Effect of Temperature on the Extraction of Biopolymer from Coconut Residue and the Performance as Drag Reducing Agent in Water Injection System: A Comparative Study

by

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Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
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Universiti Teknologi PETRONAS Bandar Seri Iskandar 31750 Tronoh Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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(PETROLEUM)

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May 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.

(AMMAR THAQIF BIN ABDUL RAHAMAN)

ABSTRACT

Synthetic and chemically synthesized polymers manufactured as Drag Reducing Agent (DRA) have been proven to be successful in minimising the effect of frictional pressure drop along injection tubing of water injection system. Several studies have been done to find alternatives on DRA sources from natural and biodegradable polymers such as Carboxymethylcellulose (CMC) which are more environmental friendly yet performing as good as the synthetic polymer which in this case is Polyacrylamide (PAM). In this study, CMC were extracted from dried grated coconut flesh after the extraction of coconut milk or so called Coconut Residue (CR) by synthesizing the cellulose under the alkali-catalysed reaction with monochloroacetic acid. 27 samples of CMC were extracted by varying the reaction parameters of temperature and the relationship with sodium hydroxide (NaOH) concentration and reaction time with the highest yield of CMC recorded at 28.991 grams at the highest reaction parameters. Drag Reducing Agent (DRA) performance were tested by utilising laboratory scale flowmeter test rig setup and manometer readings are recorded across and after the Orifice plate. From the flowmeter test, the samples extracted at the combination of highest temperature of 60°C, highest NaOH concentration of 60 wt%, and longest reaction time of 240 minutes have the highest Drag Reduction Percentage (%DR) of 21.79% as compared to Polyacrylamide which reach at best 1.28%. On the other hand, the combination of lowest extraction parameters of CMC from CR still have better %DR as compared to PAM at 2.59% and 0.86% respectively. The successful of this research indicated that the CMC extracted from CR can performed as DRA better than PAM at certain extraction parameters which will then be a kick-start to transform wastes to wealth.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Turbulent flow regime of water in water flooding system and pipeline induced drag along the inner wall of the pipeline making it difficult to flow thus increased the pressure drop along the injection tubing and the pipelines respectively. For decades, the industry has countered this issue by modifying their injection tubing and pipelines diameter to be smaller in sizes. Besides, higher pumping power pumps are installed replacing the low rated pumps to boost the injection capacity and pressure. Undoubtedly these methods have been a proven success in order to maintain the flow rate and reduced the pressure drop in the system but still not solving the presence of drag in turbulence flow along the pipelines. [1]

DRA on the other hand able to reduce the drag effect in turbulent flow by acting as a conditioner or buffer at the injection tubing and pipeline inner wall surface which in direct contact with turbulent flowing water. Introduction of conventional DRA used in industry for decades are made of polymer progressed by the study on adding polymers in solvents done by Toms in 1948 to investigate the reduction of frictional pressure drop in pipelines. [2]

In general, polymers can be divided into two (2) major groups which are synthetic polymers and organic polymers or so called biopolymers. Synthetic polymers are chemically produced which involved sophisticated equipment and complex formulation resulting higher manufacturing cost while organic polymers come from various natural resources such as plants and animals. Extensive studies and researches have been made to find the best candidates of organic polymers which will come out as a green and environmental friendly DRA yet have the ability and efficiency of synthetic polymer for drag reduction in water injection system and pipelines as well.

Organic polymers from wastes are chosen due to their abundant availability and better potential turning them from waste to money.

1.2 Problem Statement

Increased in drag in the water injection system is due to turbulence flow which substantially increase the frictional pressure drop along the injection tubing. Increased in frictional pressure drop will substantially decrease the injection tubing throughput and flow capacity. The direct effect will be less capacity of liquid flow and at the same increased the operating pressure.

Addition of high rated pumps with greater pumping power and capacity will eventually maintained the flow rate and reduced the pressure drop significantly but will results in higher operating costs due to increase in energy consumptions and maintenance costs as well with increasing number of pumps in operation simultaneously. The additional pumps will add more pressure into the injection system and thus raise the risk of injection tubing burst since the increased in pressure will stressed the integrity of the tubing itself which will be closer to the Maximum Allowable Operating Pressure (MAOP).

Proven to be a remarkable success in reducing drag in turbulent flow, extensive usage of synthetic polymers such as Polyacrylamide (PAM) in the industry however have ring the alarm of increasing risks on the adverse effects towards human and polluting the environment with the degradation of its chemical components thus increased the need to find a better replacement and greener solution.[3]

This is where the idea of utilizing conventional organic polymer wastes comes from and turning the wastes into organic DRA. Extensive researches have been done on finding the best wastes candidates and proven to be successful in their own experiments but none have been made to study the potential of coconut residues (CR) as DRA.

1.3 Objectives

The main source of organic waste material that the author decided to be tested for this research is grated coconut waste or coconut residue (CR). The objectives of this research are:

- 1) To extract Carboxymethylcellulose (CMC) from CR at different reaction temperature.
- 2) To investigate the performance of the extracted CMC as DRA when compared with commercialized synthetic DRA (Polyacrylamide) based on Drag Reduction Percentage (%) in a flowmeter experimental setup.

1.4 Scope of Study

The scope of this study will be focus more on evaluating the potential and effectiveness of biopolymer extracted from wastes in this case CMC from CR as compared to commercialized synthetic PAM in water injection system only. Due to time constraint, this study will not cover the effect of DRA with the formation or reservoir, the dynamic changes in reservoir temperature and pressure with depth as well as the chemical reaction between DRA and inner tubing wall.

Since this is an experimental study in the laboratory, the focus of the study is limited to test the effectiveness of the organic polymer extracted in the form of CMC to function as DRA with observed results on the drag reduction percentage from pressure drop across an Orifice plate with the flowmeter experimental setup. Further details of the experiment are discussed in Chapter 3. The rheology and characterization of CMC also not been carried out in this research which are the moisture content, degree of substitution (DS), and purity of CMC extracted from CR as they are not the parameter of interest in this study.

1.5 Project Relevancy and Feasibility

For the purpose of Final Year Research Project (FYP), this project is highly relevant and important not only to the oil and gas industry but also to the coconut industry in Malaysia. This is because this project is aimed to aid both industry in minimizing the frictional pressure losses in water injection tubing as well as in production pipeline while adding values to coconut industry by-products by utilizing them for the manufacturing of Drag Reducing Agent that will can be used in various industry especially oil and gas.

Furthermore, this project is highly feasible since all the equipment used is readily available in UTP besides of minimal usage of chemicals and thus low cost of overall research operation. On top of that, the same experimental procedure in this research is adopted from previous research conducted utilizing the same facilities and equipment provided in UTP. Besides that, the project timeline is within the budget and time allocated for FYP research.

CHAPTER 2 LITERATURE REVIEW

2.1 Drag Reduction and Turbulent Flow

2.1.1 Drag Reduction

The full definition of drag is something that is pulled or drawn along or over a surface, for this case the inner wall of injection tubing. [4] Drag reduction in tubing on the other hand is defined as the power recovering in the flow of fluid in the pipe by addition of small amount of chemical additives to a solvent in turbulent flow in order to decrease the pressure drop. [5] Based on Drag Reduction Theory, drag reduction occurs due to suppression of the energy dissipation by turbulent eddy currents near the pipe wall during turbulent flow. [6]

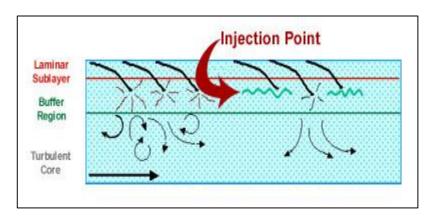


Figure 1: Schematic diagram on the drag reduction theory. [7]

Based on the study of Water Filtrate, Blatch observed a significant pressure drop when there are some additions of substances in the flowing fluid. [8] The statement of his report was further justified and confirmed by experiment conducted by Toms in 1948. A reduction of skin friction and pressure drop was observed in the experimental setup of a straight tube with added polymers in the flow at large Reynolds number. [2] Tom's

study has played an important and major role in introducing the term of drag reduction effect with extensive studies on varied source of polymers.

In oil and gas industry, drag usually exists in the streamline or flow of fluid inside the injection tubing or pipelines at high flow rate and high fluid velocity inside the tubing, which induced turbulent flow to form. When there is drag existed along the tubing inner wall which in immediate contact with the turbulence flow of fluid inside, there will be much more resistance for the fluid to flow thus contributes to the increase in pressure losses. In order to overcome the friction pressure losses, it is necessary to increase the pressure of the fluid and the easiest way to do this is pumping. [1]

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

$$DR(\%) = \frac{|\Delta P_f - \Delta P_{fDRA}|}{\Delta P_f} X 100 \tag{1}$$

Where ΔP_f , is the friction pressure drop without presence of DRA in the flowing liquid while ΔP_{fDRA} , is the friction pressure drop with the presence of DRA in the flowing liquid in the tubing. This formula has been used widely to check the direct relationship between the presence of DRA and resulting pressure drop in most of the research papers done recently. [1, 9, 10]

2.1.2 Turbulent Flow in Water Injection System

Water injection system in this case study is defined as the travelling path by the injection fluid from the injection pump to the end of injection tubing downhole in the wellbore before entering the formation. The only distinction between turbulent flow inside the injection tubing and the pipelines apart from the sizes are the position of the tubing and the pipe itself which are in vertical and horizontal position respectively. On top of that, the action of gravity favor the vertical position most. Therefore, the drag reduction theory still applies in both cases as long as there is turbulent flow occurring along the path of the flowing liquid inside the tubing or pipe.

