# **CHAPTER 1**

# **INTRODUCTION**

### **1.1 Project Background**

In the hydrocarbon production stage, the primary production is the stage when hydrocarbon is produced by the natural mechanism of reservoir. Examples of natural mechanism are natural gas cap expansion at the top reservoir or natural water drive displacing oil at bottom of reservoir. This natural mechanism energy will cause the hydrocarbon to be produce effectively without using any external energy. After producing hydrocarbon for some time, the pressure of the natural energy will decrease and the secondary recovery method would be used. The secondary recovery method relies on external energy to be supplied into the reservoir to increase the reservoir pressure so that hydrocarbon can be produce. The most common method of secondary recovery is water injection system [22].

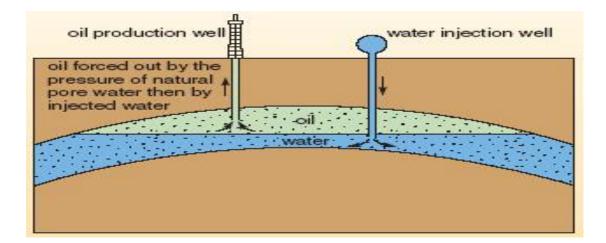


Figure 1: Water Injection System.

Source: http://imgbuddy.com/oil-reservoir.asp

In the water injection system, water flowing inside the injection well or the pipelines would create a turbulent flow because of the fluids flowing at a high velocity in large diameter tubes and when the fluids are relatively dense. This turbulent flow will produce a drag along the inner surface of the pipelines or injection wells thus will stimulate the pressure drop along it. This pressure drop will decrease the performance of water injection. The performance of water injection is crucial to the water flooding process in optimizing hydrocarbon recovery.

This pressure drop can be reduced by using several methods such as modifying the diameter of pipeline or increasing the pump capacity. But these methods are very expensive, therefore one of other alternatives to decrease the pressure drop is to use Drag Reducing Agent (DRA) by injecting it in the water. The DRA will decrease the pressure drop by reducing the turbulent flow inside the injection wells. Significant pressure drop can be accomplished without the need to increase the pumping pressure [5].

In current oil and gas practices there are three main types of DRA that have been widely used; surfactants, fibers and polymers [4]. The polymer DRA can be divided into two main parts, which is synthetic polymer and biopolymer. Synthetic polymer was produced by chemical at a higher cost while biopolymer is produced by natural resources or organic material such as plants. This project will use the biopolymer extracted from coconut husk as DRA.

### **1.2 Problem Statement**

Pressure drop in pipelines or injection wells due to turbulent flow is a major problem. The usual way to fix this problem is to increase the capacity of the pump. Increasing the capacity of the pump would increase pressure produced by the pump, thus the pressure drop will decrease. But it is constrained by increasing cost of the pump, increasing cost of energy consumption by the pump and the risk of the pressure being higher than the maximum allowable operating pressure of the pipeline (MOAP). Hence, one of other alternatives to reduce pressure drop is to use DRA to the system.

There are two types of polymer DRA, the synthetic polymer DRA is from human made chemical and the other is biopolymer DRA which is made by extracting from natural resources or organic materials. Research has shown that the synthetic polymers may have bad impacts on the environment [23]. Since, it is abundant in nature and being environmental friendly, biopolymer extracted from natural resources have the potential to be commercialized as DRA in oil and gas industries. So, this project will use the biopolymer extracted from natural resources as DRA.

As stated in Filipponi et al. (2012), the decreasing of particle size can have alterations in physical and chemical properties and make the material more soluble in water because of an increasing in surface to volume ratio. So this project will study and compare the performance between nano-scale and original-scale biopolymers extracted from coconut husk. The original scale is a product of the biopolymer synthesization, while the nano scale is a product from original scale grinded into nano particle size using planetary mill machine.

### 1.3 Objectives and Scope of Study

#### 1.3.1 Objectives

The project objectives are:

- i. To extract Carboxymethylcellulose (CMC) from coconut residues (CR).
- To compare the performance of the extracted CMC as DRA at different particle scale (nano-scale and original-scale) based on Drag Reduction Percentage (%) in a flowmeter experimental setup.

### 1.3.2 Scope of Study

The scope of study in this project is concentrating on the effects and performance of biopolymer extracted from CR as DRA at different particle size, the powder of CMC extracted from CR will be grinded into two different particle size which is original size and nano size. This research will be conducted as an experimental research and the result will be achieved by lab experiments. The study will consist of three parts, the first part is the DRA preparation. The preparation part is the extraction process of Carboxymethylcellulose (CMC) from the coconut residue (CR). The second part of this research, is to prepare the sample into two particle sizes. The original size is a product after extract CMC from CR, and the nano-scale size is a product after the original size being grinded to nano-scale using the Planetary Mill Machine. And the last part of the experiment, is to compare the performance of nano-scale particle to original-scale particle using the result of drag reduction percentage from flowmeter experimental setup.

