, mannose, rhamnose, xylose, and uronic acid. Mucilage has been postulated to have two distinct water-soluble fractions: one is a pectin with Ca^{2+} induced gel forming characteristics, and the other is a mucilage with no such properties (Sepúlveda et al., 2007). Natural polysaccharides that are water soluble can destabilise colloidal suspensions, flocculating tiny particles and lowering turbulent drag (Freitas et al., 2015).

Research by Tosif et al. (2021) stated that mucilage can be found in Aloe vera, *Salvia hispanica* seeds, *Cordia dichotoma*, *Basella alba*, *Plantago psyllium*, *Cyamopsis tetragonoloba*, Cactaceae, *Abelmoschus esculentus*, *Trigonella foenum-graecum*, *Moringa Oleifera*, and *Linum usitatissimum*. Okra mucilage had previously demonstrated promising results in the treatment of landfill leachate (Al-Samawi and Hama, 2012), biologically treated effluent (Anastasakis et al., 2009), textile wastewater (Freitas et al., 2015), tannery effluent (Agarwal et al., 2003) and sewage effluent (Agarwal et al., 2001). Based on this fact, okra, which is known to possess mucilage properties was chosen for this study.

A research based on guar gum (GG) and *Ipomoea dasysperma* (ID) seed gums as natural coagulant for the decolorization of different classes of textile dye solutions such as Direct Orange (DO), Acid Sandolan Red (ASR), and Reactive Procion Brillaint Blue RS (PBB) have been performed by Sanghi et al. (2006). According to the authors, the colour removal rates in inclining order for all the dyes with an added 5-10 mg/L dosage of GG and ID in conjunction with PAC (1 mg/L) were PAC+GG: ASR>DO>PBB and PAC+ID: ASR>PBB>DO respectively. GG was found to be more efficient than ID in decolorizing DO and ASR solutions. Hence, in conjunction with a limited dose of PAC, GG and ID seed gums were discovered to be effective coagulant aids for acid as well as procion dye.

Another research using *Alyssum* mucilage as a natural coagulant in oily-saline wastewater treatment was done by Fard et al. (2021). 40.5 mg/L of *Alyssum* mucilage can remove 84.63% COD, 96.25% turbidity and 99% surfactant respectively. Furthermore, Nougbodé et al. (2013) claimed that when optimal values (1 mL to 10 mL) were utilised to treat severely turbid surface water, the removal effectiveness ranged from 89% to 93% for turbidity and suspended particles and from 4% to 15% for noticeable colour in water. With the addition of lime and *Opuntia dillenii* mucilage, the removal effectiveness of turbidity and suspended solids increased to more than 95%, while the removal efficiency of colours increased to between 67% and 94% percent.

Increasingly reference to experimental data involving the use of okra as coagulant for water treatment has been found in literature. Starch or polysaccharide and protein contained in the mucilage are the closest explanation for okra extract's coagulation properties. There is a potential that some other coagulation active components in the mucilage also contributes to the characteristics of okra as a coagulant.

2.4 COLLOIDS AND INTERFACES

2.4.1 Characteristic of colloid

Colloidal and particle matter in domestic wastewater have a significant impact on wastewater treatment plant performance. Colloids are particles less than 1 μm in diameter that, due to their size and density, do not settle easily in water. They are made up of a variety of materials, including mineral or rock fragments, mineral precipitates, organic macromolecules, and microorganisms (e.g., protozoa, fungi, bacteria, viruses) (Ochiai et al., 2006). These colloids are mixtures with particles that are invisible to the human eye, cannot be filtered out, but can be retained within a semi-permeable membrane. The Tyndall effect occurs when the particles in a colloid are large enough to scatter light.

There are mainly two types of colloids which are hydrophilic and hydrophobic colloids. The term hydro-denotes the water phase in which the colloids present, while -phobic and -philic represent the colloids' affinity for the liquid phase. Hydrophobic refers to water repellences, whereas hydrophilic refers to a strong affinity for water molecules in the colloid's surface layers (Bratby, 2016). The characteristics of colloid dispersion are governed by particle size distribution, particle shape, and net particle interaction forces (Liu et al., 2002). Colloids are also significant in the adsorption and transfer of heavy metals and organic micropollutants.

