

**Synthesis of Biopolymer from Coconut Residue and Performance as  
Drag Reducing Agent (DRA) in Water Injection Well**

by

Tareq Moqbel Qaid Alghuribi

14672

Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)

(Petroleum)

September 2014

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

**CERTIFICATION OF APPROVAL**

**Synthesis of Biopolymer from Coconut Residue and Performance as  
Drag Reducing Agent (DRA) in Water Injection Well**

by

Tareq Moqbel Qaid Alghuribi

A project dissertation submitted to the

Petroleum Engineering Programme

Universiti Teknologi PETRONAS

in partial fulfillment of the requirement for the

**BACHELOR OF ENGINEERING (Hons)**

**(PETROLEUM)**

Approved by,

.....

(SITI SARAH SALEHUDDIN)

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

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.

---

(TAREQ MOQBEL QAID)

## ABSTRACT

Over the past 20 years, polymers have been utilized in oil and gas industries as drag reducing agent (DRA) to overcome pressure drop and optimize the flow capacity in water flooding system. Yet a new eco-friendly DRA extracted from organic materials (coconut residue) is introduced to replace polymers due to its abundance and ability to perform as effective as polymer in accelerating the flow in oil and gas systems. This research intends to describe the preparation process of CMC from coconut waste and then evaluate its effectiveness as DRA in water injection well. The result of injecting these chemicals in water injection well will reduce the number of injection wells by causing a significant improvement in the flow rate which will be able to push the hydrocarbon towards production column. At the meantime, the effect of DRA upon formation permeability is going to be investigated as well as the mechanical shear degradation of biopolymer. The effect of this new drag reducing agent extracted from coconut residue on formation permeability will be evaluated by using benchtop permeability system and observe if such permeability reduction can be induced. Mechanical degradation obstructs the implementation of biopolymer for turbulent drag reduction. In general mechanical degradation refers to the process in which the chemical chain is broken due to mechanical disturbance and result in losing biopolymer its practical usage as DRA. Thus, mechanical degradation is studied by exposing 0.5% and 1.0% solution of biopolymer to a high stirring speed and observe its influence on the viscosity of the solution with respect to time. The decrease of viscosity with time is an evident of mechanical degradation of biopolymer which is educed due to shear stirring.

## **ACKNOWLEDGEMENT**

All praised to Allah S.W.T, The Almighty, The Most Gracious and The Most Merciful. I wish to extend my deepest appreciation to everyone who contributed to the accomplishment of this Final Year Project.

First and foremost, I would like to express my thanks to my honorable supervisor, Madam Siti Sarah Salehuddin for her guidance, advices, support and encouragement throughout the entire semester of completing this project. She has motivated me to complete and be prepared to face challenges in the future career. May Allah bless her for her kindness and supports.

I also would like to thank all the lab technicians of Core Analysis Lab at Block 15 (Petroleum Engineering Department): Mr Shahrul, Mr Saiful, Ms Yeni, Block 17 (Mechanical Engineering Department): Ms. Daniel, and Block 20 (Mechanical Engineering Department): Mr. Hazri ,and Ms/ Shihadah for showing me the proper way to handle the equipment in the respective labs and their willingness to come outside of their working hours to monitor me conducting the experiment in the lab.

Not to forget to my beloved parents, family and friends for being very supportive and keen to know the outcome of my research until the completion of this report.

Last but not least, many thanks to Universiti Teknologi PETRONAS for all the equipment and devices that is provided for me in order to complete this research successfully.

# TABLE OF CONTANTS

<b>ABSTRACT</b> .....	<b>IV</b>
<b>ACKNOWLEDGEMENT</b> .....	<b>V</b>
<b>TABLE OF CONTANTS</b> .....	<b>VI</b>
<b>LIST OF FIGURES</b> .....	<b>VIII</b>
<b>LIST OF TABLES</b> .....	<b>IX</b>
<b>CHAPTER 1: INTRODUCTION</b> .....	<b>2</b>
1.1    Project Background.....	2
1.2    Problem Statement.....	4
1.3    Research Objectives.....	5
1.4    Scope of Study.....	5
<b>CHAPTER 2: LITERATURE REVIEW</b> .....	<b>6</b>
2.1    Drag Reduction Theory.....	6
2.2    Flow Regimes in Pipeline.....	7
2.3    Drag Reducing Agent Performance.....	9
2.4    DRA in Water Injection Wells.....	10
2.5    Mechanical Degradation of Biopolymer.....	11
2.6    Biopolymers.....	11
2.7    Nano-scale Applications.....	12
<b>CHAPTER 3: METHODOLOGY</b> .....	<b>14</b>
3.1    Research Methodology Procedure.....	14
3.2    Project activities.....	15
3.2.1    Synthesizing the Biopolymer.....	15
3.2.2    Materials.....	15

3.3	Extraction of Cellulose.....	15
3.4	Preparation of Carboxymethyl Cellulose (CMC).....	18
3.5	Tools and Equipment Setup .....	21
3.5.1	Sample Preparation.....	21
3.5.2	Benchtop permeability Experiment .....	22
3.5.3	DRA Mechanical Shear Degradation Test .....	25
3.6	Gantt Chart (Timeline).....	28
3.7	Project Key Milestones .....	29
<b>CHAPTER 4: RESULTS AND DISCUSSIONS .....</b>		<b>30</b>
4.1	Variables.....	30
4.1.1	Constant Variables.....	30
4.1.2	Changeable / Manipulated variables.....	30
4.1.3	Parameters of interest .....	31
4.2	CMC Extraction data.....	31
4.3	Benchtop Permeability System Test at Different Injection Rates.....	33
4.3.1	Calculation of Permeability Reduction & Recovered Permeability in Percentage.....	37
4.4	The Influence of Mechanical effects on Biopolymer Mechanical Degradation. .....	.40
4.4.2	Viscosity Reduction Calculation .....	43
<b>CHAPTER 5: CONCLUSION AND RECOMMENDATION .....</b>		<b>47</b>
5.1	Conclusion.....	47
5.2	Recommendations .....	48
<b>CHAPTER 6: REFERENCES.....</b>		<b>49</b>

## LIST OF FIGURES

Figure 1: Water flooding technique .....	2
Figure 2: Flow regimes behavior .....	7
Figure 3: Grated Coconut Flesh waste .....	12
Figure 5: Project Workflow Chart .....	14
Figure 6: Drying process of CR in oven at 50° C for 2 days .....	16
Figure 7: Mortar Grinder .....	16
Figure 8: Cooking CR with NaOH at 150°C .....	17
Figure 9: Left before rinsed with tap water; Right after rinsed with tap water.....	17
Figure 10: Solution of 40% of NaOH ,15g of CR , and 450ml isopropanol.....	19
Figure 11: Flow chart of CMC Preparation .....	20
Figure 12: Desiccators with positive displacement pump setup .....	22
Figure 13: Benchtop permeability system setup .....	23
Figure 14: Gantt chart FYP1 .....	28
Figure 15: Gantt chart FYP2.....	28
Figure 16: Project Key Milestones .....	29
Figure 17: DRA Performance at 1ml/min.....	34
Figure 18: DRA Performance at 3ml/min.....	35
Figure 19: DRA Performance at 5ml/min.....	36
Figure 20: Permeability reduction and recovered versus injection rate of Biopolymer DRA Solution.....	39
Figure 21: Effect of exposure time and stirring speed on the viscosity of 0.5% biopolymer solution .....	41
Figure 22: Effect of exposure time and stirring speed on the viscosity of 1% biopolymer solution.....	42
Figure 23: Effect of exposure time and shear rate on percentage viscosity reduction of 0.5% DRA.....	45
Figure 24: effect of exposure time and shear rate on percentage viscosity reduction of 1% DRA.....	45



## LIST OF TABLES

Table 1:Type of flow regimes .....	8
Table 2:Used Parameters in Preparing CMC and corresponding ranges .....	18
Table 3:Collected Data From The Experiment .....	32
Table 4: Observed Permeability after 60 minutes at 1,3,&5ml/min .....	37
Table 5: Summary of permeability reduction and recovered percentage .....	39
Table 6:Viscosity of 0.5% solution after exposing to different shear rate using viscometer .....	40
Table 7:Viscosity of 1% solution after exposing to different shear rate using viscometer .....	42
Table 8: Percentage Viscosity Reduction of 0.5% .....	44
Table 9: Percentage Viscosity Reduction of 1.0% .....	44

# CHAPTER 1

## INTRODUCTION

### 1.1 Project Background

Optimizing the production in oil and gas industries is very essential and has valuable effects such as maximizing the economic revenue. The production life in oil and gas industries takes place right after drilling where the three drive mechanisms will be implemented to sweep hydrocarbon towards the production column (wellbore). The first drive mechanism known as primary recovery will cause the hydrocarbon to be produced effectively by utilizing the natural energy of the reservoir. Due to the production, reservoir pressure will drop and reservoir's energy will be depleted. To maintain the production secondary recovery techniques shall be used. There are other methods which can enhance the producing wells production such as water injection method which is the most common method.

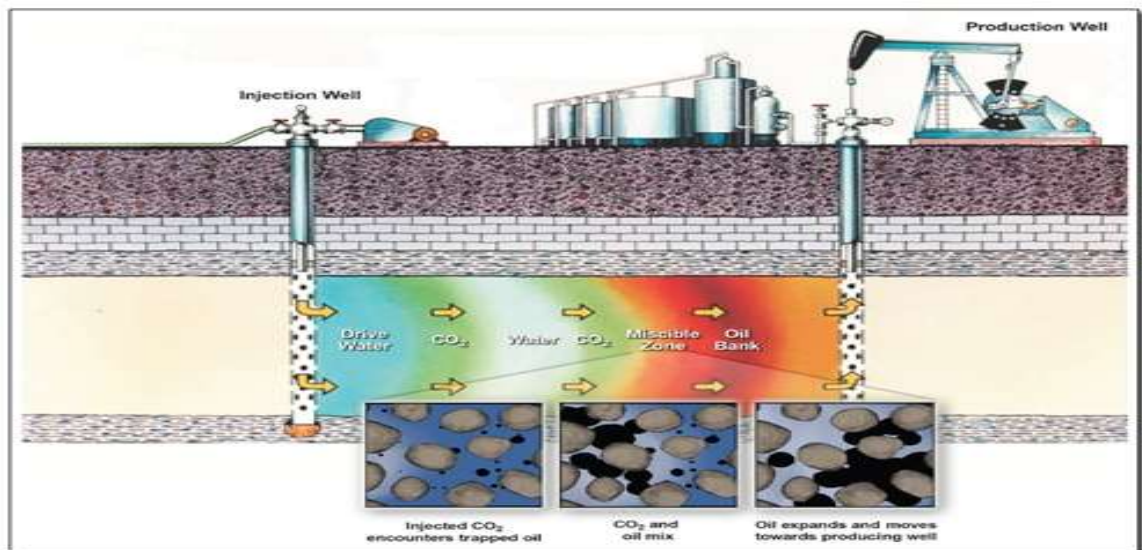


Figure 1: Water flooding technique

Source: <https://www.google.com.my/search?q=water+injection+well>

As formation gets matured the amount of produced water increases while the production of hydrocarbon decreases dramatically. The removal of the produced water during production life will be handled efficiently by introducing water flooding method in which the produced water will be injecting back into the reservoir to drive the hydrocarbon from the formation to the wellbore as well as minimizing the water production.

Drag reducing agents (DRA) are used in water injection method for the purposed described above. Water is injected to the reservoir by using a high energy pumps, yet pumps are consuming a lot of energy and require more time for installation, thus DRA is reacted with water to make a solution that can effectively enhance the flow capacity and minimize the usage of pumps in water injection systems.

As the demand of oil and gas increases dramatically, so oil and gas industries are in need to find more economic and effective ways to improve the production. Mainly, the produced hydrocarbons are transported through a large pipeline to the processing plant and refineries. The transportation of oil and gas through the line experiences serious problems due to the occurrence of turbulent flow that causes a massive pressure reduction where this reduction affects the pipeline deliverability. Turbulent flow regime has been understood as the chaotic flow that induces a drag that simply restricts the movement of the fluid. Therefore, Drag Reducing agents (DRA) are utilized since they have the ability to overcome the frictional drag and enhance the system pumpability.

