# ASSESSING THE EFFECTIVENSS OF BIOPOLYMER SYNTHESIZED FROM COCONUT HUSKS AS DRAG REDUCING AGENT IN WATER INJECTION SYSTEM

By

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Dissertation submitted in partial fulfilment of

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### **CERTIFICATION OF APPROVAL**

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Approved by,

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#### UNIVERSITI TEKNOLOGI PETRONAS

#### TRONOH, PERAK

September 2014

### **CERTIFICATION OF ORIGINALITY**

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

ERICQUELY LACIUS YUSOK

#### ABSTRACT

Drag reducing agents (DRA) are chemical agents used to reduce the pressure drop in a turbulence fluid of fluid inside a pipe, in this case the pressure drop of injection water inside the water injection pipelines from the surface facilities to the reservoir. In this research, biopolymer which derived from coconut husks effectiveness as drag reducing agent in water injection systems is assessed. The biopolymer which is Carboxymethylcellulose (CMC) was synthesized from the coconut husks cellulose by alkali-catalyzed reaction of cellulose with chloroacetic acid. The synthesized CMC effectiveness as DRA is assessed by determining the viscosity, density, pressure drop reduction and flow rate with different concentration of DRA solution. Based on the conducted experiment, viscosity and density of the DRA solution increases as the concentration of DRA solution also increases. The flow rate and pressure reduction experiment using flow loop shows that increment in the DRA solution concentration increase the pressure drop reduction. The concentration of the DRA solution however are limited to 2000ppm concentration. It is concluded that although the viscosity and density of the solution increases as the concentration of the DRA solution increases, the ability of the DRA to reduce pressure drop is uninterrupted.

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#### **CHAPTER 1**

#### INTRODUCTION

#### 1.1 Background of Study

In a water injection system, centrifugal pump(s) is often used to pump treated water into the reservoir through pipelines and the injection wells (Nelson, 2004). As the injection water travel inside the pipelines, drag is stimulated along the pipeline inner wall surface due to the turbulence flow of injection water. The stimulated drag promote pressure drop along the pipelines and injection wells (Brostow, 2008).

In 1974, Dr Toms has discovered the drag reduction effects. The effects are observed after adding DRA in the fluid inside a pipeline and it was first used in the oil and gas industry during fluid pumping for fracturing operation (McAllister, 2013). The pressure drop along inside the pipelines can also be decreased by injecting DRA in the injection water (Nelson, 2004). Researchers shows that organic polymer or biopolymer derived from natural waste such as corps waste are suitable substitute for synthetic polymer as DRA due to its abundant availability and its degradable properties.

For this research paper, it reports the experimental result of the adding CMC as DRA that has been synthesized from coconut by alkali-catalyzed reaction of cellulose with chloroacetic acid in brine solution with different CMC concentration. The research emphasis on the percentage of pressure drop reduction and the corresponding flow rate with different CMC concentration. Also central of the research project is correlating the viscosity and density of the concentration of the CMC solution.

To assess the effectiveness of the synthesized CMC from coconut husks as potential DRA for water injection system, it is required to have understanding on the flow regimes of fluid in the pipelines, behaviour of the drag reducing agents and its properties, water injection system and the synthesization of the CMC. This research projects has incorporated all the experimental results using fluid friction flowmeter, viscometer and density meter.

#### **1.2 Problem Statements**

There are ample research on the DRA in oil and gas industry mainly by Van, Jiang, and Wu (1997) on the effectiveness of DRA by assessing the degree of fluid flow drag reduction at certain flow rate and concentration. The experimental and field cases of the research done proved that the increase in concentration of the DRA increases the drag reduction. However, this research excluded the effects of the concentration towards the viscosity and density on seawater for the injection. Meanwhile, studies conducted by Li (2000) concluded that viscous fluid reduces the pump performance due to friction losses inside the pump.

In addition, recent studies on potential of organic materials as DRA has been conducted by Gurchan Singh and Jaafar (2013) and Sani and Faheem (2012) established that the organic polymer reduces pressure drop of flowing fluid in the pipeline and the magnitude of pressure reduction are affected by the concentration of DRA. Numbers of studies done on the benefits of coconut by-products in medical and food industries (Bogati, 2011) but the potential of CMC synthesized from coconuts husks are unidentified.

Nelson (2004) stated that injection of DRA in the water injection system successfully increase the flow rate of water injection of the Galley Field in the North Sea. In addition, the field case study by Indra (2003) on Kaji-KM.3 pipelines with drag reducers injection, the pipeline capacity increased up to 90%. However, the effect of the concentration to the viscosity and density of the fluid inside the pipeline are not taken account. While there is tangible success of DRA implementation of still so much have to be studied, like the potential of organic DRA itself, its solubility, molecular weight, viscosity and density.