2.4.2 Stability

The particles that stay suspended in the fluid at equilibrium characterise the stability of a colloidal system. Stability can be classified as kinetic, thermodynamic, electrostatic, or steric (depletion). Kinetic stability is correlated with the presence of an energy barrier that precludes particle coagulation. The coagulated phase has a higher free energy than the scattered phase, indicating thermodynamic stability. Electrostatic repulsion forces cause electrostatic stabilisation. Steric stability is accomplished by adding macromolecules to the system (Matusiak and Grządka, 2017).

The interaction energy of the particles in the solution characterises stability in the Derjaguin, Landau, Verwey, and Overbeek (DLVO) theory of colloidal system stability. The force-distance relationship between particles underpins colloidal stability and particle-particle interactions. The particles' interactions are classified as van der Waals attraction forces and repulsive electric double layer forces (Matusiak and Grządka, 2017). The permanent dipole-induced dipole interaction, permanent dipole-permanent dipole interaction, and induced dipole-induced dipole interaction cause Van der Waals attraction between two isolated molecules (Liu et al., 2002).

Owing thermal agitation, molecules and ions in solution move continually. Brownian motion is caused by colloidal particles being bombarded at random by molecules. Brownian motion causes particles in a fluid to vibrate consistently. This prevents particles from settling, resulting in the stability of colloidal solutions (Bratby, 2016). Surface charge properties, dispersing medium, and subsequent collision efficacy between particles/droplets/bubbles had a massive impact on the stability and rheology of particulate suspensions/emulsions (Hunter, 2001).

2.4.2.1 Surface charge and zeta potential

The surface charge influences the distribution of adjacent ions in the liquid. As illustrated in Figure 2.1, ions with opposing charges (counterions) attract to the surface, whereas ions with identical charges (co-ions) repel. This, combined with the mixing propensity of thermal motion and mutual ionic repulsion or attraction, results in the formation of an electrical double layer consisted of the charged surface and a neutralization of excess counterions over co-ions dispersed manner in the surrounding solution (Bratby, 2016).

