

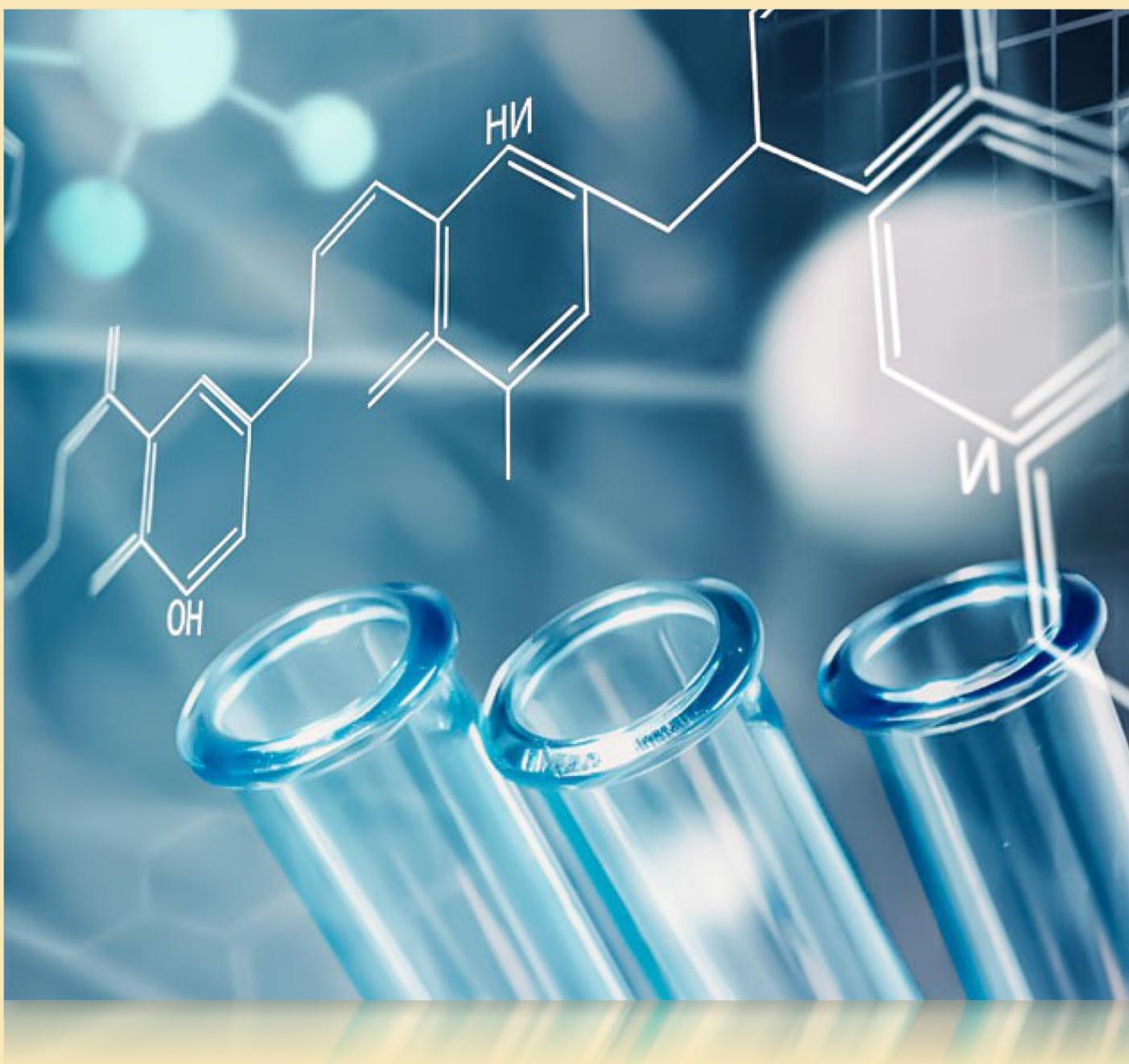
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SCIENTIFIC CONTRIBUTIONS OIL & GAS

Volume 45, No. 2, August 2022

SCIENTIFIC CONTRIBUTIONS OIL & GAS is a journal for the dissemination of information on research activities, technology engineering development and laboratory testing in the oil and gas field.