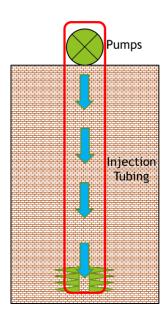


Figure 2: Water flooding system section in focus of this research.

This research is focusing on applying DRA into water injection system due to the increasing operational cost to inject fluid for secondary recovery and pressure maintenance purposes with large and high rated pumping power pumps installed. [11] This increasing costs can be minimized by the application of DRA for water injection system besides of reducing the numbers of injection wells required. [12] This statement thus denied the idea of adding more injection tubing to increase the flow capacity. [13]

Turbulent and laminar flow are the two (2) main types of flow but the main focus in this research is within turbulent flow. Turbulent flow is the condition where the Reynolds number is greater than the laminar flow. In pipelines, the turbulent flow occurs when Reynolds number exceeds 2300 [1] which is slightly lower than Brostow's rule-of-thumb whom quoted to mentioned in his research that whenever Reynolds number calculated is greater than the following

$$N_{Re} > 1.7 \ x \ 10^4 s^{-1} \tag{2}$$

thus turbulent flow is formed inside the pipelines. [14] Nevertheless, both of them arrived on the same generalization of the turbulent flow region based on the worldwide acceptable general Reynolds number which is shown below

$$N_{Re} > \frac{Du_{av}\rho}{\eta} \tag{3}$$

where D is the pipe diameter, u_{av} is the average flow velocity and ρ is the fluid mass density. However, the interchangeability of Reynolds number with correction factors is dependent on the type of fluid flow in the pipelines whether they are Newtonian or Non-Newtonian fluid. Turbulent flow is merely important as to get better results on the study of drag reduction when adding polymers into the flow. [1, 9, 15]

2.2 Drag Reducing Agent (DRA)

DRA as the name is the agent that acts in the turbulence flow in pipelines by reducing the drag along the inner wall of the injection tubing or pipelines with resulting pressure drop where most of the time polymers are used as the medium since Toms's findings in 1948.

2.2.1 DRA Solubility and Degradation

Different types and sources of DRA materials being tested usually polymers will have different rate of solubility and degradation. Therefore, several precautions have been identified by previous researches which one (1) of them clearly stated that DRA solution must be injected at the discharge of the booster pump to avoid polymer degradation. [9]

DRA solubility on the other hand is highly affected by the types of solvent in used, temperature of the solvent as well as the size of the DRA particle itself. Previous studies have confirmed that rate of DRA degradation from polymer increased with the increase in temperature of the solutions. [16, 17] Recent study on the effect of temperature towards DRA showed significance influence on polymer solubility in the fluid, degradation of the polymer as well as apparent viscosity of liquid [1] thus confirmed the previous studies as well.

Different process or method of extraction of CMC will also determine the degree of substitution (DS) when mixed with solvent such as water.[18]

2.2.2 DRA Sources and Performance

The most common DRA been used in the industry nowadays are comprised of synthetic polymers such as CMC [15], Polyacrylamide (PAM) and Polyethylene oxide.[16] CMC is chosen in this research since it is possible to extract the CMC from agricultural wastes of plants and fruits. [15] Synthetic polymers are manufactured by linking basic structural units together. [19] Polymers identified with high molecular weight are good potential of drag reducers.

Based on the research on the measurement of drag reduction in polymer added turbulent flow, the drag reduction effect tends to increase with increasing molecular weight of the polymer and Reynolds number [10] which had proven the previous research on a field test using high molecular weight polymer also getting the same trends in their results on oil soluble DRA. [6]

A review made few years later on Drag reduction in flow: Review of applications, mechanism and prediction which clearly line up a few findings been agreed upon which one (1) of them mentioned upon drag reduction is directly proportional to the molecular mass, M of the polymer, regardless of liquid (solvent) type. [14]

According to the book of Thermoplastic Starch, some natural polymers can be classified into amylose, cellulose, chitin and chitosan, proteins and gelatin. [20] Byproduct of grated coconut flesh [21] is a natural polymer which indicates a potential to be a green and environmental friendly DRA since they are biodegradable materials with high cellulose contents.

2.2.3 Carboxymethylcellulose (CMC)

Carboxymethylcellulose or in short is CMC was produced from the modification of natural polymer known as cellulose.[15] Sometimes, CMC can be defined as the derivative of cellulose group formed by the reaction of acid and alkali such as sodium hydroxide (NaOH) and monochloroacetic acid where this acid function is to etherify the CMC besides of neutralizing the effect of alkali from NaOH. Higher concentration of NaOH increases the Degree of Substitution (DS) of carboxymethyl group on the

cellulose backbone thus result in higher molecular weight. On top of that, the substitution process created a strong inter-molecular bond between carboxymethyl group and hydroxyl group which results in higher mechanical properties thus less mechanical degradation. [22]

Grated coconut flesh waste or so called coconut residue (CR) is the leftovers of coconut meat after extraction of milk by mechanical squeeze is done. CR is chosen because of its high cellulose contents which could be beneficial as a potential DRA. [15] This is confirmed by a study conducted recently which claimed that the CR contains as much as 72.67% and 72.33% of cellulose for total dietary fiber (TDF) in original and water washed CR samples, respectively. [23]

2.2.4 Nano-scale DRA

Nanomaterial is an object that has at least one (1) dimension in the nanometre scale where nanometre scale is conventionally defined as 1 to 100nm. [24] According to the book entitled Springer Handbook of Nanotechnology, nanoparticle is defined as an aggregate of atoms bonded together with a radius between 1 and 100nm which typically consist of 10-10⁵ atoms. [25] The interest on nano-scale DRA comes from the nature for example self-cleaning properties of lotus leaf [25] and less friction structure of shark skin. [26]

Another factor which drives the author to focus on the nano-scale DRA is because some researchers already been successful in their studies and it show that it is not impossible. The common synthetic polymer been applied and studies repetitively is the PAM which some have managed to transform PAM into nanoparticles by inverse-emulsion polymerization technique. [27] Besides that, there is another study on extraction of oil from coconut waste showing good indicator to yield more oil when the particle size is smaller. [21] Their research has thus confirmed the previous studied which concluded that rate of extraction increases with the decrease in the size of particle. [28]

Since it will be not effective to inject nanomaterial or nanoparticle into the turbulent flow of water inside the pipeline being studied, they will be mixed with water or any suitable solvents thus forming a new kind of nanofluid. [29] New nanomaterials showed high performance in polymer nano composites due to their high aspect ratio and high surface area of the dispersed nano-sized particles. [30] Therefore, it is expected that when the wastes are prepared in nanoparticles, they will have changes in both physical and chemical properties such as more soluble in water and higher shear strength as compared to their bulk materials since they have an increased surface-to-volume ratio. [24]

CHAPTER 3 RESEARCH METHODOLOGY

3.1 Introduction

The main focus of this research methodology comprises of the laboratory experimental investigation in order to achieve the research objectives which are mentioned in the first chapter. The experimental investigation of this research are divided into several subsections which are arranged in ascending order from sample preparation to sample evaluation for easier repeatability with other raw materials to be tested. They are encompasses of:

i) Biopolymer extraction

→ The process of synthesizing the biopolymer from grated coconut residue is adapted from previous research of The Study of Drag Reduction Ability of Naturally Produced Polymers from Local Plant Sources. [15]

The original idea of extracting the CMC from natural by-products is formulated by the Center of Excellence for Polysaccharides Research, Friedrich Schiller University of Jena, Germany. [15]

ii) Flowmeter test

→To determine and assess the drag reducing ability of the produce biopolymer.

In the first subsection of the experiment, the method of producing the biopolymer will include the extraction of cellulose and synthesizing the cellulose extracted to obtain

CMC. [15] All the equipment and chemicals used are listed within the same subsection of samples preparation. The samples prepared will be use throughout the experiment of this research. The following subsection describes the step-by-step procedures on the study of drag reduction utilizing a laboratory scale flowmeter experimental setup by observing the changes in pressure drop across an Orifice plate from manometer readings.

3.2 Biopolymer Preparation

A detail procedures on the process of extracting biopolymer from raw solid waste and synthesizing the CMC from the extracted biopolymer will be presented within this subsection. All equipment and chemical solutions used also will be presented chronologically with the process being conducted.

