WATER TREATMENT USING COAGULANT FROM MORINGA OLIEFERA SEEDS

By

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LIST OF ABBREVIATIONS

SEM	Scanning Electron Microscopy
FTIR	Fourier Transform Infrared
MOSC	Moringa Oliefera Seed Coagulant
NTU	Nephelometric Turbidity Units

RAWATAN AIR MENGGUNAKAN KOAGULAN DARI BENIH MORINGA OLIEFERA

ABSTRAK

Masalah air yang telah menjadi isu global di seluruh dunia. Pada masa kini, banyak sumber-sumber air yang telah tercemar dengan bahan kimia berbahaya. Oleh itu, rawatan air sisa diperlukan. Salah satu proses dalam rawatan air adalah koagulasi dan flokulasi. Secara amnya, aluminium sulfat digunakan sebagai koagulan dalam rawatan sisa air tetapi bahan ini dikatakan akan memberi beberapa kesan sampingan kepada manusia. Untuk menggantikan penggunaannya koagulan semula jadi amat diperlukan. Koagulan semula jadi datang daripada tumbuhan seperti Moringa Oliefera. Serbuk Moringa Oliefera yang telah diekstrak akan digunakan sebagai koagulan semula jadi. Serbuk ini akan dicirikan menggunakan SEM dan FTIR manakala serbuk selebihnya akan digunakan sebagai koagulan semula jadi. Untuk mengetahui keupayaan Moringa Oliefera koagulan, eksperimen ujian balang telah dijalankan untuk mengetahui parameter yang mempengaruhi keberkesanan koagulan ini.

Dari ujikaji yang dijalankan, ia menunjukkan bahawa serbuk Moringa Oliefera yang diekstrak mengandungi hidrokarbon dengan gelang aromatik dan amina alifatik yang terdiri daripada sebatian nitro. Dalam uji kaji baling ujian, penyingkiran kekeruhan bahan koloid dalam air adalah lebih tinggi apabila kadar pencampuran dan masa pencampuran adalah 30 rpm selama 40 minit dengan 96.8% penyingkiran. Untuk kepekatan yang tinngi, penyingkiran adalah paling tinggi dengan dos optimum 50mg/L koagulan yang menghasilkan nilai 95.6% penyingkiran. Akhir sekali, pada kadar

kepekatan air yang berbeza, penyingkiran kepekatan adalah lebih tinggi bagi pH optimum 7 dan 8 dengan 93.8% dan 96.7% penyingkiran. Oleh itu penggunaan koagulan semula jadi ini boleh digunakan sebagai pengganti penggunaan koagulan konvensional dalam rawatan air.

ABSTRACT

Water related problem became global issue around the world. The usages are important in our daily life. Nowadays, many of the water sources were polluted with hazardous material. Therefore, the treatment of wastewater was needed. One of the processes in water treatment was coagulation and flocculation. Conventionally, aluminium sulphate was use as coagulant but it was said that would give some side effect. To replace it the use of natural coagulant was needed. Natural coagulant from plant like Moringa Oliefera was studied to know it composition and its ability to act as coagulant. The powder of Moringa Oliefera that has been extracted will be use as natural coagulant. This powder will be characterized using SEM and FTIR while the remaining powder will be use as natural coagulant. To know the ability of Moringa Oliefera coagulant, jar test experiment was conducted to know the parameter affecting this coagulant.

From the experiment, it shows that extractes Moringa Oliefera powder contain hydrocarbon with aromatic ring and aliphatic amines that consist of nitro compound. During jar test experiment, the turbidity removal of colloidal material in the water was higher when the mixing rate and time of mixing was 30 rpm for 40 minute with 96.8% removal. While at higher turbidity, the removal was highest with optimum dosage of 50mg/L of coagulant with 95.6% removal. Lastly, at different range of water turbidity the turbidity removal were higher for optimum pH 7 and 8 with 93.8% and 96.7% removal. Therefore the use of this natural coagulant can be use as the replacement of conventional coagulant use in water treatment.

CHAPTER ONE

INTRODUCTION

1.1 Study Background

Water treatment was important process that needed in treating water sources. Water treatment was needed to neutralize water sources either from industrial discharge or groundwater sources. Effluent come out from the industries might contain some hazardous material or chemical that can affect water source or environment if it not been removed before being discharge out. Sometimes, groundwater sources also contaminate with some harmful microorganism and turbidity (American Water Work Association, 2003). Therefore effective water treatment process was needed to make sure harmful material did not contaminate water resources.