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SCIENTIFIC CONTRIBUTIONS OIL & GAS

Volume 45, No. 2, August 2022

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PREFACE

Dear Readers,

Scientific Oil and Gas Contribution this edition proudly presents five manuscripts discussing the interesting topics in oil and gas industries. In this edition, we discuss the use of polymer solution is expected to increase the viscosity value of the displacement fluid so that it can form a “piston-like” to increase the volumetric sweep efficiency of the light oil reservoir.

Other topic in this edition, alcohol has the potential to be used as an alternative to fossil fuels to reduce total emissions from spark-ignition (SI) engines. The impact of a mixture of 20% methanol and ethanol in gasoline on the compatibility of Ethylene Propylene Diene Monomer (EPDM) and polyamide materials, which are used as fuel hoses in SI vehicles.

Briefly, polymers are often used to increase oil recovery by improving sweeping efficiency. The screening was carried out as a first step in evaluating the test parameters of several polymers of the Hydrolyzed Polyacrylamide (HPAM) type in fluid and sandstone reservoir rocks.

Next, the process hydrocracking methyl ester of palm oil into fractions biogasoline by faujasite catalyst of fly ash impregnated with nickel have been made. Preparation for faujasite synthesis of fly ash can be done by removing organic compound and refluxing HCl. Synthesis do by melting the fly ash which has been prepared with NaOH 1: 1.2 and in aging for 8 hours and in the hydrothermal autoclave for 24 hours.

The last topic discusses wax deposit is one of the major flow assurance experienced in the process of oil production and transportation from sub- surface to surface. Large amounts of data are required to perform modeling using existing thermodynamic models such as carbon number data from HGTC.

The Editorial Board and the Publisher Council would like to thank Reviewers, Expert Editors, Editors, and Authors who have contributed results of their research to the 2nd edition of Scientific Contribution Oil and Gas

Jakarta, August 2022
Best regards,

Editorial Board

ABSTRACTS

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Dadan DSM Saputra, Bayu D Prasetyo, Hes-tuti Eni, Yudha Taufantri, Ghifahri Damara, Yusuf D Rendragraha, Testing Center for Oil and Gas LEMIGAS, Jl. Ciledug Raya, Kav. 109, Cipulir, Kebayoran Lama, Jakarta Selatan 12230 Indonesia, Email: dadan.saputra@esdm.go.id

Investigation of Polymer Flood Performance in Light Oil Reservoir: Laboratory Case Study

Scientific Contributions Oil & Gas, August 2022, Volume 45, Number 2, pp. 81-86.

ABSTRACT

The use of polymer solutions in the application of chemical EOR injection technology has a role in increasing oil recovery efforts by improving oil mobility in porous media. The addition of the polymer solution is expected to increase the viscosity value of the displacement fluid so that it can form a "piston-like" effect to increase the volumetric sweep efficiency of the light oil reservoir. The polymer used in this study was HPAM using 3 concentrations, namely 500 ppm, 1000 ppm, and 1500 ppm conducted at a temperature of 70 °C. The rheology test of the polymer included concentration vs temperature and shear rate vs viscosity. Thermal stability testing of polymer for 7, 14, 30, 60, and 90 days at 70 °C was done to determine the stability of the polymer solution. Filtration testing was conducted with the criteria of FR <1.2. The static adsorption test has been done with the standard limit of adsorption value <400 µg / gr. Polymer injectivity test using 3 variations of injection rates and coreflooding test were conducted to determine the reduction of Sor in reservoirs due to polymer displacement. From the polymer testing stage, it was found that HPAM polymers at 3 concentrations were compatible with the injection. This is indicated with the clear solution for 3 concentrations at room temperature and 70 °C. The rheology test results showed that the polymer solution with 3 concentrations was decreased in viscosity with the addition of the shear rate value. In the thermal stability test, the viscosity value of the HPAM with 500 ppm was relatively constant. The value

