SCALE STUDY: THE GREEN INHIBITORS FOR SILICA POLYMERIZATION

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Dissertation submitted

in partial fulfillment of the requirements for the

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CERTIFICATION OF APPROVAL

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Approved by,

_____

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

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.

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NOR AZMAN BIN NOR AZMI

ABSTRACT

This project is entitled The Green Inhibitors for silica polymerization aimed to identify and evaluate the efficiency of potential green inhibitors from natural extract to be compared with the commercial green inhibitor such as polyaspartate. This project generally has the purpose of providing the alternatives in mitigating the silicate scale in the reservoir which undergone ASP flooding recovery method. Silicate scale deposition during the ASP flooding is considered as the serious problem as it may cause severe damage to the reservoir as well as the equipment used during the production. There is no threshold scale inhibitor identified to completely prevent the silicate scale deposition. But, conventional scale inhibitor has been used to effectively prevent or delay the silicate scale formation for several years. Unfortunately, the usage of conventional scale inhibitor is not biodegradable and may harm the aquamarine environment which has led to the invention of green inhibitor. The usage of green inhibitor is to maintain the integrity of oil industry as well as to abide by the environmental regulations. In this project, several green inhibitors from plants have been identified such as Psidium Guava and Barley which have the inhibitory effects towards scaling. Therefore, experimental procedure is done to evaluate these potential green inhibitors as well the commercial green inhibitor to determine the efficiency of the inhibitors toward silica polymerization in brine solution. The proposed experimental method for this project is the static model test which illustrates the condition of the reservoir with the presence of the suggested green inhibitors. The objective of the experiment is to evaluate the efficiency of the green inhibitor by examining the efficiency of inhibitors with the prepared brine solution containing silica. The results of this project showed that the proposed green inhibitors have the inhibitory capability to delay the scale formation. But the overall process can be improved to enhance the results.

ACKNOWLEDGEMENT

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CHAPTER 1

1.0 INTRODUCTION

This project is related to the problem raised by the application of tertiary enhanced oil recovery method. The severity of the EOR method application is analyzed in the beginning of the project to define the root cause of the problem in order to enhance mitigation alternatives in future application.

1.1 Background of Study

The increasing demand of hydrocarbon as the main sources of energy in the world has urged the industry to develop various methods to improve the recovery of hydrocarbon which is named as Enhanced Oil Recovery (EOR) methods. Alkaline/Surfactant/Polymer (ASP) is one of the methods designed to lower the interfacial tension (IFT), water wet the formation, and decrease water mobility to recover residual oil (Wyatt et al. 2002). This EOR method has enable high increment in the oil recovery and therefore is used in numerous oil fields like Daqing oilfield in China and Southern Alberta. The use of alkali in this EOR method is also considered as the cost effective method as alkali is less costly compared to other equivalent levels of surfactant (Jennifer et al., 2012)

Despite the successful results of ASP usage in recovering hydrocarbon, the alkaline based fluid injection has caused a major problem towards the hydrocarbon production of the related oilfields where the formation of silicate scale has been discovered to be deposited in various locations including the production equipments and the near wellbore area. This silicate scale is originally induced by the high pH of ASP flooding in the reservoir where quartz silica from the rock formation dissolved in the high pH ASP flood and becoming stable. As the ASP flood flows deeper into reservoir and encounters the formation fluid and connate water, the pH of ASP flood is decreased and triggers the formation of silica. The further formation of the scale itself is complex as it involves various factors including silica concentration in the

formation, the temperature of the reservoir and also the present of other common minerals such as calcium and magnesium.

The conventional ways to eliminate scale development can be divided into two: mechanical method and chemical method (Umar & Said, 2013). Mechanical method involves removing the scale physically by drilling or reaming but this method obviously has the most disadvantages in term of the cost expense and formation integrity. Therefore, chemical method has become the largest area where chemical reaction between inhibitors and formation fluid is studied to inhibit the silicate scale formation in the reservoir especially in ASP flooding oilfields.

1.2 Problem Statement

At the early phase of ASP flooding, the silica dissolution occurs in which silica in the formation dissolve into the high pH alkaline flood typically above 10.5 (Jennifer et al., 2012). It is understood that silica dissolution is the initiator of the silicate scale formation in the overall process. However, the idea to cater the problem by adjusting the pH of alkaline solution cannot be simply done. This is because it will basically diverge from the main purpose of injecting ASP which is alkaline based solution to enhance the hydrocarbon recovery.

The later stage is the polymerization of silica when the ASP water flood containing silica encounters connate water which consists of neutral pH 7 of formation fluid. The neutralization of ASP flood water will reduce the solubility of silica and in later process polymerize which further forms colloidal silica nanoparticles. The inhibition of silica scale formation during this stage might be more reasonable because of the stable neutralized pH of the solution in the production well. Therefore, this will lessen variable parameters that need to be considered to develop the best inhibitor for silicate scale formation should be more effective.

Conventional scale inhibitors have been developed based on various organic and inorganic compounds as well as polymers such as phosphonates and phosphates that have high suitability to effectively prevent further growth of silicate particles and

delayed the scale formation (Darling & Rakhspal, 1998). Despite the great functionality of these compounds, they have been recognized as poor ecotoxicity and non-biodegradable scale inhibitors. Therefore, the usage of green inhibitors in oil and gas industry has been the real concern in order to meet the minimum requirements of the environmental regulations.

There are several polymers that have been used as green inhibitors such as Polyacrylic acid (PAC), thermal Polyaspartate (TPA), ammonium difluoride (Darling & Rakhspal, 1998) as well as Polysaccharide. These alternatives need to be tested and studied to determine the most effective green scale inhibitor that can inhibit the polymerization of silica scale.

1.3 Objective

The objectives of this project are:

1) To study the characteristics of existing green inhibitors
2) To identify other potential green inhibitors from natural extract (plant) based on the characteristic studied from existing green inhibitors
3) To test the effectiveness of potential green inhibitors compared to the existing green inhibitors

1.4 Scope of Study

The scope of study for this project are:

1) Deep research to identify the alternative green inhibitors from natural plant based on the characteristics of existing green inhibitors.
2) Setting-up the procedure to extract the natural inhibitors from the identified plant.
3) Setting-up experiment to test and compare the alternative green inhibitors with the commercial green inhibitors.
4) The potential green inhibitors are specifically tested to prevent or delay silica polymerization where effect of other bivalent cation scale formation is excluded.

3

The experiment will use the listed green inhibitors and potential green inhibitors for the overall experiment:

1) Polyaspartate
2) Psidium Guava
3) Barley

This is a lab experiment study that examines the effectiveness of listed inhibitors in the same operating parameters and conditions. Proposed green inhibitors are selected as they contain inhibitory element according to research done.