DRA are classified to three types which are polymers (either natural or synthetic polymers), surfactants, and fibbers. Toms (1949) was the first observer who observed the drag reduction phenomenon and explained its functionality in dampening turbulent flow, and optimizing the production across the system [1]. Practically, injecting small amount of additives to the fluid reduces the pressure drop and improves the flow rate without changing the pipeline conditions [2]. DRA performance is dependent upon several parameters, namely, concentration, viscosity, solubility, and molecular weight of the chemical additives. These parameters are considered the main factors that can control the effectiveness of the DRA at any case.

## 1.2 Problem Statement

Apparently, pipelines are the remarkable and essential tools in oil and gas industries, thus production of hydrocarbon is transported through pipelines laying from the surface of producing wells to the processing plants. The presence of turbulent flow in the line has considered a complex problem which leads to a massive pressure drop inside the pipe that cause a dramatic reduction in oil and gas production. This problem has become a controversial concern in oil and gas industries. Meanwhile, installation of several pumps across the line would have the ability to optimize pipeline capacity, but unfortunately usage of pumps is not encouraged due to the high energy consumption by pumps which might lead to negative effects, such as the pump pressure might exceed the maximum allowable pressure of the pipe, plus it is unaffordable. Numerous researches and studies have been done in order to find out the desirable solution that can replace the usage of pumps efficiently.

Considerably, DRA that extracted from synthetic polymers is introduced in oil and gas industries by injection them into the system in order to enhance flow throughput and minimize pressure drop in the line. The drag reducing agents (DRA) are declared as the best replacer for pumps, due to the potential they have in improving the flow rate of the system by destroying turbulent flow regime. As the use of synthetic polymer as DRA increases extensively, some undesirable effects emerge and approved to be harmful to our environment due to their chemical compositions. Therefore, the biopolymers extracted from organic minerals are introduced as DRA by which they have been proven through experimental studies to be the convenient alternative for synthetic polymer DRA since they are abundant in nature, and environmental friendly drag reducing agent.

Interestingly, this project will make a use of natural waste as DRA to resolve the production problem either during water flooding techniques or during production from the producing wells into the processing plants.

### **1.3 Research Objectives**

This study aims to study the effectiveness of natural polymer as DRA by executing the following objectives effectively.

1. Extraction of Carbomethyl Cellulose (CMC) from coconut residue.
2. To investigate the effect of DRA on formation permeability in water injection well.
3. To study the mechanical effects on mechanical shear degradation of biopolymer as DRA.

### **1.4 Scope of Study**

The scope of this study is focusing more on evaluating the potential and effectiveness of biopolymer extracted from wastes, and its effects on permeability in water injection wells. The scope of this research is to conduct experimental studies by using core flooding experiment, benchtop permeability system and viscometer devices. The used medium will be water in the pipe. Based on the laboratory data, the effectiveness of organic polymer can be evaluated and traced using many parameters. The experiment will be carried on and repeated by changing the parameters in order to achieve a better reading which will help in making a good conclusion about each objective. Changing parameters will manipulate a significant role in testing mechanical degradation. The limitation of this study is the unavailability of crude oil to be used as medium in the test pipe, so it is replaced by water since DRA are soluble in water.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Drag Reduction Theory**

Generally, drag is a type of friction or force which is created in a pipe as fluid movement resistance. In the same word, drag can be defined as any force that acts in opposite motion to the flowing fluid. According to Karami & Mowla.(2012) the frictional drag is created by the interaction between the pipe internal wall and eddies which creates a disturbance force causing a reduction in the production rate [2]. The behavior of turbulent flow induces due energy loss across the system causing difficulties to the acceleration of the flow, so the production rate will have a low speed due the reduction in pressure. The decrease of the flowing liquid (oil or water) in the pipeline is one of the challengeable obstacles in petroleum industries, so it is resolved by injecting drag reducing agent through the line resulting in optimizing the pipeline capacity. Initially, Drag reducing agent was first introduced by Tom.B.(1948) who illustrated that the addition of slight quantity of chemical additives into a turbulent flow solvent would have the ability to decrease the frictional force by over 70%. The Potential of DRA (rag reducing agent) has successfully implemented to improve the flow capacity throughout the pipeline [1]. The drag reducing agent in crude oil pipeline transportation was firstly used in Trans- Alaskan line in which the drag reducing agent was injected to the pipeline which results in a significant increment in pipeline deliverability [3].

(De Gennes, 1990 & DH. Lee 2010) observed that drag reducing is not created due to the pure viscous impact of dilute polymer solution inside the system [4]. The reduction is generated once the fluid flows in disorder manner causing reduction in both flow rate and pressure simultaneously [5]. Campbell (2001) clarified that drag reduction is

basically a term utilized to describe the changes in the behavior of turbulent flow after introducing DRA [6] . Energy loss is the main reason behind the reduction of pressure of the liquid in the conduit in the direction of flow [7]. During the operation drag reducing agents act as a shock absorber and utilize its potential to interfere the turbulent burst.

## 2.2 Flow Regimes in Pipeline

Generally, the flow behavior can be described as laminar and turbulent flow. Laminar flow is a stabilized flow which flows in a uniform manner whereas the turbulent flow is unstable flow and flowing aggressively due to the eddies disturbance [8]. Moreover, turbulent flow regime is where the flowing fluid is flowing in a chaotic manner and always presented in highly disorder way. In the pipelines, there is a buffer region where the laminar flow regime is triggered into turbulent flow. Lumley (1973) interpreted that the buffer layer exists between laminar and turbulent regions in which the turbulence penetration will encounter difficulties and results in slippage inside the pipeline [9].

Numerous studies have interpreted that, drag reducing agent (DRA) is only feasible to be used in turbulent regimes through which frictional lose is emerged and leads to a pressure drop as well as flow rate reduction along the pipeline. As shown in figure 1, the transition region describes the fluid that flows in non-uniform manner due to the presence of some disturbances which changes the behavior of the flow regime into turbulent flow.

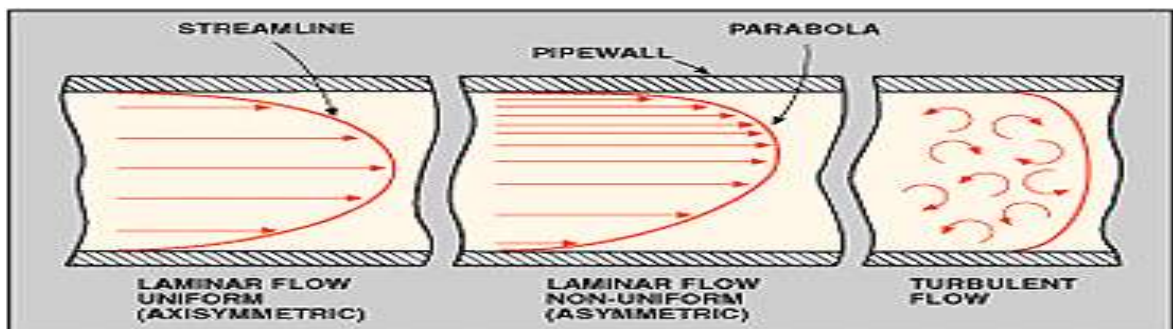


Figure 2: Flow regimes behavior

The transition flow is dependent on the characteristic of the fluid such as density and viscosity as well as system geometry. To distinguish between laminar and turbulent flow

regimes it is essential to use the Reynolds number parameter (Re). Reynolds number is known as the ratio of inertial forces to viscous forces. The determination of Reynolds number is done by applying the following equation:

$$Re = \frac{\rho v d}{\mu} \dots\dots\dots(1)$$

Where,

$\rho$  = density of the fluid ,

$v$  = flow velocity

$d$  = Internal diameter of the pipe,

$\mu$  =viscosity of the presented fluid.

In most of experimental and theoretical calculation, flow in pipe is laminar if Reynolds number (Re) is less or equal to 2300, turbulent if Re bigger than 4000, and it is considered as transitional regime in between.

Table 1:Type of flow regimes

Reynolds No	Type of flow regime
$Re \leq 2300$	Laminar flow
$2300 \leq Re \leq 4000$	Transitional flow
$Re \geq 4000$	Turbulent flow

Indra Prasetyo (2003) reported that, performance of drag reducing is more effective if the degree of the turbulence is higher, so it means the drag reducing performance boosts as the viscosity and velocity of the fluid increases [10]. Hence, the effectiveness of drag reducing agent is depending on Reynolds number value, so high Reynolds number will leads to influential and efficient performance of drag reducing.



### 2.3 Drag Reducing Agent Performance

Drag Reducing Agent (DRA) is a highly molecular weight chemical that is derived either from Synthetic polymers or organic waste materials known as biopolymers. DRAs are very active agents which extracted from natural waste materials such as banana peels, flesh coconut husk, and etc. Subsequently, those wastes are verified to be very practical DRA for several reasons; they are less time consuming, eco-friendly, having high biodegradation, and effectively soluble in aqueous medium (either water or oil). The reason beyond converting natural waste materials into DRA is because they possess a considerable amount of cellulose that turns to carboxymethylcellulose through etherification process using sodium hydroxide [14].

DRA has been introduced to industries because of their high mechanisms and benefits in reducing the operational cost by removing the unnecessary power pumps, and reducing the drag to evolve the efficiency across pipeline. DRA is very effective and quick in lowering pressure drop at any porous medium of both liquid (oil & water) and gas velocities [19]. The addition of drag reducing agents is efficient in decreasing the slug frequency as well as the thickness of liquid film which definitely diminishes frictional pressure losses within the line and improve flow rate accordingly [11]. As reported slug frequency occurs in the presence of high liquid velocity, so slug will decrease by injection DRA chemical additives leading to slug frequency of 0 slug/minute.

Mechanically, DRA is introduced to delay the occurrence of turbulence across the pipeline. As it has been proven by many researchers drag reducing agent effectiveness increases with increasing the concentration of the additives. A typical concentration of 40ppm to 100ppm is capable of destroying the turbulent flow and reducing pressure drop by over 60% [12].

Drag reduction is defined as the difference between pressure differential without DRA to the pressure drop with DRA, divided by the pressure drop without DRA. Hence, the percentage of drag reduction (DR %) is determined by using the following formula:

$$\%DR = \frac{\Delta P_{without\ DRA} - \Delta P_{with\ DRA}}{\Delta P_{without\ DRA}} \times 100\% \dots\dots\dots (2)$$

Where,  $\Delta P_{without\ DRA}$  ,is the pressure drop before injection DRA, and  $\Delta P_{with\ DRA}$ ,is pressure drop right after injection DRA.

## 2.4 DRA in Water Injection Wells

Water flooding is one of the tertiary drive mechanism techniques and it is widely used through which water is injected through injection wells to the hydrocarbon zone. As water injection continues vertical flood will be generated, sweeping the hydrocarbons toward wellbore of producing wells. [15] elaborated that, DRA has the ability to reduce the number of the needed injection wells .Also, Chevron Texaco Company used DRA in Galley field to increase the rate of water injection and the result was observed by injecting 45ppmv of DRA the achievable water injection rate would be 40000b/d. DRA was mixed with water and used in water flooding treatment to increase the viscosity of water which will affect the mobility ration of water; hence oil will be more likely to flow towards production column [18].

The purpose of injecting water into the formation is to maintain formation pressure as well as pushing the hydrocarbon out from the reservoir to the production column. Therefore, H.A.Al-anazi,et al.(2006) mentioned in their paper that, DRA is favorable with biocides to be used in the seawater injection system to increase reservoir pressure which implies in maintaining oil production at a high level [13].

Commonly, DRAs are injected into the injection wells to see their effectiveness in water flooding. As not many studies have focused on DRA effects on the formation during injection operations. For instance, if it damages the formation, it would not be commercially feasible to be exercised.

## **2.5 Mechanical Degradation of Biopolymer**

Degradation decreases the efficiency of polymer and biopolymer additives because they are highly dependent on the effectiveness of molecular weight [20]. Drag-reducing Biopolymers are found to lose their effectiveness when subjected to an intense turbulence field for sufficient amounts of time. These losses are usually associated with chain breakage where they turn to weak. However, if aggregation enhances drag reduction, it follows that degradation should also occur because of the breakup of agglomerates. Several studies used viscosity measurements to detect that changes in the average molecular weight accompanies changes in the effectiveness of a polymer which is known as mechanical degradation [22].