of the FR for HPAM 500 ppm is 1.1, HPAM 1000 ppm is 1.07 and HPAM 1500 ppm is 1.03. The results of the static adsorption test showed the lowest HPAM value of 500 ppm was 156 µg/gr. In the injectivity test results, the resistance residual factor (RRF) values at injection rates of 0.3, 0.6, and 1 cc/min were 0.8, 1.04, and 1.12. The RRF value was close to 1, indicating that after injection of 500 ppm of HPAM tended to not experience plugging. Polymer flooding shows the oil recovery factor (RF) of water injection is 39% OOIP, and RF after polymer injection with 0.35 PV with flush water is 13.5% OOIP or 22% Sor. Knowing the behavior of HPAM polymer with various concentrations to be used for chemical EOR injection, it could provide advantages for future implementation in the light oil reservoir in Indonesia

(Author)

Keywords: filtration, injectivity, light oil, polymer flooding, rheology.

Nurmajid Abdurrojaq, Rizal Zaelani, Belva Adam Haley, Nur Allif Fathurrahman, Riesta Anggarani, Cahyo Setyo Wibowo, Maymuchar, Fuel and Lubricant Technology, Department of Product Application Technology, Testing Center for Oil and Gas LEMIGAS, Jl. Ciledug Raya, Kav 109, Cipulir, Kebayoran Lama, Jakarta Selatan, Indonesia, Email: nurmajid99@gmail.com

The Effect of Methanol-Gasoline (M20) and Ethanol-Gasoline (E20) Blends on Material Compatibility

Scientific Contributions Oil & Gas, August 2022, Volume 45, Number 2, pp. 87-94.

ABSTRACT

Alcohol has the potential to be used as an alternative to fossil fuels to reduce total emissions from spark-ignition (SI) engines. The impact of a mixture of 20% methanol and ethanol in gasoline on the compatibility of Ethylene Propylene Diene Monomer (EPDM) and polyamide materials, which are used as fuel hoses in SI vehicles, is presented in this study. The immersion test

methodology was employed to study the influence of both types of alcohol on gasoline blend to compatibility properties i.e., hardness and weight change. Based on the result, EPDM and polyamide materials have different characteristics of material compatibility with E20 and M20. Tests on M20 and E20 fuel samples on EPDM material show a higher effect on hardness by 5-9% than pristine gasoline. Additionally, there was no change in the weight of the polyamide material in the RON 90, E20, and M20 test samples. However, there was a change in the hardness of the polyamide material by 6-11% in RON 90, E20, and M20 fuels. Moreover, there was no change in the FTIR spectrum, indicating that there was no dissolution of the EPDM and polyamide materials into the test fuel for 6 weeks of immersion.

(Author)

Keywords: Ethanol, methanol, compatibility, EPDM, polyamide.

with criteria FR value < 1.2 , screen factor test, and adsorption testing using the static method with a standard limit of adsorption value $< 400 \mu\text{g}/\text{gr}$ and polymer injectivity test. From these tests, scoring (range 0-100) was carried out to determine polymer candidates in polymer flooding testing. The F1 polymer candidate for the sandstone reservoir was obtained with a score of 82.25. From the scoring results, the selected F1 polymer candidate has a concentration value of 2000 ppm. For thermal degradation, the polymer F1 2000 ppm experienced degradation of 15.5%. The results of the F1 2000 ppm polymer static adsorption test were $54.8 \mu\text{g}/\text{gr}$. With the RRF = 1 value indicating rock permeability after injection of polymer F1 2000 ppm, it tends not to experience plugging due to injection of polymer solution.

(Author)

Keywords: Polymer, sandstone, rheology, injectivity.

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Scientific Contributions Oil & Gas, August 2022, Volume 45, Number 2, pp. 95-100.