Therefore, this studies is taking account the effect of the coconut synthesized DRA concentration towards the viscosity and density of the injection water. This research also include the percentage of the pressure drop reduction in a piping system by conducting experiment.

#### 1.3 Objectives and Scopes of Study

#### 1.3.1 Research Objectives

The following are the objectives of this research:

- (a) To synthesize Carboxymethylcellulose (CMC) from coconut husks.
- (b) To observe the changes in viscosity and density with different concentration of CMC solution.
- (c) To observe the pressure drop reduction and flow rate increment of flowing fluids with different concentration of CMC solution.

#### 1.3.2 Scope of Study

The synthesization of CMC from the coconut husk are conducted according to standard procedure of alkali-catalyzed reaction of cellulose with chloroacetic acid with fixed reaction temperature and reaction time. The experimental procedure are according to Gurchan Singh and Jaafar (2013).

Thorough experimental studies were conducted to assess the effectiveness of the synthesized CMC from coconut husks by measuring the change of density and viscosity of the CMC solution with its corresponding concentration. This parameters are measured using viscometer. The temperature of this parameters measurement are fixed at ambient temperature.

The change in pressured drop in a pipeline were also observed and measured using fixed diameter with different concentration using fluid friction flowmeter with the solution in loops. The assumptions of conducting the experiment are the flow regime in the flow is turbulence flow, of constant flow rate of the whole flow loop and at fixed temperature (room temperature). The CMC solution are also assumed to be homogeneous solution for all of the conducted experiment.

#### **CHAPTER 2**

#### LITERATURE REVIEW

#### 2.1 Drag Reduction

Drag is defined as something that retards motion, action or advancement ("Drag," 2014). Meanwhile according to Britannica (2014), drag is defined as a force that is exerted by a fluid stream on any obstacle in its path or felt by an object moving through a fluid. In a pipeline, drag or frictional drop is known as the result of the resistance encountered by a flowing fluid coming into contact with the pipe walls (McAllister, 2013). Drag reduction is defined as the increase of the pump ability of a fluid caused by the addition of additives into the fluid (Indra, 2003).

There are generally two types of flow in the pipeline, there are laminar and turbulence flow ("What Are Drag Reducers?," 2014). The type of flow can be quantified using dimensionless Reynolds number (Indra, 2003). The Reynolds number can be determined using the following equation:

Equation 1: Reynolds Number

$$Re = \frac{\rho u d}{\mu}$$

Where,  $\rho$  is the fluid density,  $\mu$  is the fluid dynamic viscosity, u is the fluid velocity, and d is the pipe diameter (Genick Bar–Meir, 2011).

Fluid with Reynolds number less than 2000 is a laminar flow and fluid with Reynolds number more than 4000 is a turbulence flow. Meanwhile fluid with Reynolds number between 2000 to 4000 is a transitional flow (Indra, 2003).

In a laminar flow of fluid, the observed pressure drop cannot be changed unless the physical properties of the fluid is changed but in a turbulence flow of fluid the pressure loss can be reduced by solving DRA into the fluid (Truong, 2001).

#### 2.1.1 Drag Reduction Mechanisms

In a turbulent flow regime inside a pipeline, the fluid molecules is moving in a random manners, resulting to the loss of energy as swirling currents and other indiscriminate motions. Therefore, as turbulence in the flow increases, the more energy is lost, thus pressure drop along the flow increases ("What Are Drag Reducers?," 2014).

According to the Universal Law of Wall, for rough and smooth pipe surface, there are two over-all flow in the pipe: the turbulent core and the wall layer ("Chapter 12 - Turbulence," 2012). The wall layer is assumed to comprise all type of flow regime where velocity varies with the distance from the pipe wall to the centre, assuming that the fluid molecule is a point. Meanwhile the turbulent core is the zone where eddy currents of the fluid and random motions of the fluid molecule ("Chapter 12 - Turbulence," 2012; McAllister, 2013).

The wall layer itself is consisting of laminar sublayer, buffer region and turbulent sublayer (Veldman) as shown in Figure 1.



Source: Boundaries Layer in Fluid Dynamic

Figure 1: Flow regime inside a pipeline

The laminar layer is presumed to have laminar flow and the point of velocity increases as it move away from the wall but there is no crossflow of the point in this layer. In the turbulent sublayer, its inner boundary is the turbulent core and the velocity varies logarithmically from the wall, and the crossflow is large and nearly as great as the crossflow at the turbulent core. The pressure loss at this zone is contributed by the turbulent shear generated. Meanwhile in the buffer zone, the point velocity variation with the point position variation is not established and it is presumed that it should be same as the laminar sublayer at the outer boundary and the same as the turbulent sublayer at the inner boundaries. The pressure loss in this layer is contributed by the fluid density and viscosity (McAllister, 2013; Veldman).