Over a century, water plays biggest part in human life. From age ago dependency of human toward water can be shown. Most of human civilization also started near the water source mostly near the river. Water is important not only as transportation medium, but it also been use as drinking and food source. Beside human, others living organisms also need water as their food sources and habitat.

In water treatment there are many process that can be use to remove unwanted material from water. This unwanted material either dissolves material or undissolve material need to remove from water. One of the processes is using a coagulant. The purpose of using coagulant in water treatment is to remove colloidal and small particle that settle down slowly in sedimentation unit. Besides coagulant, to improve the process of the flocculation coagulant agent also been add to aid this process. By using coagulant agent, these small particles are essentially coated with chemically sticky layer that allow them to agglomerate together and settle down in a certain period of times (Chris Binnie et al, 2002).

For conventional water treatment process, the two main steps in this process were flocculation and coagulation step and disinfection step. Besides this two steps there were other combine process also needed such as sedimentation and filtration. These two other units were helped in remove the sludge and filter the remaining sludge. All of this were combined together to treat water based on quality of water sources such as turbidity, the microorganism presented and others like the cost of treatment and the availability of the chemical used (Bodlund, 2013).

In coagulation unit, the most common coagulant that been use nowadays are aluminum sulfate and iron salt. These two kinds of coagulant also are called as alkaline agent. The use of alkaline agent might be useful to help these salts to consume alkalinity of water. This means that the coagulant use have a positive charge while the colloidal particles in water contain negative charge. By coagulation, flocs will formed and settled down under gravity caused of charge neutralization (Pritchard, Craven, Mkandawire, Edmondson, & O'Neill, 2010).

The use of this kind of chemical coagulant was said would give harmed toward human if they drink from water treated with this coagulant. The flocs which settle down was said contained with aluminum if aluminum salts was used as coagulant and caused aluminum accumulation in environment. While some others coagulant also said could induce and caused Alzhemier's disease and carcinogenic effects (Muthuraman & Sasikala, 2014). To prevent something like these happen the use of organic coagulant should be use.

Organic coagulant can be extract or produce from microorganism, animal or plant (Narasiah, 1998). It was said that polymeric coagulant can be cationic, anionic and non-ionic while natural coagulant was referred as polyelectrolytes. Besides that, natural coagulants mostly contain protein or polysccaharides (Yin, 2010).

The one that been subjected to use in this study is moringa oleifera seed. This tropical plant was said that originated from Northern India. This tree also been said to have uses and have high contain of nutrition in it (Kumar, Rubha, Manivasagan, Babu, & Balaji, 2012). This plant was widely use as food and medicinal sources also caused of the nutrient contain in every part of this plant (Yin, 2010).

1.2 Problem Statement

Water source is important source for human life. In their everyday life always related to water. Nowadays some serious problems were emerged that related to water source which water pollution. Pollution of water has been serious problem for entire world. Many water sources have been polluted caused of human hand.

The discharge from industries always channeled straight to water source without proper treatment. Beside that although the treatment has been done it also can have some hazardous matter in the discharge. This is because some use in the water treatment might use the usage of chemical substance. This also can cause the pollution. Because of that more organic based treatment were needed to avoid this problem. One of the treatments of water is coagulant and flocculation process.

This process is using aluminium salts as a coagulant to bind colloidal matters in the water to flocs together and settle down to the bottom of the tanks. The use of this kind of compound was said that would cause some effect for human health. To replace the use of this compound the study using natural source of compound are needed. Therefore, some study about this natural compound must be conducted to know the effectiveness of natural coagulant for water treatment to replace the conventional compound use nowaday.

1.3 Research Objectives

The experiment objectives are:

i. To identify the characteristic of Moringa Oliefera powder after extraction

ii. To measure turbidity removal of $CaCO_3$ using Moringa Oleifera coagulant

iii. To study the parameters that affecting Moringa Oliefera coagulant

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

The quality of water nowadays has become a concern topic throughout the worlds. Water source mainly river stream has been a placed from the industries to discharge all their waste. Some industries might treat their discharge first before been release to water source but some industries also might not treat their discharge. Some of this treated discharge also sometimes may contain slightly higher harmful material in it even been treated before. Although the harmful material is slightly but if there too much of the discharge, this material will accumulated in the water and then can cause water pollution. Besides that the application of conventional water treatment also uses some chemical in their process. Some of this chemical might leave some trace in water discharge after been treated. Part of this process is coagulation. For coagulation, aluminium substance was used in the process but this chemical also said can give harm to human. Therefore there are needs of replacement of this chemical with something natural from plant or other organism that can act same with this chemical but not harmful as that chemical.