This study deals with the experimental data for mechanical degradation and the performance of drag reducing polymers. Two solutions of biopolymer with high, different molecular weights were exposed to shear degradation using viscometer at which it setup at high string speed for 1 hour.

## **2.6 Biopolymers**

Biopolymers are generated from natural materials that are renewable and have the potential to dissolve within a short timescale. Biopolymers have been used in many essential and beneficial applications since they are abundant and diverse in the nature. Apparently, synthetic polymers are used widely in various applications, so due to their extensive usage they have become a concern in industries because of their slow destructions and degradations, thus natural polymers are likely to be the suitable replacer.

Banana peels and flesh coconut residue are type of the natural waste materials. As explained by [16], fresh banana peels is represented by 40% of the total weight of the banana itself. The banana peels are locally collected and they are degraded or destructed within 2-10days but it depends mostly on the season of the year [17].

Grated coconut flesh, mainly consists of cellulose, and some carbohydrates. Cellulose is useful and it is found in plants such as flesh coconut, banana peels, and etc. Flesh coconut is chosen as the source of Carboxymethylcellulose as it contains carbohydrate and crude fiber which will produce more cellulose through the reaction of Carboxymethylcellulose. Figures 3,&4 show flesh coconut waste and powder.



Figure 3: Grated Coconut Flesh waste & Powdered Grated Coconut

Source: <https://www.google.com.my/search?q=flesh+coconut+waste>

## 2.7 Nano-scale Applications

Nano-scale technology is a modern technology divaricated from nanotechnology in which it is extremely small tool used to construct system with very negligible dimensions that come on order of nanometer; hence 1nm is equivalent to billions of meters  $10^{-9}$  m. Nanotechnology is used in many applications, such as physical, chemical, and biological applications. According to K.P.Hoelscher,et al. (2012) nanotechnology is introduced in drilling fluid to improve the durability as well as enhancing the stability near the wellbore [15]. Also, they identified that in shale formation the permeability is quite low, so the addition of nanoparticle into drilling mud will reduce the leak off and it will have the ability to plug the shale pores and control water loss. In this study the author measure the organic DRA in Nano-scale in order to investigate the solubility of

the additives in water solvent and compare it with the DRA measure by the normal scale. As a result, the Nano-scale biopolymer shall have high solubility in the solvent that rapid in overcoming the frictional loss across the system efficiently.

## **CHAPTER 3**

### **METHODOLGY**

The methodology is a tool that describes methods and materials applied to a certain field of study in order to collect necessary data. This chapter aims to cover the work flow, along with the project activities, milestone, Gantt chart, materials, equipment, and apparatus effectively.

#### **3.1 Research Methodology Procedure**

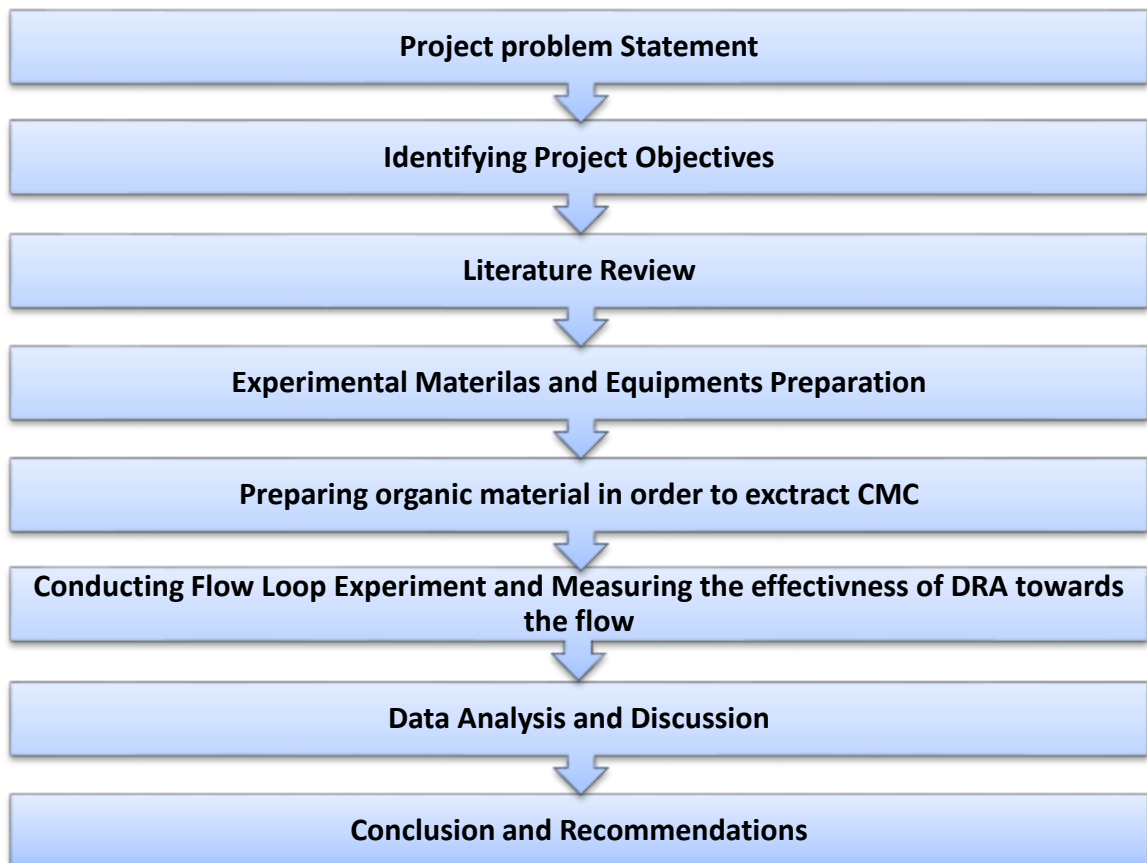


Figure 4: Project Workflow Chart

## **3.2 Project activities**

### **3.2.1 Synthesizing the Biopolymer**

Synthesizing biopolymer from organic minerals such as coconut residue is not an easy task to handle since not many studies have been done on this topic. The process for the preparation of biopolymer from coconut residue is basically divided into two stages; first stage involves cellulose extraction, and the second stage interpret the synthesis process of CarboxyMethyl Cellulose (CMC) from coconut residue (coconut waste).

### **3.2.2 Materials**

Coconut residue will be collected from local source as a waste and then it will be sold in the market. Therefore, the coconut residue is purchased from a shop in Taman Maju. The needed chemicals for synthesis of carboxymethylcellulose (CMC) were also purchased from a chemical company known as Irama Canggih Sdn Bhd. The required chemicals for synthesizing CMC are as listed below:

- i. Sodium hydroxide pellets AR QREC S5158-1-1000
- ii. Isopropanol AR QREC PR141-1-2500
- iii. Ethanol 96% denatured AR QREC E7045-1-2500
- iv. Methanol AR QREC M2097-1-2500
- v. Chloroacetic acid
- vi. Acetic acid AR QREC A1020-1-2500

## **3.3 Extraction of Cellulose**

- i. Basically, coconut residue will be rinsed with water for the purpose of cleaning and then it will undergo drying process either naturally by exposing it to sun for 3-4 days or artificially by placing it in an oven at 50° C for 2 days.



Figure 5: Drying process of CR in oven at 50° C for 2 days.

- ii. The dried CR will be grinded to a fine powder by using Mortar Grinder. The grinding process will be done in batches where the grinder is set up at 3 minutes for each batch.



Figure 6: Mortar Grinder

- iii. The grinded CR will then be cooked with 1M of NaOH in a 2 liters beaker at 150°C and stir for 1 using a magnetic stirrer (200rpm). While monitoring the mixture, it was observed that it turns to a reddish mixture due to the reaction between CR and NaOH at 150°C. The objective of cooking CR with NaOH, is to remove the undesirable products that increase the adhesion of the powder.





Figure 7: Cooking CR with NaOH at 150°C

- iv. After the red slurry is obtained, it will be filtered by using tea bag filter to remove the powder from the liquid phase. The suspended powder will be washed with plenty of water until the red color is gone and it turns to its originated color.



Figure 8: Left before rinsed with tap water; Right after rinsed with tap water.

- v. The obtained residue will be dried in an oven at 121°C for some time (30 minutes, 24 hours) so it will be ready to be synthesized to obtain the CMC. This drying process is to ensure the moisture has been eliminated completely. The dried powder will be kept in a tight container before synthesizing CMC.

### 3.4 Preparation of Carboxymethyl Cellulose (CMC)

For synthesizing CMC, there are three parameters that should be controlled as in table 2. By manipulating with these parameters, the author would be able to figure out at which time, temperature and concentration of NaOH, a higher mass of CMC will be produced.

Table 2: Used Parameters in Preparing CMC and corresponding ranges

Parameters	Range
Reaction Temperature (C°)	50-60
Reaction Time (min)	60-240
NaOH Concentration (m/v %)	20-50

By manipulating with these parameters, a good conclusion will be made on what is the best value of three parameters that can result out in a higher mass of CMC. Therefore, the author is trying to be more productive and efficient by selecting the best parameters values that can lead to optimum results of CMC mass.

Preparation of Carboxymethyl Cellulose is prepared through two reactions as follows:-

- ✓ Alkalization Reaction
- ✓ Carboxymethylation Reaction.

#### **Procedure:**

- i. 15.0g of cellulose obtained in the previous step, 50ml of NaOH of 40% concentration, and 450ml of isopropanol were mixed in a beaker using a magnetic stirrer for 30 minutes. And then 18g of chloroacetic acid is added to the solution to initiate the Carboxymethylation reaction and the solution is stirred again for 30 minutes. This step is important to ensure that the biopolymers are mixed completely with chemicals and distilled water until it behaves as homogenous.

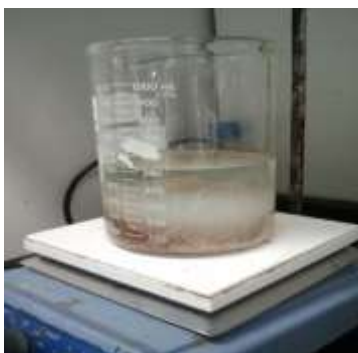


Figure 9: Solution of 40% of NaOH ,15g of CR , and 450ml isopropanol

- ii. The mixture prepared in the previous step is heated in the oven at 50°C for 60 minutes. The beaker is covered with foil to prevent evaporation during the heating process. Right after this step the solution was filtered by using sieve to separate solid phase from liquid phase. After that, the solid phase is suspended in 100ml methanol of 70% v/v overnight. Glacial acetic acid was added into the beaker to neutralize the suspended solids in methanol solution and then it is filtered using a filter funnel with a filter paper.
- iii. The sample is suspended in 300ml of ethanol of 70% v/v for 10 minutes to remove the unwanted products. Afterward, the sold phase will be washed with methanol until it looks clean. The product will undergo a drying process in an oven for 24hours at 55 ° C. And then will be grinded to very fine powder and finally CMC is produced.