3.2.1 Biopolymer Synthesis

This part will show all the method for cellulose extraction from raw waste of CR and synthesizing the cellulose into CMC along with all the equipment and materials required throughout the entire processes. CR is the leftovers of grated coconut meat after the coconut milk is extracted from it by subjecting the grated meat to physical treatment such as compression. This method is found to be more feasible thus chosen due to the minimal requirement on the amount of chemicals used besides of it relied mostly on the raw organic material available on the CR itself.

A. Materials

1 kilogram of CR is collected from a coconut milk processing shop located in the vicinity of Universiti Teknologi PETRONAS (UTP), Perak, Malaysia. CR is used due to the ease to find abundant source for free since they are considered as solid waste besides of claimed to have a very high cellulose contents of 72.67% and 72.33% for total dietary fiber (TDF) in original and water-washed CR samples, respectively. [23] Therefore, CR is chosen to be the main subject sample for this study. Throughout the preparation and analysis of CMC in this section, 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). [15] The chemicals required for the extraction and synthesis of CMC are listed as follow:

- 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 for synthesis MERCK 412
- vi) Acetic acid AR QREC A1020-1-2500

All the chemicals are purchased from chemical supplier nearby which is Irama Canggih Sdn Bhd, Ipoh, Perak with 99% purity. Volume and amount required for each chemicals are described during the process.

B. Cellulose Extraction

i) CR is rinsed with plenty of tap water until clean before oven-dried for 30 minutes at 121°C (250°F). The tap water is acquired from the laboratory of UTP to ensure consistency and repeatability with other raw materials later on.



Figure 3: Washed CR was oven-dried for 30 minutes.



Figure 4: CR after dried in the oven for 30 minutes at 121°C (250°F).

ii) The dried CR were then grinded to less than 20mm in size by utilizing the Cole Parmer mortar grinder. The container of the grinder can only be filled with less than 50 grams of CR at a time. The grinder was set at 3 minutes for each run.

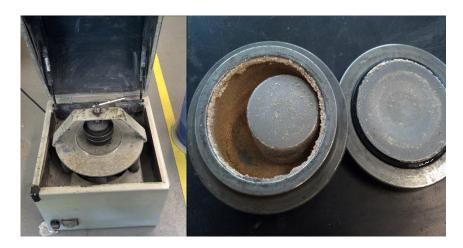


Figure 5: Cole Parmer Mortar Grinder. [15]

The use of mortar grinder aided for preparing the CR sample in fine powder form due to its fast and powerful grinding mechanism. By completing the grinding activities, the CR sample have been reduced in sizes from coarse-shaggy structure to fine powder form.

After grinding with mortar grinder, the powder CR was kept in air-tight container to prevent it absorbing moisture as shown in Figure 6.



Figure 6: Powder CR was kept in air-tight container.

iii) Dried CR was then mix and cooked with 1M of NaOH in a beaker at 150°C hot plate temperature for 1 hour using a magnetic stirrer (200RPM). The amount of CR added to the NaOH solution should not too much which will be difficult for the magnetic stirrer to stir the thick mixture. This step was done to remove any impurities and undesirable products from the CR. After mixing, it was observed that the mixture will turn from brownish mixture to a reddish-purple mixture and this changes occurred faster when heated on the magnetic stirrer.



Figure 7: CR powder cooked with 1M NaOH at 150°C mixture temperature.

iv) After cooked and stirred at the same time, reddish-purple slurry was formed and filtered using tea bag to prevent very fine slurry from slipped into the sink. The filtered solution was removed while the residues left in the tea bag were rinsed with plenty of tap water until the reddish-purple colour is gone as shown in Figure 8.



Figure 8: Reddish-purple colour is gone after rinsed with tap water.



Figure 9: (Left) After rinsed with tap water; (Right) Before rinsed with tap water.

v) After the residues were filtered and rinsed, the clean residues were oven-dried once again in the oven for 30 minutes at 121°C (250°F). The process of synthesizing CMC requires an equipment which in this case is the oven where almost all the moisture of the CR are dried up before it is possible to extract to cellulose. The oven also crucial to heat up the mixture to a specific temperature for reaction to occur. Dried residues were then kept in another air-tight container to prevent moisture absorption.



Figure 10: Residues were oven-dried for 30 minutes at 121°C (250°F).

C. Carboxymethylcellulose Synthesis from Coconut Residue

Three (3) sets of mixture consist of 15.0g of residues obtained in the previous step, 50ml of NaOH with varying concentrations (20%, 40% and 60%) were mixed in three (3) separate beakers containing 450 ml of isopropanol using overhead stirrer for 30 minutes at 200 rpm. 18.0g of monochloroacetic acid was measured by Fz-200i and electronic weighting scale (accuracy of 0.001g and maximum capacity of 220g) and then added into the three (3) beakers to initiate the carboxymethylation reaction. Magnetic stirrer was used to stir the initiated solutions for 30 minutes at 200 rpm. This step is essential to ensure the biopolymer are mixed thoroughly with water as the solvent until the polymer solutions are visibly homogeneous [15] as shown in Figure 11.



Figure 11: Mixtures of cooked CR with monochloroacetic acid, isopropanol and varied sodium hydroxide concentrations.

- ii) Prior to heating the mixtures of polymer solutions prepared in the previous step in the oven at varied reaction temperature (50°C, 55°C and 60°C) and different heating time period (60 minutes, 150 minutes and 240 minutes), the beakers were covered with aluminium foil to avoid evaporation during the entire heating process. At the end of this step, sieve was used to separate and remove the solution phase while the solid phase is kept aside before suspending it into 100ml of methanol (70% v/v). Glacial acetic acid was poured into the beaker to neutralize the suspended solids in methanol solution. A small funnel with a new filter paper was used to filter the neutralized solids from the solution.
- iii) A series of rinsing and washing the leftovers on the filter paper were conducted with five (5) repetitions; the filtrate will be suspended in 300ml of ethanol (70% v/v) for 10 minutes to remove unwanted byproducts before washing with 300ml of absolute methanol and filtered for the next cycle. The final filtrates were dried at 55°C in the oven for a total of 24 hours in the period of three (3) days with eight (8) hours continuous oven operation. Finally, a total of 27 varied samples of CMC were obtained after completing the 24 hours period. The samples were kept in air-tight container and labeled accordingly after

further grinded into fine powder with mortar grinder. The samples were weighted with electronic balance and recorded in Table 7 in Chapter 4.

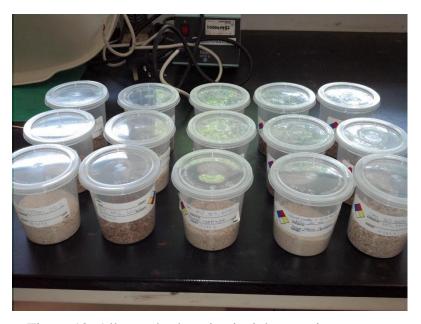


Figure 12: All samples kept in air-tight container.

3.3 Flowmeter Test

Focused mainly on the drag reduction study, every biopolymer samples prepared in powder form were mixed into solution using tap water as the based solvent for the testing on drag reduction performance by utilizing a laboratory scale flowmeter experimental setup.



Figure 13: Flowmeter experimental setup with Orifice plate.

3.3.1 Samples Preparation for Flowmeter Test

The sample preparation to be run with the flowmeter were prepared based on the minimum requirement by the flowmeter setup. The flowmeter can only works with minimum of 20 liters of water to ensure fully-water bearing flow inside all the pipes and test section with no bubbles.

Samples preparation comprise of 1 liter of water and 8 grams of DRA sample in this case Polyacrylamide and CMC extracted from CR at varied reaction parameters. When 8 grams of sample mixed with 1 liter of water, the solution will have a concentration of 8000 ppm. The prepared 8000 ppm DRA solution in the lab was then mixed into the flowmeter tank containing 19 liters of water to have a total of 20 liters of mixture with diluted concentration from 8000 ppm to 400 ppm.

The following equation was used for all 27 samples assuming the tap water have a mass of 1 kilogram per liter.

Concentration of DRA (ppm) =
$$\frac{Mass\ of\ DRA}{Volume\ of\ distilled\ water} \times 10^6$$
 (4)

For the sample preparation on this subsection, the same methods from previous subsection were applied to mix the DRA in water where a magnetic stirrer was used and set at a constant low speed at 200 rpm for 30 minutes until homogenous mixture was visible to minimize the destruction of the polymer chain. The prepared solutions were then transferred into the glass bottle with cap and labeled respectively.

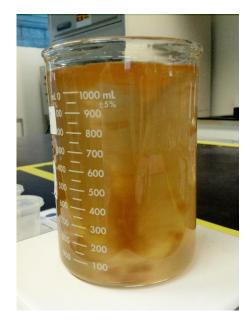


Figure 14: 1 litre of water mixed with 8 grams of CMC extracted.