2.2 Coagulant

Coagulant was use for the removal of suspended solid. It said that ability of coagulant to coagulate water is related to its charge and it size of synthetic polymer. It is same with natural coagulant where the protein contain in organic coagulant will be one of the factors that help in coagulating activity in water treatment.

By coagulation, the nonsettleable solid in water will floc together to becoming a large and heavy solids. Coagulant adding and mixing into water caused physical-chemical reaction between coagulant agents with the particle in water. These nonsettleable solids did not settle down because of two reasons:

1. Particle size
2. Natural forces between particles

Particle size can be divided into three which are suspended solids, colloidal solids and dissolved solids. Suspended solid was held in suspension by natural action of flowing water because of its small size and less dense. Colloidal solids were very fine silts, bacteria, color-causing particles and viruses. Colloidal solids cannot be seen by naked eyes but its effect can be seen as color or turbidity in water. Lastly, for dissolved solids, any particles of organic or inorganic matter that dissolved in water. These solids cannot be seen by naked eyes and can cause public health or aesthetic problems.

Table 2.1: Natural settling rate for small particles

Particle Diameter, *mm*	Representative Particle	Time Required to Settle in 1-ft (0.3-m) Depth
		Settleable
10	Gravel	0.3 seconds
1	Coarse sand	3 seconds
0.1	Fine sand	38 seconds
0.01	Silt	33 minutes
		Considered Nonsettleable
0.001	Bacteria	55 hours
0.0001	Color	230 days
0.00001	Colloidal particles	6.3 years
0.000001	Colloidal particles	63-year minimum

Sources:Water Ttreatment 3rd edition (American Water Work Association, 2003)

In coagulant process there are two chemical used which were coagulant and coagulant aids. Most of coagulant contain positive charge ion to neutralize the negative charge of solids in water. Aluminum and iron were two type of chemical component used for coagulant because of the ion contains of these to chemical have more positive charge which was trivalent ions. While coagulant aid was a chemical added during coagulation process to improve coagulation, reduce amount of coagulant needs and build stronger and more settleable flocs.

2.3 Factor affecting coagulant

The effectiveness of coagulant and coagulant aids also related to the characteristics of water. All of these characteristics were needed to observe the turbidity removal of the coagulant. The characteristics of water and its affect can be seen on the table 2.2 below:

Table 2.2: Characteristics of water and their affect on coagulation process

CHARACTERISTIC	AFFECT
Temperature	- Lower temperature cause poor coagulation - More chemical or longer time needed
pH	- Coagulation process been disturbed at higher and lower value of pH - Optimal pH varies depends on coagulant use
Alkalinity	- Low alkalinity limit reaction of alum and ferric to forming complex aluminum or iron hydroxide result to poor coagulation
Turbidity	- Proper floc difficult to form at lower turbidity - Floc also difficult to accumulate
Color	- Color caused by organic compound react with chemical coagulant and result to poor coagulation.

Sources:Water Ttreatment 3rd edition (American Water Work Association, 2003)

The other factors affecting the process was the flash mixing and time of mixing. Mixing should be turbulent enough to make sure the coagulant dispersed throughout the chamber. For the timing, to develop heavy floc particles some time would be taken. Some would only take only a few minute while others can take up to 1 hour.

The last factor was zeta potential measurement of the particulate matter. Zeta potential was use to measure the excess number of electrons found on the surface of all particulate matter. The charge magnitude would be use to determine whether the particles in suspension would repelled each other and remained in suspension or agglomerate and settle. Repelling force was greater if zeta potential was more negative. Zeta potential was acceptable to the particle for range in size from ultramicroscopic to particle that visible to the naked eyes. Zeta potential should be zero to make sure the particle settle down properly. Zeta meter was used to measure zeta potential. General degree of coagulation can be shown in the table 2.3 below:

Table 2.3: degree of coagulation at varies zeta potential range

Average Zeta Potential	Degree of Coagulation
+3 to 0	Maximum
–1 to –4	Excellent
–5 to –10	Fair
–11 to –20	Poor
–21 to –30	Virtually none

Sources:Water Ttreatment 3rd edition (American Water Work Association, 2003).