Figure 10: Produced CMC from Coconut Residue

✓ The preparation of CMC is summarized as in the following flow chart:

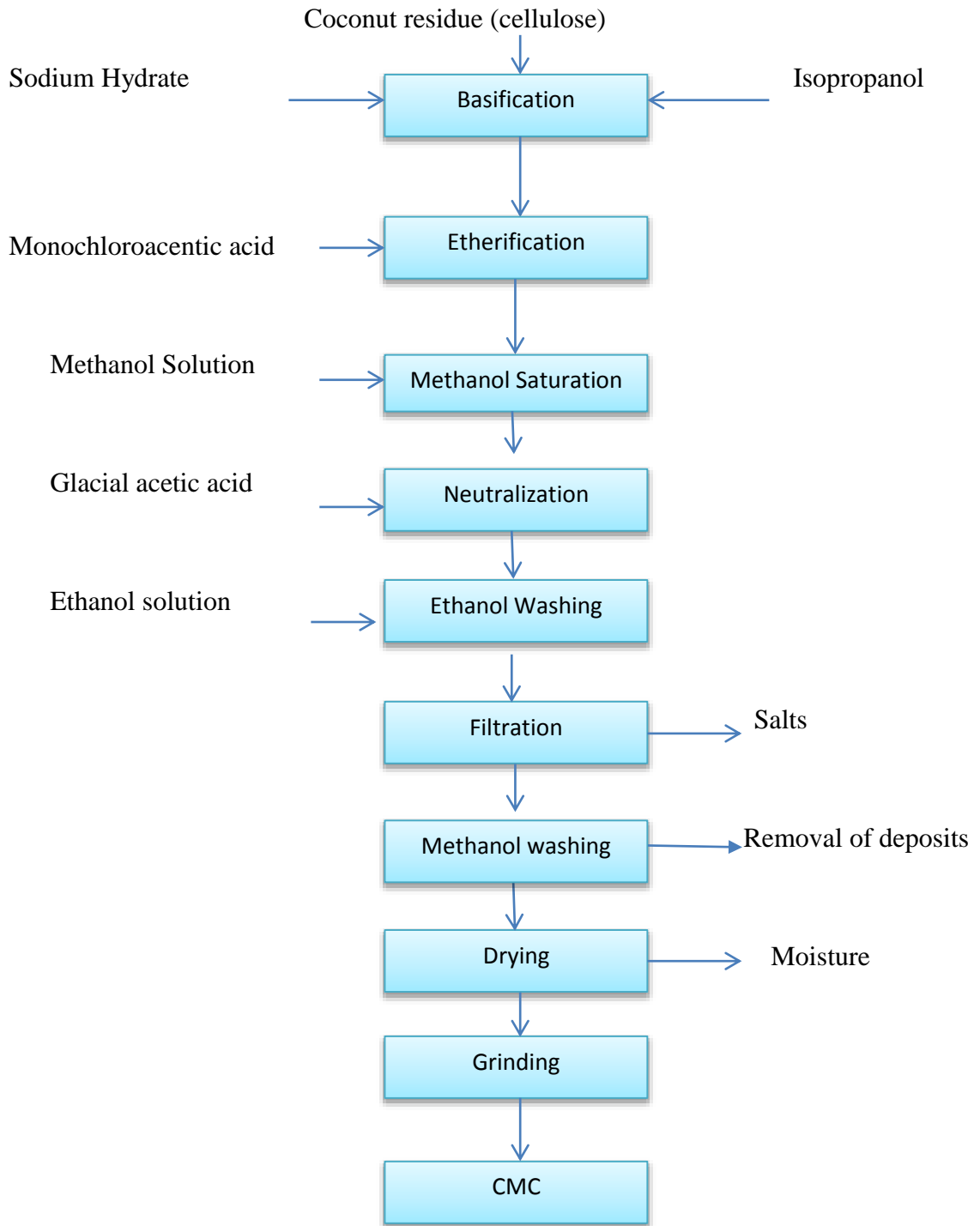


Figure 11: Flow chart of CMC Preparation

### 3.5 Tools and Equipment Setup

#### 3.5.1 Sample Preparation

Solution of DRA is prepared by adding CMC powder that is extracted from coconut residue with distilled water. By manipulating with mass, different concentrations would be achieved.

##### ✓ Brine Solution

To prepare a brine solution that has salinity of 10000ppm, it is required to use the following formula:

$$\text{Concentration (ppm)} = \frac{\text{mass of brine(g)}}{\text{volume of distilled water (ml)}} \times 10^6$$

$$\text{Concentration (ppm)} = \frac{10g}{1000ml} \times 10^6$$

$$\text{Concentration (ppm)} = 10000\text{ppm}$$

##### ✓ DRA solution

To prepare a solution of DRA that has concentration of 50ppm in order to be used in permeability test. Basically, it can be prepared as follows;

$$\text{Concentration (ppm)} = \frac{\text{mass of DRA(g)}}{\text{volume of distilled water (ml)}} \times 10^6$$

$$\text{Concentration (ppm)} = \frac{0.05g}{1000ml} \times 10^6$$

$$\text{Concentration (ppm)} = 50\text{ppm or } 0.05\%$$

After that, the DRA solution (biopolymer) is mixed under a medium shear rate using standard magnetic stirrer for about 8 hours, in order to create a homogenous solution of DRA solution. New DRA solution is prepared before each run to avoid any effects caused by shift time degradation between each run.

### 3.5.2 Benchtop permeability Experiment

The core samples that is chosen in this experiment are Barea sandstone cores. There are 3 core samples with length of 3inches and diameter of 1.5 inches. These core samples were saturated for around 8 days using Desiccator system to ensure that the cores are fully saturated.

- **Desiccator with vacuum pump**

The desiccator setup is as in figure 12. The core samples are immersed in 1000ml beaker containing 600ml brine of 10000ppm salinity. After that the positive displacement pump I switched on to start saturation process.



Figure 12:Desiccators with positive displacement pump setup

- **Bench Top Permeability System Specification**

BPS-805 is system designed to test perform permeability tests at pore pressure up too 5000psi and confining pressure to 9950psi. It consists of low pulsation HPLC pump to deliver fluid at flow rates from 0.01ml/min to 10ml/min. Also it has a vertical hassler core holder which can accommodate core sample of 1.5 inch to 4 inches. Having a manual bypass valve to balance pressure on the transducer. A dome-loaded backpressure regulator is used to keep downstream elevated pressure as desired. Pressure transducer was used to measure the pressure drop across the core.

- Positive Displacement Pump
- Core Holder can accommodate a core with 3in length and diameter of 1.5in to 4 in
- Pressure Transducer
- Back pressure valve
- Reservoir and core valve
- Inline and bypass valve
- Personal computer to display the reading



Figure 13: Benchtop permeability system setup

### **3.5.2.1 Materials**

- ✓ Sandstone core sample
- ✓ Organic polymer as DRA
- ✓ Brine
- ✓ Distilled water

### **3.5.2.2 Benchtop Permeability System Experiment Procedure**

1. The three Core samples were saturated in brine solution containing salinity of 10000ppm. The saturation process was conducted by using desiccator equipment and positive displacement pump to ensure that the cores are fully saturated with brine. For a better result core samples were saturated for 8 days.
2. DRA solution is prepared by mixing 0.05g of DRA with 100ml of brine. After mixing the DRA with brine, the solution undergoes mixing process for at least 8 hours using magnetic stirrer in order to generate a broken solution that can be categorized as homogenous solution.
3. New DRA solution was prepared before each run to avoid shift time degradation.
4. The core will be deposited into the holder in permeability bench system, and then it will be flooded with brine at three different flow rates 1ml/min, 3ml/min, and 5ml/min for a duration of 60 minutes for each run.
5. First core sample is placed in the core holder and then the pump is switched on to flood the core with brine solution at 1ml/min for 60 minutes.
6. The second run will be executed right after the first run at which the core sample will be flooded with brine containing 50ppm of DRA at the same flow rates for 1 hour.



7. After that, the core will be flooded with brine in reverse direction (backflow process) at a flow rate of 8ml/min for 15 minutes to restore the permeability back and then it will be flooded again with brine at 1ml/min for 1 hour in order to achieve the final permeability of the core.
8. To continue the test for the remaining two core samples, step 5, 6, and 7 should be repeated only injection rate will be changed to 3ml/min for the second core sample, and 5ml/min for the third core sample. Finally permeability vs time plot will be generated on PC screen for each run.

### **3.5.3 DRA Mechanical Shear Degradation Test**

Mechanical degradation of biopolymer is studied using viscometer device. The use of viscometer is basically used to measure the viscosity of any solution at different shear rates. To conduct the test successfully user is required to run each sample immediately after preparation to avoid chemical degradation. The required volume of DRA solution for each run must not be less than 500ml, because it says in viscometer manual that any volume less than 500ml should be avoided.

#### **3.5.3.1 Sample Preparation**

In this experiment 6 sample must be prepared, 3 of these samples having a concentration of 0.5% of DRA, and the remaining 3 samples contain a concentration of 1% of DRA (biopolymer). The 0.5% and 1% solutions are prepared as follows:

##### 0.5% DRA solution

$$\text{Concentration (\%)} = \frac{\text{mass of DRA (g)}}{\text{Volume of distilled water}} \times 100\%$$

$$\text{Concentration (\%)} = \frac{0.5\text{g}}{100\text{ml}} \times 100\%$$

$$= 0.5\%$$

### 1.0% DRA solution

$$\text{Concentration (\%)} = \frac{\text{mass of DRA (g)}}{\text{Volume of distilled water}} \times 100\%$$

$$\begin{aligned}\text{Concentration (\%)} &= \frac{1g}{100ml} \times 100\% \\ &= 1.0\%\end{aligned}$$

### **3.5.3.2 Equipment Used**

- Viscometer
- 800ml beaker
- Magnetic stirrer

### **3.5.3.3 Viscometer Test Producer:**

1. Switch on the viscometer and connect the spindle to the viscometer itself.
2. Put the solution into 800ml beaker and then lower the spindle until it fully immersed in the solution. After that setup the desired shear rate you want to use and then click the button again to save your set up.
3. The first run is executed by putting a 500ml of 0.5% solution of Biopolymer into 800ml beaker and then setup the shear rate at 500rpm and click on button to start the test. The original viscosity of the solution is noted down at 0rpm. The test for each sample is conducted for 1 hour, but the author is used to note the viscosity value of the solution after 15 minutes (i.e. after 15 min, 30 min, 45min, and 60min).
4. The second run will be executed as same as the first run in step 3, the only thing will be changed is the shear rate at which it will be changed to 1000rpm.

5. The third run will follow the same procedure as in the previous steps only shear rate will be changed to 1500rpm.
  
6. Afterward the new solution of 1% concentration will be used and it will be executed at three different shear rates, 500rpm, 1000rpm, and 1500rpm. This means step 3, 4, and 5 will be repeated to complete the test successfully. For example, first run of 1% concentration will be tested at 500rpm, followed by second run at 1000rpm, and finally third run at 1500rpm. The original viscosity of the solution will be determined at 0rpm, then after disturbance with these 3 shear rates viscosity will be taken after each 15 minutes for a duration of 1 hour.

### 3.6 Gantt Chart (Timeline)

NO	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project	■	■	■											
2	Preliminary Research Work - Conducting online research - Literature review				■	■	■	■							
3	Submission of Extended Proposal								●						
4	Proposal Defense - Preparation for presentation									■					
	Lab Reservation to conduct the testing - Purchasing Chemicals										■				
5	Project work Continues											■	■		
6	Submission of Interim Draft														●
7	Submission of Interim Report														●

Figure 14: Gantt chart FYP1

No	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Project Work Continues - Preparing Progress report	■	■	■	■	■	■	■							
2	Submission of Progress Report								●						
3	Project Work Continues - Evaluate the results - Explore the discussion									■	■	■	■		
4	Pre-SEDEX											●			
5	Submission of Draft Report												●		
6	Submission of Dissertation													●	
7	Submission of Technical Paper													●	
8	Oral Presentation														●
9	Submission of Project Dissertation (Hard Copy)														●

Figure 15: Gantt chart FYP2

Remarks:

●	Deliverability
■	Work Progress

### 3.7 Project Key Milestones

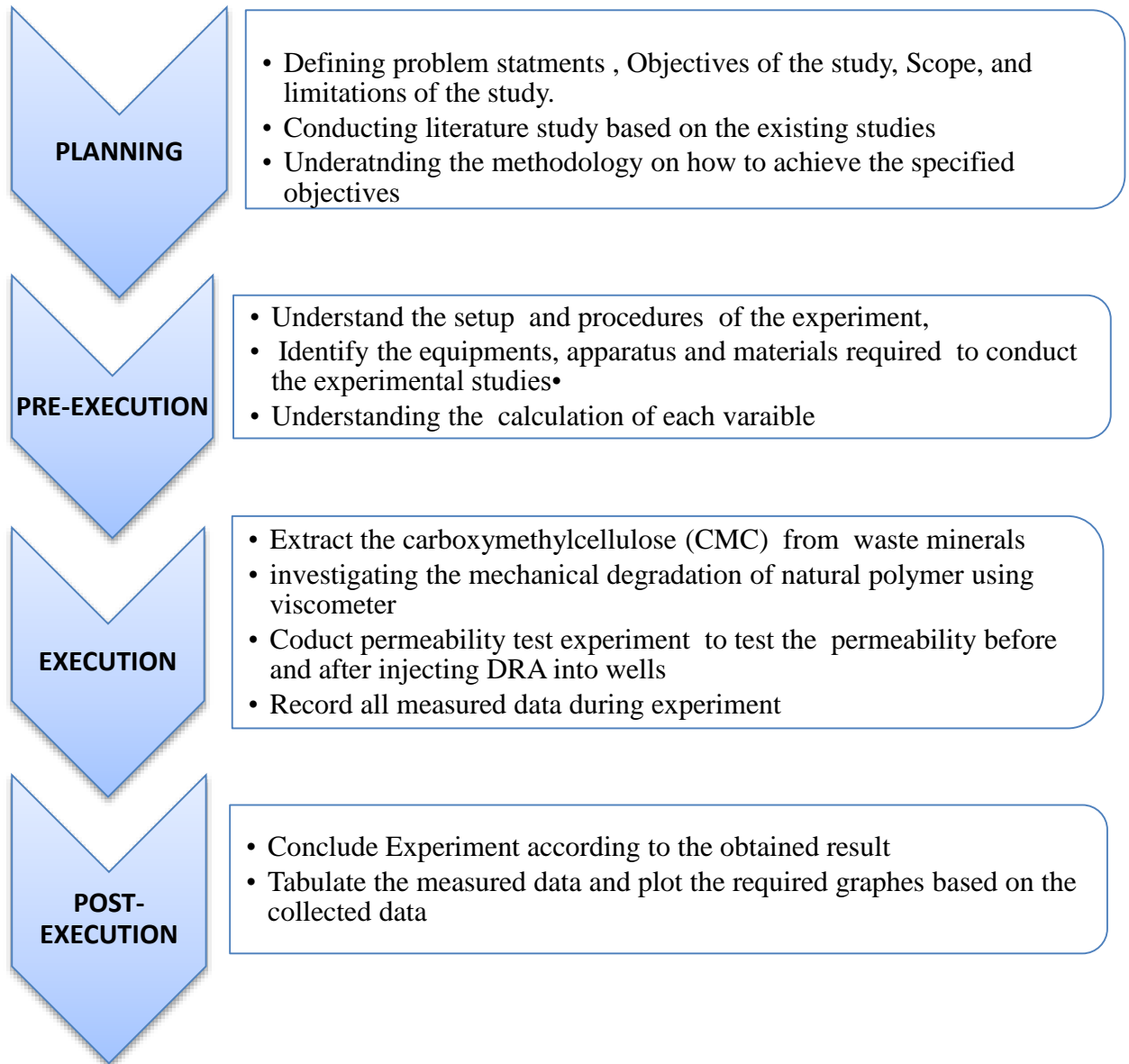


Figure 16:Project Key Milestones

## **CHAPTER 4**

### **RESULTS AND DISCUSSIONS**

This chapter illustrates all obtained results from experimental studies and laboratory activities. The results are going to be generated after running the samples; however there are some results which are calculated manually. Before conducting any test, it is very essential to define the variables to be used in the experimental studies in order to target the study to the desired objectives effectively. As dealing with experiment work there must be some variables that are unchangeable and some are changeable as listed below.

#### **4.1 Variables.**

##### **4.1.1 Constant Variables**

- i. Volume of brine used during permeability test (1000ml)
- ii. Mass of DRA added to the brine solution (0.05g)
- iii. Concentration of brine 10000ppm (1%)
- iv. Permeability test time (1hour for each run)
- v. Concentration of NaOH (40%)
- vi. Mechanical degradation test time ( 1 hour for each run)

##### **4.1.2 Changeable / Manipulated variables**

- i. Injection rate in permeability system (1cc/min, 3cc/min, 5cc/min)
- ii. DRA reaction time ( 60 minutes, 120 minutes)
- iii. DRA heating temperature ( 50°C , and 55°C)
- iv. Shear rate of Viscometer (500rpm, 1000rpm, and 1500rpm)
- v. Concentration of used samples during degradation test (0.5%, and1%)

### **4.1.3 Parameters of interest**

- i. Extracted mass of CMC (in grams)
- ii. Permeability before introducing DRA and after introducing DRA
- iii. Viscosity of the DRA solution before/after exposing to mechanical stirring speed to evaluate mechanical degradation of biopolymer.

## **4.2 CMC Extraction data**

The extraction procedure of CMC is explained in detail in the methodology. This chapter is going to show the obtained results after finishing the test and describe the significant behind changing the three parameters which are concentration of NaOH, reaction temperature, and reaction time. Meanwhile, it was observed that at certain reaction temperature and reaction time different mass of CMC is produced. However, there was a study conducted about synthesizing biopolymer from coconut residue which gave the author a general sense of the values of the three parameters that can result in a higher mass of CMC. According to Ammar, (2014) a 40% concentration of NaOH is more likely to produce a high mass of CMC, so it should be used as the typical concentration during extraction process in order to save time and enable the author to have sufficient time for executing the remaining objectives. Yet the author is going only to manipulate with reaction temperature and time as summarized in the following table.

Table 3: Collected Data From The Experiment

Run No	Concentration Of NaOH(%)	Reaction Temperature (°C)	Reaction Time (min)	Extracted Mass of Carboxy-methylcellulose (g)
1	40	50	60	26.872
2	40	50	60	27.100
3	40	50	60	23.665
4	40	50	120	22.030
5	40	55	60	19.511
6	40	55	240	24.745

The above table 3 summarizes the achieved CMC mass in each run at certain condition. It can be clearly seen that, the concentration of NaOH does not change in all 6 runs, because it has been proven in previous study by UTP student to be the applicable concentration that high CMC mass can be collected at temperature of 50°C, and time of 60 minutes; regardless to temperature and reaction time. The table also shows that as the heating time increases from 60 to 120 minutes the extracted mass of CMC will be less. Similarly, if reaction temperature increases to 55°C while the time remains constant, the extracted mass of CMC will drop to about 19.511g due to Cellulose degradation which usually degrades as temperature increases. Another reason behind the reduction of produced mass of CMC might be due to the degree of substitution of samples. For the first and second samples the author does not change any of the parameters, but the surprise was that the produced mass at the first run is 26.872g while at the second run is 27.100g of CMC. This is most probably, due to the oven temperature it was not constant at 50 °C ,since other people are using it at the same time, so when they open the oven and put their samples this will actually affect the temperature value to reduce a bit and result out in a slight different in the collected reading of CMC mass. As a result, the lower the reaction temperature and time, the higher mass of CMC will be obtained.



### **4.3 Benchtop Permeability System Test at Different Injection Rates**

The evaluation of the effect of DRA on permeability of formation was conducted by utilizing BenchTop Permeability system. The procedure of conducting this test was explained in details in the methodology. However, there are some factors that shall be considered before executing the test. First factor, it is required to measure the used core samples permeability, so the author did measure the core samples permeability before running the experiment, and found out these cores have a low permeability ranging from 8md to around 27md. Second factor that is highly required, is about core samples saturation where the three cores undergoes a saturation process for 8 days using desiccators to ensure each core sample was 100 percent saturated for achieving better results. Once adding 0.05g of DRA into brine solution, it must be exposed to stirring process using magnetic stir for at least 8 hours to create a homogenous solution.

In this section, first core sample was flooded with brine at 1ml/min injection rate for one hour, afterward it was flooded again with DRA solution at the same injection rate, and then back flow process was run at high injection rate of 8ml/min for some time. Followed by brine injection rate at 1ml/min in order to obtain the final permeability reading. Same procedure will be repeated for core 2, and 3 but at different injection rate 3ml/min, and 5ml/min respectively. After 1 hour, all permeability readings against time will be recorded automatically by bechtol permeability system.

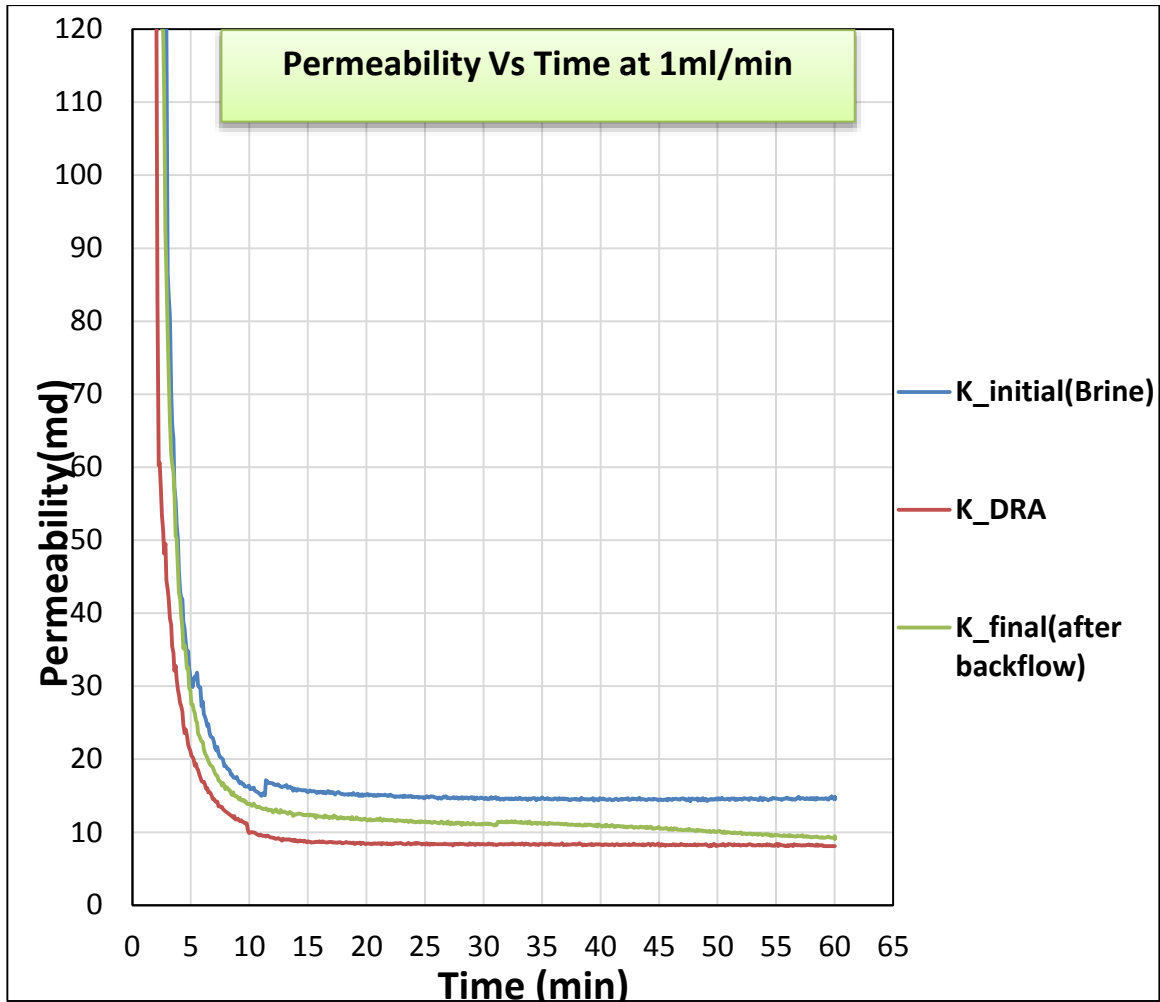


Figure 17:DRA Performance at 1ml/min

Figure 18 shows that, once the core is initially flooded with brine at 1ml/min, the stabilized permeability was recorded at 14.565md after 1 hour of the test. However, it dropped to a value of 8.119md after flooding the core with DRA solution at the same injection rate. This indicates there is going to be a permeability reduction if DRA is injected at 1ml/min. The core is then reversed and back flow process was carried out at 8cc/min for 15 minutes in order to restore the permeability. Afterwards, the core is flooded with brine again for 1 hour at 1ml/min to obtain the final permeability; hence final permeability was recorded to be 9.122md. This shows around 15.51% of permeability was restored back. As the permeability stabilizes pressure profile becomes constant.