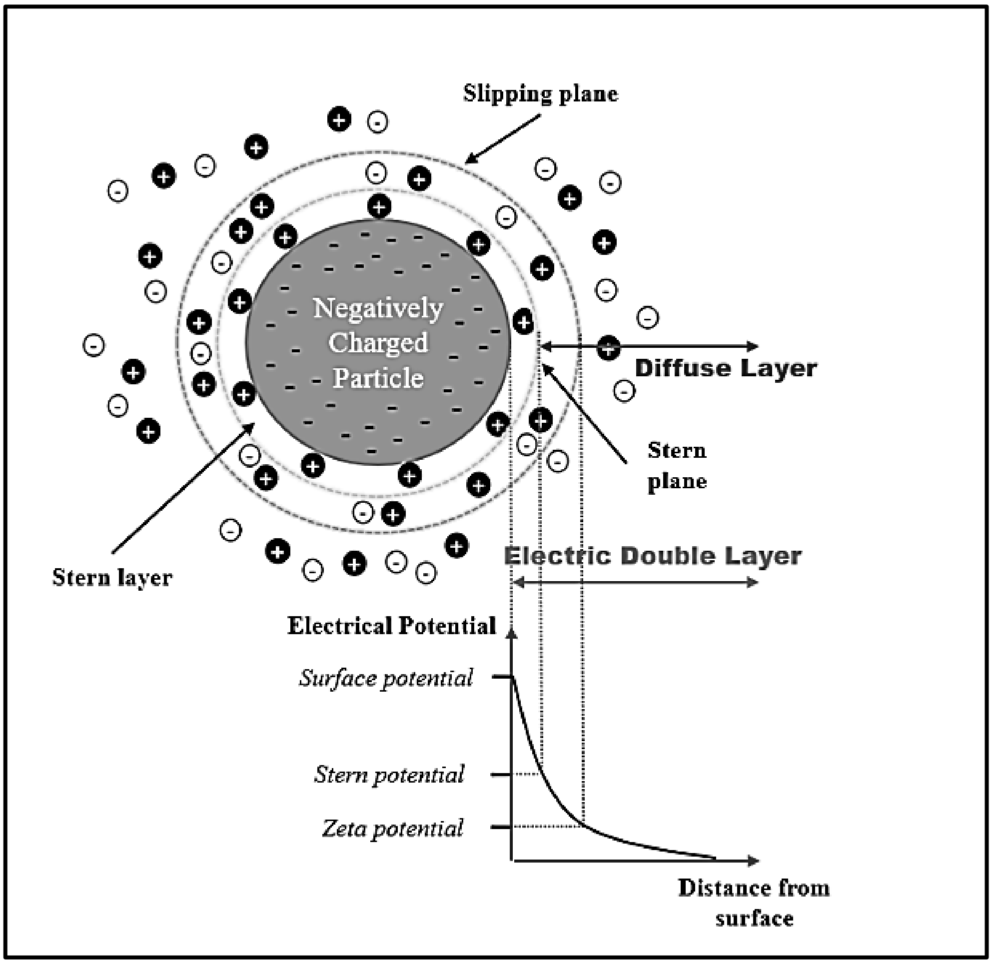

Figure 2.1: Conceptual representation of the electrical double layer (Sheng et al., 2022).

The structure of the double layer is composed mainly of the Stern layer and the Gouy layer. Partially desolvated ions adsorbed by electrostatic attraction or specifically adsorbed ions may be present within the Stern layer, forming the inner Helmholtz plane. The outer Helmholtz plane is comprised of a first layer of hydrated counterions that are likewise attracted by electrostatic attraction. The Stern layer of uniform thickness to the hydrated radius ion without charge isolates the centre of the closest counterions from the surface charge (Liu et al., 2002).

The residual counterions and co-ions are dispersed in the Gouy layer. A shear plane within the Gouy layer shows the threshold of fluid motion's ability to sweep counterions away from the surface. Ions within the shear plane move with the particle, whereas ions outside of it move freely and are significantly affected by fluid and thermal motion (Liu et al., 2002). The Gouy layer of counterions surrounding the particles spreads out over great distances at low ionic strength. Particles repel one other when Gouy layers overlap, and repulsion emerges at a wide separation. The diffuse layer is less extensive at high electrolyte concentrations, and particles must approach relatively close before repulsion is detectable.

When electric charges are electrostatically charged, the potential at the colloid surface drops to zero at the suspension's centre. The zeta potential is the potential at a specific location from the surface when the shear plane is formed. The degree of electrostatic or charge repulsion/attraction between particles is measured by zeta potential, which is one of the fundamental features known to determine stability. The stronger the potential, the higher the repulsive force and the greater the stability of the colloidal particle (Ghernaout et al., 2015).

Typically, the boundary between stable and unstable suspensions is fixed at +30 or -30 mV. Particles with zeta potentials ranging from -30 mV to +30 mV are expected to have sufficient repulsive force to offer higher physical colloidal stability (Zetasizer Nano User Manual, 2013). Unfortunately, a low zeta potential value may lead to particle aggregation and flocculation by virtue of the van der Waals attractive forces exerting on them. These can contribute to physical instabilities. Even if the particles are distributed, if their density is greater than the dispersion, they will eventually sediment, forming a close packed bed.

The most important factor regulating zeta potential is pH. A zeta potential value is practically meaningless without the given pH. Not only are zeta potential measurements essential for confirming double layer theories, but they are also crucial for colloid stability, ion adsorption studies, and particle surface characterisation (Zetasizer Nano User Manual, 2013).

2.4.2.2 Point of zero charge (PZS) and isoelectric point (IEP)

The point of zero charge (PZC) and isoelectric point (IEP) have developed specifications for identifying metal oxides in aqueous dispersions, characterizing ion adsorption (surface excess), coagulation stability, dispersion rheological characteristics, and so on (Kosmulski, 2016). Particles in water produce a surface charge that attracts counterions, resulting in the formation of the electrical double layer.