1.5 Relevancy of the project

The relevancy of the project can be determined by looking at how beneficial this project for future usage. The silica scale problem that emerges in the oil industry brings a lot of misfortune loss in the hydrocarbon production process. Silica scale inhibitor is being developed by various parties to enhance the recovery system. Yet, there is no absolute inhibitor that is classified as the threshold inhibitor for silica scale problem which can completely prevent the scaling of silica mineral. Other than that, the conventional inhibitor used in the current procedure is harmful and toxic to the ecosystem. By doing this project, it can provide the world with the alternatives silica scale inhibitor which can further developed in the future in order to determine the threshold inhibitor for silica scale. Meanwhile, the concept of having natural extract as the alternative inhibitor will definitely provides the solution to save the environment from more harm and toxic chemical waste.

For the author point of view, the project can provide the understanding of the actual hydrocarbon production condition to prepare him for the working life in the future. The fundamental understanding of scaling in oil production is important to enable the student to be innovative and be more focus to provide the solution for the best hydrocarbon recovery condition in the future.

1.6 Feasibility of the project

5

. Based on the study done, the project can be done considering there is no shortage of equipment or chemicals in the future work plan. The experiments can be done in the laboratory facilities provided in the university while the chemicals are available for usage. The test and analysis of the project are expected to be done within 2-3 months period. The basic fundamental procedure on how to reach the objective of the project is already cleared but there might be several alterations in the future for more accurate results and analysis.

CHAPTER 2

2.0   LITERATURE REVIEW

Various papers and books have been reviewed closely to gather the important information on silicate scale formation and ASP application in the industry. The information are divided into categories for better understanding as included in this part.

2.1 Mechanism of Silicate Scale Formation

Silicate scale is the major scale deposition found in the production well and surface facilities which applied the tertiary recovery of ASP flooding (Wyatt et al. 2002). The formation silicate scale is initiated by the high pH of ASP flood which dissolved the quartz in the rock formation. The pH of the dissolution is indicated to be typically 11 and above (Arensdorf et al. 2011). The dissolved quartz is recognized as monomeric silica. The reaction of dissolution of silica (Bashbar, Elraies, & Osgouei, 2013) is shown in figure below:

$$SiO_4 \text{ (quartz)} + NaOH \text{ (alkaline)} \longrightarrow Si(OH)_3O^-Na^+ \text{ (monomeric silica)}$$
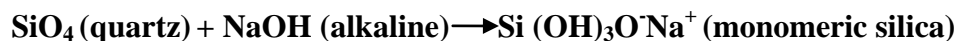
As the ASP flood flow to the production well, it encounter the connate water which neutralize the pH of the flood. The solubility of monomeric silica decrease significantly which leads to the polymerization of silica and forms colloidal silica (Arensdorf et al. 2010). According to (Amjad and Zuhl 2008), the polymerization of silica is rather complicated to be understood. Yet the agreed process that is likely to proceed as shown below:

$$Si(OH)_4 + OH^- \longrightarrow (OH)_3SiO^- + H_2O$$

$$(OH)_3SiO^- + Si(OH)_4 \longrightarrow (OH)_3Si - O -- Si(OH)_3 \text{ (dimer)} + OH^-$$

$$Dimer \longrightarrow Cyclic \longrightarrow Colloidal \longrightarrow Amorphous Silica \text{ (scale)}$$
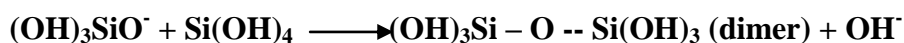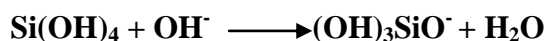
Connate water in the production well contains other minerals such as Mg^+ that can bridge the colloidal silicate particles and forms magnesium silicate scale which is more stable and harmful.

2.2 Mechanism of Silicate Scale inhibition

Scale inhibition is basically the chemicals that prevent or delay the formation of scale when injected into the scaling water (Umar & Said, 2013). The inhibition mechanism that are widely adapted in the inhibition process are the absorption of chemicals onto crystal surface of precipitate to retard the further growth of it or by adhering to metal surface to prevent the deposition of scale on it (Conne, 1983). The first mechanism is mainly targeting to cater the scale problem at the earlier stage to ensure that scale formation is not overlooked until it becomes severe. According to (Demadis, Stathoulopoulou, & Ketsetzi, 2007) the silicate inhibition can be illustrated as shown below:
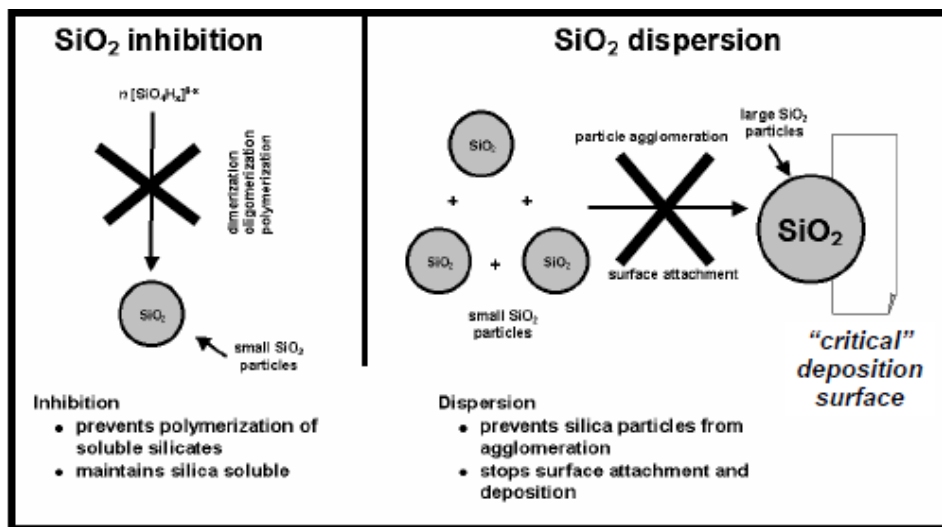
Figure 1 Two major approach for silica inhibition
(Demadis, Stathoulopoulou, & Ketsetzi, 2007)

The inhibition mechanism shown above is mainly applied in the water treatment system. However it shows the importance of preventing the polymerization of soluble silicate in the scaling water also can be related to the oilfield condition. The prevention of the silicate polymerization can be referred back to the crystals growth retardation which is the cause of chemicals induction into the system. Other than

that, the injection of inhibitor also will enable the maintenance of silica solubility in the scaling water which prevents it to be precipitated.