### **CHAPTER 2**

### LITERATURE REVIEW

### 2.1 Drag

Drag was defined as a force that was exerted by a fluid flow on any obstacle in its path or felt by an object moving through a fluid [1]. Drag also can be defined as something that hinders motion, action or advancement [2]. And as stated in McAllister (2013), drag or frictional drop in pipelines is known as the result of the resistance encountered by a flowing fluid interaction with the pipeline walls.

There are two general types of flow, which is laminar and turbulent. The turbulent flow is the major problem in oil and gas industries as this flow will produce extra drag. The diagram in Figure 1 below shows a characteristic of turbulent flow in a pipeline that has three portions of the fluid flow which are laminar sub layer, buffer region and turbulent core [13].

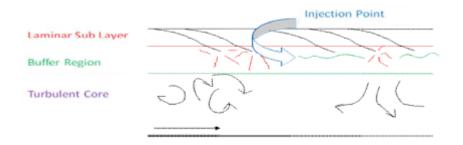


Figure 2: Segments of Turbulent Flow in Straight Pipeline [13].

As reported by Suan (2009), figure 1 shows the turbulent core in a pipe. It is the major area and it consist of most of the fluid flow in the pipelines. This is the zone of the swirlflow and random waves of turbulent flow. The nearest to the pipeline wall is the laminar sub layer. In this zone, the fluid flow alongside in sheets. The buffer region is between the laminar layer and the turbulent core. At the buffer region the flow will start to change into cyclone flow form and oscillate, it moving faster as the flow get closer to the turbulent core. After that, the flow will get unstable and break up as the fluid flow

into the turbulent region. This discharge of fluid flow into the turbulent region is called a "turbulent burst" which effects in a loss of energy. Drag reducing agent will interfere with the bursting process, therefore minimize the turbulent bursts. This effect will reduces frictional pressure loss resulting in fewer loss of energy.

This form of flow can be measured by using dimensionless Reynolds number [15]. The dimensionless Reynolds number can be calculated by equation below:

Reynolds Number (Re), Equation:

### Re=<u>ρud</u> μ

Where;  $\rho$  - fluid density,

- μ fluid viscosity,
- u -fluid velocity, and
- d -pipe diameter.

Fluid flow with Reynolds number less than 2000 is a laminar flow and fluid flow with Reynolds number more than 4000 is a turbulent flow. Then fluid flow with Reynolds number between 2000 and 4000 is a transitional flow [15].

### 2.2 Drag Reducing Agent (DRA)

Drag reducing agent is usually used as an additive in pipelines for purposes to reduce turbulent in the pipe. Commonly used in petroleum industries pipelines, the DRA can increase the pipeline performance by decreasing the turbulent and thus letting a more efficient flow of oil. As published by Toms (1948), the injection of slight quantities of high molecular weight long-chain polymers addicted to a single phase fluid flow can cause big reductions in the frictional pressure resistance at the wall.

Drag reduction in turbulent flow can be achieved by several types of additives for example surfactants, fibers, and polymers. This drag reducing additives can dramatically affect the turbulent structures of the flowing fluid, increase the flow rate and decrease the energy consumption [4]. Drag reducing agent is the additive which can reduce drag in turbulent flow with a small amount of concentration, with contrast to the drag in turbulent flow of the pure solvent. These small amounts of concentrated interruptions commonly show insignificant effect in laminar flows [5].

Drag reduction percentage (DR %) can be calculated by the widely accepted general formula of:

$$DR(\%) = \frac{\left|\Delta P_f - \Delta P_{fDRA}\right|}{\Delta P_f} X \mid 100$$

Where  $\Delta P_f$ , is the pressure drop without DRA in the flowing fluid while  $\Delta P_{fDRA}$ , is the pressure drop with DRA in the flowing fluid in the tubing. This formula has been used widely to check the direct relationship between the presence of DRA and resulting pressure drop in most of the research papers done recently [6, 7].

#### 2.3 Polymers as Drag Reducing Agent (DRA)

Polymer are the most commonly considered and most working drag reducing agent systems [4]. Adding of a slight quantity of polymer to a turbulent flow can significantly decrease the quantity of drag a fluid generates in pipelines. Polymers are frequently used because of important aspects such as that they hold a high molecular weight, chain flexibility, and long linear chain structure. It is also important that polymers are very soluble in the fluid [17].

Table 1 [10] provides a list of the common polymer uses as DRA. Previous studies on polymer drag reduction indicate that the most polymers meeting parameters described above are able to reduce the drag in a turbulent flow.

| Water-soluble polymers        | Hydrocorbon soluble polymers  |
|-------------------------------|-------------------------------|
| Polyethylene oxide (PEO)      | Polyisobutylene (PIV)         |
| Polyacrylamide (PAM)          | Polystyrene (PS)              |
| Guar gum (GGM)                | Polymethylmethacrylate (PMMA) |
| Xanthan gum (XG)              | Polydimethylsiloxane (PDMS)   |
| Carboxymethyl cellulose (CMC) | Polycisisoprene (PCIP)        |

Table 1: Most Widely Used Drag Reducing Polymer agents [10].