2.4 Moringa Oleifera

Moringa oleifera plants belong to family *Moringaceae*, a single genus family of shrubs and trees. All part of the plant contains many nutrients and can be used widely in every field of study. For water purifying agent not all part of this plant can be use as a coagulant (Ndabigengesere, Subba Narasiah, & Talbot, 1995). This can be show from the table 2.4 below. From this table we can see that only dried pods of moringa oliefera that can be use as coagulant. The picture of dried pod moringa oliefera was shown in the Figure 2.1 below while in the Figure 2.2 was a picture of moringa oliefera seeds. Besides as natural coagulant, Moringa Oliefera tree also have many other use such as a medicine, fertilizer, and contain high nutrition (Kumar et al., 2012). This paper also said that each part of this plant can be use for different field. Each part of the plant contain active agent that can be used

Forms of Moringa	Coagulation activity
Green pods:	
- Whole pods	absent
- Seeds	absent
- Bark of green pods	absent
- Dried green pods	absent
Dried pods:	
- Whole pods	absent
- Non-shelled seeds	
- Unfiltered	present
- Filtered	present
- Residual solids	absent
- Shelled seeds	
- Unfiltered	present
- Filtered	present
- Residual solids	absent
Bark of pods	absent
Bark of seeds	absent

Table 2.4: coagulant activities of different moringa oliefera forms
*Sources from (*Ndabigengesere et al.,

Figure 2.1: Moringa oliefera dried pods

Figure 2.2: Moringa oliefera seed from its pods

There were many study have been done by using moringa oliefera as coagulant agent.

Most of this papers study about the effectiveness of this coagulant in water treatment. Characteristic like temperature, pH and turbidity of water always subjected as study (S. a Muyibi, Salleh, Salleh, & Gombak, 2009)(Pritchard et al., 2010). Besides that, this coagulant also been compared with others kind natural coagulant like tannins, cactus and nirmala seeds (Yin, 2010).

CHAPTER THREE

MATERIALS AND METHODS

3.1 Introduction

Experiment procedure will be explain in this chapter, all step will be explain from after Moringa Oliefera seed been extract to remove its oil content to how to prepare coagulant from residue of Moringa Oliefera extracted. Before that, some of extracted Moringa Oliefera also was characterized using SEM and FTIR. Experimental set-up to test the coagulant was done at Unit Operation Laboratory of School of Chemical Engineering.

3.2 Overall experiment

This experiments consist only 5 steps which shown in Figure 3.1. Firstly Moringa Oliefera pods been dried for certain period of times. After that obtain seeds from the pods and crush it until it become powder. Extract oil from this powder using Soxhlet method using heptane. Collect the supernatant of this process and characterize this component using FTIR and SEM to find it molecular weight and structure before been purify using phosphate buffer. After been purified moringa cake than been use as coagulant and been tested for jar test. Lastly, characterize again after coagulation using FTIR and SEM

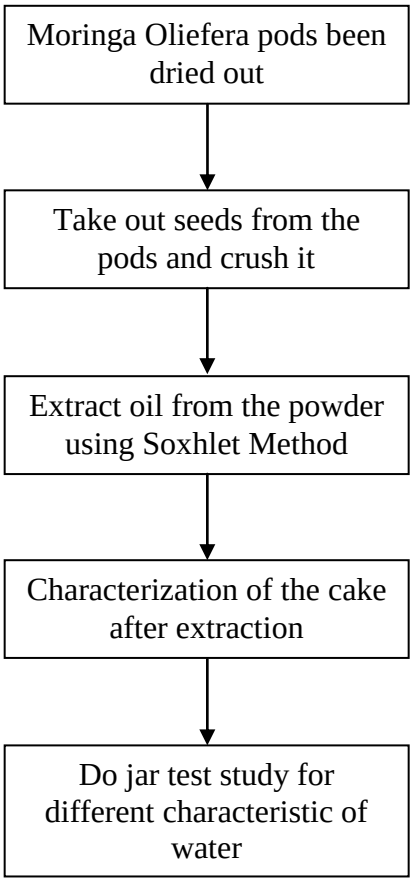


Figure 3.1: Flow diagram of overall process of the

3.3 Moringa Oleifera characteristic after extraction

Moringa Oleifera seed was collected from it shell pod and grounded into fine powder using mortar and pestle. After that, the powder of the Moringa Oleifera was extracted using Soxhlet method using heptane. After extraction, this solution then been filtered out to separate the oil and moringa oliefera cake. Some of the cake then was analyzed using FTIR and SEM to know their compound and molecular weight.