2.3 Scale Inhibitors

The common scale inhibitor or conventional scale inhibitor normally used are the phosponate-based inhibitor in the oil and gas industry. However the usage the phosponate-based chemical is quite harmful to the environment (Darling & Rakhspal, 1998). The inhibitor is in fact will be released to the environment once the inhibition is done. It has become the emerging concern to the world that the toxicity and negative impact of the discharged chemicals to the open sea may have endangered the marine ecosystem (Wilson D., 2010). In order to abide to the environmental regulations and to keep producing hydrocarbon without costing the ecosystem, environmental friendly green inhibitors are developed and used in scale inhibition process. Even though the usage of green inhibitors is still in the early stage, there are several environmental acceptable and reliable green inhibitors that have been used in the production well. The most popular green inhibitor discovered is the polyaspartate. This poly aspartic acid is discovered to be naturally biodegradable, possess good adsorption and desorption properties (Amjad Z., 2008). Listed below are Polyaspartate and possible green sources of scale inhibitor.

Table 1 Green Sources of Scale Inhibitor

PASP	1) Main component for inhibiting ability: Carboxylate group. 2) Synthesized from Aspartic acid through Thermal Polycondensations with or without acid catalyst.
Barley	1) Barley contains several vitamins and minerals including niacin (Vitamin B3), thiamine (Vitamin B1), selenium, iron. 2) Contained high content of inulin which possibly useful potential for scale inhibitor.

Psidium Guajava	1) Used in various fields.
	2) Often used as medicinal substances in curing inflammation, diarrhea and cancer.
	3) Contain various group of component that may be beneficial as antiscalant.

Based on the literature review, it is difficult to identify the exact mechanism of the scaling problem as the process is involving complicated brine chemistry which cannot be monitored closely. The scale inhibition process can be applied in the general way without specifying the exact scaling mechanism to be catered. Besides that, the scale inhibitors studies done showed that no threshold inhibitors have been developed to fully inhibit the silicate scale formation; therefore this project can be the platform to enhance the development of silicate scale inhibitor for the oil and gas industry application.

CHAPTER 3

3.0   METHODOLOGY

The methodology of this project includes the methods and procedures used to complete the experimental work done. Besides that, the overall progress and flow of activities throughout this project is also included. This part is important to ensure the smooth progress of the project within the time allocated.

3.1 Project Experimental Procedure

3.1.1 Compatibility test

The compatibility test is conducted as the preliminary test to examine the effects of green inhibitors in different concentrations towards the formation of silicate scale. The test conducted is considered as the base to experiment to examine the compatibility of the sample and the solvent as well as the compatibility of green inhibitors prepared mixed with the brine solution.

3.1.1.1 Sample and Brine Water Preparation

- *Preparation of Psidium Guajava and Barley sample*
  All samples (figure 6) were cleaned with running tap followed by rinsing it with distilled water. The grains are dried overnight for 12 hours and then in a Memmert oven at 50°C for 8 hours. The dried samples were grinded into powdery form using an electrical blender. All the samples powders were passed through a sieve to separate the coarse particles. The samples are stored in clean bottles for further use.
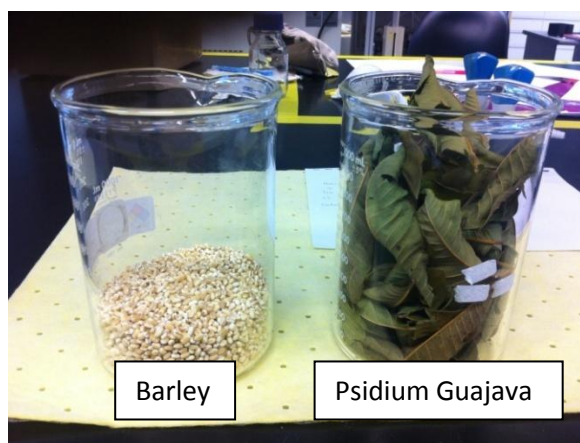
Figure 2 Green samples

- *Preparation of green inhibitors extracts*

  The selected concentrations of green extracts are 100ppm, 1000ppm, 10000ppm and 50000ppm. Green samples are weighted to 0.01g, 0.1g, 1g and 5g and added to 100 ml of distilled water respectively. The prepared solution is stirred for 30 minutes using a magnetic stirrer.

- *Preparation of ASP, formation anion and formation cation water:*

  1) A selected amount of NaCl is weighed and added into 1000ml beaker. The salt is dissolved by the addition of adequate distilled water. It is stirred for 15 minutes.

  2) The additional salts as listed below are weighed into separate containers, dissolved and stirred before being added to 1000ml beaker containing the NaCl.

  3) The solutions prepared are transferred to 1000ml flask and mixed.

  4) The mixed solution of ASP water is then diluted to 1 L in 1000ml volumetric flask and stirred for 30 minutes.

  5) The pH of the solution is adjusted to 7 using 10% HCl or 10% NaOH. The prepared solution of ASP, formation anion and formation cation water is mixed at a ratio of 2:1:1 respectively.

  6) The final solution is the blank solution to be tested with different concentrations of green inhibitors.

  7) The procedures are repeated for formation cation and formation anion.

Table 2 Solution for Compatibility Test (Arensdorf et al, 2010)

	g/L		
	ASP Water	Connate anion water	Connate cation water
NaCl	2.5	2.4	3.5
KCl	0.08		0.16
NaHCO3	2.8	5.6	
Na2SO4	0.3	0.6	
Na2SiO3-5H2O	14.2		

3.1.1.2 Compatibility Test Procedure

1) Different concentrations of each green inhibitor (100 ppm, 1000 ppm, 5000 ppm, 10000 ppm) is to be tested into the 4 other blank solution (mixed solution of ASP, formation anion and formation cation water) to observe the scaling reaction.
2) The test is conducted at 25°C and 85°C
3) The turbidity of the solution is measured by using observation.

Table 3 Table 5 Compatibility Test result table

Scale Inhibitor	Concentration (ppm)	Initial observation	Observation after 24 hours
Garlic extract	100		
	1000		
	10000		
	50000		
Grains extract	100		
	1000		
	10000		
	50000		

3.1.2 The Static Bottle Test

The static bottle test method is used to evaluate the efficiency of green inhibitors to prevent the silica polymerization and inhibit the silicate scale precipitation. The method consists of three steps which are:

1. Determine the efficiency of each green inhibitor based on turbidity measured using turbidimeter.
2. Determine the efficiency of green inhibitor based on the absorbance measurement of silicate in the solution examined by using spectrophotometer.

For each green inhibitor, a synthetic brine solution is prepared to imitate the brine solution in the production well. This is the preparation of the blank solution to prove the formation of scale in the absent of inhibitors.

3.1.2.1 Sample and ASP Water Preparation

- *Preparation of Psidium Guajava and Barley sample*
  All samples were cleaned with running tap followed by rinsing it with distilled water. The grains are dried overnight for 12 hours and then in a Memmert oven at 50°C for 8 hours. The dried samples were grinded into powdery form using an electrical blender. All the samples powders were passed through a sieve to separate the coarse particles. The samples are stored in clean bottles for further use.