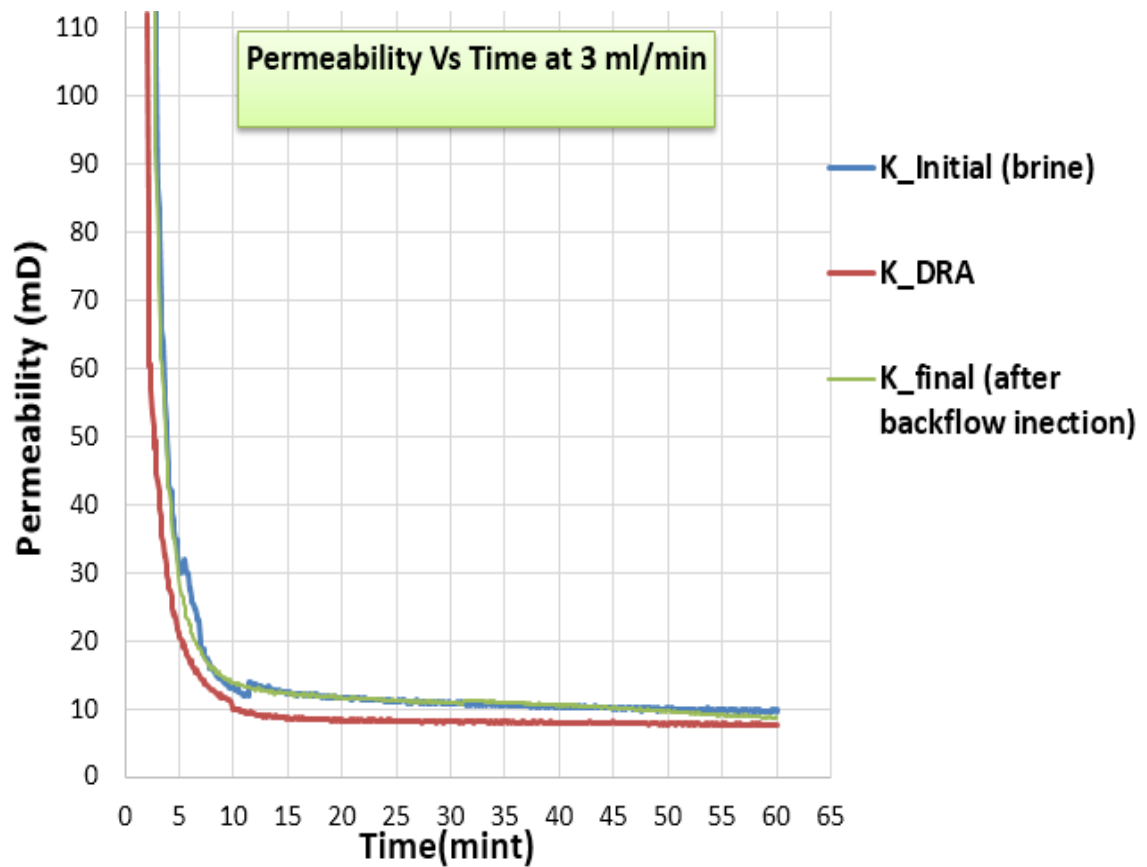


Figure 18:DRA Performance at 3ml/min

According to the figure 19 above, the core sample was flooded continuously with brine at 3ml/min for exactly 1 hour. After one hour it can be seen that, the permeability reading becomes constant at 10.131md. Then the core was flooded with DRA solution at the same injection rate at which permeability reduced to 7.65md. A reduction of 25.37% on permeability is observed. To achieve the final permeability core was reversed and a back flow process was carried out at 8ml/min for 15 minutes in order to restore the permeability as illustrated in the above graph. The core was then flooded with brine again at 3ml/min for 1 hour and a final permeability was stabilized at 8.347md which indicates around 30.34% of permeability is recovered. As a result of this graph as injection rate increases permeability reduction percentage decreases.

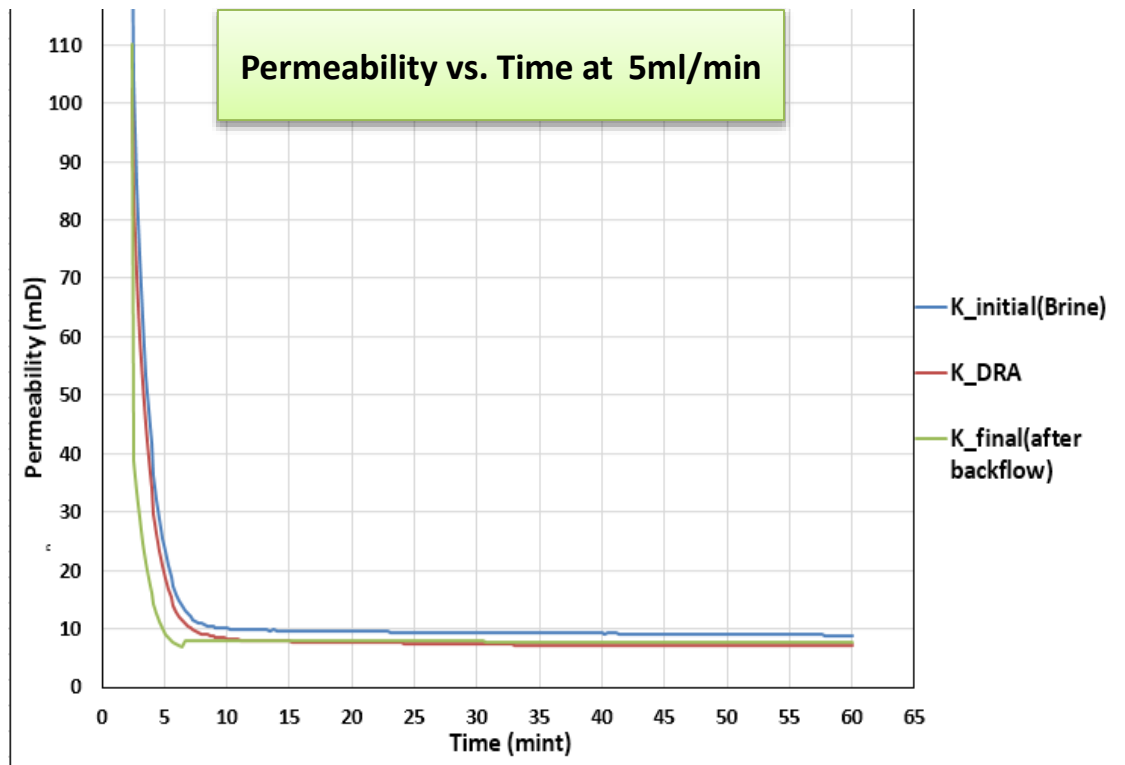


Figure 19:DRA Performance at 5ml/min

The above figure 20 illustrates the permeability reading at injection rate of 5ml/min where 3 test were carried out for 1 hour. First run, the core was flooded with brine at 5ml/min until a stabilized value of permeability is detected at 8.739md. Second run, the same core was flooded with DRA solution at the same injection rate and a reduction of permeability is observed. After 1 hour of flooding the core with DRA the permeability decreased to 7.037md. To restore permeability back, a backflow process is utilized at injection rate of 8ml/min for 15 minutes. After the inverse flooding process the core was flooded with brine again at 5ml/min and the final permeability was recorded to be 7.607md after 1 hour. Only 19.47% of permeability reduction is attained, however a higher recovered permeability value of 43% is observed. This means, permeability reduction can be overcome by increasing the injection rate of DRA, so at a higher injection rate there will be insignificant permeability reduction substantially.

### 4.3.1 Calculation of Permeability Reduction & Recovered Permeability in Percentage

Table 4: Observed Permeability after 60 minutes at 1,3,&5ml/min

	Injection Rates		
	1ml/min	3ml/min	5ml/min
<b>K_Intital</b>	14.586	10.131	8.739
<b>K_DRA</b>	8.119	7.560	6.760
<b>K_final</b>	9.122	8.347	7.607

Permeability Reduction and Permeability Recovered Calculation

#### For 1ml/min

$$K_{REDUCTION} = \frac{K_{initial} - K_{DRA}}{K_{initial}} \times 100\% \quad \dots \dots \dots (3)$$

$$K_{REDUCTION} = \frac{14.586 - 8.119}{14.586} \times 100\%$$

$$K_{reduction} = 44.34\%$$

$$K_{recovered} = \frac{K_{final} - K_{DRA}}{K_{initial} - K_{DRA}} \times 100\% \quad \dots \dots \dots (4)$$

$$K_{recovered} = \frac{9.122 - 8.119}{14.586 - 8.119} \times 100\%$$

$$= 15.51\%$$

**For 3ml/min**

$$K_{REDUCTION} = \frac{K_{initial} - K_{DRA}}{K_{initial}} \times 100\%$$

$$K_{REDUCTION} = \frac{10.131 - 7.560}{10.131} \times 100\% \\ = 25.378\%$$

$$K_{recovered} = \frac{K_{final} - K_{DRA}}{K_{initial} - K_{DRA}} \times 100\%$$

$$K_{recovered} = \frac{8.347 - 7.560}{10.131 - 7.560} \times 100\% \\ = 30.61\%$$

**For 5ml/min**

$$K_{REDUCTION} = \frac{K_{initial} - K_{DRA}}{K_{initial}} \times 100\%$$

$$K_{REDUCTION} = \frac{8.739 - 7.037}{8.739} \times 100\% \\ = 19.476\%$$

$$K_{recovered} = \frac{K_{final} - K_{DRA}}{K_{initial} - K_{DRA}} \times 100\%$$

$$K_{recovered} = \frac{7.61 - 6.76}{8.74 - 6.76} \times 100\% \\ = 43.00\%$$

Table 5: Summary of permeability reduction and recovered percentage

Injection Rates (ml/min)	K_reduction	K_recovered
1	44.337	15.510
3	25.378	30.611
5	19.476	42.789

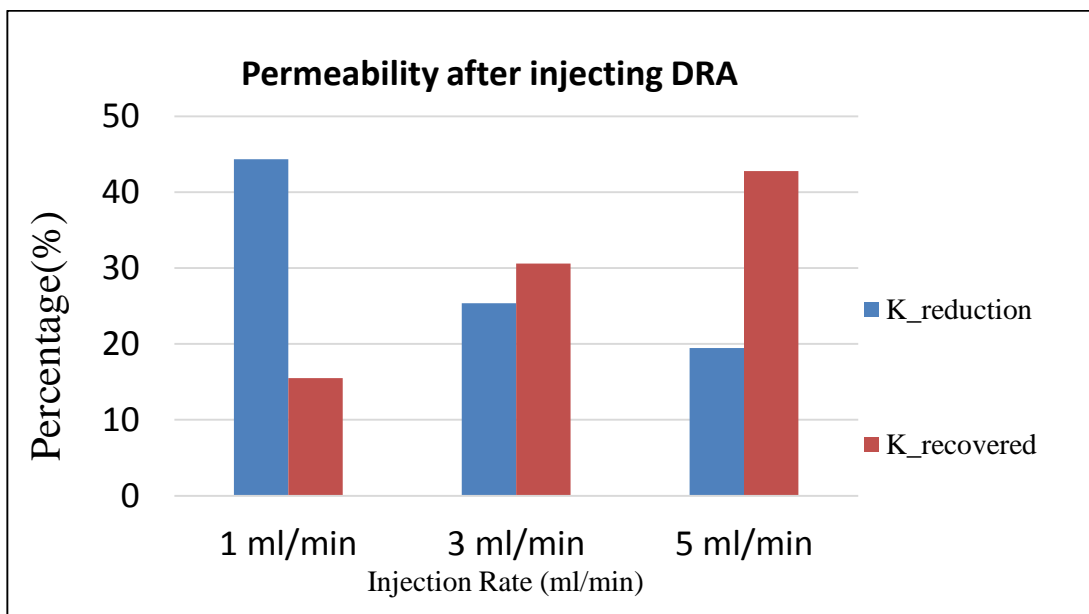


Figure 20: Permeability reduction and recovered versus injection rate of Biopolymer DRA Solution

According to the above bar chart, it can be clearly seen that, permeability reduction is a function of injection rate. For instance, the highest permeability reduction is achieved at 1ml/min injection rate which is around 44.34%, whereas the lowest permeability reduction was observed at 5ml/min injection rate which is 19.47%. In contrast, the recovered permeability is dependent on the injection rate where it raised to 43% at 5ml/min injection rate, followed by 30.34% at 3ml/min, and finally it dropped to 15.54% at 1ml/min. As a result, permeability reduction decreases with increasing the injection rate of the system, and the vice versa for the recovered permeability.