DRA presumed that it does not act at the turbulent core but at the wall layer (McAllister, 2013). DRA is assumed that it does not act in the turbulent core but, at the wall layer. Researcher concluded that DRA may inhibit the action of crossflow that is either generated at the laminar layer or the buffer zone. It then move inward into the turbulent sublayer and core and reduce the crossflow number, size and velocity or the DRA absorb the crossflow energy and return to the flow reducing either the crossflow number, size or velocity. Anyhow, no researchers really know which effect actually occurred. Therefore, it is concluded that: DRA act to reduce turbulence, DRA seems to act in the laminar sublayer or the bufferzone by effectively enlarging the zone or their flow, thus total friction is reduced without changing the friction factor (Bushnell, 1990; Little et al., 1975; McAllister, 2013; "What Are Drag Reducers?," 2014).

Drag Reduction in a given length of tube and flow rate has been quantified as:

Equation 2: Drag Reduction Percentage

$$\% DR = \frac{\Delta P - \Delta P_{DRA}}{\Delta P} \times 100$$

Where %DR is the percentage of drag reduction,  $\Delta P$  is the pressure drop of the fluid without solving DRA into the fluid, and  $\Delta P_{DRA}$  is the pressure drop of the fluid with DRA in the fluid (Karami & Mowla, 2012; Little et al., 1975; Truong, 2001).

#### 2.1.2 Drag Reducing Agents

Drag reducing agent defined as any materials that have the ability to reduce drag that causes pressure drop of a fluid inside a pipeline or conduit. In other words, any materials that can reduce the pressure drop in a pipeline (Bushnell, 1990; Little et al., 1975). DRA are generally categorized into three different category: polymer, surfactant and suspended solid (Nor'Aqilah, 2012).

Surfactant DRA are viscoelastic wormlike micelles have been found to be able to reduce drag in the oil and gas pipelines (Yang, 2002). The structure and dynamic properties of the micelle extend the surfactant into different types which are anionic soaps solution, non-ionic surfactant, zwittertionic surfactant and cationic surfactant (Jacques, Bin, & Hans-Werner; Yang, 2002). These micelles structure of surfactant have the ability to self-repair after their structure are altered due to high shear in a flowing conduit (Abdul-Hadi & Khadom, 2013).

Meanwhile, suspended DRA are any materials that are mixed or injected into a liquid and it can be easily added and removed from the liquid. It is also proved to be mechanically stable. There are two common suspended solid used as DRA: fibers and granular particles (Hameed, Hamied, & Shnain). It is proved that the increase in the suspended solid concentration increases the drag reduction (Derevich, Eroshenko, & Zaichik, 1985).

According to Nor'Aqilah (2012) polymer DRA can be further divided into two different categories: synthetic polymer and organic polymer or biopolymer. Synthetic polymer are polymers that is derived from petroleum products (Kaur, 2013). Natural polymers or biopolymers are defined as polymers formed under natural condition during the growth cycle of all organisms. It is formed within the organism cells by complex metabolic processes. Biopolymer is practically applied three main areas which is agricultural, medical and good packaging (Mitrus, Wojtowicz, & Moscicki, 2009). Research of the practical application of biopolymer as DRA is ongoing due to the abundance and degradable properties of biopolymer (Kaur, 2013).

#### 2.2 Carboxymethyl Cellulose Synthesization

Cellulose is one of the most abundant naturally occurring biopolymer and it is commonly found in the cell walls of plants. CMC have wide range of application is flocculation, drag reduction, textiles, foods, papers and detergents, oil well drilling and drugs. CMC is a cellulose derivatives formed by reaction of chloroacetic acid and sodium hydroxide (Biswal & Singh, 2004). Sodium carboxymethyl groups (CH2COONa) are introduced to plant cellulose molecule which produce CMC and promote solubility to water (Kamide et al., 1985).