- *Plant samples extraction using Soxhlet Extraction*
  The powdered samples were extracted using soxhlet extractor to improve the extraction. The samples were measured and placed into the extraction thimble. The thimbles were placed into extraction chamber and suspended above ethanol solvent. The soxhlet extraction method included a condenser that converted solvent evaporates when heated into liquid. The liquid form is mixed with samples powder to produce green inhibitors extracts.
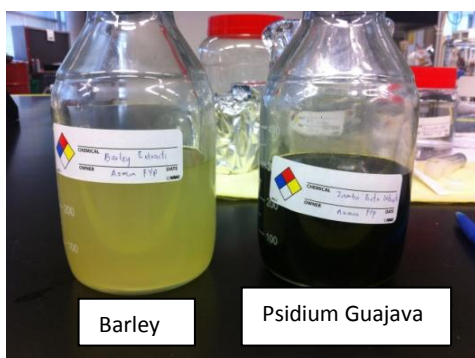
Figure 3 Green samples extracts

Figure 4 Soxhlet Extraction experiment

Preparation of PASP/Green extracts blends

1) Different concentrations PASP/Green extracts blend is prepared as below
   o 1% PASP + 3% Green extracts
   o 1% PASP + 5% Green extracts

2) In order to prepare 1% PASP + 3% Green extracts sample; 1g of PASP is added to 3g of green extracts in a flask. The flask is filled with distilled water until it is 100g. .

3) The procedure is repeated for another concentration using 5g of green extracts.

4) A 4% and 6% of PASP solutions is prepared to be compared with the 1% PASP + 3% Green extracts and 1% PASP + 5% Green extracts throughout the experiment.

5) The PASP/Green extracts blend is classified as below:
   o PASP + Psidium Guajava   = SI 1
   o PASP + Barley   = SI 2
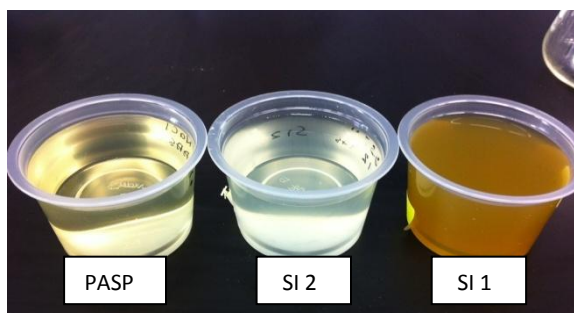
Figure 5 PASP and PASP/Green extracts

- *Preparation of ASP, formation anion and formation cation water:*
  1) A selected amount of NaCl is weighed and added into 1000ml beaker. The salt is dissolved by the addition of adequate distilled water. It is stirred for 15 minutes.
  2) The additional salts as listed below are weighed into separate containers, dissolved and stirred before being added to 1000ml beaker containing the NaCl.
  3) The solutions prepared are transferred to 1000ml flask and mixed.
  4) The mixed solution of ASP water is then diluted to 1 L in 1000ml volumetric flask and stirred for 30 minutes.
  5) The pH of the solution is adjusted to 7 using 10% HCl or 10% NaOH.
  6) The procedures are repeated for formation cation and formation anion water.

Table 4 Solution for Static Bottle Test (Arensdorf et al, 2010)

| | g/L | | |
	ASP Water	Connate anion water	Connate cation water
NaCl	2.5	2.4	3.5
KCl	0.08		0.16
NaHCO3	2.8	5.6	
Na2SO4	0.3	0.6	
Na2SiO3-5H2O	10.7		
MgCl2-6H2O			0.5
CaCl2-2H2O			0.6
BaCl2-2H2O			0.06
pH	10.8	6.8	

- Additional salts are added to enhancing the scaling reaction so that the inhibitors can be tested.

3.1.2.2 Static Bottle Test Procedure

The experimental procedure is listed as below:

1) The brine solution is prepared according to a ratio of 2:1:1 mixture of ASP water, formation cation water and formation anion water respectively.

2) 5 ml of formation anion water is added to a beaker of 50 ml,. The green inhibitor to be tested is added at this point.

3) 5 ml of formation cation water is added later into the beaker and the mixed solution is shaked thoroughly.

4) 10 ml of ASP water is later added to the solution and mixed well by shaking.

5) The procedure is repeated for each green inhibitors prepared earlier.

6) The bottle of each mixture is marked for later experiment and analysis process.

7) The prepared mixture is then heated in an oven at 80°C to imitate the condition of reservoir.

8) The static bottle test is completed by applying turbidimeter test and spectrophotometer test.

Figure 6 Mixture of brine
solution and green inhibitors

Figure 7 ASP, formation cation
and formation anion water

- *Turbidimeter Test*

The solution prepared for static bottle test is examined by using a Hach 2100Q portable turbidimeter. The equipment is used to measure the turbidity of the solution as a measure of precipitation of silicate scales in the solution. The solution prepared is placed into the cell sample and examined by using the turbidimeter to detect the amount of absorbed light which is recorded in turbidity measurement with the unit NTU. The turbidity of the solutions are determined at three times: t=0 h, t=2 h and t=24 h.The results are filled into a table for better analysis.

Table 5 Turbidimeter Results table

	SI	NTU/Hours			
		0	1	2	24
1% & 3%	PASP				
	SI 1				
	SI 2				
	Blank				

- o SI 1 = extract blend of PASP + Psidium Guajava
- o SI 2 = extract blend of PASP + Barley

Figure 8 Hach 2100Q Turbidimeter

- *Spectrophotometer Test*

The spectrophotometer used in this project is the T60U (UV-Visible) that has a wavelength range of 190nm – 1100nm. The main function of this equipment used in static bottle test is to measure the absorbance of wavelength transmitted through the solution sample. The absorbance measured is a dimensionless measurement which can be used to calculate the efficiency of green inhibitors towards inhibiting the scale formation in the form of precipitation. The procedures for testing by using spectrophotometer are listed below:

1) A liquid holder is filled with solution to be tested until the full indicator.
2) A blank liquid holder is placed into the cell holder at cell no 1.
3) The solution liquid holder is placed into the cell holder at cell no 2.
4) A wavelength range of 380nm is set. This is considered as the visible wavelength.
5) Zero button is pressed to start calibration.
6) Run button is pressed to start the absorbance measurement of test solution.
7) The results are tabulated for further analysis

Table 6 Absorbance measurement table example

	SI	Absorbance/Hour			
		0	1	2	24
1% & 3%	PASP				
	SI 1				
	SI 2				
	Blank				

The efficiency of green inhibitor can be calculated by using the formula below:

$$\% \, Efficiency = \left[1 - \frac{(At - Ao)}{(Abt)}\right] x \, 100$$

Where:

- At = Absorbance in brine solution at t= 1,2,24 hrs
- Ao = Absorbance in brine solution at t=0 hrs
- Abt = Absorbance of blank solution at time = t

3.1.3 Materials and Tools

This project requires potential inhibitors which derived from local natural resources. Besides that, certain chemicals are also needed for the preparation of the solutions.