#### 4.4 The Influence of Mechanical effects on Biopolymer Mechanical Degradation

The influence of mechanical effects on biopolymer that is utilized as DRA in this study was investigated by exposing the solution of 0.5% concentration and 1% concentration of DRA to a high mechanical stirring speed of 500rpm, 1000rpm, and 1500rpm using viscometer device. During exposing each sample to these shear rates viscosity values were noted down after each 15 minutes where the test of each sample is last for one hour. The prepared sample of 0.5% is stirred at stirring speed of 500rpm for a duration of one hour and the effect of the shear rate on viscosity is observed and recorded as summarized in the following table.

Table 6: Viscosity of 0.5% solution after exposing to different shear rate using viscometer

Time(min)	Viscosity (cp) at 500rpm	Viscosity (cp) at 1000rpm	Viscosity (cp) at 1500rpm
0	54.62	54.62	54.62
15	53.41	43.48	30
30	52.03	35.11	21.53
45	49.36	30.84	18.78
60	47.56	28.07	12.09



By utilizing the data in the above table, the following plot of viscosity versus time is generated.

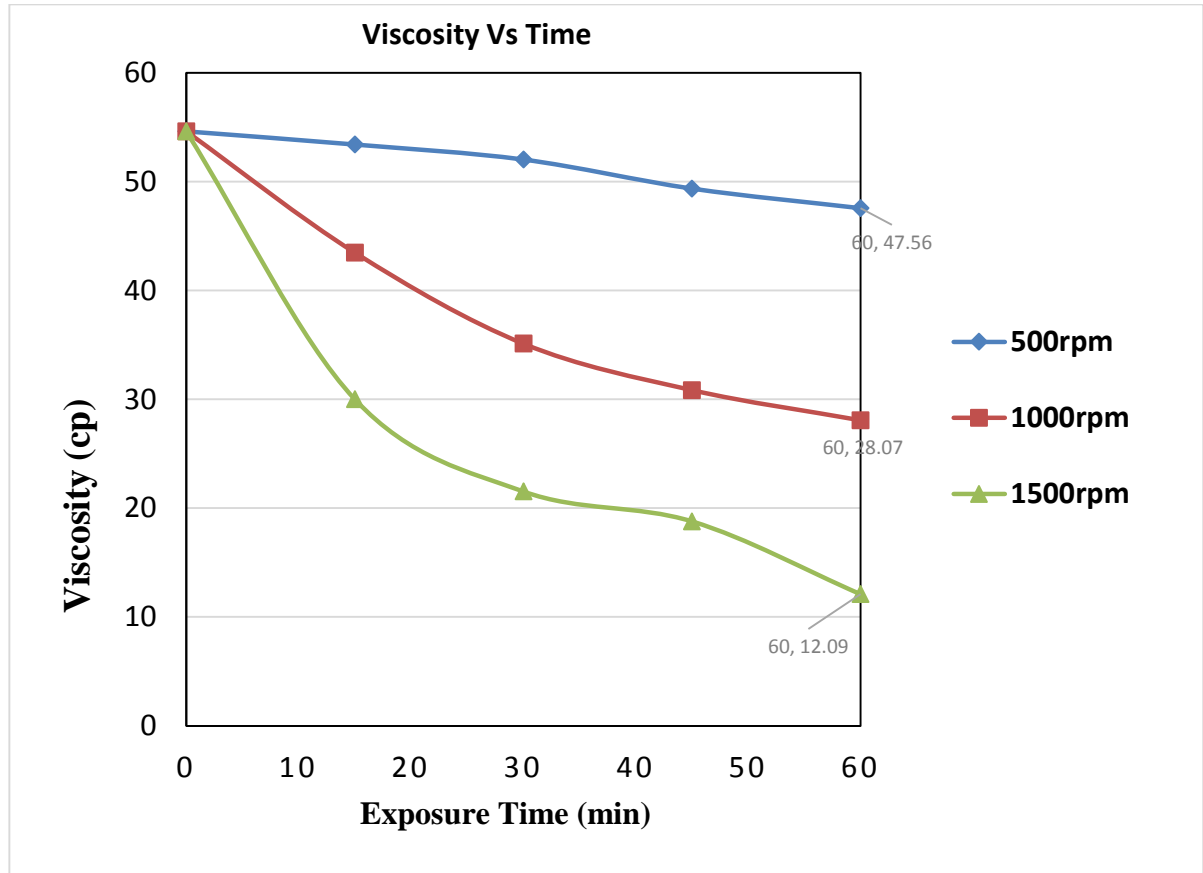


Figure 21: Effect of exposure time and stirring speed on the viscosity of 0.5% biopolymer solution.

From figure 22, the original viscosity of 0.5% solution is 54.62 cp before disturbance. After exposing the sample to viscometer of 500rpm shear rate the viscosity decreases gradually to 47cp due to mechanical degradation. As the stirring speed of viscometer increases a high reduction on viscosity will be achieved. For example, at shear rate of 1000rpm viscosity dropped to 28cp and once the shear rate changed to 1500rpm the viscosity reduced dramatically to 12cp due to the high stirring speed which impacts the rheological behavior of biopolymer and causing a breakage in the chemical chain of the biopolymer.

Table 7:Viscosity of 1% solution after exposing to different shear rate using viscometer

Time(min)	Viscosity (cp) at 500rpm	Viscosity (cp) at 1000rpm	Viscosity (cp) at 1500rpm
0	113.03	113.03	113.03
15	106.81	70.08	48.64
30	96.69	57.15	34.29
45	91.25	54.76	23.17
60	87.07	40.23	16.09

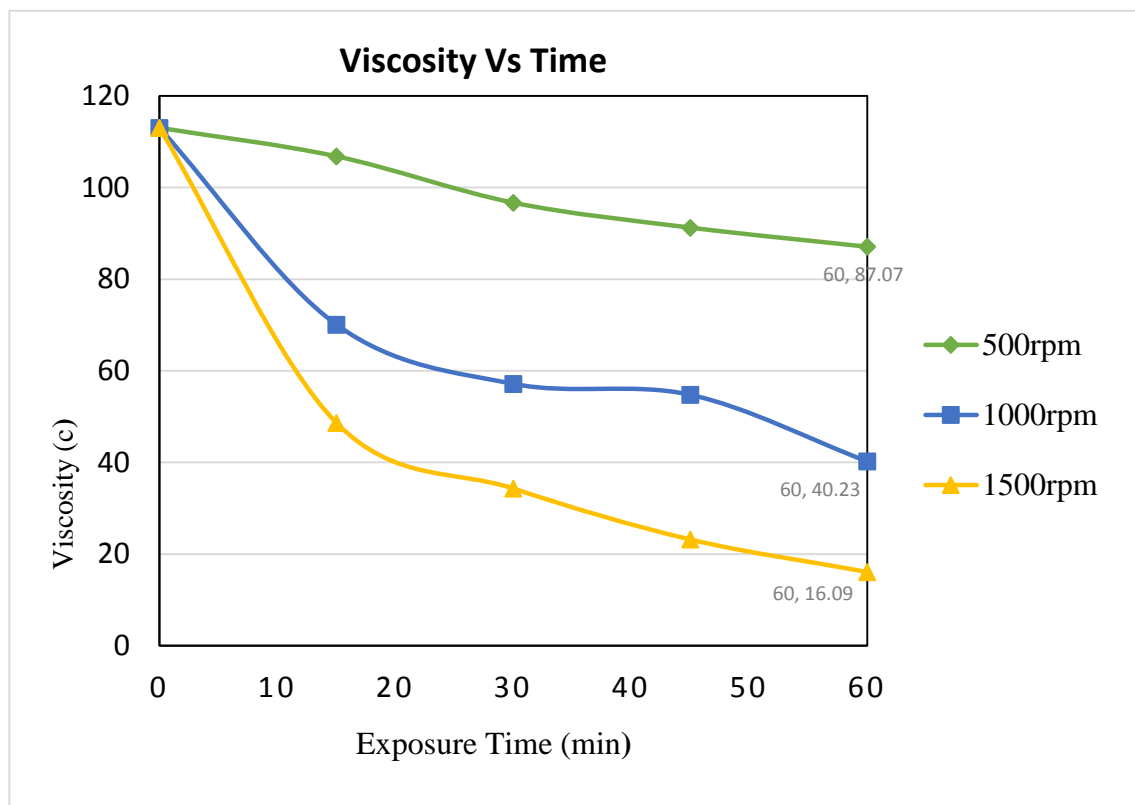


Figure 22: Effect of exposure time and stirring speed on the viscosity of 1% biopolymer solution.

The decrease of biopolymer solution (of 1% ) viscosity with increasing exposure time of shearing is illustrated as in figure 23. The experiment runs were carried out on 1.0% (w/v) concentration of biopolymer at three different shear rate 500rpm, 1000rpm, and

1500rpm, so it can be finalized from this figure a gradual lowering of viscosity at 500rpm is detected with exposure time due to mechanical shear degradation. At 1000rpm and 1500rpm a considerable reduction of viscosity is observed as exposure time increases due to the disturbance caused by viscometer which results out in a mechanical degradation of biopolymer continuously. After exactly 60min the viscosity at 1000rpm dropped dramatically to 40.23cp. Similarly, at 1500rpm the viscosity of the solution changed dramatically from 113.03 cp to 16.09 cp after 60 minutes. This is can be conclude by saying that as the molecular weight concentration increases a faster mechanical degradation will result out as investigated above for 0.5% and 1.0% concentrations.

#### 4.4.2 Viscosity Reduction Calculation

Viscosity reduction percentage %RV is calculated by using the following equation;

$$\%RV = \frac{\mu_{before} - \mu_{after}}{\mu_{before}} \times 100 \% \dots\dots\dots (5)$$

Where:

$\mu_{before}$  = viscosity of solution before degradation

$\mu_{after}$  = viscosity of solution after degradation

#### **For solution of 0.5% concentration**

$$\%RV = \frac{54.62 - 53.41}{54.62} \times 100 \%$$

$$\%RV = 2.22\%$$

Therefore, the viscosity reductions of 0.5%, and 1.0% solutions are calculated and summarized as in the following tables:

Table 8: Percentage Viscosity Reduction of 0.5%

<b>Time</b>	<b>%RV @ 500rpm</b>	<b>%RV @ 1000rpm</b>	<b>%RV @ 1500rpm</b>
0	0	0	0
15	2.215306	20.39546	45.07506
30	4.741853	35.71952	60.5822
45	9.630172	43.53717	65.61699
60	12.92567	48.60857	77.86525

Table 9: Percentage Viscosity Reduction of 1.0%

<b>Time</b>	<b>%RV@ 500rpm</b>	<b>%RV @1000rpm</b>	<b>%RV @1500rpm</b>
0	0	0	0
15	5.502964	37.99876	56.96718
30	14.45634	49.4382	69.66292
45	19.26922	51.55269	79.50102
60	22.96735	64.40768	85.76484

From the data calculated in Table 8 and 9, the two plots below will be generated to describe the viscosity reduction due to mechanical effects on the prepared solution of 0.5% concentration of DRA.