### 2.4 Carboxymethylcellulose (CMC)

Carboxylmethylcellulose (CMC) is a water-soluble biopolymer achieved by mixing the carboxymethyl groups along the cellulose chain. CMC is usually synthesized by the alkali catalyzed reaction of cellulose with chloroacetic acid [11]. CMC also has many widespread of applications such as drag reduction, textiles, foods, papers and detergents, oil well drilling and drugs. CMC is a cellulose products shaped by reaction of chloroacetic acid and sodium hydroxide [12]. Cellulose was considered as biopolymer that can be found vastly in fruit. Therefore, the usage of CMC extracted from CR is environmentally friendly and sustainable.

### 2.5 Nano-scale Drag Reducing Agent

Nano-scale are termed by something having a measurement in nanometers [15]. Nano particles also were defined as an aggregate of atoms bonded together with a radius between 1 and 100nm which typically consist of 10-105 atoms [16]. There was a study of drawing out oil from coconut wastes shows better display to yield more oil when the particle size is decreased [17]. According to Filipponi et al. (2012), when the material were prepared in nano particles, it will have alterations in both physical and chemical properties such as more soluble in water and higher shear strength as compared to their bulk materials since they have an increased surface-to-volume ratio.

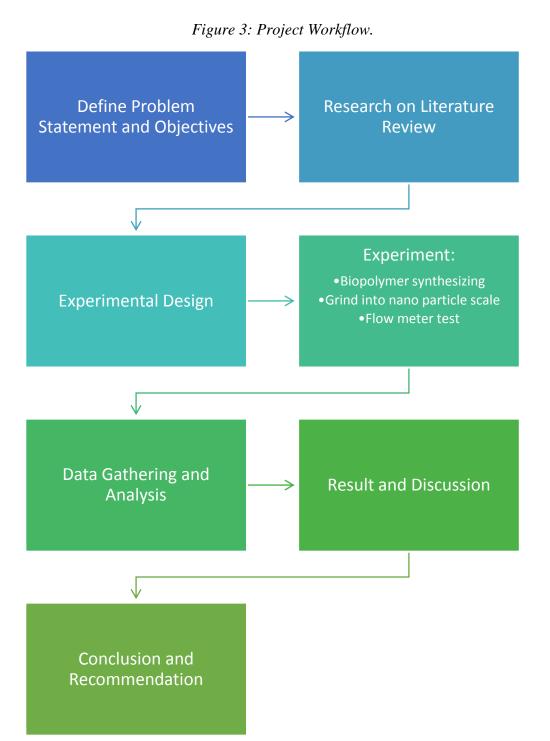
### 2.6 Coconut Residue

Coconut residues (CR) are coconut content remains after the extraction of coconut milk. Research done by Mirhosseini (2010) shows, cellulose content of the grated coconut is 72.6% which make it a good candidates for extracting CMC. Moreover, one of the top ten coconut producing countries in the world is Malaysia and have a total coconut production of 23,000MT in 2014 [24]. Because coconut is one of the fruits that is easy to acquire and have a high cellulose contents, CR is selected as a fruit to extract CMC in this research.

# **CHAPTER 3**

# METHODOLOGY

# **3.1 Project Methodology**



# **3.2 Project Experiments**

# 3.2.1 Biopolymer Synthesization

## **3.2.1.1 Materials and Chemical**

- Coconut Residue
- Sodium hydroxide pellets AR QREC S5158-1-1000
- Isopropanol AR QREC PR141-1-2500
- Ethanol 96% denatured AR QREC E7045-1-2500
- Methanol AR QREC M2097-1-2500
- Chloroacetic acid for synthesis MERCK 412
- Acetic acid AR QREC A1020-1-2500
- Distilled Water
- Tap Water

# **3.2.1.2** Tools and Apparatus

- Cole Parmer Mortar Grinder
- Oven
- Electronic Weight Scale
- Hotplate Magnetic Stirrer
- Beaker
- Conical Flask
- Measuring Cylinder
- Filter Funnel

# **3.2.1.3 Procedure of Cellulose Extraction**

i. Coconut residue (CR) was washed with water until cleaned and then put in an oven to dry the CR at 120°C for 24 hours.



Figure 4: Washed coconut residue was oven-dried for 24 hours.

ii. Then 40g of dried CR was cooked at 100°C with 1 L of 1M of NaOH (40g of NaOH diluted in 1 L of distilled water) in 2 L beaker under continuous stirring using a hotplate magnetic stirrer for one hour to eliminate undesired products in the CR. After a while, the mixture will turn from brown color to dark reddish color shown in figure 5.



Figure 5: Cooked CR with 1M of NaOH solution at 100 °C for 1 hour.

iii. The dark red slurry was filtered using filter funnel to separate the CR from the NaOH solution and the CR was washed with tap water until the dark reddish color turn nearly as it original color.



Figure 6: CR after being wash with water.