Materials needed:

- Barley grains - Barium Chloride , $BaCl_2$ - Fresh Jambu Batu (Psidium Guajava) - Calcium Chloride dihydrate, $CaCl_2.2H_2O$ - Potassium Chloride, KCl	- Magnesium Chloride, $MgCl_2.6H_2O$ - Sodium Chloride, NaCl - Sodium Bicarbonate, $NaHCO_3$ - Sodium Silicate, Na_2SiO_3 - Sodium Sulphate, Na_2SO_4

Equipment needed:

For extraction: - Heater stirrer - Electronic scale - Aluminium foil - Spatula - Beakers - Sieve - Glass bottles - Electrical grinder - Soxhlet Extractor - Memmert Oven	For experimentation - Turbidimeter - Spectrophotometer - Syringe - Glass bottles - Oven **Figure 9 T60U Spectrophotometer**

3.2 Phases of project activities
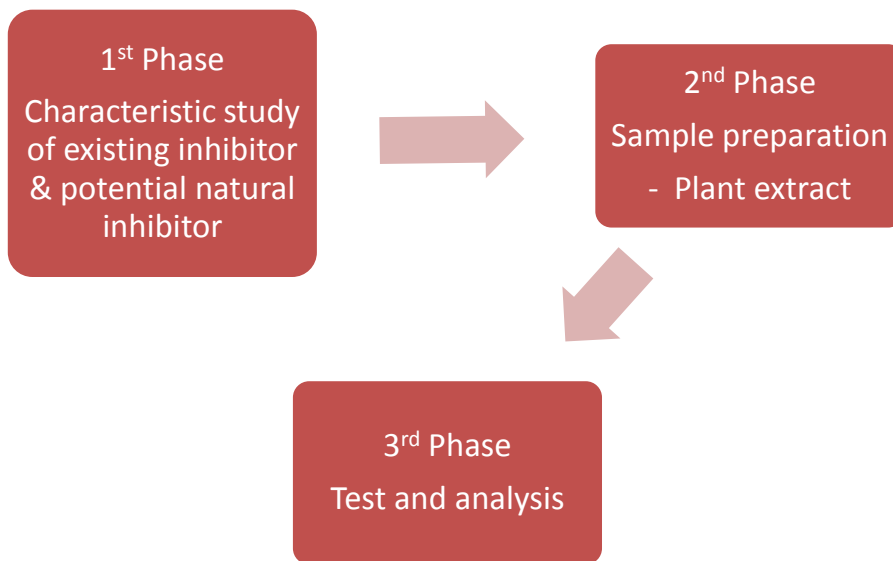
Figure 10 Phases of project

3.3 Project Flow Chart
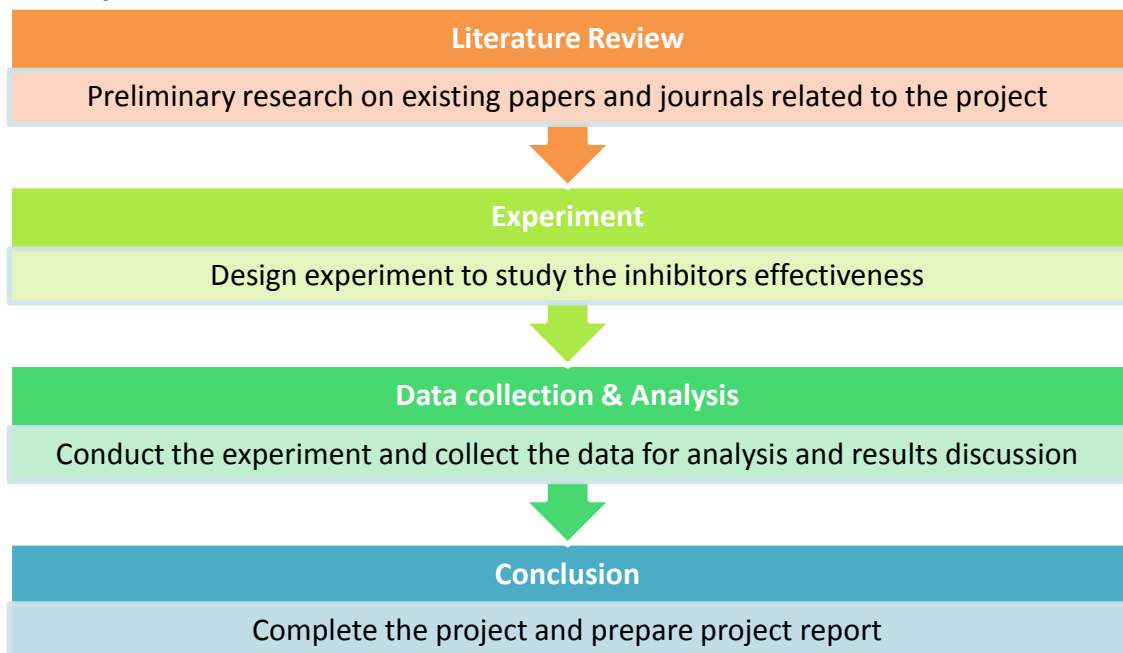
Figure 11 Project Flow Chart

3.4 Project Gantt Chart of FYP

Table 7 FYP 1 Gantt Chart

Detail/Week	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						■								
Proposal Defence								■	■					
Project work continues										■	■	■		
Submission of Interim Draft Report													■	
Submission of Interim Report														■

Table 8 FYP 2 Gantt Chart

Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Project Work continues	■	■	■	■	■	■	■								
Submission of Progress Report								■							
Project Work Continues									■	■	■	■			
Pre - SEDEX										■					
Submission of Draft Final Report											■				
Submission of Dissertation (soft)												■			
Submission of Technical Paper												■			
Viva														■	
Submission of Project Dissertation													■		

3.5 Key Milestone

Proposal Defence (week 9)

Input study acquisition & Experiment familiarization and planning (week 10 - 12)

Improvise report draft submission (week 13)

Completed interim report submission (week 14)

Proceed with 2nd semester – Project implementation and completion(week 1 - 14)

Figure 12 Key Milestone of FYP 1

Submission of Progress Report (week 8)

Project works continues until completion (week 9 - 12)

Submission Of Dissertation - soft copy and submission of Technical Paper (week 12)

Viva (week 13)

Submission of Project Dissertation - hardbound (week 15)

Figure 13 Key Milestone of FYP 2

CHAPTER 4

4.0 RESULTS AND DISCUSSION

The results of both compatibility test and static bottle test are included in this part. The results are analyzed and relates to the objectives of this project.