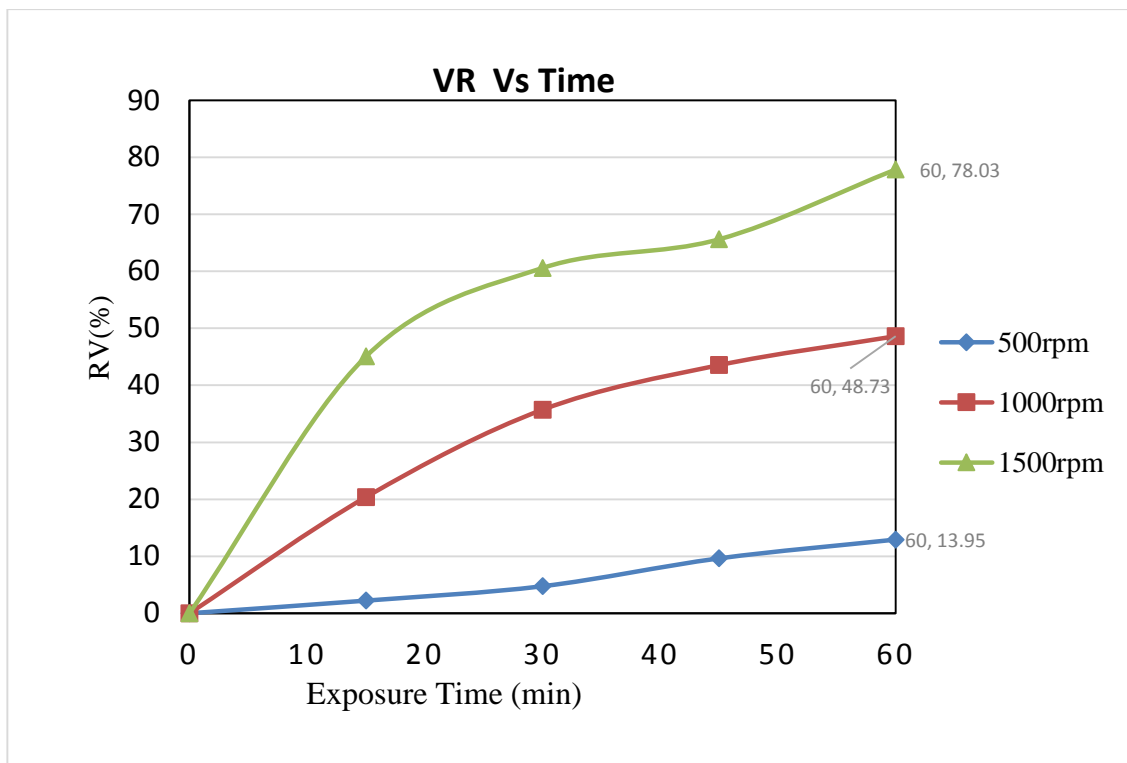


Figure 23:Effect of exposure time and shear rate on percentage viscosity reduction of 0.5% DRA.

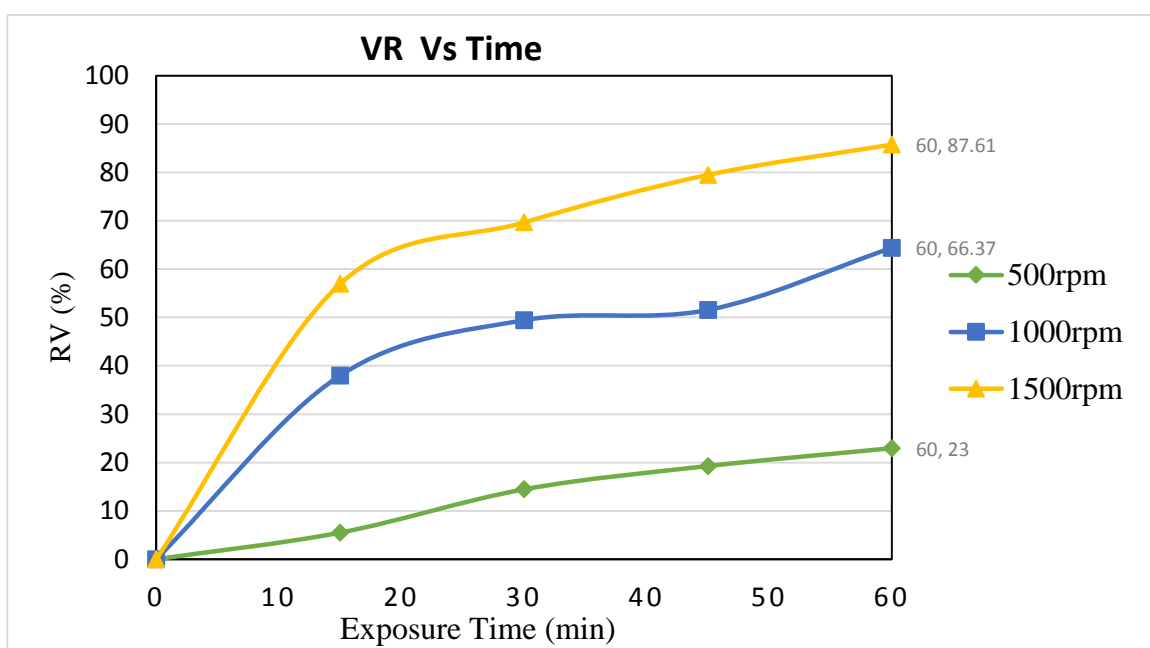


Figure 24:effect of exposure time and shear rate on percentage viscosity reduction of 1% DRA.

Biopolymer as a water soluble is proven to be an effective drag reducing agent (DRA) in turbulence flow regime. Therefore, it is essential to investigate the impact of mechanical forces on drag reducing agent performance. The above figures (24, & 25) show the percentage lowering of the viscosity is dependent on shear rate (stirring speed of viscometer) and exposure time. The reduction of viscosity of 0.5% DRA at 500rpm, 1000rpm and 1500rpm are 13.95%, 48.73%, and 78.03% respectively. At 1.0% concentration of DRA the percentage viscosity reduction jumped rapidly from 0% to 87.61% at shear rate of 1500rpm, this indicates that any solution of high concentration of biopolymer is more likely to undergo a faster mechanical degradation. The above two figures prove that biopolymer has a poor mechanical stability in turbulent flow, so it will lose its efficiency of acting as drag reducing agent faster to avoid this some chemicals should be mixed with biopolymer in order to enhance their mechanical stability in turbulent flow and last effective for longer time. The reduction in viscosity is higher as the shear rate is higher. For instant, the percentage viscosity reduction at 1500rpm jumped up suddenly to almost 57% after the first 15 which shows biopolymer is highly affected by mechanical disturbances towards mechanical degradation with time. As a result of the above two figures, mechanical degradation rate increases as stirring speed of viscometer and exposure time increase.

The obtained percentage value shows a gradual decrease of the viscosity with exposure time and stirring speed due to degradation of organic polymers. As the controlled parameter will the angular speed, so degradation at high stirring speed occurs more rapidly than that at low stirring speed, thereby indicating that when the stirring speed increases, mechanical degradation plays an important role.

## CHAPTER 5

### CONCLUSION AND RECOMMENDATION

#### 5.1 Conclusion

Introducing DRA into oil and gas industries has played an important role in resolving pressure reduction problems in water injection well and dampening turbulent flow. The Extracted CMC from coconut residue know as biopolymer has been proven to be as effective as synthesizing polymer in minimizing pressure drop and enhancing the productivity of the system. In this study, it has been observed that the amount of produced CMC from coconut residue is controlled by the reaction parameters such as temperature, time and concentration of sodium hydroxide temperature in which different CMC mass was collected with the change of reaction temperature and reaction.

In the other hand, DRA helps in optimizing the water capacity in water injection well; however it cause some reduction in permeability at the same time. Precaution need to be taken to ensure that the reduction in permeability is negligible. As a result from permeability test, it has been observed permeability reduction is a function of injection rates; high injection rate has low reduction in permeability and a higher permeability can be recovered as injection rate increases.

Mechanical degradation refers to the chemical process by which the effectiveness of biopolymer as DRA is weakened by mechanical stirring forces since these action will result in breakage in biopolymer chain. The mechanical degradation of biopolymer was studied by exposing two dilute solutions of 0.5% and 1.0% to high mechanical stirring. It was observed that the susceptibility of biopolymer to degradation increases with increasing the concentrations, stirring speed and the exposure time. As a result of this test a gradual decrease of viscosity is observed with time progression due to the poor

mechanical degradation of biopolymer. The efficiency of biopolymer as DRA will decrease dramatically with time due to shear degradation of biopolymer molecules once exposed to a turbulent flow process.

## **5.2 Recommendations**

As working in this research, there are some limitations and some other difficulties which have been experienced by the author. For future continuation of research and development purposes with regards to this research the author has finalized the following recommendations that can be considered for furthering this study in the future.

1. Studying the morphology of CMC by using FESEM in order to figure out its rheological behavior.
2. Identifying how long it takes for DRA to degrade across the pipeline, and determining the length of the pipeline that each injected batch of DRA can still act as effective agent in overcoming the frictional forces.
3. Injection skid should be added up to the equipment at the injection point to avoid any back flow of the DRA.
4. Using CT scan for the core samples in order to observe the interior behavior of the formation permeability reduction as time goes.
5. Mechanical degradation should be evaluated at different temperatures and compare it with mechanical degradation covered in this study at ambient conditions.
6. To determine the best point in the pipeline where the turbulent force happens so that the performance of DRA is known.
7. Usage of Sodium lauryl ether sulfate with Organic polymers to enhance their mechanical degradation.



## CHAPTER 6

### REFERENCES

1. Toms, B.A., 1948. Some observations on the flow of linear polymer solution through straight tube at large Reynolds numbers. In: Proceedings First Congress in Rheology, North Holland, Amsterdam, vol.2, pp. 135-141.
2. Karami, H.R., and Mowla, D. (2012). Investigation of the effects of various parameters on pressure drop reduction in crude oil pipelines by drag reducing agents. Journal of Non-Newtonian fluid mechanics. Shiraz, Iran.
3. Burger, E.D., and Wahl, H.A. (1982). 'Flow increase in the Trans-Alaska pipeline through use of polymeric drag reducing additives'. SPE 9419, Journal of Petroleum Technology.
4. De Gennes, P. 1990. Introduction to Polymer Dynamics. Cambridge University Press.
5. Lee, D.H. 2010. Drag Reduction by Dilute Polymer Solution. University of Michigan.
6. Campbell, S.E., and Jovancicevic, V., "Performance Improvements from Chemical Drag Reducers", SPE 65021 (paper presented at the 2001 SPE International Symposium on Oilfield Chemistry, Houston, Texas, 13-16 February 2001).
7. Kelland, M.A., "Drag-Reducing Agents," in Production Chemicals for Oil and Gas Industry, CRC Press, 2003.
8. Cengel, Y., & J. M. (2006). 'Fluid Mechanics: Fundamentals and Applications'. McGraw Hill.

9. J. Lumley, J. "Drag reduction in turbulent flow by polymer additives". Z, 263 (1973) Drag reduction in turbulent flow by polymer additives
10. Prasetyo.I.(2003).”Drag reducer increases pipeline capacity to accommodate the successful of field development” .Presented at SPE Asia Pacific oil and gas conference and exhibition.Jakarta,Indonesia.
11. Kang.C,and Jepson.W.P.(1999).Multiphase flow conditioning using drag-reduction agents.SPE 56569.Annual Technical Conference and exhibition in Houston,Texas.
12. Joseph and J. A. Ajiienka (2010).”Use of Drag Reducer in Improving Water Injectivity in Ukpokiti Field”., Niger Delta, Leonardo Electronic Journal of Practices and Technologies.177-188
13. H. A. Al-Anazi, M. G. Al-Faifi, F. Tulbah, and J. Gillespie (2006).”Evaluation of Drag Reducing Agent (DRA) for Seawater Injection System”: Lab and Field Cases.100844. (SPE Asia Pacific Oil & Gas Conference and Exhibition, Adelaide,Australia, 11–13 September)
14. Gurchran,H.K,& Jaafar,A.(2013).”The study of drag reduction ability of naturally produced polymers from local plant source” .international petroleum technology conference.Beijing,China.IPTC 17207.
15. Katherine.P.H,Guido.D.S,Meghan.R,and Steve.Y.(2012). “application of nanotechnology in drilling fluids”.M-I SWACO. SPE 157031
16. Tchobanoglous, G., Theisen, H. and Vigil, S.A. 1993. Integrated solid waste management: Engineering principals and management issues, p. 3-22. New York: McGraw-Hill.
17. Pascual.B. 2009. Biopolymers, plastic of the future. Sarrià Chemical Institute.
18. Jennifer .N. 2003. “Optimising production using drag reducing agents in water injection wells”. ConocoPhillips Specialty Products Inc.
19. B.C, Lester., 1985 . Basics of drag reducing. Oil and Gas Journal, February, P. 51-56.
20. Culter, J.D., Zakin, J.L., Patterson, G.K.: "Mechanical Degradation of Dilute

21. Solutions of High Polymers in Capillary Tube Flow ", J. Appl . Polym. Sci., 19, p.32253240, 1975.
22. Matthew W. Liberatore, Seunghyun Baik, Anthony J. McHugh and Thomas J. Hanratty" Turbulent drag reduction of polyacrylamide solutions: effect of degradation on molecular weight distribution", J. Non-Newtonian FluidMech. 123 (2004) 175–183.