Parameter Analysis of Polymer on Sandstone Reservoir in Indonesia: an Experimental Laboratory Study

ABSTRACT

Polymers are often used to increase oil recovery by improving sweeping efficiency. The screening was carried out as a first step in evaluating the test parameters of several polymers of the Hydrolyzed Polyacrylamide (HPAM) type in fluid and sandstone reservoir rocks. The test was carried out using a reservoir fluid classified as light oil (35°API) and at a reservoir temperature (60°C). The HPAM polymers used are A1, F1, F2, F3, and P1 polymers. The test parameters carried out on these 5 types of polymer (A1, F1, F2, F3 dan P1) include a compatibility test for formation water. The rheology polymer test includes concentration vs Tres, and shear rate vs viscosity which aims to determine the type of polymer solution being tested is a non-Newtonian or pseudoplastic fluid group. Thermal stability test of polymer for 60 days to determine the stability of the polymer solution and whether it is degraded or stable. Filtration testing

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Scientific Contributions Oil & Gas, August 2022, Volume 45, Number 2, pp. 103-114.

Converting Catalytic Palm Oil (MEPO) to Produce Biogasoline Using Zeolite Faujasite Catalyst From Fly Ash with Nickel Impregnation (Ni)

ABSTRACT

The process hydrocracking methyl ester of palm oil into fractions biogasoline by faujasite catalyst of fly ash impregnated with nickel have been made. Preparation for faujasite synthesis of fly ash can be done by removing organic compound and refluxing HCl. Synthesis do by melting the fly ash which has been prepared with NaOH 1: 1.2 and in aging for 8 hours and in the hydrothermal autoclave for 24 hours. The character faujasite using XRD and Si / Al ratio produces crystallinity main peak of 67% and Si / Al ratio of 1.65. Hydrocracking process using a variety of 4 catalyst used fly ash leaching results, faujasite, Ni-Faujasite 2%, and Ni-Faujasite 4%. Test the activity and selectivity of the catalyst to produce liquid product analyzed by GC-MS with the best catalyst was Ni-Faujasite

4% to yield 42.34% of the activity and selectivity of biogasoline fraction of 7.12%. The impregnation of the nickel catalyst is made by soaking in salt of nickel and then oxidation using O₂ gas and reduction using H₂ gas. The impregnation of nickel will affect the character of the catalyst so that the activity and selectivity of the catalyst is changed. The impregnation of nickel 4% on faujasite successfully done with nickel content of 3.71%, increasing Si / Al ratio of 2.27 and an acidity of 0.0035 mol / g.

(Author)

Keywords: Faujasite, impregnation, nickel, hydrocracking, biogasoline.

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Scientific Contributions Oil & Gas, August 2022, Volume 45, Number 2, pp. 115-125.

Laboratory Studies for The Development of a Demulsifier in Handling Production Fluid Emulsions in The “SRG” Field.

ABSTRACT

The “SRG” Oil Field is located in the South Sumatra basin, and the oil produced is classified as heavy oil and generally water-oil emulsion occurs. As a result of the formation of this emulsion which will cause corrosion of equipment in the field. The samples that have been taken in the field are then investigated in the laboratory of PT Farca Risa Sejahtera. First, perform BS&W testing on GS-belimbing and GS-11 oil samples to determine the water content and deposits present in the oil. The second is to determine the ratio of the amount of oil and formation water to be used in subsequent tests. The third selection of demulsifiers for formulation materials is based on the ability of water drop, clear water and interface. The four demulsifier formulations combine the demulsifiers that pass the selection into 5 formulas with the hope of uniting the advantages and covering each other’s shortcomings of each demulsifier that passes the selection. The fifth test is overtreated to determine the appropriate dose for the use of a predetermined demulsifier formula. Emulsion sample testing was also carried out on CGS oil samples (GS-belimbing oil and GS-11) plus the oil present in the pits. The