The following is the synthesis reaction of CMC based on (Biswal & Singh, 2004; Gurchan Singh & Jaafar, 2013);

Equation 3: Synthesis of CMC

$$\begin{split} & [\mathrm{C}_{6}\mathrm{H}_{7}\mathrm{O}_{2}(\mathrm{OH})_{3}]_{n} + \\ & \mathrm{nNaOH} \rightarrow [\mathrm{C}_{6}\mathrm{H}_{7}\mathrm{O}_{2}(\mathrm{OH})_{2}\,\mathrm{ONa}]_{n} + \mathrm{nH}_{2}\mathrm{O} \end{split}$$

$$\begin{split} & [\mathrm{C}_{6}\mathrm{H}_{7}\mathrm{O}_{2}(\mathrm{OH})_{2}\mathrm{ONa}]_{n} + n\mathrm{ClCH}_{2}\mathrm{COONa} \rightarrow \\ & [\mathrm{C}_{6}\mathrm{H}_{7}\mathrm{O}_{2}(\mathrm{OH})_{2}\mathrm{OCH}_{2}\mathrm{COONa}]_{n} + n\mathrm{NaCl} \end{split}$$

#### 2.3 Water Injection

Water is injected into a reservoir for two main reason: waterflooding and waterpressure maintenance (Bradley & Gipson, 1987). Water-pressure maintenance is defined as a process of injecting water into an oil-producing reservoir to supplement the indigenous natural energy to the reservoir and to improve the field oil-producing characteristics before the economical productive limit are reached (Bradley & Gipson, 1987). Waterflooding is a secondary recovery method by which water is injected into the reservoir trough an injection well to obtain additional oil recovery through movement of reservoir oil to a producing well. This recovery method is done after the reservoir has approached its economically productive limit by primary-recovery methods (Porges, 2006). In general, the more water is being injected into the reservoir, subsequently the more oil can be produced, until water breakthrough occurs (Nelson, 2004). To transport water into the formation, water is usually pumped using centrifugal pump(s) which usually located on the production platform and it is transported through small-bore piping (typically 6-8 inch) (Nelson, 2004). Through the transportation of water to the reservoir, pressure loss occur due to friction loss and due to fittings and flanges ("Pipeline Pressure Loss," 2014). The amount of water can be injected is limited by the following factors: the capacity of injection pump(s), the pipelines and injection tubing capacity, and the reservoir characteristics (Nelson, 2004; "Waterflood design," 2014). Typically, DRA is injected at the downstream of the injection pump(s) and by injecting DRA pressure losses along the transportation of water is reduced (McAllister, 2013; Nelson, 2004). The reduction of pressure loss promote the increment of water flow rate can be pumped until the maximum allowable operating pressure of the water injection system is reached (Nelson, 2004).



Figure 2: Simplified Process Flow Diagram of Water Injection

Along the transportation of water injection from the pumping station to the reservoir, the pressure drop due to the turbulence flow and the fluid fraction of the fluid. The injection water will go through the flowlines, fittings, choke, flanges and tubing before exactly reaching the reservoir. These causes pressure drop and decreases the water injection flow rate (Nelson, 2004).

#### 2.4 Drag Reduction in Water Injection System

There are successful field case studies on drag reduction in water injection system. Based on the case studies Brent Alpha offshore field, the volume of water injected increases to 34% with concentration of DRA up to 100 ppm. Meanwhile the in Gyda oil field, rate of seawater injection to the researvoir increases to 50 % with concentration of DRA up to 80 ppm and no adverse effects on the DRA mixed with the seawater (Al-Anazi, Al-Faifi, Tulbah, & Gillespie).

Meanwhile, the field case study by Indra (2003) on Kaji-Semoga field in Indonesia shows another successful reduction of drag inside field pipelines. The pipeline capacity was increased up to 90% with the injection of DRA and effect in accelerated oil production. In Inda field located in Nigeria, DRA are proved to increase the capacity of pipelines and reduce drag inside the pipelines. It is proved that DRA is a economically reasonable options to increase to pipeline capacity (Ibrahim & Braimoh, 2005).

Meanwhile based on the field case study by Nelson (2004), her paper concluded that DRA increase water injection rate, increase oil production rates, extend the field production life, increase the overall recoverable reserves, an effective way to save energy but at the same time increase water injection and environmentally friendly.

### **CHAPTER 3**

### METHODOLOGY

### 3.1 Project Methodology



Figure 3 Research Methodology

#### 3.2 Project Experimental Procedure

#### 3.2.1 Synthesis of Carboxymethyl Cellulose from the Coconut Husks

The following procedure is adapted from previous research of "The Study of Drag Reduction Ability of Naturally Prcoduced Polymers from Local Plant Source" by Gurchan Singh and Jaafar (2013) and is originally formulated by the Center of Excellence for Polysaccharides Research, Friedrich Schiller University of Jena, Germany.