3.4 Preparation of Moringa Oliefera based Coagulant

Extracted Moringa Oliefera powder was weight on electronic balance. Weight about 4g of powder. Then put the powder into 500mL beaker. Adding 200mL of distilled water into the beaker before mixing it for a while until powder mix well with water. After mixing, leave the solution for 30 minute before filter it using filter paper. Fluid obtain from the filtration was use as coagulant. The mixture of Moringa Oliefera powder and distilled water gave 0.02g/mL concentration of Moringa Oliefera coagulant. A few milliliter of this coagulant then were use as a dosage to carry out the next experiment to study the parameters that affecting Moringa Oliefera coagulant in water treatment. Solution of Moringa Oliefera based coagulant can be show on the figure 3.2 below

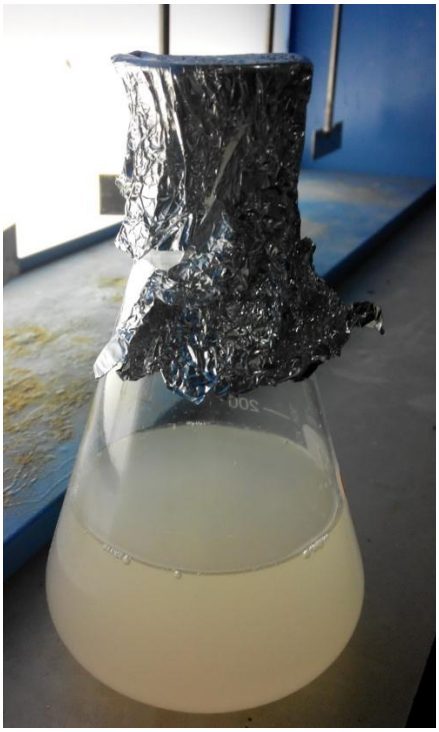


Figure 3.2: Moringa Oliefera based coagulant solution

3.5 Jar test study

There were few jar test study been conducted to study the turbidity removal using Moringa Oliefera coagulant at different parameters for treatment of water. The set up of experiment was as figure 3.2 below.

Figure 3.3: Set up of the experiment

3.5.1 Jar test studies on turbidity removal based on mixing time and mixing rate

Four different 1000mL beaker were filled up with water. Same amount of $CaCO_3$ been added into the beaker to have the same concentration for each beaker. The weight of $CaCO_3$ in the beaker is 1g. The solution was stirred for about one minute first before the initial turbidity of each beaker been taken. After record the initial turbidity, each beaker been stirred with same mixing rate which is 150 rpm for one minute for rapid stirring. Mixing rate then been slowed down to 20 rpm for slow stirring. After 10 minutes, take out one beaker from stirrer and leave it out for 45 minute to let colloidal

material in the solution sediment to the bottom of the beaker. Final turbidity reading will be taken after 40 minutes. This procedure been done repeatedly for each 10 minutes for each beaker. For different mixing rate, the procedure been repeated again with another mixing rate which is 30 rpm, 40 rpm and 50 rpm. Final turbidity reading been tabulated into table.

3.5.2 Jar test studies on turbidity removal based on $CaCO_3$ turbidity and coagulant dosage

Sample was prepared with different concentration of $CaCO_3$ in 1000mL beaker. Each beaker was fill with 0.01g, 0.02g, 0.04g, 0.06g and 0.08 g of $CaCO_3$. 1000mL of distilled water was added into the beaker. The solution then been stirred for about one minutes before turbidity reading been taken from each beaker. After that, 1mL or 20mg/L of coagulant solution been added into the beaker. The solution then been stirred at 150 rpm for one minute before the mixing rate been slowed down to optimum mixing rate and mixing time from previous experiment. Stirrer then been stopped and the solution was left out for 40 minutes to allow colloidal matters to settle down. Turbidity reading of each beaker then been taken. The process was repeated with different dosage of coagulant ranging from 30mg/L to 50 mg/L. Final turbidity reading then been tabulated into the table.