- iv. The obtained product was oven-dried at 60°C for 24 hours. This was to ensure that the powder is completely dried.
- v. After the oven-dried process the cellulose powder was obtained.

# **3.2.1.4 Procedure of CMC Synthesizing**

- i. Consist of two reactions: alkalization and carboxymethylation.
- Alkalization reaction: 100 mL of 60% NaOH mix with 900 ml of isopropanol solution was added with 30 g of cellulose powder under continuous stirring using magnetic stirrer for half an hour.

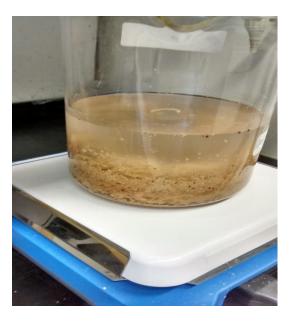


Figure 7: Solution of 100 ml of 60% NaOH, 15 g of cellulose powder and 900 ml of isopropanol.

iii. Carboxymethylation reaction: 36 g of chloroacetic acid was added into the mixture while continuous stirring for another half an hour.

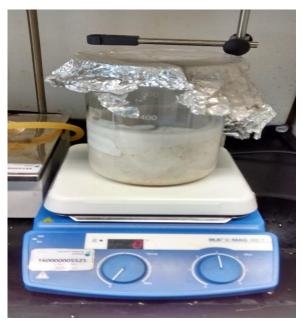
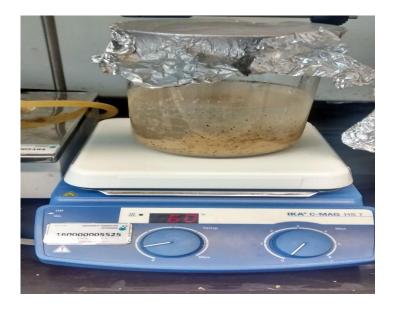


Figure 8: Solution of 100 ml of 60% NaOH, 15 g of cellulose powder, and 900 ml of isopropanol added with 36 g of choloroacetic acid.

iv. Then the solution was heated for 4 hours reaction time at 60°C reaction temperature. To avoid evaporation during the entire reaction process the beaker was covered with aluminum foil.



*Figure 9: The solution after heated for 4 hours at 60 °C.* 

- v. The mixture was filtered and suspended in 100 ml 70% v/v methanol for 24 hours.
- vi. The mixture then was neutralized using 100 ml glacial acetic acid.
- vii. Undesired products was removed by washing process with 300 ml of 70% ethanol and 300 mL of absolute methanol.
- viii. Finally the product was put through a drying process in an oven for 24 hours at  $60 \,^{\circ}\text{C}$ .
- ix. Carboxymethylcellulose (CMC) powder was obtained.



Figure 10: Washing process, left washed with ethanol and right washed with methanol.



Figure 11: CMC obtained.

### **3.2.2 CMC Grinding Process**

The next step after the production of CMC, is to grind the CMC into finer particle scale. This process is done by using Planetary Mill Test Setup to grind the CMC and using Particle Size Analyzer to check the particles size of grinding product and check the original synthesize product.

# **3.2.2.1 Materials and Chemical**

• CMC powder

# **3.2.2.2 Tools and Apparatus**

- Pulverisette 5 Planetary Mill
- Grinding Jar
- Grinding Ball; 1 cm, 2 cm and 3 cm diameter size.
- Mastersizer 2000 Particle Size Analyzer

# 3.2.2.3 Planetary Mill Test Parameters Set Up.

This grinding process of CMC is affected by three main parameters which are the rotation speed, weight ball to powder ratio and grinding time [25]. The parameters set for this test was recorded as table 2.

| Parameters                  | Description                      |
|-----------------------------|----------------------------------|
| Rotation Speed              | 300 rpm                          |
| Weight Ball To Powder Ratio | 7 to 1                           |
| Grinding time for 1 cycle   | 6 mins operation and 3 mins stop |
| Total grinding time needed  | 30 hours = 200 cycle             |

Table 2: Planetary Mill test parameters.

# **3.2.2.4 Planetary Mill Test Sample Preparation.**

The sample needs to be prepared in a ratio of 7 to 1, weight of ball to weight of CMC. The grinding ball have 3 different diameter sizes and weight. Table 3 shows the diameter and weight of this grinding balls.

| Diameter | Weight |
|----------|--------|
| 1 cm     | 3 g    |
| 2 cm     | 22 g   |
| 3 cm     | 75 g   |

Table 3: Grinding ball diameter size and weight.

The calculation of needed grinding balls for 20 grams of CMC:

The ratio is 7 to 1,

So for 20 g of CMC the weight of the balls are 7 X 20 g = 140 g

Total ball needed,

| Diameter | Ball needed | Weight |
|----------|-------------|--------|
| 3 cm     | 1 X 75 g    | 75 g   |
| 2 cm     | 2 X 22 g    | 44 g   |
| 1 cm     | 8 X 3 g     | 24 g   |
| Total    | Weight      | 143 g  |

Table 4: Grinding ball calculation.