#### 3.2.1.1 Materials

i. Coconut Husks

5 kilograms of pressed coconut husks (waste) are collected from coconut milk processing shop located within five kilometres radius of Universit Teknologi PETRONAS.

ii. Chemicals for the Synthesis

Throughout the preparation and synthesis of the coconut husks CMC, all the chemicals used are in the form of Analytical Reagent (AR) grade or equivalent due to high purity as compared to Standard Laboratory Reagent (SLR) (Kaur, 2013). The chemical required during the synthesis of CMC from the coconut husks are as follow:

- a. Sodium hydroxide pellets AR QREC S5158-1-1000
- b. Isopropanol AR QREC PR141-1-2500
- c. Ethanol 96% denatured AR QREC E7045-1-2500
- d. Methanol AR QREC M2097-1-2500
- e. Chloroacetic acid for synthesis MERCK 412
- f. Acetic acid AR QREC A1020-1-2500

The listed chemicals are purchased from Irama Canggih Sdn. Bhd, Ipoh Perak.

- **3.2.1.2** Synthesis Procedure (Cellulose Extraction)
  - i. The collected coconut husks were washed thoroughly with tap water to remove it from any contaminations.
  - The product in (i) were then oven-dried for 5 hours at 50°C with open air movement out of the oven to make sure the coconut husks are not burnt and are fully dried.



Figure 4: Washed coconut husks was oven-dried for 5 hours.

iii. The dried coconut husks were then grinded by using Cole Parmer mortar grinder to reduce its size to powder form (≤ 1mm). A 50 gram of coconut husk were grinder for 3 minutes for each run due to the limited space of the grinder container. The grinded coconut husks are kept in air-tight container to kept it dry and prevent it from absorbing moisture.



Figure 5: Cole Parmer Mortar Grinder



Figure 6: The coconut husks before grinded



Figure 7: The coconut husks after grinded

iv. 50 grams of the product in (iii) is then mixed and cooked with 10 gram of NaOH pellets and 800ml of distilled water in a beaker at 150°C of 300RPM for 1 hour using a hot plate magnetic stirrer. This step is taken to remove any impurities and undesirable from the coconut residue. Based on observation, the mixture turn reddish colour.



Figure 8: Coconut husks cooked in NaOH solution

v. The product in (iv) is then filtered using tea bag and the filtered slurry was washed with running tap water for 10 minutes until the reddish colour of the slurry changed to the original colour.



Figure 9: Washing the cooked coconut husks

- vi. The product in (v) is then left to be oven-dried for 30 minutes at 121°C. The coconut husks are ensured to be fully dried by allowing air to be circulated out of the oven. This is to ensure the cellulose are extracted from the coconut husks.
- **3.2.1.3** Synthesis of Carboxymethyclcellulose from the coconut husk celluloses.
  - 15.0 gram of the product in 3.1.1.1 9 (vi) was then mixed in a beaker with 50 ml of 60% NaOH solution, 450 ml of ml of isopropanol using magnetic stirrer for 30 minutes at 200 rpm.
  - 18.0 of monocholoroacetic acid then added into the solution in (i) and was stirred for 30 minutes at 200 rpm. The coconut husks are ensured to be mixed thoroughly in the solution to ensure the carboxymethylation reaction are successful.
  - iii. The mixture in (ii) is the heated at 60°C for 60 minutes in the oven.The beaker was covered with aluminium foil to avoid evaporation during the reaction process.



# Figure 10: Coconut husk alkali-based reaction with chloroacetic acid

iv. The mixture (iii) was then filtered using sieve to remove the solid phase from the solution phase. The filtrate was then suspended with 100ml of 70% v/v methanol in a beaker for 10 minutes before adding 100ml of glacial acetic acid into the beaker for neutralization. The mixture was then filtered using a sieve, the filtrate was collected.

- v. The filtrate in (iv) was suspended in 300 ml of 70% v/v methanol for 10 minutes to remove unwanted byproducts. The mixture was filtered using sieve and suspended and stirred with 300 ml of absolute methanol for 3 minutes. The mixture was the filtered using sieve.
- vi. The filtrate in (v) is then oven-dried for 24 hours at 55°C. The dried product was then grinded using Mole Parmer mortar grinder to reduce the CMC obtained into powde r form and it was kept in an air-tight container to prevent absorption of moisture.
- vii. The synthesis of CMC was repeated for several times until the total mass of the CMC reach the desirable amount.

#### 3.2.2 Flow Loop Experiment

#### **3.2.2.1** Description of Circulating Flow Loop System

Figure 9 represent the schematic diagram of the flow system apparatus used in the experiment, which consist of tank of solution ( $0.88 \times 0.88 \times 0.88 \text{ m}3$ ), centrifugal pumps (flow rate up to 4.2m3/hr: Power = 5hp) which used to circulate solution from the tank through the pipes, flowmeter (4.2 m3/hrmaximum flow rater), ball choke valve to control flow rate and direction of solution flow rate through the system, pressure gauge and PVC pipes with outside diameter of 33.5mm.