six BS&W tests after using the new formula. GS-belimbing has a production rate of ±22,000 BFPD with a water cut value obtained from the separator test in the field and validated by the BS&W test in the laboratory of ±92%, the value of oil production in GS Belimbing is ±1760 BOPD. While the GS-11 has a production rate of ±33,000 BFPD with a water cut value of ±91%, the value of oil production in GS 11 is ±2970 BOPD. While the CGS has a fluid production rate of ± 58,000 BFPD with a water cut of ± 90%, the value of oil production at the CGS is ± 5800 BOPD. Formula code H5 with a composition of 10% (F-13; water drop) plus 10% (1030; interface) and 80% (F-16; clear water) which was selected for GS-belimbing. The formula with code A1 which has a composition of 80% F-8 plus 10% 1030 and 10% F-16 was chosen for the GS-11. For the CGS, the S5 formula is 10% (F-16 clear water) plus 10% (1030; interface) and 80% (F-8; water drop). The results of the BS&W test after the new formula showed that there was no water in the oil in the centrifuge tube and it was stated that the BS&W value was close to 0%. There are 3 demulsifier products from the formulation, namely HAS-1 for GS-belimbing, HAS-2 for GS-11, and HAS-3 for CGS plus pit. The amount of HAS-3 demulsifier that needs to be injected into the CGS is 7.31 gallons per day (GPD). The number of HAS-1 demulsifier injected into GS Belimbing was 2.22 GPD, while the number of HAS-2 demulsifier injected into GS-11 was 3.74 GPD..

(Author)

Keywords: Crude oil, Demulsifiers, Bottle test, Formulation, Dose, Overtreat



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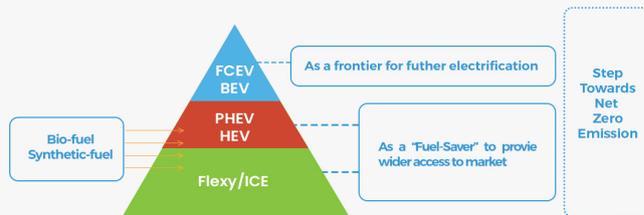
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ZONE 1

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ZONE 2

Contains information about vehicle technologies, in line with Multi-Pathway approach which every technology contribute to reduction of carbon emissions.

ZONE 3

Example green ecosystem implementation in life, to preserve the environment.

ZONE 4

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Investigation of Polymer Flood Performance in Light Oil Reservoir: Laboratory Case Study

Dadan DSM Saputra, Bayu D Prasetyo, Hestuti Eni, Yudha Taufantri,
Ghifahri Damara, Yusuf D Rendragraha

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ABSTRACT - The use of polymer solutions in the application of chemical EOR injection technology has a role in increasing oil recovery efforts by improving oil mobility in porous media. The addition of the polymer solution is expected to increase the viscosity value of the displacement fluid so that it can form a “piston-like” effect to increase the volumetric sweep efficiency of the light oil reservoir. The polymer used in this study was HPAM using 3 concentrations, namely 500 ppm, 1000 ppm, and 1500 ppm conducted at a temperature of 70 °C. The rheology test of the polymer included concentration vs temperature and shear rate vs viscosity. Thermal stability testing of polymer for 7, 14, 30, 60, and 90 days at 70 °C was done to determine the stability of the polymer solution. Filtration testing was conducted with the criteria of FR <1.2. The static adsorption test has been done with the standard limit of adsorption value <400 µg / gr. Polymer injectivity test using 3 variations of injection rates and coreflooding test were conducted to determine the reduction of Sor in reservoirs due to polymer displacement. From the polymer testing stage, it was found that HPAM polymers at 3 concentrations were compatible with the injection. This is indicated with the clear solution for 3 concentrations at room temperature and 70 °C. The rheology test results showed that the polymer solution with 3 concentrations was decreased in viscosity with the addition of the shear rate value. In the thermal stability test, the viscosity value of the HPAM with 500 ppm was relatively constant. The value of the FR for HPAM 500 ppm is 1.1, HPAM 1000 ppm is 1.07 and HPAM 1500 ppm is 1.03. The results of the static adsorption test showed the lowest HPAM value of 500 ppm was 156 µg/gr. In the injectivity test results, the resistance residual factor (RRF) values at injection rates of 0.3, 0.6, and 1 cc/min were 0.8, 1.04, and 1.12. The RRF value was close to 1, indicating that after injection of 500 ppm of HPAM tended to not experience plugging. Polymer flooding shows the oil recovery factor (RF) of water injection is 39% OOIP, and RF after polymer injection with 0.35 PV with flush water is 13.5% OOIP or 22% Sor. Knowing the behavior of HPAM polymer with various concentrations to be used for chemical EOR injection, it could provide advantages for future implementation in the light oil reservoir in Indonesia.