Figure 12: The sample preparation according to the ratio.

# **3.2.2.5 Procedure of Planetary Mill Test.**

- i. The main power was switched "ON". The green light and control panel will be working indicating the machine is operational.
- ii. After that, 20 g of CMC powder and 140 g of grinding ball were put in the grinding jar.
- iii. The hood was opened and the jar was placed at designated arms.
- iv. The screw at jar designated arms was tightened.
- v. The rotation speed 300 rpm, running time 6 mins operation and 3 mins stop for 1 cycle, and the total cycle was set at the control panel.



Figure 13: The grinding jar tightened lock in the designated arms and the parameters were set in the control panel.

- vi. Start button was pressed and the hood would be locked and the mill machine would run.
- vii. After finishing grinding, the main switch of the machine was switched off.
- viii. The mill will be unlocked after switching off.



Figure 14: The grinding product after 30 hours.

# 3.2.2.6 Procedure of Mastersizer 2000 Particle Size Analyzer.

i. The Particle Size Analyzer and the computer was switched on.



Figure 15: Particle Size Analyzer equipment set up.

ii. The 5 g of CMC powder was put at designated area inside the equipment.



Figure 16: CMC powder inside the Particle Size Analyzer equipment set up.

- iii. Than the equipment was start from the computer.
- iv. The percentage particle size result was obtained in the computer.

### **3.2.3 Fluid Friction Experiments**

# **3.2.3.1** Materials and Chemical

- Tap water
- CMC powder

### **3.2.3.2** Tools and Apparatus

- Beaker
- Test setup for Fluid Friction
- Electronic Weight Scale
- Measuring Cylinder

### **3.2.3.3 Procedure of Sample Preparation**

- i. 8 g of CMC was added to 1L of water under continuous stirring using magnetic stirrer to get 8000 ppm concentration.
- ii. This solution was filtered to remove any impurities.
- iii. Solution will be diluted with 39 L of tap water to get 200 ppm concentration.

### **3.2.3.4 Procedure of Fluid Friction Test**

- i. Tank of fluid friction was filled with 40 L of tap water.
- ii. The pump was switched on and the liquid level in manometer is kept to stabilize after 1 minute and record the initial reading of the atmospheric pressure.
- iii. The flow control valve was slowly opened until the liquid flow rate reach 1 m<sup>3</sup>/hr
  by monitoring the variable area fluid friction.
- iv. The manometer readings at the point across and away from the Orifice plate were observed and recorded after letting the reading stabilized for 1 minute.

- v. Than steps iii and iv were repeated using flow rate 2 m<sup>3</sup>/hr
- vi. Then the pump was switched off and the flow control valve was fully opened to let all of the fluid drained out into the tank below.
- vii. After that the drain valve was fully opened at the side of the tank to drain all water from the tank and to prepare for the new run.
- viii. The fluid friction setup was flushed once with clean water after each sample run to remove any leftovers inside the system.
- ix. All the steps were repeated using CMC solution samples.



Figure 17: Fluid friction equipment set up.

# 3. 3 Experimental Designs

| Biopolymer (CMC) Extraction                 |                                  |  |  |  |  |  |  |  |
|---|----------------------------------|--|--|--|--|--|--|--|
| Reaction Temperature                        | 60 °C                            |  |  |  |  |  |  |  |
| Reaction Time                               | 240 min                          |  |  |  |  |  |  |  |
| NaOH Concentration                          | 60%                              |  |  |  |  |  |  |  |
| Mass of Dried Coconut Residue for 1 batch   | 30 gram                          |  |  |  |  |  |  |  |
| Expected Mass of CMC extracted from 1 batch | 27 gram                          |  |  |  |  |  |  |  |
| Mass of CMC needed                          | 16 gram                          |  |  |  |  |  |  |  |
| Total batch for CMC needed                  | 1                                |  |  |  |  |  |  |  |
|   |                                  |  |  |  |  |  |  |  |
| Planetary Mill (nano-scale g                | rinding)                         |  |  |  |  |  |  |  |
| Speed                                       | 300 rpm                          |  |  |  |  |  |  |  |
| Weight Ball to Powder ratio                 | 7 to 1                           |  |  |  |  |  |  |  |
| Grinding time for 1 cycle                   | 6 min operation and 3 min rest   |  |  |  |  |  |  |  |
| Total grinding time needed                  | 30  hours = 200  cycle           |  |  |  |  |  |  |  |
|   |                                  |  |  |  |  |  |  |  |
| Fluid Friction Experim                      | ent                              |  |  |  |  |  |  |  |
| Volume of DRA Solution                      | 40 Liter                         |  |  |  |  |  |  |  |
| Concentration of DRA Solution               | 200 ppm                          |  |  |  |  |  |  |  |
| Samples                                     | 2 (nano and original scale size) |  |  |  |  |  |  |  |
| CMC mass needed for 1 sample                | 8 gram                           |  |  |  |  |  |  |  |
| Total mass CMC needed                       | 16 gram                          |  |  |  |  |  |  |  |

Table 5: Experimental Design Parameters.