The point of zero charge (PZC) describes the solution conditions (pH value) at which the surface density of positive charges (cation) equals that of negative charges (anions) (Rey et al., 2011). The isoelectric point (IEP) is the pH at which zeta potential of a molecule or surface is equal to zero, $\xi = 0$. The molecule or surface must be amphoteric, which means it must have both acidic and basic functional groups in order to obtain a sharp isoelectric point. If the specific sorption of ions and counterion dissociation are ignored, PCS corresponds with IEP (Gulicovski et al., 2008).

PZC is correlated to the charge on the particle's surface and is strongly reliant on the material's pH; it influences a broad range of colloidal material characteristics, including stability, electrolyte interaction, suspension rheology, and ion exchange capacity (Khan et al., 2007). The colloidal particle ionises carboxylic and amine groups in the solution by supplying a favourable mixture of H^+ the and OH^-. Since both ionised groups neutralise each other, the colloid is deemed neutral and has reached the isoelectric point. The zwitter ion is the colloid's related ion.

When pH is adjusted by adding a base, the additional OH- neutralises the acid extremity of the zwitter ion (i.e., the NH_3^+); the zwitter ion disappears, and the entire colloidal entity becomes negatively charged. If the pH is reduced by the addition of H^+ ions, the inverse condition is met. The influx of H^+ ions neutralise the zwitter ion's base extremity (the COO^-); the zwitter ion disappears, and the entire colloidal entity becomes positively charged. Based on this interpretation, a hydrophilic colloid can achieve a primary charge of either negative or positive as a function of pH, as shown in Equation 2.3 and 2.4 (Ghernaout et al., 2015).

$$MOH_{(surf)} + H^+{}_{(aq)} \leftrightarrow MOH_2{}^+{}_{(surf)} \tag{2.3}$$

$$MOH_{(surf)} + OH^- \leftrightarrow MO^-{}_{(surf)} + H_2O_{(l)} \tag{2.4}$$

Electrophoresis is an electric field-induced mobility of charged species in a supporting medium (a liquid or a hydrophilic gel). It is utilized to segregate charged species from tiny inorganic or organic ions to charged biopolymers including DNA or proteins, as well as whole cells, chromosomes, or microbes (Gas, 2005). When an electric current is applied across an electrolyte, suspended charged particles are drawn to the oppositely charged electrode. The particles' viscous forces attempt to impede this motion. When these two opposing forces attain equilibrium, the particles move at a steady velocity (Zetasizer Nano User Manual, 2013).

2.4.3 Destabilization of colloids

Coagulation, aggregation, and flocculation all contribute to colloidal system instability. The charge of the scattered particles is neutralized, resulting in colloidal particle aggregation. Van der Waals forces or other intermolecular forces hold the particles together. Several researchers have examined the mechanism of colloidal instability in suspension (Ghernaout et al., 2015; Amran et al., 2018; Maćczak et al., 2020). According to Ghernaout et al. (2020), there are four coagulation mechanisms: double layer compression, charge neutralization, adsorption and interparticle bridging, and precipitate enmeshment (sweep flocculation).

Coagulation via electrical charge neutralization can be performed by either reducing the zeta potential of the colloids or flooding the medium with abundance oppositely charged ions (Nemerow, 2007). Owing to the elevated salt concentration, ionic strength, or zeta potential, the potential energy barrier decreases, and particle interaction becomes more likely to take place. When particles collide, the barrier progressively diminishes, and particles can adhere. The particles are believed to be entirely destabilized and to coagulate swiftly (Liu et al., 2002).

Coagulants are capable to disrupt negatively charged colloids and dissolved materials in aqueous solution. Metal salts and polymers are the two most commonly used coagulants. Metal salts destabilize negatively charged colloids by neutralizing their charge with cationic hydrolysis products and incorporating contaminants into an amorphous hydroxide precipitate ('sweep flocculation') (Duan and Gregory, 2003). Polymers can be used to generate particle aggregation and relying on the adsorption of the polymer on the particle surface, they can function either by charge neutralization or polymer bridging (Maćczak et al., 2020).

2.5 COAGULATION AND FLOCCULATION MECHANISM

In a domestic wastewater treatment plant, raw water is treated using chemical and physical processes such as coagulation-flocculation, sedimentation, and filtration. Coagulation-flocculation, being the primary treatment stage, is critical in controlling