4.1 Compatibility Test

4.1.1 Results

Based on the laboratory tests, the results for compatibility test are tabulated as below:

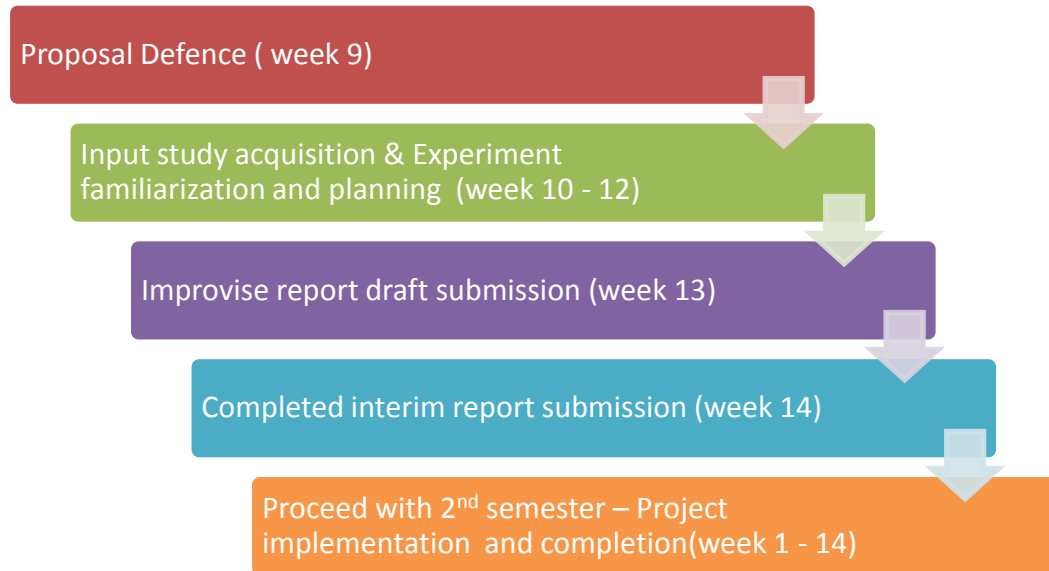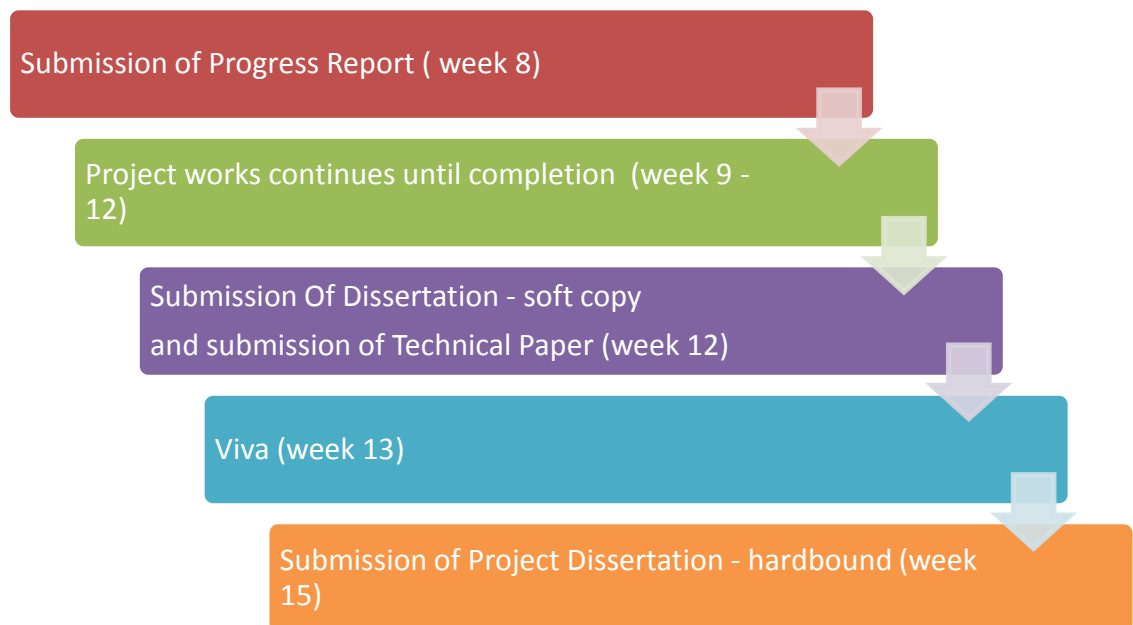Table 9 Compatibility Test Results at 25 °C

25°C	Scale Inhibitor	Concentration (ppm)	Initial observation	Observation after 24 hours
	Blank Solution	-	Hazy solution	Hazy solution
	Psidium Guava	100	Clear solution	Hazy solution
		1000	Clear solution	Hazy solution
		10000	Clear solution	Clear solution
		50000	Clear solution	Hazy solution
	Barley extract	100	Clear solution	Hazy solution
		1000	Clear solution	Hazy solution
		10000	Hazy solution	Hazy solution
		50000	Hazy solution	Hazy solution

Table 10 Compatibility Test Results at 85 ºC

85°C	Scale Inhibitor	Concentration (ppm)	Initial observation	Observation after 24 hours
	Blank Solution	-	Hazy solution	Hazy solution
	Psidium Guava	100	Clear solution	Hazy solution
		1000	Clear solution	Hazy solution
		10000	Clear solution	Hazy solution
		50000	Clear solution	Hazy solution
	Barley extract	100	Clear solution	Hazy solution
		1000	Clear solution	Hazy solution
		10000	Clear solution	Hazy solution

		50000	Clear solution	Hazy solution

4.1.2 Discussions

Table 9 and Table 10 show the compatibility of all green inhibitors (Psidium Guajava extract and Barley extract) in different concentrations of synthetic brine water with the absence of mg^+ and Ca^+ ions to avoid quick silicate formation. The tests conducted at two temperatures show different results that may be due to the nature of the brine chemistry that adversely impacts the scale inhibitor performance in some ways. The test conducted at 85°C shows consistent trend of extracts compatibility compared to another test. From Table 10, it is shown that the extracts are compatible with all concentrations of brine solution at the early observation and cloudy solution is observed after 24 hours due to precipitation of scale. The test conducted at 25°C shows incompatibility of mixture at high concentration of brine (5000ppm, 10000ppm) and all the mixtures show cloudiness at later time. By comparing the two different test temperatures, it was analyzed that the higher temperature of 85°C will create a better condition for the reaction of green inhibitors with brine solution in order to inhibit the scale formation. This temperature is chosen to imitate the condition of reservoir with high temperature. The haziness of solution observed after 24 hours shows the precipitation of silicate scale in the existence of green inhibitors. These results explained that there are no threshold inhibitors at any condition that can completely inhibit the formation of silicate scale. Therefore, the focus of further experiment is to find the best inhibitor that can delay the formation of scale and to be compared with the performance of conventional green inhibitor such as polyaspartate (PASP).