3.5.3 Jar test studies on turbidity removal based on pH

Sample was prepared with different concentration of $CaCO_3$ in 1000mL beaker. Each beaker was fill with 0.01g, 0.02g, 0.04g, 0.06g and 0.08 g of $CaCO_3$. 1000mL of distilled water was added into the beaker. Initial pH value been taken from each beaker before the value been adjusted with NaOH or HCl. The pH value of each solution been adjusted until pH 5. The solution then been stirred for about one minutes before turbidity reading been taken from each beaker. Suitable dosage of coagulant from previous experiment was used. Sample then been mixed at 150 rpm for one minute before been slow down to optimum mixing rate and mixing time. The solution then been leave out for 45 minute before turbidity reading been taken. The experiment was repeated again using different value of pH ranging from 6, 7, 8 and 9. Final turbidity reading then been tabulated into the table.

CHAPTER FOUR

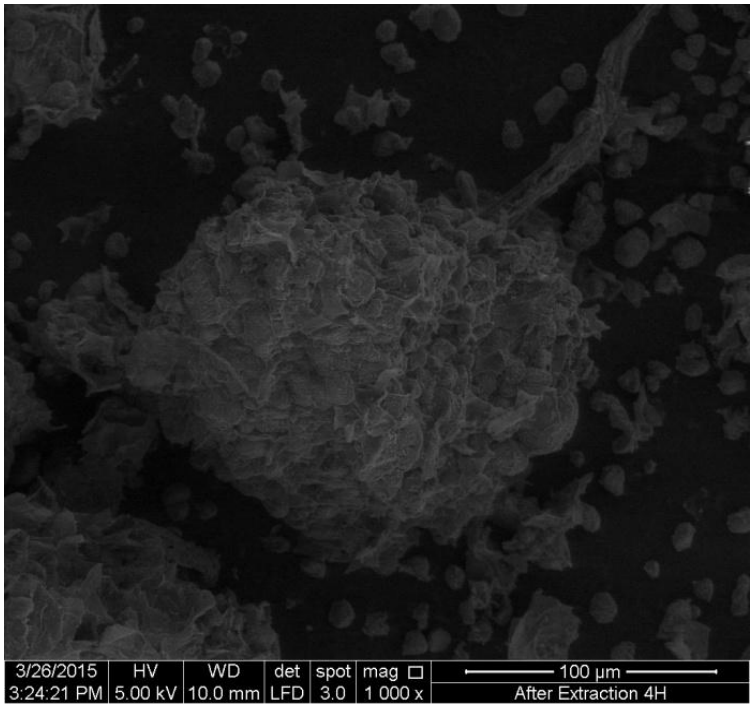
RESULTS AND DISCUSSION

4.1 Introduction

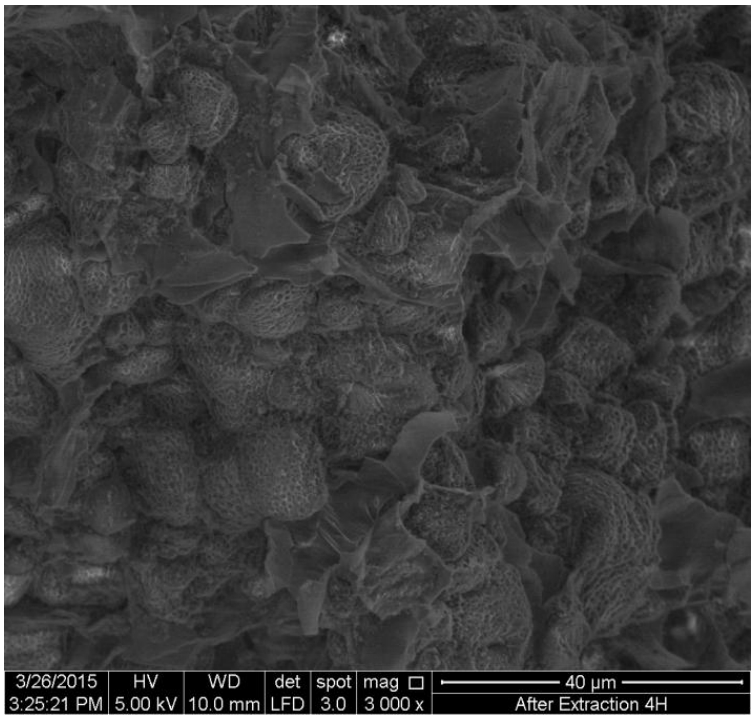
This chapter represents results and discussions on the parameter that been affecting the turbidity removal of water using Moringa Oliefera coagulant. In this discussion, each result will be discuss based on the parameter that been studied. The characterization of the surface morphology and the composition of components on Moringa Oliefera after extraction on its surface were obtained by using Scanning Electron Microscopy (SEM). The samples were analyzed by SEM with different magnifications in order to get a better view on the Moringa Oliefera extracted powder's surface. After that, the samples were analyzed using Fourier Transform Infra Red (FTIR) spectroscopy in order to determine the chemical composition on the surface.