Figure 11: Schematic diagram of the flow system

#### 3.2.2.2 Flow Loop Experimental Procedure

- i. 6 grams of CMC were mixed with 1 litre of distilled water and stirred using magnetic stirred until it is fully mixed into the water.
- ii. For the flow loop test, add the 1 litre of CMC solution into 29 litres distilled water in the flow loop tank.
- iii. The concentration of the solution are calculated using the following formula:

Equation 4: Concentration of CMC Solution in ppm

 $Concentration (ppm) = \frac{Mass of CMC (gram)}{Volume of Solution (ml)} \times 10^{6}$ 

Thus, for 6 grams of 30000ml CMC solution, the concentration is 200ppm.

 iv. The operation is started by pumping the solution through the flow loop and the choke valve is opened until the flowmeter measure at stabilized rate of 1 m3/hr and the flow loop are left circulated for 15 minutes to ensure no air inside the pipe.



Figure 12: Flowmeter

v. The pressure on the gauge are recorded.



#### Figure 13 Pressure measurement at different points

vi. The procedure are repeated with different concentration of the CMC of 400ppm, 600ppm, 800ppm, 1000ppm, 1200ppm, 1400ppm, 1600ppm, 1800ppm and 2000ppm.

#### 3.2.3 Measurement of Viscosity and Density using Rheometer

- i. 100ml of CMC solution of 400ppm, 800ppm, 1200ppm, 1600ppm and 2000ppm from the flow loop experiment were collected for viscosity and density measurement.
- 11.4 ml of the solution of different concentration were loaded into the rheometer. The rheometer were connected to a computer and the viscosity and density of the solution were obtained. Note: The Rheometer is an automated measuring equipment.

#### 3.3 **Project Key Milestones**



Figure 14: Project Key Milestones

### 3.4 Project Gantt Chart

Table 1 and 2 are the Gantt chart of this project.

WEEK	4	5	6	7	8	9	1	1	1	1	1
ACTIVITIES							0	1	2	3	4
Research on Project											
FYP Proposal											
FYP Proposal Defence											
<ul> <li>Study on Experimental Procedure</li> <li>Flow Loop Experiment</li> <li>CMC Synthesis</li> <li>Measurement of Viscosity and Density</li> </ul>											
Lab Booking											
Gathering of Material											
Experiment : Synthesis of CMC											
Interim Report											

Table 1: Final Year Project I Gantt Chart

WEEK ACTIVITIES	1	2	3	4	5	6	7	8	9	1 0	1 1	1 2	1 3	1 4
Lab Booking														
Flow Loop Test Experiment														
Viscosity and Density Measurement														
Progress Report Submission														
Analysing Experiment Results														
Pre-SEDEX														
Final Report (Draft 1)														
Submission of Dissertation														
Submission of Technical Paper														
Viva														
Report Submission														

 Table 2: Final Year Project II Gantt Chart

### 3.5 List of Equipment, Apparatus and Chemicals

### 3.5.1 List of Equipment

No.	Equipment	Purpose
1	Cole Parmer Mortar Grinder	To grind the coconut husks to powder form.
2	Magnetic Stirrer / Hot Plate	To stir and heat up mixture simultaneously at desired speed and temperature.
3	Weighing Scale	To measure the weight of materials.
4	Flowloop	To measure pressure drop in a pipe.
5	Drying Oven	To dry up moisture from involved materials.
6	Rheometer	To measure the viscosity and density of the solutions.

### Table 3: List of Equipment

### 3.5.2 List of Apparatus

No.	Apparatus	Purposes
1	Glass Beaker of different capacity	To store the solutions and mixture for heating, stirring, reaction and drying processes.
2	Stopwatch	To measure time for each process that require time monitoring.
3	Measuring cylinder	To measure volume of solutions or chemicals accurately

4	Metal Sieve (mesh size < 0.2mm)	To separate solid phase from liquid phase.
5	Air-tight containers	To store dried coconut husks and CMC and prevent from moisture absorption by the materials.