4.2 Static Bottle Test

4.2.1 Turbidity Experiment

The turbidity experiment is a part of static bottle test to analyze the formation of silicate scale in term of precipitation. The test is analyzed using turbidimeter.

4.2.1.1 Results and discussions

<u>1% PASP + 3% Extracts Blends</u>

Table 11 Turbidity measurement of mixture (1%PASP + 3%Extracts blends)

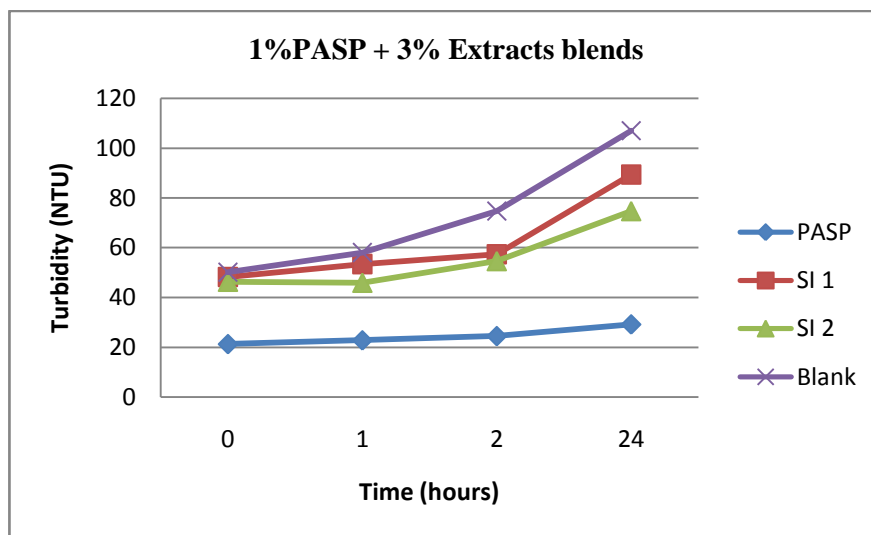|	SI	NTU/Hours			
		0	1	2	24
1% & 3%	PASP	21.3	22.8	24.5	29.2
	SI 1	48.2	53.4	57.3	89.4
	SI 2	46.3	45.9	54.6	79.5
	Blank	50.2	58.1	74.7	107

Figure 14 Graph of turbidity measurement of 1%PASP + 3% extracts blends

<u>Discussions</u>

PASP tested in this experiment significantly showed a good performance of a scale inhibitor. Based on the turbidity measured, it is able to delay the precipitation until 24 hours where the turbidity is not reaching the turbidity of blank (without inhibitor)

at any time. On the other hand, SI 2 showed better delaying performance compared to SI 1. SI 2 turbidity level is lower than blank sample (t=0) as well as SI 1 between 0 – 2 hours. This indicates a better delaying ability of SI 2 compared to SI 1.

<u>1% PASP + 5% Extracts Blends</u>

Table 12 Turbidity measurement of mixture (1%PASP + 5%Extracts blends)

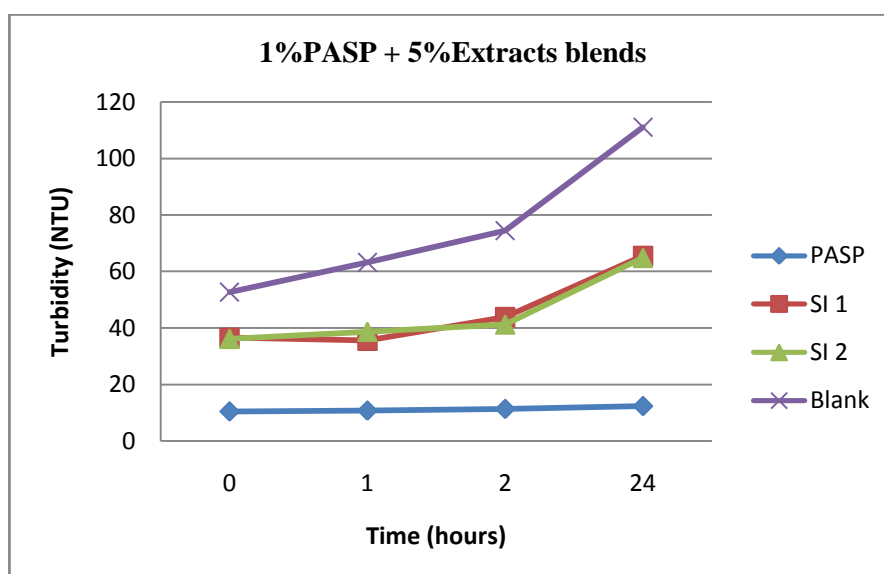|	SI	NTU/Hours			
		0	1	2	24
1% & 5%	PASP	10.4	10.7	11.3	12.3
	SI 1	36.5	35.5	43.8	65.5
	SI 2	36.1	38.6	41.2	64.8
	Blank	52.7	63.2	74.4	111

Figure 15 Graph of turbidity measurement of 1%PASP + 5% extracts blends

<u>Discussions</u>

PASP showed the best performance as shown previously in lesser inhibitor concentration. As the concentration of the extracts increase, the performance of both SI 1 and SI 2 showed significant improvement in term of delaying ability. Both inhibitors turbidity levels are obviously lower than the blank at hour 0, 1 and 2. These indicate good inhibition ability of both inhibitors at higher concentration. Based on the trend displayed, SI 1 and S1 2 are assumed to reach the turbidity level of blank (t=0) at a time of more than 6 hours. Eventhough both inhibitors showed

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similar performance, SI 2 is slightly better with lower turbidity measured throughout the experiment compared to SI 1.