3.3.2 Flowmeter Test Procedures

The laboratory scale flowmeter test setup have the following components attached together in a closed-loop system which are:

- i) 40 liters tank capacity
- ii) Centrifugal pump
- iii) Manometers with 8 pressure points readings
- iv) Venturi test section
- v) Variable Area Flowmeter

- vi) Orifice plate test section
- vii) Flow Control Valve

The laboratory flowmeter test setup was used since according to the theory, high flow velocity across the Orifice plate will produced a turbulent flow at the back of the plate. Since DRA will only works in a turbulent flow condition, this flowmeter test setup is the best and simplest way to test the performance of DRA by measuring the amount of fluctuation on the manometers before, after and away from the Orifice plate which in this case were labeled as point 6, 7 and 8 respectively.

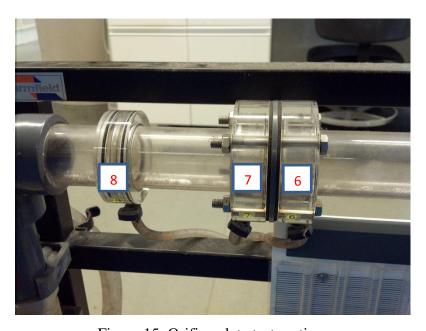


Figure 15: Orifice plate test section.

Procedures:

- 1. Tank was filled with 20L of water with all the flowmeter loop drained out of any liquid leftover.
- 2. The centrifugal pump was switched on with intake valve to pump and flow control valve both full open to ensure full circulation of water into the system without traces of bubbles.
- The flow control valve was fully closed and the pump was immediately switched off after no all bubbles were removed from the system as well as inside the manometer.

- 4. The manometer was bleed off by loosening the bleeding screw on top of the manometer to have all the liquid readings of manometer equally leveled below the manometer scale of 0 and the bleed screw was tighten immediately.
- 5. The pump was switched on and the liquid level in manometer was kept to stabilize after 1 minute and recorded in Table 8 as initial reading of the atmospheric pressure.
- 6. The flow control valve was then slowly opened to reach liquid flow rate of 20L/min by monitoring the variable area flowmeter.
- 7. The manometer readings were observed and recorded after let to stabilize for 1 minute at point 6, 7, and 8 respectively in Table 8 which are the point across and away from the Orifice plate.
- 8. The pump was switched off and flow control valve was fully opened to let all liquid drain out into the tank below.
- 9. The drain valve at the side of the tank was fully opened to drain all water from the tank to prepare for the new run.
- 10. The experiment were repeated with Polyacrylamide and the rest of 27 samples of CMC extracted where the tank was filled with only 19 liters of water and added with 1 liter of mixture prepared in lab respectively for every consecutive runs. The flowmeter setup was flushed once with clean water after each sample run to remove any leftovers inside the system.

For the flowmeter test, fully-water bearing was ran first to get the baseline data for the manometer readings. Polyacrylamide and all 27 samples were run separately to observe the best DRA with the highest drag reduction percentage is at which parameters being studied. Pressure drop across the Orifice plate will be observed from the manometer readings across and away from the plate at points 6, 7 and 8. All manometer readings during the test were recorded and tabulated in Table 8 and 9 in Chapter 4.



Figure 16: (Left) Variable area flowmeter was set to 20L/min. (Right) Manometer readings was taken at points 6, 7, and 8 respectively.

3.4 Key Milestones

Planning

- Conduct research based on existing study to identify the problem, objectives and appropriate methodology to achieve the objectives
- Understanding the concept and the scope of study

Pre-Execution

- Understanding the setup and procedures of the experiment
- Study the calculation of variables

Execution

- Synthesizing the biopolymer
- Conduct the flowmeter test on the CMC extracted for DRA performance by observing pressure drop across Orifice plate
- Record all the measured variables and resulting data

Post Execution

- Tabulate the data in tables and plot the graph respectively
- Conclude the outcome of the experiment based on the results obtained

Figure 17: Key Milestones of the research.

Project Schedule Literature Review Research on: -Drag in pipelines -Turbulent Flow and Reynolds Number -Drag Reducing Agent (DRA) -Organic polymers -Environmental issue with synthetic DRA Project Objectives Materials Selection and Screening Process Extraction of Carboxymethylcellulose Flowmeter Test for DRA Performance Results and Discussion Conclusion and Recommendation

Figure 18: The process flow chart of the overall project

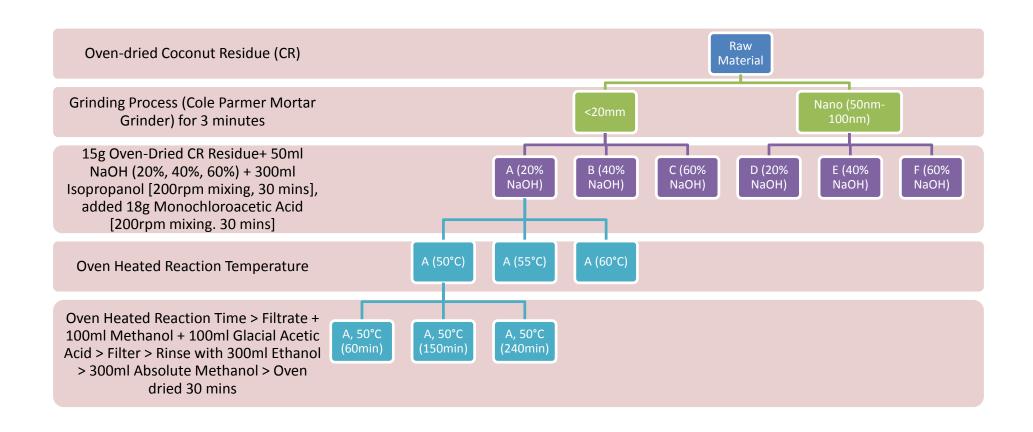


Figure 19: Carboxymethylcellulose preparation and extraction flowchart.

3.5 Gantt Chart

Table 1: Gantt chart for FYP 1.

| FYP 1 | | | | | | | We | ek | | | | | | |
|--|---|---|---|---|---|---|----|----|---|----|----|----|----|----|
| Activities | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Selection of project topic | | | | | | | | | | | | | | |
| Preliminary research work | | | | | | | | | | | | | | |
| Submission of Extended Proposal Defence | | | | | | | | | | | | | | |
| Proposal Defence | | | | | | | | | | | | | | |
| Project work continues: -Lab booking -Chemicals purchasing | | | | | | | | | | | | | | |
| Submission of Interim Draft Report | | | | | | | | | | | | | | |
| Submission of Interim Report | | | | | | | | | | | | | | |

Table 2: Gantt chart for FYP 2.

| FYP 2 | Week | | | | | | | | | | | | | | |
|---|------|---|---|---|---|---|---|---|---|----|----|----|----|----|----|
| Activities | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| Project work continues: -Synthesizing the biopolymer -Sample preparation -Rheological study -Gravity driven flow test | | | | | | | | | | | | | | | |
| Submission of Progress Report | | | | | | | | | | | | | | | |
| Project work continues: -Tabulate data and result -Construct graph -Conclude experiment based on the result obtained | | | | | | | | | | | | | | | |
| Pre-EDX | | | | | | | | | | | | | | | |
| Submission of Draft Report | | | | | | | | | | | | | | | |
| Submission of Dissertation (soft bounded) | | | | | | | | | | | | | | | |
| Submission of Technical Paper | | | | | | | | | | | | | | | |
| Oral Presentation | | | | | | | | | | | | | | | |
| Submission of Project Dissertation (hard bounded) | | | | | | | | | | | | | | | |

3.6 Equipment, Apparatus, Chemicals and Software

Table 3: List of equipment used.

| No. | Equipment | Purpose |
|-----|---|--|
| 1. | Cole Parmer Mortar Grinder | To grind the materials to a finer powder sizes of less than 20mm. |
| 2. | Magnetic stirrer with variable hot plate and digital thermometer | To continuously stir the mixtures while heating up the solutions to a desired temperature. |
| 3. | Drying/Heating Oven | To dry up moisture of the prepared powders and filtrate. |
| 4. | Fz-200i A&D Electronic Weighting Scale (accuracy 0.001g and max. capacity 220g) | To accurately measure the weight of the powders and water |
| 5. | Laboratory Scale Flowmeter Experimental Setup with Orifice Plate and Manometer | To do flow test on the effects of injected DRA into the turbulence flow water system |

Table 4: List of apparatus used.