4.2 Scanning Electron Microscopy

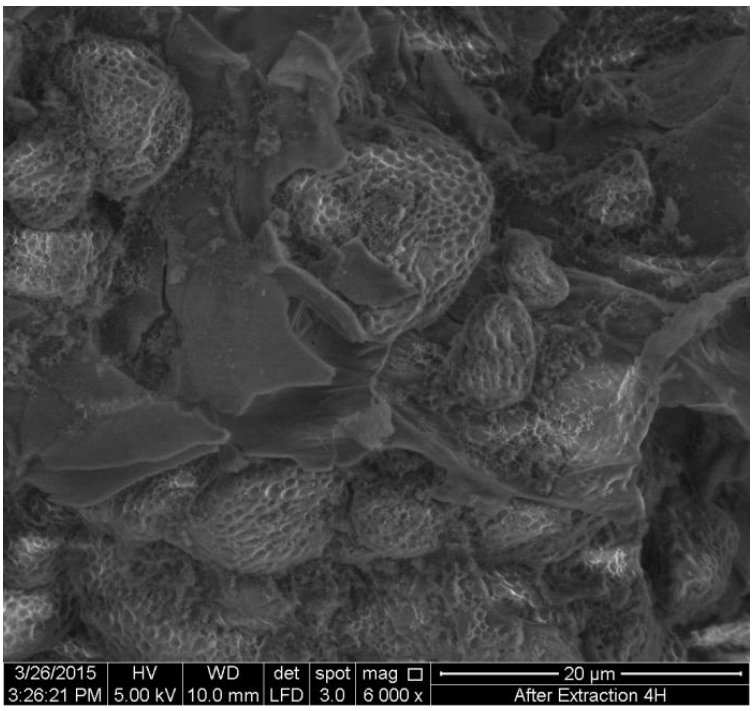
After Moringa Oliefera seed been extracted from its oil, the remaining powder of Moringa Oliefera powder will be characterized using SEM to know the morphology of the surface of the powder. The SEM image can be seen from figure 4.1 below:


3/26/2015 | HV | WD | det | spot | mag
3:24:21 PM | 5.00 kV | 10.0 mm | LFD | 3.0 | 1 000 x
100 µm
After Extraction 4H

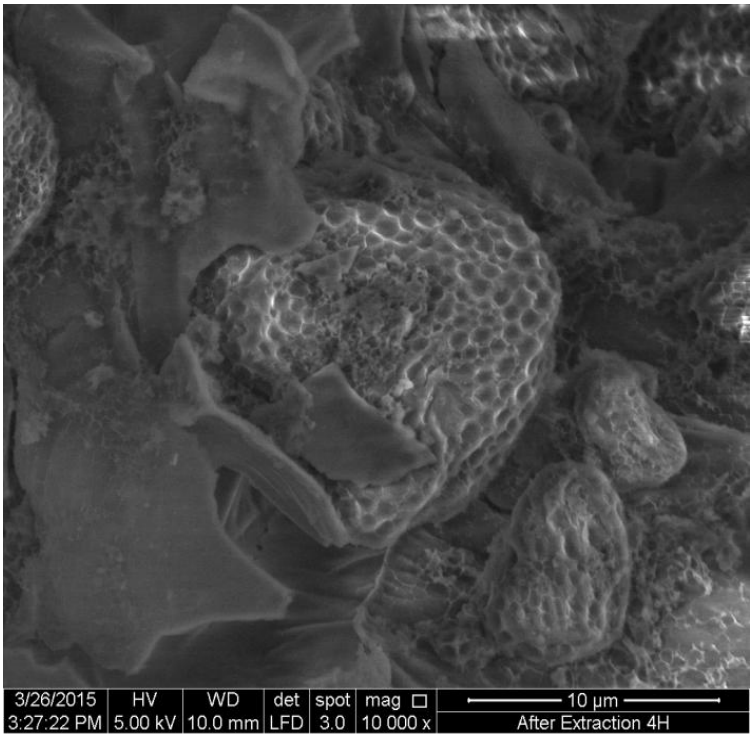
(a)


3/26/2015 | HV | WD | det | spot | mag
3:25:21 PM | 5.00 kV | 10.0 mm | LFD | 3.0 | 3 000 x
40 µm
After Extraction 4H

(b)



(c)



(d)

Figure 4.1 SEM image of Moringa Oliefera powder surface after extraction at magnification of (a) 1000x, (b) 3000x, (c) 6000x, and (d) 10000x.