# **3.4 Gantt Chart**

| Week                  | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|-----------------------|---|---|---|---|---|---|---|---|---|----|----|----|----|----|
| Selection of Title    |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Project               |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Literature Review/    |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Analysis Project      |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Submit Extended       |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Proposal              |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Proposal Defence      |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Project Work:         |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| -Acquired the         |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Chemical              |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| -Book the lab         |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Submit Interim Draft  |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Improving Interim     |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Draft                 |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Submit Interim Report |   |   |   |   |   |   |   |   |   |    |    |    |    |    |

Table 6: FYP I Gantt Chart.

| Week<br>Activities         | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|----------------------------|---|---|---|---|---|---|---|---|---|----|----|----|----|----|
| Project work               |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| continues:                 |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| -Synthesizing the          |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| biopolymer                 |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| -Sample preparation        |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| -Grinding CMC process      |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| -Fluid friction test       |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Submit Progress            |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Report                     |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Project                    |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| WorkContinues:             |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| -Formulate data and result |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| -Plotting graph            |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| -Data analysis and         |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| conclusion                 |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Pre-SEDEX                  |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Submit Final Draft         |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Report                     |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Submit Dissertation        |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| (Soft Bound)               |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Submit Technical           |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Paper                      |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Viva                       |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Submit Project             |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Dissertation (Hard         |   |   |   |   |   |   |   |   |   |    |    |    |    |    |
| Bound)                     |   |   |   |   |   |   |   |   |   |    |    |    |    |    |

Table 7: FYP II Gantt Chart.

# **CHAPTER 4**

# **RESULTS AND DISCUSSION**

#### 4.1 CMC Synthesization

In this extraction of CMC from coconut husk process, there were several constant variables that have been used. These constant variables were the optimum parameters to synthesize the CMC based on the previous UTP student result. The constant variables were as follow:

- i. NaOH concentration is 60 %
- ii. CMC reaction time is 4 hours
- iii. CMC reaction temperature is 60 °C.

There was no manipulated variable as the objective of this experiment is to produce or synthesize the CMC. The following assumptions were made during the synthesizing process:

- i. The final products from the synthesization are successful.
- ii. The products were also free from any impurities.

Throughout the project, 3 batches of CMC have been successfully produced, and the mass of CMC produces are presented in the following table.

| Batch<br>No. | CR initial<br>mass (g) | 60 % wt<br>NaOH (ml) | Isoproponol<br>(ml) | Chloroacetic<br>Acid (g) | CMC<br>produced (g) |
|--------------|------------------------|----------------------|---------------------|--------------------------|---------------------|
| 1            | 30                     | 100                  | 900                 | 36                       | 28.47               |
| 2            | 30                     | 100                  | 900                 | 36                       | 27.98               |
| 3            | 30                     | 100                  | 900                 | 36                       | 28.23               |

Table 8: Mass of produced CMC.

From table 8, the average of CMC produced after synthesization process is 28.22 g. This shows that around 5.93% loss of CMC from the initial. This occurred during the transferring between beakers and the filtration for washing process. The total of 84.68 g of CMC were prepared for others experiments.

### 4.2 CMC Grinding Process

In this CMC grinding process there are two separated test involves, the Planetary Mill test for grinding the CMC and the Particle Size Analyzer for checking the particle size of grinding product. The following are several constants for the whole process.

- Rotation speed is 300 rpm
- Weight ball to powder ratio is 7:1
- Grinding time is 30 hours.

For the Planetary Mill test, the mass before and after the grinding process were recorded and tabulated in table below. The total of 39.76 g of CMC were produced for the next experiments.

| Batch No. | Mass Before (g) | Mass After (g) |
|-----------|-----------------|----------------|
| 1         | 20              | 19.85          |
| 2         | 20              | 19.91          |

Table 9: Mass of CMC before and after the grinding.

For the Particle Size Analyzer Test, 2 time tests were conducted using random 5 g of CMC from the grinding product and the CMC synthesize product. Assumption were made that the rest of CMC powder particle size are the same based on average of the 2 test result. Figures below shows the result of the 2 test.

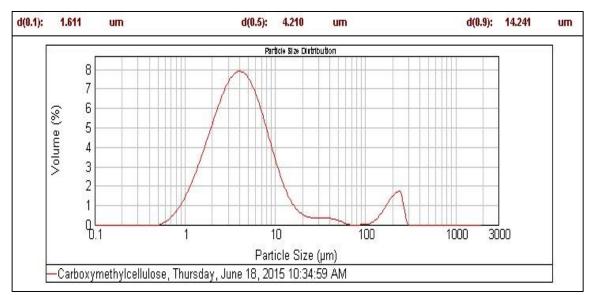


Figure 18: First test, particle size vs volume.

Table 10: First test, data of the smallest particle to the biggest particle and the volume.