| No. | Apparatus | Purpose |
|-----|-----------------------------------|--|
| 1. | Glass beakers | To store the mixtures and solutions |
| | (variable size; small, medium and | and for heating and stirring activities. |
| | large) | |
| 2. | Glass bottle with lid | To store the mixtures and solutions |
| | (variable size; small, medium and | and for heating and stirring activities. |
| | large) | |
| 3. | Aluminium foil | To cover the beakers as to avoid |
| | | evaporation of the heated solutions |
| | | |
| 4. | Filter funnel | To filter the filtrate out of the |
| | (with 3mm holes) | solutions. |
| | | |
| 5. | Filter papers | To filter the filtrate out of the |
| | | solutions. |
| | | |
| 6. | Stopwatch | To measure the time for each test of |
| | | each sample |
| | | |
| 7. | Measuring cylinder | To measure the amount of solutions or |
| | | solvents to be use accurately |
| | | |

| 8. | Metal sieve | To separate solid phase and liquid phase |
|----|----------------------|---|
| 9. | Air-tight containers | To store the filtered powders to maintain minimal moisture content. |

Table 5: List of chemicals/solvents used.

| No. | Chemical/Solvent | 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 |

Table 6: List of software used.

| No. | Software | Purpose |
|-----|----------------------------|--|
| 1. | Microsoft Office Word 2010 | To write up the reports |
| 2. | Microsoft Excel 2010 | Database for experimental data, plot graphs and calculations |
| 3. | Microsoft Power Point 2010 | Draw schematic diagram and editing pictures |

CHAPTER 4

RESULTS AND DISCUSSION

At the end of the experiments conducted for this research, the results recorded were divided into two (2) sections to make it convenient and not confusing for future reference. All the variables and assumptions made throughout the research experiment were also listed in this chapter.

4.1 Variables

4.1.1 Constant Variables

- i. Volume of solution in the mixing tank (20 L)
- ii. Volume of prepared DRA solution (1 L)
- iii. Diameter of the orifice (0.02 m)
- iv. Pipe diameter (0.024 m)
- v. Total pipe length of the flow meter
- vi. Concentration of DRA mixed in the tank (400 ppm)
- vii. Water temperature at room temperature (25°C)

4.1.2 Manipulated Variables

- i. Concentration of NaOH (20 wt%, 40 wt% and 60 wt%)
- ii. Reaction time (60 minutes, 150 minutes and 240 minutes)
- iii. Reaction temperature (50°C, 55°C and 60°C)
- iv. Type of DRA mixed in the tank (PAM and 27 samples of CMC extracted from the CR)

4.1.3 Responding Variables

- i. Mass of CMC extracted (grams)
- ii. Liquid level in manometer at point 6, 7 and 8

4.2 Assumptions Made

- i. The DRA solutions prepared prior to flowmeter test were mixed completely with the water in the tank. This assumption is important because complete solubility of DRA in the solution is required to ensure drag reduction.
- ii. The liquid flow inside the flowmeter experimental setup is in conduit flow. As mentioned in the literature review, the DRA acts as dampener in between the flowing liquid and along the pipe internal wall.
- iii. The flow after the Orifice plate is turbulent flow regime. The water flowing at high velocity after the Orifice plate opening due to constriction in size. The fluctuation of the manometer reading across and away from the Orifice plate indicate the existence of pressure drop from turbulent flow.

4.3 Yield of Carboxymethylcellulose Extracted

In this result section, the amount of mass yield (g) of CMC extracted from CR at various reaction temperature, sodium hydroxide concentration and reaction time were tabulated in Table 7 accordingly.

The result were sorted by the smallest reaction temperature of 50°C to 60°C followed by smallest concentration of sodium hydroxide of 20 wt% to 60 wt% and lastly by the reaction time from 60 minutes to 240 minutes respectively in the table.

From the table, three (3) graphs have been plotted to show the relationship between the reaction temperature during extraction of CMC from CR with the changes in sodium hydroxide concentration and reaction time as well which have different results on the yield mass of CMC extracted in Figure 20, 21 and 22.

The reason behind the different sets of reaction parameters applied during the extraction of CMC from CR is because there is no research available up to the completion of this research on the study of CMC extraction from CR. Therefore, the author found that it necessary to include the changes in sodium hydroxide concentration as well as the reaction time apart of focusing on the reaction temperature only.

This is because it is understood that different tested materials for the extraction of CMC will have different sets of reaction parameters that suited best for the materials itself. [31, 32] Therefore, the results from this research will aided as base datasets for future research and optimization on the extraction of CMC from CR.

Table 7: Mass of CMC extracted from CR.

| No | Reaction Temperature (°C) | Reaction Concentration NaOH (wt%) | Reaction Time (mins) | Mass of Carboxy- methylcellulose Extracted (g) |
|----|------------------------------|---|-------------------------|---|
| 1 | 50 | 20 | 0 | 15 |
| 2 | 50 | 20 | 60 | 15.361 |
| 3 | 50 | 20 | 150 | 15.76 |
| 4 | 50 | 20 | 240 | 14.771 |
| 5 | 50 | 40 | 0 | 15 |
| 6 | 50 | 40 | 60 | 27.014 |
| 7 | 50 | 40 | 150 | 22.417 |
| 8 | 50 | 40 | 240 | 23.465 |
| 9 | 50 | 60 | 0 | 15 |
| 10 | 50 | 60 | 60 | 20.521 |
| 11 | 50 | 60 | 150 | 24.26 |
| 12 | 50 | 60 | 240 | 21.492 |
| 13 | 55 | 20 | 0 | 15 |
| 14 | 55 | 20 | 60 | 13.965 |
| 15 | 55 | 20 | 150 | 14.851 |
| 16 | 55 | 20 | 240 | 14.563 |
| 17 | 55 | 40 | 0 | 15 |
| 18 | 55 | 40 | 60 | 18.822 |
| 19 | 55 | 40 | 150 | 20.409 |
| 20 | 55 | 40 | 240 | 25.497 |
| 21 | 55 | 60 | 0 | 15 |
| 22 | 55 | 60 | 60 | 25.426 |
| 23 | 55 | 60 | 150 | 19.896 |
| 24 | 55 | 60 | 240 | 26.491 |
| 25 | 60 | 20 | 0 | 15 |
| 26 | 60 | 20 | 60 | 12.57 |
| 27 | 60 | 20 | 150 | 8.978 |
| 28 | 60 | 20 | 240 | 13.588 |
| 29 | 60 | 40 | 0 | 15 |
| 30 | 60 | 40 | 60 | 19.527 |
| 31 | 60 | 40 | 150 | 23.728 |
| 32 | 60 | 40 | 240 | 25.679 |
| 33 | 60 | 60 | 0 | 15 |
| 34 | 60 | 60 | 60 | 25.106 |
| 35 | 60 | 60 | 150 | 21.621 |
| 36 | 60 | 60 | 240 | 28.991 |

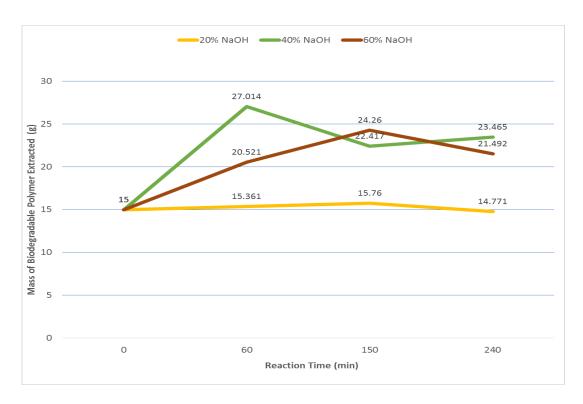


Figure 20: Mass of CMC extracted at 50°C reaction temperature.

From the graph shown in Figure 20, at reaction temperature of 50°C, the highest amount of CMC extracted from CR is 27.014 grams with 40% sodium hydroxide (NaOH) concentration by weight (wt%) and 60 minutes reaction time in the oven. The lowest CMC yield recorded at 50°C reaction temperature is 14.771 grams with 20 wt% of NaOH concentration and 240 minutes of reaction time.

The pattern on the graph shown that at this reaction temperature of 50°C, all samples with 20 wt%, 40 wt% and 60 wt% of NaOH concentration were increasing in CMC yield with increasing reaction time up to 150 minutes given the initial mass of 15 grams each samples. However, this trend is not followed by the sample with 40 wt% NaOH concentration since the yield of CMC was reaching the peak of 27.014 grams at 60 minutes but immediately declining as the reaction time increased to 150 minutes.

On the other hand, the other samples with NaOH concentration of 20 wt% and 60 wt% were also declining in CMC yield as the reaction time increased more than 150 minutes at the yield of 14.771 grams and 21.492 grams respectively while the previous sample at 40 wt% NaOH starts to increase in the yield mass up to 23.465 grams.