From figure 4.1(a) we can see that the surface of each particle of Moringa Oliefera powder surface after been extracted to remove oil from Morina Oliefera powder. This surface contains many pores that distribute evenly on the surface of the particle. As in Figure 4.1 (d), the size of the pore is less than 10µm.

4.3 FTIR analysis

For FTIR analysis, extracted powder that been dried out and being added with KBr will be analysis with FTIR at wavenumber range from 400 to 4000. The result of the analysis can be shown as in figure 4.2 below.

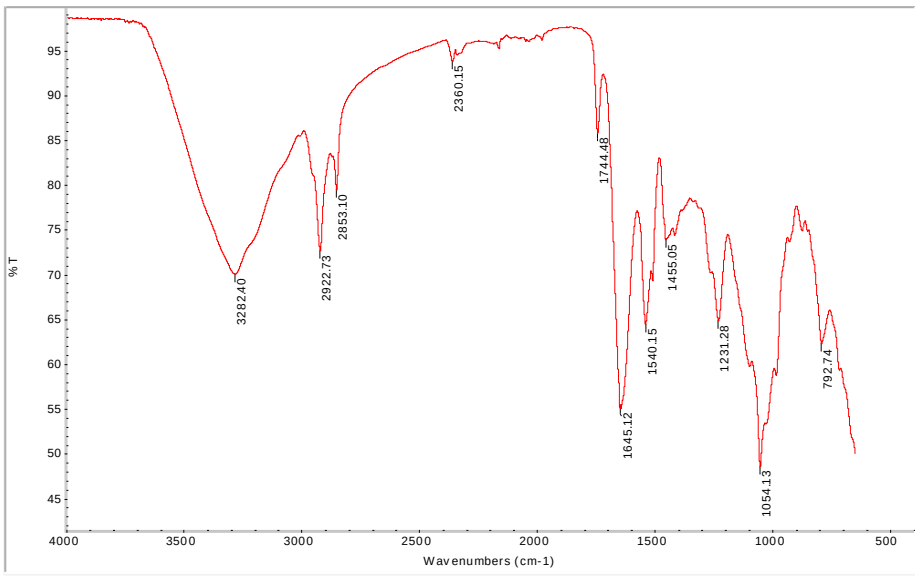


Figure 4.2 FTIR analysis graph of Moringa Oliefera powder after extraction.

From the graph of Figure 4.2, the strong peak of wavelength can be shown at wavelength 1645.2 cm^{-1} and 1054.13 cm^{-1}. This band show that Moringa Oliefera powder mainly contain of –C=C– stretch bond. This kind of bond was same as

functional group of alkenes. Beside that the other band at wavenumber 1054.13 cm^{-1}, the characteristic of the powder also contain of C–N stretch which is represent aliphatic amines functional group.

Then for the other band of graph peak, the wavenumber values are 3282.28 and 2927.09. This shows that this powder also contain of functional group alkynes terminal and C–H stretch bond that represent alkanes group. Lastly, the others lower peak at wavenumber 791.82 and 1538.81 show that the present of aromatic ring or 1°, 2° amines and nitro compound functional group. Therefore Moringa Oliefera consists of hydrocarbon with aromatic ring and aliphatic amines that consist of nitro compound.

4.4 Study on slow mixing rate and time of mixing

For jar test experiment, some of the procedure involve in this process was the mixing rate of the coagulant dosage with kaolin mixture and the time of mixing for suspended solid in the mixture to sediment at the bottom of the beaker. When adding Moringa Oliefera seed coagulant (MOSC) into the solution, the mixture will mix at rapid spin for around one minute before the mixture were spin at slow speed at certain time.

From the Muyibi & Evison, (1995) ,Muthuraman et al, (2013), Ng et al., (2012) and other papers the rapid mixing rate for jar test experiment are between 100 rpm to 200 rpm for 30 to 60 seconds while for slow mixing rate are at 20 rpm to 45 rpm for about 10 minutes to 30 minutes. After this two stage of stirring, the mixture than left for 40 minute to sediment. The mixture were stirred rapidly to make sure the coagulant was mix well with kaolin solution while slow stirring for the coagulant to floc together with