4.2.2 Spectrophotometer Experiment

4.2.2.1 Results and discussions

1% PASP + 3% Extracts Blends

Table 13 Absorbance measurement (1%PASP + 3%Extracts Blend)

	SI	Absorbance/Hours			
		0	1	2	24
1% & 3%	PASP	0.093	0.093	0.095	0.102
	SI 1	0.236	0.272	0.328	0.376
	SI 2	0.224	0.243	0.252	0.316
	Blank	0.242	0.295	0.345	0.389

Table 14 Efficiency Calculation (1%PASP + 3%Extracts Blends)

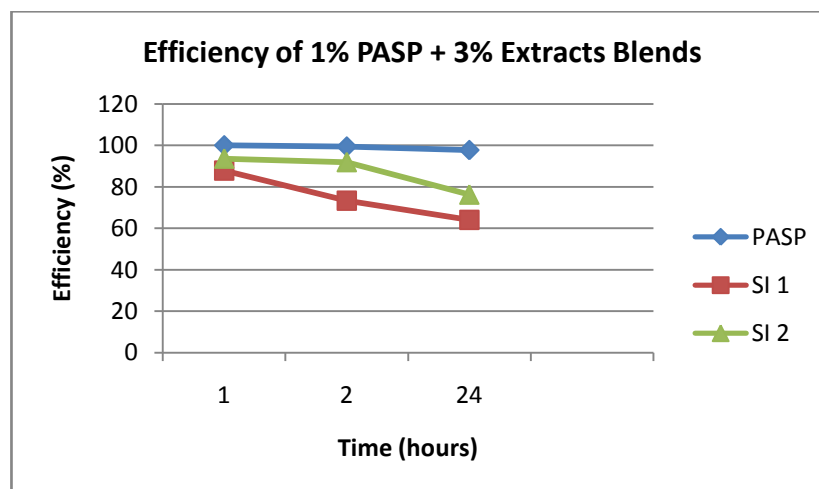|	SI	Efficiency (%)		
		1	2	24
1% & 3%	PASP	100	99.4	97.7
	SI 1	87.8	73.3	64
	SI 2	93.6	91.9	76.3

Figure 16 Efficiency Graph of (1%PASP + 3%Extracts Blends)

Discussions

The absorbance measurement indicates the concentration of silicate precipitation in the solution. The higher the absorbance values indicate higher precipitation formed in the solution. The absorbance values of SI 1 and SI 2 are significantly higher than the absorbance values of PASP. This shows that both proposed inhibitors are lacking in inhibiting the precipitation of scale. On the other hand, the efficiency calculation shows a good result for each inhibitor. The efficiency results can be used to identify

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the delaying ability of the inhibitors. SI 2 is observed to have better efficiency compared to SI 1. But, both inhibitors are unable to inhibit the scale formation during the early reaction.

1% PASP + 5% Extracts Blends

	SI	Absorbance/Hours			
		0	1	2	24
1% & 5%	PASP	0.041	0.041	0.044	0.049
	SI 1	0.193	0.226	0.283	0.323
	SI 2	0.191	0.21	0.218	0.281
	Blank	0.242	0.295	0.345	0.389

Table 15 Absorbance measurement (1%PASP + 5%Extracts Blend)

Table 16 Efficiency Calculation (1%PASP + 5%Extracts Blends)

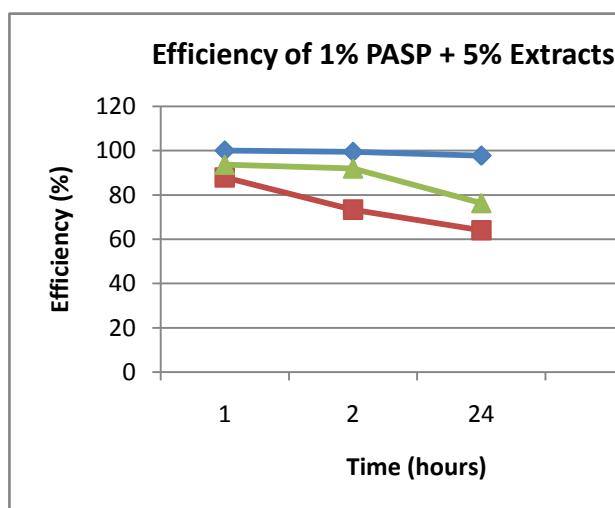|	SI	Efficiency (%)		
		1	2	24
1% & 5%	PASP	100	99.1	97.9
	SI 1	88.8	73.9	66.6
	SI 2	93.6	92.2	76.8

Figure 17 Efficiency Graph of (1%PASP + 5%Extracts Blends)

Discussions

Higher concentrations of PASP + Extracts Blends are still not able to compete with the conventional inhibitor in terms of inhibiting the scale formation at first hand. But the higher concentrations of proposed inhibitors used has show slight improvements in delaying the precipitation of silicate scale. For both concentration of proposed inhibitor used, SI 2 consistently shown better delaying ability compare to SI 1. After 24 hours, SI 2 recorded 76.8 % of efficiency compared to 66.6% of SI 1.

CHAPTER 5

5.0 CONCLUSION AND RECOMMENDATION

The overall conclusions and recommendations of this project are described briefly in this part.

5.1 Conclusion

Barley and Psidium Guajava extractions are selected to be the proposed inhibitors from the early stage of this project based on the research done. Both green plants contain identified components that may have the inhibitory effects toward scale formation. The green plants are examined by using compatibility test and static test method to identify their ability to inhibit or delaying the formation of silicate scale in the presence of ASP water. Various equipment namely turbidimeter and spectrophotometer are used in the evaluation process to examine the performance of the green plants towards scale inhibition. The compatibility test done shown that both green plants are compatible with all the different concentrations of brine solution when the test is done at 85°C. This indicates that the inhibitors will have better performance in reservoir compared to the surface. The performances of the green plants are then compared with the conventional green inhibitor which is Polyaspartate (PASP) through static bottle test. PASP showed the best performance throughout the experiment. Based on the test, Barley showed better inhibiting capability compared to Psidium Guajava where Barley has better delaying ability throughout the 24 hours of test. The performance of both inhibitors showed a significant improvement in their delaying capability as the concentration of the green plants extracts are increase.

In conclusion, the results show that both Barley and Psidium Guajava extracts have the potentials to be developed as an alternative for silicate scale inhibitor based on the laboratory tests done to examine both extracts performance compared to the existing conventional green inhibitor.

5.2 Recommendations

In order to further develop the alternative scale inhibitors, further research and more experiments will have to be performed on the listed green plants. The recommendations for further study of the potentials green inhibitors are as below:

1) The proposed inhibitors need to be tested at more different concentrations.
2) The proposed inhibitors need to be tested at more range of reservoir concentrations.
3) The observation need to be done at more frequent timing
4) Research needs to be done to study on additives required to further inhibit scale formation.
5) Researches and experiments need to be done to identify the active component of both alternative green inhibitors. This is to ensure a proper classification of each inhibitor can be done and enhancement can be applied to improve the performance of the scale inhibitor.
6) More sample of green plants need to be examined and analyzed to provide a wide alternative of green inhibitors for the industry.
7) Despite the delaying ability of the inhibitors, the capability to efficiently inhibit the scale formation at the early phase needs to be improved for more effective result.

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