### 3.5.3 List of Chemicals

### Table 5: List of Chemicals

No.	Chemical	Purity/Grade	Supplier
1.	Distilled water	100%/SLR	UTP Lab
2.	Sodium hydroxide pellets AR QREC S5158-1-1000	99%/AR	Irama Canggih Sdn Bhd
3.	Isopropanol AR QREC PR141-1-2500	99%/AR	Irama Canggih Sdn Bhd
4.	Ethanol 96% denatured AR QREC E7045-1-2500	99%/AR	Irama Canggih Sdn Bhd
5.	Methanol AR QREC M2097-1-2500	99%/AR	Irama Canggih Sdn Bhd
6.	Chloroacetic acid for synthesis MERCK 412	99%/AR	Irama Canggih Sdn Bhd
7.	Acetic acid AR QREC A1020-1-2500	99%/AR	Irama Canggih Sdn Bhd

#### **CHAPTER 4**

#### **RESULT AND DISCUSSION**

#### 4.1 Synthesis of the CMC from Coconut Husks

In synthesising the CMC from the coconut husk, the following assumptions made, the following are the assumptions:

- i. The conducted procedure of synthesis of the CMC are successful and the final products are pure biopolymer.
- ii. The final products or the CMC are free from any impurities.

In synthesis of the coconut husk cellulose by alkali-catalyzed reaction of cellulose with chloroacetic acid, there is no manipulated variables as the sole aim for this experiment is to produce desired amount of CMC.

The following variables are remained constant throughout the synthesis:

- i. Reaction Time
- ii. Reaction Temperature
- iii. Reaction Concentration of NaOH

For each run of the synthesis, the same procedure a repeated until the total amount of the CMC reached the desired amount for the flowmeter experiment and the viscosity and density measurement.

#### 4.2 Viscosity and Density Measurement

The viscosity and density of the solution are measured at 26°C room temperature, and kept constant throughout the measurement. The pressure are also kept at atmospheric pressure of 101 kPa or 14.7 psi throughout the measurement.

The solution are assumed to be homogeneous where the CMC powder a fully dissolved in the brine solution.

Table 6 are the measured viscosity and density of the CMC solution with different concentration from the automated rheometer.

Mass of CMC(gram)	Concentration (ppm)	Viscosity (Cp)	Density (g/cm3)	
0	0	100.38	0.9998	
12	400	114.28	1.0192	
24	800	127.45	1.0211	
36	1200	142.12	1.0318	
48	1600	154.89	1.0399	
60	2000	169.17 1.0409		

 Table 6: Viscosity and Density Measurement with Corresponding Concentration of CMC

#### 4.3 Flow Loop Pressure Drop Result

The following assumption are made for the flow loop experiment:

- i. The solution prepared for the flow meter are homogeneous and the CMC powder fully mixed in the brine solution.
- ii. The flow inside the flow loop pipe are turbulence flow and there are no presence of air inside the pipe.
- iii. The effect of friction in the experiment is neglected due to same diameter of pipe used throughout the flow loop experiment.

The following table is the result of the pressure drop in the flow loop system with different concentration of CMC.

Mass of CMC (gram)	Concentration (ppm)	P1 (cm)	P2 (cm)	ΔP = P1-P2	%DR	%FI
0	0	26.9	14.1	12.8	-	-
6	200	26.7	14.4	12.3	3.90625	20.20
12	400	26.4	14.5	11.9	7.03125	30.24
18	600	26.2	14.5	11.7	8.59375	35.01
24	800	26.1	14.7	11.4	10.9375	42.06
30	1000	26.1	14.8	11.3	11.7188	44.41
36	1200	26.1	14.8	11.3	11.7188	44.41
42	1400	26.1	14.9	11.2	12.5	46.77
48	1600	26	15	11	14.0625	51.51
54	1800	26	15	11	14.0625	51.51
60	2000	25.8	15.2	10.6	17.1875	61.20

Table 7: Percentage of Drag Reduction

4.4 Effect of CMC Concentration



Figure 15: Graph of concentration versus Viscosity



Figure 16: Graph of concentration versus Density

Based on the graph above, the density of the CMC solution increases with the increasing of the CMC concentration with up to 2000ppm. The same trends are observed for the viscosity of the CMC solution, where the increment of the concentration result to increment of its viscosity. Therefore, we could conclude that the concentration of CMC as DRA effect the physical properties of the distilled water. This result contradict to the work by (Usui & Saeki, 1993) and (Mowla & Naderi, 2006) on effect of DRA concentration on the physical properties of the flowing fluid inside the pipelines.

The viscosity and density of the solution increases approximately at average of 8.5% at 400ppm increment of the solution concentration.



Figure 17: Concentration versus Pressure Drop and %DR



#### Figure 18: Concentration versus %DR and %FI

Figure 13 shows the effect of CMC concentration on the percentage of drag reduction. The percentage of the drag reduction, %DR are calculated using the following equation by McAllister (2013) of,

$$\% DR = \frac{P_{Base} - P_{DRA}}{P_{Base}} \times 100$$

Where for this experiment, the  $P_{Base}$  is the pressure drop of flowing solution inside fixed length and diameter of pipe with no DRA in the solution and  $P_{DRA}$  is the pressure drop of flowing solution inside the pipe with certain concentration of DRA injected into the solution.