Keywords: filtration, injectivity, light oil, polymer flooding, rheology.

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INTRODUCTION

The use of polymer solutions in the application of chemical EOR injection technology has a role in increasing oil recovery efforts by improving oil mobility in porous media. The addition of the polymer solution is expected to increase the viscosity

value of the displacement fluid so that it can form a “piston-like” effect to increase the volumetric sweep efficiency of the light oil reservoir. (Sheng, 2010; Seright et al, 2008; Shah and Schechter, 1977; Jamaloei et al, 2011).

The polymer screening and performance testing must be done before pilot scale implementation in the oil fields. The partially hydrolyzed polyacrylamide (HPAM) polymer was used in this study. Several tests have to be passed to make sure the HPAM polymer performance in reservoir conditions meets the criteria which will improve the oil recovery of the mature fields. Several tests which must be done were screening tests or rheology evaluations such as compatibility tests, viscosity vs. shear rate tests, thermal stability tests, filtration tests, and static adsorption tests. The injectivity tests and coreflooding tests were also carried out to know the performance of the polymer injection into the native and/or synthetic core. The purpose of this study is to investigate the performance of the HPAM polymer injection in increasing oil recovery in the light oil reservoir.

METHODOLOGY

This research study begins to understand the rheological properties of the polymeric material and provides more insight into the adequacy of polymer HPAM from its behavior through porous media (reservoirs). HPAM concentrations of 500, 1000, and 1500 ppm were tested in this study.

The material of HPAM was mixed in brine water that has a designated salinity of roughly 2800 ppm (see Table 1 for brine water composition). The experiment consists of several tests, starting with testing compatibility, shear test, filtration, adsorption, thermal degradation, injectivity, and coreflooding (Poettman and Hause, 1978; Lemigas, 2008; Veerabhadrapappa et al, 2011). All the tests were carried out at 70°C as the light oil reservoir temperature.

Table 1
Brine water composition

Parameter	Value	Unit
ANION		
Chloride	709.06	mg/L
Bicarbonate	1037.31	mg/L
Sulfate	0.07	mg/L
Carbonate	120.02	mg/L
Hydroxide	0.00	mg/L
KATION		
Sodium	891.1	mg/L
Calcium	20.04	mg/L
Magnesium	15.19	mg/L
Iron	0.00	mg/L
Barium	0.00	mg/L
Total Dissolve Solid	2792.81	mg/L
pH	8.71	

- **Compatibility test**
The solubility of HPAM with various concentrations was visually observed at both room and 70 °C temperature to investigate the phase solution, color changing, and precipitation.
- **Shear test**
All the rheological experiments were performed on Brookfield DVIII with UL adaptor. For each test (viscosity vs concentration and viscosity vs shear rate), the polymer solutions were prepared with varying concentrations. Concentrations ranged from 500 ppm to 1500 ppm of polymeric material in brine. The viscosity vs concentration of HPAM was measured using a shear rate of 7 rpm and the viscosity vs shear rate was measured using a shear rate from 50 rpm to 250 rpm.
- **Filtration test**
A filtration test was conducted to evaluate whether the polymer solution has free of aggregates which could lead to formation plugging. The measurement of the filter test is pumped through a 3 µm membrane with a differential pressure of 2 bars.
- **Adsorption test**
The polymer solutions were prepared to conduct a static adsorption test according to Recommended Practice (RP 63).
- **Thermal degradation test**
Tests were performed for 3 months at 70 °C temperature under anaerobic conditions in sealed glass ampoules.
- **Injectivity test**
HPAM injectivity test was run at a concentration of 500 ppm, the temperature of 70 °C, and slow injection rates of 0.3 cc/min, 0.6 cc/min, and 1 cc/min.
- **Coreflooding test**
During the flooding experiment, the injection rate of the displacing fluids was controlled at 0.3 cc/min with polymer injection of 0.35 PV.