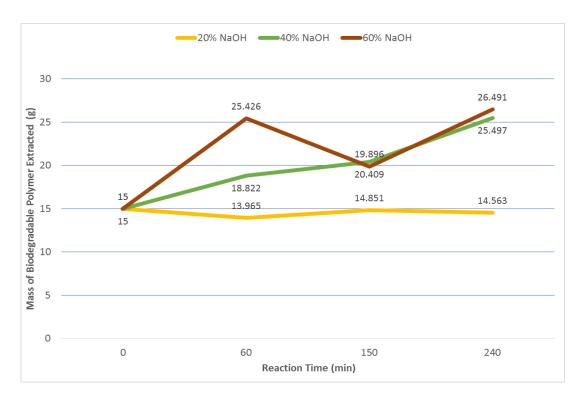


Figure 21: Mass of CMC extracted at 50°C reaction temperature.

Based on the graph shown in Figure 21 above, at reaction temperature of 55°C, the highest yield of CMC extracted from CR is 26.491 grams with NaOH concentration of 60 wt% and reaction time of 240 minutes. The lowest yield recorded for this reaction temperature is 13.965 grams with reaction parameters of 20 wt% NaOH concentration and 60 minutes of reaction time.

The pattern on this graph is slightly different from the previous graph shown on Figure 20 where only samples with NaOH concentration of 40 wt% and 60 wt% shows increasing yield at shorter reaction time of 60 minutes with 18.822 grams and 25.426 grams respectively with initial sample of 15 grams each. The sample with 20 wt% of NaOH concentration shows a slight decreased in the amount of CMC yield at 60 minutes reaction time with only 13.965 grams which is the smallest amount recorded at the reaction temperature.

On the other hand, the sample with 40 wt% shows no sign of decreasing in CMC yield at reaction temperature of 55°C even with longer reaction time up to 240 minutes as compared to previous graph with maximum yield of 25.497 grams.

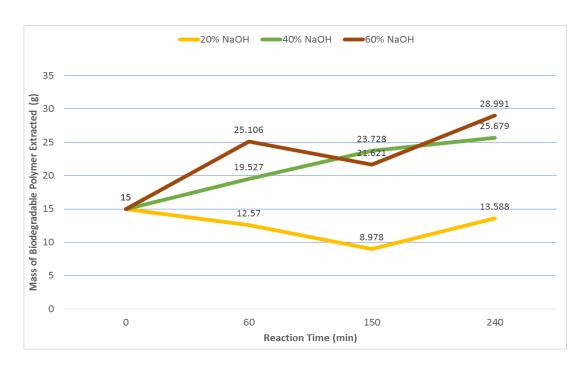


Figure 22: Mass of CMC extracted at 50°C reaction temperature.

From the graph shown in Figure 22 above, at reaction temperature of 60°C, the highest yield of CMC recorded is 28.991 grams at the peak NaOH concentration of 60 wt% and 240 minutes reaction time. However, the lowest yield of CMC recorded at this reaction temperature fluctuated down to 8.978 grams with NaOH concentration of 20 wt% and reaction time of 150 minutes.

The pattern can be observed from the above graph resembles the patterns described in the previous graph in Figure 21 where both samples with NaOH concentration of 40 wt% and 60 wt% were increasing in the yield of CMC with increasing reaction time up to 240 minutes with the amount of CMC peak to 25.679 grams and 28.991 grams respectively. The fluctuation pattern for sample with 60 wt% of NaOH also the same at 55°C reaction temperature where the yield fluctuated at 150 minutes with the resulting yield of 21.621 grams from 25.106 grams at shorter reaction time of 60 minutes.

On the other hand, the yield for sample with NaOH concentration of 20 wt% is not at par with the rest of the samples since it fluctuated down steeply to only 8.978 grams at 150 minutes but started to increase again at longer reaction time up to 240 minutes.

From the observation and analysis done for the three (3) graphs, the yield of CMC from CR does affected with the reaction temperature during the extraction process. It is also observed that the highest yield of CMC can be gained with the highest sets of reaction temperature at 60°C, with NaOH concentration of 60 wt% and longest reaction time of 240 minutes.

For the effects of reaction temperature, this is proven by the results measured and recorded on the CMC yield at 60 wt% NaOH concentration and 240 minutes of reaction time, the yield increased from 21.492 grams at 50°C to 28.991 grams at 60°C. This shows an increase of 34.89% yield of CMC with only 10°C increased in reaction temperature. This increase may be due to the fact that there is a better reaction environment for the carboxymethylation of the CR while the decrease of yield at higher reaction temperature could possibly due to the cellulose degradation. [33]

However, the amount of yield does not signify that particular reaction parameters will yield the best sample that have the performance as a good DRA during the flowmeter test. On the other hand, these parameters should be identify as the main manipulated variables that will varied the yield of CMC extracted from CR for the upcoming researches.

Besides that, the changes in reaction parameters and the extraction method of CMC will determine the different characterization of the CMC extracted itself such as the Degree of Substitution (DS). [32, 33] Effect of reaction temperature and the concentration of NaOH during extraction of CMC also will have significant impact on the rheology of CMC solution when mixed with different types of solvents such as the viscosity. [34]

Since the result from this section could not conclude at which reactions parameters are the best to resembles a good DRA, the results from the flowmeter test will give a better insight whether the yield of CMC from the highest reaction parameters will give the highest Drag Reduction Percentage (%DR) or vice versa. All the results from flowmeter test were shown in the following section 4.2.

4.4 DRA Performance by Flowmeter Test

In this result section, the manometer readings from the flowmeter test of water and various solutions are recorded and tabulated in Table 8 and Table 9 respectively. The manometer readings data were recorded at point 6, 7 and 8 and the table also shown the calculated differential readings of the manometer from the initial level and the final level between point 6 to point 7 (X) and point 6 to point 8 (Y). X means the pressure drop or difference in manometer reading across the Orifice plate while Y is located further at 0.1 m away after the Orifice plate.

The Drag Reduction Percentage (%DR) were calculated for all the samples run within the flowmeter test by using the differential reading from the manometer itself without calculating the amount of pressure drop. This is because the amount of pressure drop is equal to the differential reading from the manometer liquid level and thus pressure drop can be replaced directly with the liquid level from manometer readings only in the formula.

The initial reading of manometer recorded to be at 322mm which is the atmospheric pressure in the Block 20 Fluid Mechanics Lab at ground level. The ambient temperature recorded in the lab is around 25°C. When the flowmeter was run with fully-water bearing, the manometer readings shows some fluctuation in the water level at the three pressure points taken; point 6, 7 and 8 and recorded to be 304mm, 188mm and 226mm respectively. The reading from fully-water run was used as based data for calculation of %DR as the replacement for pressure drop without addition of DRA.

All formulas used for this flowmeter test and calculations made from the data recorded were shown in the following subsection 4.4.1.

Table 8: Manometer readings recorded at point 6, 7, and 8 during flowmeter test.

| No. | Type of Mixture | Reac | tion Parameters | | Manometer Reading | | | Differentia | l Calculated | Calculated | Calculated |
|-----|-----------------|---------------------|-----------------------------|----------------|---|--|--|------------------------------|------------------------------|--|--|
| | | | | | | | | | Y | Drag | Drag |
| | | Temperature (°C) | Concentration NaOH (wt%) | Time (mins) | Before Orifice (mm) - Point 6 | After Orifice (mm) - Point 7 | Away After Orifice (mm) - Point 8 | Point 6 - Point 7 (mm) | Point 6 - Point 8 (mm) | Reduction Percentage (%DR) at X | Reduction Percentage (%DR) at Y |
| - | Initial Reading | - | - | - | 322 | 322 | 322 | 0 | 0 | 0.00 | 0.00 |
| A | Tap Water | - | - | - | 304 | 188 | 226 | 116 | 78 | 0.00 | 0.00 |
| В | Polyacrylamide | - | - | - | 304 | 189 | 227 | 115 | 77 | 0.86 | 1.28 |
| 1 | CMC extracted | 50 | 20 | 60 | 304 | 185 | 226 | 119 | 78 | 2.59 | 0.00 |
| 2 | from CR | 50 | 20 | 150 | 304 | 186 | 229 | 118 | 75 | 1.72 | 3.85 |
| 3 | | 50 | 20 | 240 | 304 | 187 | 229 | 117 | 75 | 0.86 | 3.85 |
| 4 | | 50 | 40 | 60 | 304 | 189 | 229 | 115 | 75 | 0.86 | 3.85 |
| 5 | | 50 | 40 | 150 | 304 | 190 | 238 | 114 | 66 | 1.72 | 15.38 |
| 6 | | 50 | 40 | 240 | 304 | 191 | 241 | 113 | 63 | 2.59 | 19.23 |
| 7 | | 50 | 60 | 60 | 304 | 190 | 240 | 114 | 64 | 1.72 | 17.95 |
| 8 | | 50 | 60 | 150 | 304 | 192 | 240 | 112 | 64 | 3.45 | 17.95 |
| 9 | | 50 | 60 | 240 | 304 | 193 | 241 | 111 | 63 | 4.31 | 19.23 |

Table 9: Manometer readings recorded at point 6, 7, and 8 during flowmeter test.