For the first test (figure 18 and table 10), 10 % of the CMC particles are smaller than 1.611  $\mu$ m, 50 % of the CMC particles are smaller than 4.21  $\mu$ m and 90% of the CMC particles are smaller than 14.241  $\mu$ m. The smallest particles size is 0.6  $\mu$ m or 600 nm and the biggest particles size is approximately 300  $\mu$ m.

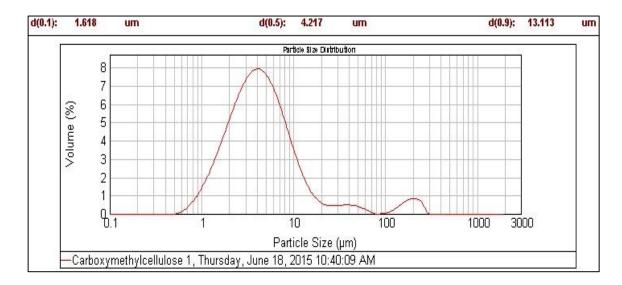


Figure 19: Second test, particle size vs volume.

| Stze (µm) | Volume In % |
|-----------|-------------|-----------|-------------|-----------|-------------|-----------|-------------|-----------|-------------|-----------|-------------|
| 0.010     | 000         | 0.105     | 0.00        | 1.096     | 1.87        | 11.482    | 2.05        | 120.226   | 0.26        | 1258.925  | 0.00        |
| 0.011     | 000         | 0.120     | 000         | 1.259     | 2.51        | 13,183    | 1.43        | 138,038   | 0.46        | 1445.440  | 000         |
| 0.013     | 0.00        | 0.138     | 0.00        | 1.445     | 3.22        | 15,136    | 0.97        | 158,489   | 0.65        | 1659.587  | 000         |
| 0.015     | 000         | 0.158     | 0.00        | 1.660     | 397         | 17.378    | 0.65        | 181.970   | 0.76        | 1905.461  | 000         |
| 0.017     |             | 0.182     | 0.00        | 1.905     | 4.74        | 19.953    | 0.47        | 208,930   | 0.72        | 2187.762  | 0.00        |
| 0.020     | 000         | 0.209     | 0.00        | 2.188     | 5.47        | 22,909    | 0.40        | 239.883   | 0.50        | 2511,536  | 000         |
| 0.023     |             | 0.240     | 000         | 2.512     | 6.13        | 26.303    | 0.39        | 275.423   | 0.00        | 2884.032  | 0.00        |
| 0.026     |             | 0.275     | 0.00        | 2,884     | 6.65        | 30.200    | 0.42        | 316.228   | 0.00        | 3311.311  | 000         |
| 0.030     |             | 0.316     | 0.00        | 3,311     | 7.01        | 34,674    | 0.44        | 363.078   | 000         | 3801.894  | 0.00        |
| 0.035     |             | 0.363     | 000         | 3,802     | 7.15        | 39,811    | 0.43        | 416,369   | 000         | 4365.158  | 000         |
| 0,00      | 122210003   | 0.417     | 20019       | 4,365     | 7.15        | 45.709    | 0.65        | 478.630   | 0.00        | 5011.872  |             |
| 0.046     | 0.00        | 0.479     | 0.00        | 5012      | 10,0045     | 52.481    | 840-21      | 549.541   | 1001202     | 5754,399  |             |
| 0.052     | 000         | 0.550     | 000         | 5,754     | 6.69        | 60.255    | 0.28        | 630.967   | 000         | 6606.934  | 1 States    |
| 0.060     | 0.00        | 0.631     | 0.09        | 6.607     | 6.12        | 69,183    | 0.17        | 724.436   | 0.00        | 7585.776  | 0.00        |
| 0.069     | 000         | 0.724     | 0.24        | 7.586     | 5.39        | 79.433    | 0.02        | 831.764   | 0.00        | 8709.636  | 000         |
| 0.079     | 0.00        | 0.832     | 0.53        | 8.710     | 4.53        | 91.201    | 000         | 954,993   | 0.00        | 10000     | 0.00        |
| 0.091     | 000         | 0.965     | 0.87        | 10.000    | 3.65        | 104.713   | 0.02        | 1096.478  | 000         |           |             |
| 0.105     | 0.00        | 1.096     | 1.32        | 11.482    | 2.80        | 120.226   | 0.11        | 1258.925  | 010         |           |             |

*Table 11: Second test, data of the smallest particle to the biggest particle and the volume.* 

For the second test (figure 19 and table 11), 10 % of the CMC particles are smaller than 1.618  $\mu$ m, 50 % of the CMC particles are smaller than 4.217  $\mu$ m and 90% of the CMC particles are smaller than 13.113  $\mu$ m. The smallest particles size is 0.6  $\mu$ m or 600 nm and the biggest particles size is approximately 300  $\mu$ m.