Meanwhile, Figure 13 shows the effect of CMC concentration on the percentage of flow rate increment. The flow rate increment, %FI are calculated using the following equation referring to Darby (2001) of

Equation 6: Flow Rate Increment Percentage

$$\%FI = \left(\frac{1}{1 - \left(\frac{\%DR}{100}\right)^{0.55}} - 1\right) \times 100$$

Where for this equation, the only needed parameters is the %DR of the solution at certain concentration of CMC solution.

Based on both Figure 12 and Figure 13, it shows that as the concentration of CMC solution increases the %DR and %FI also increases. This support and is the same as the result by Karami and Mowla (2012).



Figure 19: Graph of concentration versus %DR and Viscosity

Correlating effects of the DRA concentration on the physical properties of viscosity and density to the %DR and %FI, it shows that at concentration of CMC from 200ppm to 2000ppm the changes of the viscosity and density of the %DR does not affect the efficiency of drag reducing effects of the CMC as DRA. Although the injection of CMC increases the viscosity and density of the solution, it does not affects the efficiency of the flow loop pumps, this is because the flow rate of the solution from point P1 to P2 of the flow loop does not decreases as the concentration increases. Therefore, for concentration of 200ppm to 2000ppm the viscosity and density changes does not affect the performance of CMC as DRA at the same temperature and assuming that the flow regime inside the pipe is turbulence flow.

Based on Figure 14, the flow rate increased up to 61.20% with 2000ppm which lower than the %FI on the experiment conducted by Karami and Mowla (2012). The test that they conducted reach more than 61% FI at 250ppm using surfactant as DRA. The difference of concentration variation of this experiment and by Karami and Mowla (2012) are more than 70% which if CMC to be implemented as DRA on field, it will be uneconomical. This may contradict to the assumption made on the degree of success of the synthesizing the CMC from the coconut husks, where the synthesized coconut husks cellulose are 100% CMC.

#### **CHAPTER 5**

#### **CONCLUSION AND RECOMMENDATION**

#### 5.1 Conclusion

For this project, it is proven that CMC can produced from coconut husks by synthesizing its cellulose using alkali-catalyzed reaction of cellulose with chloroacetic acid. This add value to the coconut husk as potential DRA as currently coconut husk are either to be thrown away or as animal feed in Malaysia. Therefore, the first objective is achieved.

Based on the measurement of viscosity and density of the solution at different concentration of CMC, it proven that the amount of CMC in the solution effect this physical parameter at fixed temperature and pressure. The relation of viscosity and density to concentration of CMC is as the concentration of CMC increases, so does the viscosity and density. However, this conclusion is only limited at concentration of 400ppm to 2000ppm and assuming that the CMC solution is homogenous.

In addition, the %DR and %FI also affected by the concentration. In which, the increment of the CMC concentration increases the %DR and %FI. The %FI is increased up to 61.20% with 2000ppm of CMC concentration. This objective achieved by assuming the flow inside the pipe is turbulence flow and the CMC solution is homogenous.

Therefore, based on the experimental results, for CMC solution of concentration 200ppm to 2000pm, the changes in viscosity and density of the solution does not affect its ability of reducing drag inside the pipeline.

#### 5.2 Recommendation

#### 5.2.1 Study the Morphology of CMC Synthesized from the Coconut Husks

To determine the properties of the CMC polymer, it is important that the molecular shape and the way of the molecules are arranged in a solid is studied. The study of its morphology will give a clear picture on how the macroscopic properties of this material changes in the solution prepared and its changes after it being used as DRA.

#### 5.2.2 Study on Different Organic Waste

The study on different organic waste on their potential to be used as DRA. A comparative studies on the produced CMC from different organic waste will add values on these wastes at the same time supporting green technology.

#### 5.2.3 Study on the Solubility of the CMC

Based on observation, the CMC takes a certain time to fully mix into the solution. Therefore, the study on the solubility of the CMC will give a better picture of the best way on preparing the solution in term of time, temperature and pressure.

#### 5.2.4 Develop a Field Case Study

A pilot study on CMC as DRA should be conducted to assess the effectiveness of the CMC in the field. The study should include economical consideration and the compatibility of the CMC as DRA.

#### 5.2.5 Study of the effect of CMC Concentration to the Formation Core

The CMC of different concentration injected into the formation may cause changes in the properties of the formation sand. Therefore, studying the compatibility of CMC on the formation sand will ensure no damage occurred due to the CMC.

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