| | Table 9: Manometer readings recorded at point 6, 7, and 8 during flowmeter test. | | | | | | | | | | |
|-----|--|------------|------------------|-------|---------|------------|-------------------|-----------|---------|----------------------|---------------|
| No. | Type of Mixture | Reac | ction Parameters | | Mar | ometer Rea | ding | Differ | | Calculated | Calculated |
| | | | | | | | | Calcu | | Drag | Drag |
| | | | | | | | , | X | Y | Reduction | Reduction |
| | | Temperatur | Concentration | Time | Before | After | Away | Point 6 - | Point 6 | Percentage (% DR) of | Percentage |
| | | e (°C) | NaOH (wt%) | (mins | Orifice | Orifice | After | Point 7 | - Point | (%DR) at X | (%DR) at Y |
| | | | |) | (mm) - | (mm) - | Orifice | (mm) | 8 (mm) | A | • |
| | | | | | Point 6 | Point 7 | (mm) - Point 8 | | | | |
| | | | | | | | 1 Unit o | | | | |
| 10 | CMC extracted | 55 | 20 | 60 | 304 | 186 | 227 | 118 | 77 | 1.72 | 1.28 |
| 11 | from CR | 55 | 20 | 150 | 304 | 185 | 227 | 119 | 77 | 2.59 | 1.28 |
| 12 | | 55 | 20 | 240 | 304 | 187 | 230 | 117 | 74 | 0.86 | 5.13 |
| 13 | | 55 | 40 | 60 | 304 | 189 | 234 | 115 | 70 | 0.86 | 10.26 |
| 14 | | 55 | 40 | 150 | 304 | 190 | 230 | 114 | 74 | 1.72 | 5.13 |
| 15 | | 55 | 40 | 240 | 304 | 191 | 241 | 113 | 63 | 2.59 | 19.23 |
| 16 | | 55 | 60 | 60 | 304 | 190 | 238 | 114 | 66 | 1.72 | 15.38 |
| 17 | | 55 | 60 | 150 | 304 | 194 | 242 | 110 | 62 | 5.17 | 20.51 |
| 18 | | 55 | 60 | 240 | 304 | 194 | 242 | 110 | 62 | 5.17 | 20.51 |
| 19 | CMC extracted | 60 | 20 | 60 | 304 | 185 | 228 | 119 | 76 | 2.59 | 2.56 |
| 20 | from CR | 60 | 20 | 150 | 304 | 186 | 230 | 118 | 74 | 1.72 | 5.13 |
| 21 | | 60 | 20 | 240 | 304 | 187 | 228 | 117 | 76 | 0.86 | 2.56 |
| 22 | | 60 | 40 | 60 | 304 | 190 | 237 | 114 | 67 | 1.72 | 14.10 |
| 23 | | 60 | 40 | 150 | 304 | 190 | 239 | 114 | 65 | 1.72 | 16.67 |
| 24 | | 60 | 40 | 240 | 304 | 192 | 230 | 112 | 74 | 3.45 | 5.13 |
| 25 | | 60 | 60 | 60 | 304 | 191 | 238 | 113 | 66 | 2.59 | 15.38 |
| 26 | | 60 | 60 | 150 | 304 | 192 | 240 | 112 | 64 | 3.45 | 17.95 |
| 27 | | 60 | 60 | 240 | 304 | 195 | 243 | 109 | 61 | 6.03 | 21.79 |

From the data recorded in Table 8 and Table 9, three graphs have been plotted each to visualize the relationships on Drag Reduction Percentage (%DR) across Orifice plate (X) and 0.1 m away after Orifice plate (Y).

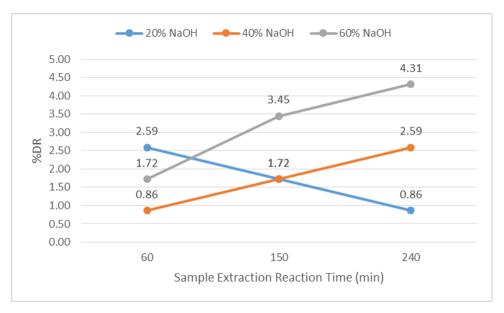


Figure 23: Calculated Drag Reduction Percentage (%DR) across orifice plate (X) for samples extracted at 50°C.

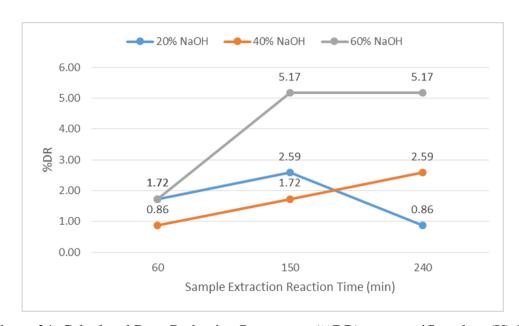


Figure 24: Calculated Drag Reduction Percentage (%DR) across orifice plate (X) for samples extracted at 55°C.

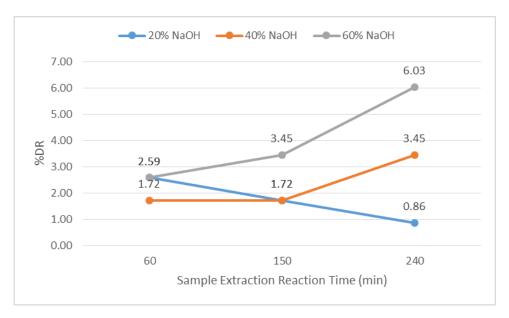


Figure 25: Calculated Drag Reduction Percentage (%DR) across orifice plate (X) for samples extracted at 60°C.

From the three graphs shown in Figure 23, 24 and 25, at different extraction temperature of the CMC from CR, the %DR were increasing with respect to increase of temperature, NaOH concentration and reaction time. However, this pattern was not followed by the sample with the lowest NaOH concentration of 20 wt% since from the graph, the %DR is declining from with increasing reaction time and temperature.

The amount of %DR is small due to the fact that the turbulent flow is not fully formed inside and across the Orifice plate and thus the DRA is not fully functioning at X which is across point 6 and point 7.

The highest %DR recorded was 6.03% which is the sample extracted at 60°C reaction temperature, highest NaOH concentration of 60 wt% and longest reaction time of 240 minutes while the lowest %DR recorded for test section X is 0.86% at various reaction parameters.

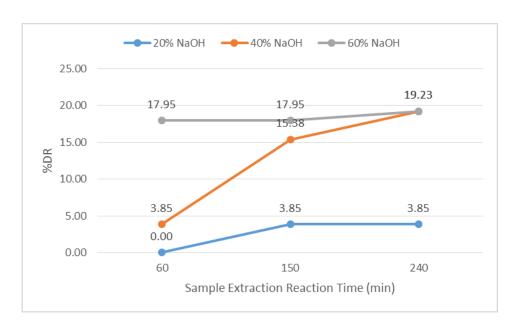


Figure 26: Calculated Drag Reduction Percentage (%DR) 0.1m away after orifice plate (Y) for samples extracted at 50°C.

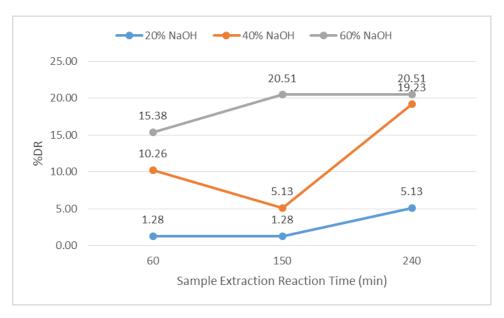


Figure 27: Calculated Drag Reduction Percentage (%DR) 0.1m away after orifice plate (Y) for samples extracted at 55°C.

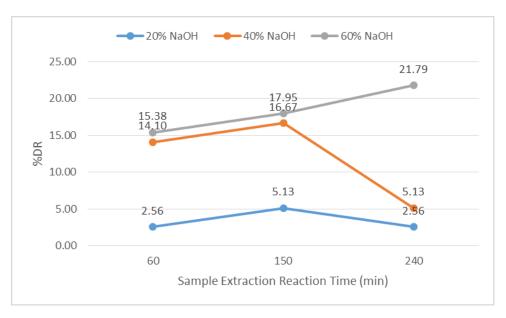


Figure 28: Calculated Drag Reduction Percentage (%DR) 0.1m away after orifice plate (Y) for samples extracted at 60°C.

From the three graphs shown in Figure 26, 27 and 28, at different reaction temperature of the CMC extraction from CR at 50°C, 55°C, and 60°C, the %DR were increasing for each samples tested in the flowmeter at test section Y where the differential manometer reading were recorded at point 6 and 8 with an exceptional for samples extracted at lower parameters of reaction temperature, NaOH concentration and reaction time.

For example, the inconsistency of the increasing pattern can be seen on the graph in Figure 27 of 55°C reaction temperature where the %DR is increasing for sample with extraction parameters of 40% NaOH from 150 minutes to 240 minutes but somehow decreasing in %DR as shown in the graph of Figure 28 for the sample of same NaOH concentration and time parameters but at higher extraction temperature of 60°C.