RESULTS AND DISCUSSION

Polymer flooding is intentionally conducted to reduce the relative permeability of water in the reservoir, therefore can improve the production of oil, as well as enlarge the swept volume of the reservoir. Recent popular material of polymer that is assured to accommodate oil fields is HPAM. HPAM has most

often been used to achieve a more favorable mobility ratio and improve macroscopic sweep in chemical EOR by increasing the viscosity of the water. When dissolved in fluid, the polymer solutions have a viscosity that depends on many aspects: concentration, molecular weight, temperature, and salinity (LEMIGAS, 2008; Levitt and Pope, 2008).

In this study, the investigation of polymer flood has been performed using light crude oil. The characteristic of light crude oil has been shown in Table 2. Based on the result, the °API of crude oil was approaching 28 and this has to do with designing the compatible HPAM type. Besides, the water analysis demonstrates roughly 2800 ppm salinity brine.

Table 2
Characteristic of light crude oil

Determination	Method	Result	Unit
Density at 15°C	ASTM D. 5002	0.8792	g/cm ³
°API Gravity	ASTM D. 5002	27.5588	-
Kin. Viscosity at 70°C	ASTM D. 445	6.9884	cSt
Pour Point	ASTM S. 5853	45	°C
Asphaltene	IP. 143	0.374	%wt
Total Acid Number	ASTM D. 664	0.0156	mg KOH
Saturated	Chromatography Column	52.20	%wt
Aromatic	Chromatography	16.04	%wt

The compatibility of the polymer solution was first conducted at both room and 70 °C temperatures. This presents in Figure 1, with the good result of the clear phase solution, the color of the solution was not changing, and no precipitation, which is essential to obtain distinctly sufficient chemicals.

The rheological properties of the HPAM polymer



Figure 1
Compatibility polymer solution at room temperature (a) and 70°C (b)

solution were evaluated by measuring the apparent viscosity vs concentration and viscosity vs shear rate. This experiment is one of the most prominent screenings of an injected chasing fluid during the chemical flooding process. Figure 2 demonstrates variation shear rate from 50 rpm to 250 rpm was

conducted on the viscosity of 3 concentrations of the polymer at 70 °C temperature. This result presents HPAM is generally classified as a non-Newtonian fluid because the viscosity changes when the shear rate was applied. Thus, the type of fluid rheology is pseudo-plastic fluid. In this desired condition, pseudo-plastic fluid was known as shear thinning, in which viscosity decreases as the shear rate increases. The experiment result of viscosity vs concentration demonstrated the viscosity increasing steadily with increasing polymer concentration at 70 °C temperature. This behavior greatly contributes to the shear thickening of the HPAM solution when the polymer flows at a high shear rate in porous media.

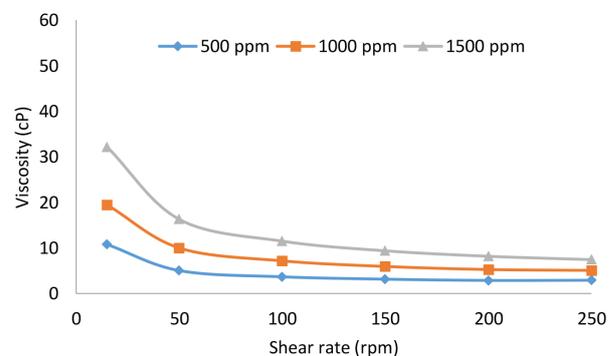


Figure 2
Effect of polymer on shear rate test results

The filtration test was performed to determine whether the polymer can flow through the rock pores and to evaluate the effect of debris. Figure 3 informs a volume plot graph against the time of the polymer with 3 concentrations. Each concentration solution ensured that polymer hydration had been achieved.