From both of the test, the particles size are very similar the range of particles size which is from 600 nm to 300  $\mu$ m. The average of percentage volume form both test result were summarize in table below.

| Range of Particle size (600 nm – 300 µm)                         |
|--|
| $D(0.1) - 10$ % of the particles are smaller than 1.615 $\mu$ m  |
| $D(0.5) - 50$ % of the particles are smaller than 4.214 $\mu$ m  |
| $D(0.9) - 90$ % of the particles are smaller than 13.677 $\mu$ m |
| Table 12: Summary of both test result from grinding process.     |

For the third test, this test is to find the size of particles from the original CMC synthesize product. The average range of particle sizes were summarize in the table below.

Range of Particle size (200 
$$\mu$$
m – 1.3 mm)

D(0.1) - 10 % of the particles are smaller than 282.7 µm

D(0.5) - 50 % of the particles are smaller than 703.7  $\mu$ m

D(0.9) - 90 % of the particles are smaller than 1362.3 µm

#### **4.3 Fluid Friction Experiments**

In this fluid friction experiments the drag reduction performance were tested between samples of 200 ppm from nano particle size CMC and 200 ppm from original particle size CMC. The following formula were used to calculate the drag reduction percentage.

$$Drag \ Reduction \ (\% DR) = \frac{\Delta P_{water} - \Delta P_{DRA}}{\Delta P_{water}} \ x \ 100\%$$

Where:

 $\Delta P_{water}$  = Differential manometer liquid level without DRA (tap water)

 $\Delta P_{DRA}$  = Differential manometer liquid level with DRA

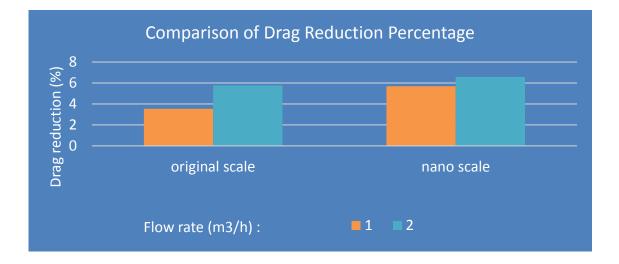
The drag reduction percentage were calculated and the result were summarized in table below.

Table 13: Summary of test result from original CMC synthesize.

| Flow<br>Rate | Sample    |      |                               |      |                           |      |
|--------------|-----------|------|-------------------------------|------|---------------------------|------|
| (m3/h)       | Tap Water |      | 200 ppm CMC Original<br>Scale |      | 200 ppm CMC Nano<br>Scale |      |
|              | ΔP (mm)   | % DR | $\Delta P (mm)$               | % DR | ΔP (mm)                   | % DR |
| 1            | 93.2      | -    | 89.9                          | 3.54 | 87.9                      | 5.69 |
| 2            | 97.3      | -    | 91.7                          | 5.76 | 90.9                      | 6.58 |

### Table 14: Summary of fluid friction result.

From data summarized in table 14, graph below were plotted to visualize the comparison between the CMC original scale and CMC nano scale.



*Figure 20: Comparison of drag reduction performance with different particle size and flow rate.* 

From graph shown in figure 20, the nano scale sample have better performance than the original scale. This proved that when the material were prepared in nano particles, it will have alterations in both physical and chemical properties [18]. And as the flow rate increasing the performance also increased. This is because of high flow rate results a high friction force thus will lead to high pressure drop.

# **CHAPTER 5**

## **CONCLUSION AND RECOMMENDATION**

### **5.1 Conclusion**

From this project, it can be concluded that the CMC has been successfully synthesized from an organic waste which is coconut residue. The synthesization process was completed by using the optimum parameters, 60% wt of NaOH, 60°C of reaction temperature and 4 hours of reaction time. The average of 28.22 g per batch were successfully produced from the initial 30 g of coconut residue cellulose powder.

The grinding process was successful as the range of particles that have been produced was from 600 nm to 300  $\mu$ m. The grinding process used Planetary Mill at parameters of 300 rpm of rotation speed, 7:1 ratio of weight of ball to powder, and 30 hours of grinding time. And the process of checking particles sizes used the Particle Size Analyzer equipment. The particle size can get smaller by increasing the rotation speed, increase the weight of ball to powder ratio and increase the grinding time.

For the fluid friction results it can be concluded that the nano particle size solution has better performance than the original particle size solutions. The fluid fractions experiment used 40 liter of solution and the concentration used for the solution were 200 ppm. This prove the literature review Filipponi et al. (2012), about nano particles that have alterations in chemical or physical because of increasing surface to volume ratio.

In conclusion, all of the objectives for this project were successfully achieved. The nano particle sizes can have better performance on the drag reduction.

### **5.2 Recommendation**

- i. Study different types of organic waste and compare the performance with the coconut residue.
- ii. For grinding into nano scale particles sizes it is recommended to use other type of Planetary Mill. For example in UTP have Planetary Mill version 5 but the latest version is version 7. So for the next study can try and search Planetary Mill version 6 or 7.
- iii. Study and compare the mechanical degradation of original particles sizes and nano particle sizes.
- iv. Study the chemical alterations of the CMC after being grinded into nano particle sizes.
- v. Compare the performance of nano particles sizes to original particle sizes in its effects regarding the reduction on permeability.

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