The highest %DR reduction recorded in the test section 0.1 m away after the Orifice plate (Y) is 21.79% at 60°C reaction temperature and the lowest is 0%. This may due to the fact that turbulent flow is fully formed further away after the Orifice plate and not across it thus the CMC is functioning as DRA at the best performance in this test section.

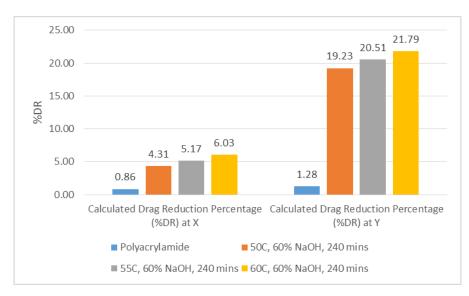


Figure 29: Drag Reduction Percentage (%DR) of samples with highest extraction parameters at different test section.

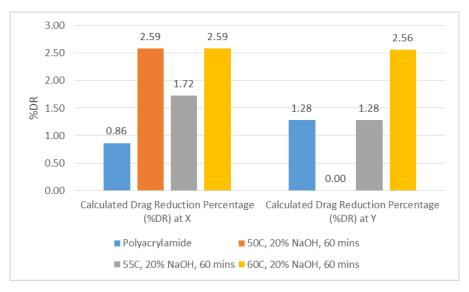


Figure 30: Drag Reduction Percentage (%DR) of samples with lowest extraction parameters at different test section.

From the comparison made of the two graphs shown in both Figure 29 and Figure 30, at highest extraction parameters of the CMC sample from CR, the %DR is greater than the Polyacrylamide (PAM) itself at both test section X and Y; across the Orifice plate and 0.1 m away from the Orifice plate. The highest %DR recorded for PAM is 1.28% while for the CMC sample is 21.79%. This may be due to the fact that the PAM is being degraded during pumping into the flowmeter test setup since the pump used was centrifugal pump.[9] On the other, the CMC extracted from CR can withstand mechanical degradation from the pump.

4.4.1 Calculation for Concentration of DRA Solution

To determine the concentration of DRA solution to be mixed in the tank, the formula from equation (4) was used as shown below:

$$Concentration \ of \ DRA \ (ppm) = \frac{Mass \ of \ DRA}{Volume \ of \ distilled \ water} \times 10^6$$

$$\frac{8 \ grams}{20000 \ mL} \times 10^6 = 400 \ ppm$$

From the above calculation, it shows that the DRA concentration when mixed into the tank will be 400 ppm.

4.4.2 Reynolds Number

In order for the DRA to work in reducing the drag and frictional pressure drop, the liquid flow inside the pipe, fittings and fittings of the flowmeter must be in turbulent flow regime. Calculation of Reynolds number is important to determine the type of flow regime which is the range greater than 5000 (Nre > 5000). The general formula for Reynolds number calculation used is as follow:

$$N_{Re} = \frac{\rho v D}{\mu} \tag{5}$$

Where $\rho = \text{Density (kg/m}^3)$,

v = Velocity (m/s),

D = Diameter (m),

 μ = Viscosity (kg/m.s).

From the flowmeter test experimental setup, the following parameters were recorded:

Flow rate: $20 \text{ L/min} = 3.33 \times 10^{-4} \text{ m}^3/\text{s}$

Diameter of orifice: 0.02 m

Orifice area: 3.1 x 10⁻⁴ m²

Density of water: 1000 kg/m³

Absolute viscosity = $8.937 \times 10^{-4} \text{ Pa.s}$

The velocity of the flow inside the flowmeter is determined by calculation which is derived from the flow rate and area. The calculation is shown as follow:

$$V = \frac{Q}{A} = \frac{3.33 \times 10^{-4}}{3.1 \times 10^{-4}} = 1.074 \, m/s$$

By knowing the velocity, Reynolds number can be calculated by entering all the values from into the formula shown in equation (5):

$$N_{Re} = \frac{1000 \times 1.074 \times 0.02}{8.937 \times 10^{-4}}$$

$$N_{Re} = 24034.9 \gg 5000$$

Since the value of calculated Reynolds number is 24034.9 which is greater than 5000, the flow inside the flowmeter is proved to be turbulent flow regime and hence the testing of DRA is valid using the experimental setup.

4.4.3 Calculation of Drag Reduction

The Drag Reduction Percentage (%DR) calculated in the table were calculated using the following formula:

$$\%DR = [|(\Delta P1 - \Delta P2)|/\Delta P1] \times 100$$
 (6)

Where:

For X (across Orifice plate):

 $\Delta P1 = \text{Differential manometer liquid level (Point 6 - Point 7)}$ without DRA (tap water)

 $\Delta P2$ = Differential manometer liquid level (Point 6 – Point 7) with DRA

For Y (0.1 m after Orifice plate):

 $\Delta P1 = \text{Differential manometer liquid level (Point 6 - Point 8) without DRA (tap water)}$

 $\Delta P2$ = Differential manometer liquid level (Point 6 – Point 8) with DRA

Taking example of Polyacrylamide, the manometer reading at point 6, 7 and 8 are 304mm, 189mm and 227mm respectively.

For X (across the Orifice plate):

 $\Delta P1 = \text{Differential manometer liquid level (Point 6 - Point 7) without DRA (tap water)}$

= 304mm - 189mm

=116mm

 $\Delta P2$ = Differential manometer liquid level (Point 6 – Point 7) with DRA (Polyacrylamide)

= 304mm - 227mm

=78mm

Therefore, the %DR for Polyacrylamide across the Orifice plate (X) is calculated to be:

%DR =
$$[|(\Delta P1 - \Delta P2)|/\Delta P1] \times 100$$

= $[|(116 - 78)|/116] \times 100$
= 0.86%.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From this study, it is now proven that CMC can be produce as a value added product from CR which is a waste or by-product of the coconut industries that is presently utilized as animal feed in Malaysia thus meet the first objective of this research. In the experiment carried out for this research, the amount of CMC extracted is acceptable ranging from as low as 8.987 grams to largest of 28.991 grams as recorded. However, the drawback from this research are the purity and degree of substitution of the produced CMC from CR which are affected by the reaction parameters such as temperature, time and concentration of sodium hydroxide were not covered and identified.

On the other hand, the outcome from this research also concluded that the CMC extracted from CR can function well as DRA which then meet the second objective for this research. This can be seen from the results that there is significant pressure reduction as compared to fully water bearing flowing across the Orifice plate which is the baseline. The performance of the CMC tested with flowmeter experimental setup is at par and better than the performance of PAM which has been used widely in the oil and gas industry since the samples can withstand mechanical degradation of the centrifugal pump as compared to PAM.

Apart from that, the results from the laboratory scale flowmeter test setup is a valid equipment to test the performance of DRA apart from utilizing Double Concentric Cylinder (DCC) Rheometer and flow loop which have been recommended in previous researches.

5.2 Recommendations

Due to time limitation in completing this research, there are several parameters have not been covered and tested from this research. In addition to that, the following recommendations listed are what can be done in the upcoming research on producing DRA from organic wastes.

5.2.1 Explore Various Organic Wastes Available

Different sources and types of organic wastes should be tested and undergone the same research methodology as of this research in order to have consistent results and reliability for comparisons and references in future researches.

5.2.2 Compilation of Tested DRA Sources

A standard DRA databases need to be put in place where researchers all around the globe can make use of and update it from time to time depending on which methodology of producing organic DRA from wastes are used and what kind of tests have been done upon it.

5.2.3 Include Dynamic Reservoir Temperature and Pressure

Apart from time constraint, the lack of high technology experimental setup to test the produced DRA from organic waste is another factor this parameter could not be included in this research. This part of the study should focus more on the thermal and mechanical degradation of the DRA as to be treat as when injecting the DRA down the injection tubing in field case which are continuously exposed to changes in temperature and pressure.

5.2.4 Chemical Reaction between Produced DRA and Inner Wall Tubing

This part of the study should consider several other factors such as pH values, temperature and materials reactivity which may lead to the reaction between the DRA and the inner wall of the tubing itself thus causing less effective drag reduction performance.

5.2.5 Reaction between Formation Rocks and Produced DRA

This is another interesting part of the study which is not able to be covered in details for this research. In this study, one should focus on the reaction of the reservoir rocks once the produced DRA get in contacts and how it response towards it.

5.2.6 Variations of Produced DRA Concentrations

The amount of DRA produced need to be sufficient to varied the concentrations during testing with flow loop. This is because at higher concentration of DRA, the %DR will increase substantially.

5.2.7 Approach on Producing Nano-scale DRA

Size of DRA play an important role with the Degree of Substitution (DS) and thus will determine the solubility of the produced DRA. Besides, there is no research have been done on the effects and performance of Nano-scale DRA.

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