The value of the FR for HPAM 500 ppm is 1.1, HPAM 1000 ppm is 1.07, and HPAM 1500 ppm is 1.03. This result rapidly indicates HPAM is acceptable as it does not tend to plug porous media in the reservoir because the requirement of the filtration ratio (FR) value was below 1.2.

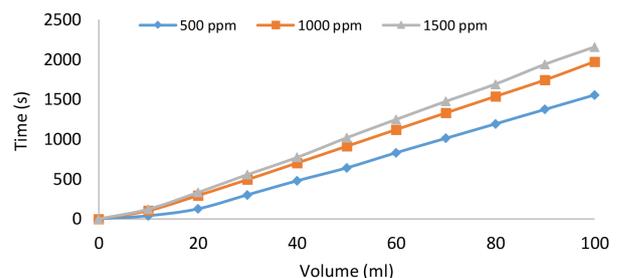


Figure 3
Filter test using 3 µm membrane results

Table 3
Core characteristic

Core	D (inch)	L (inch)	W (gr)	ϕ	Ka (mD)
a	1.5	1.9	100.0	0.30	1302.1
b	1.5	1.8	98.3	0.27	1539.7
c	1.5	1.7	93.9	0.26	1493.4

The thermal degradation test was conducted for 3 months at 70 °C temperature to investigate the remaining viscosity of HPAM. The result is presented in Figure 4. The viscosity of 500 ppm maintains a constant value in the last 30 days, and the rest after 3 months of the aging period, decreases slowly from

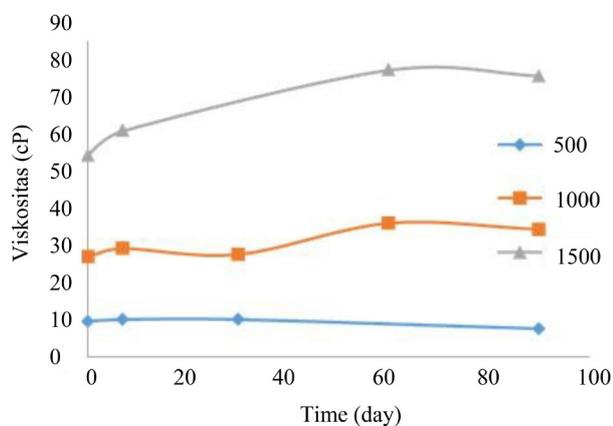


Figure 4
Thermal degradation test results

10 cP to 7 cP with a viscosity retention percentage of 30%. Comparatively, the remaining viscosity of the two concentrations left was demonstrated at 1000 ppm and 1500 ppm with increasing the number viscosity due to the changing of the colloidal system solution, likely through a hydrolysis reaction. Thus, more effective preparations should be developed to improve their thermal degradation.

To understand the performance of the polymer to the rocks, injectivity and coreflooding tests were carried out. The characteristic of the rocks that were used is sandstone native core plugs with a permeability range of 1500 to 2500 mD and an average porosity of 0.26.

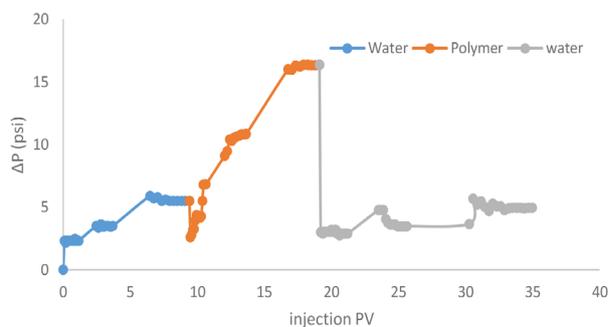


Figure 5
Injectivity test results

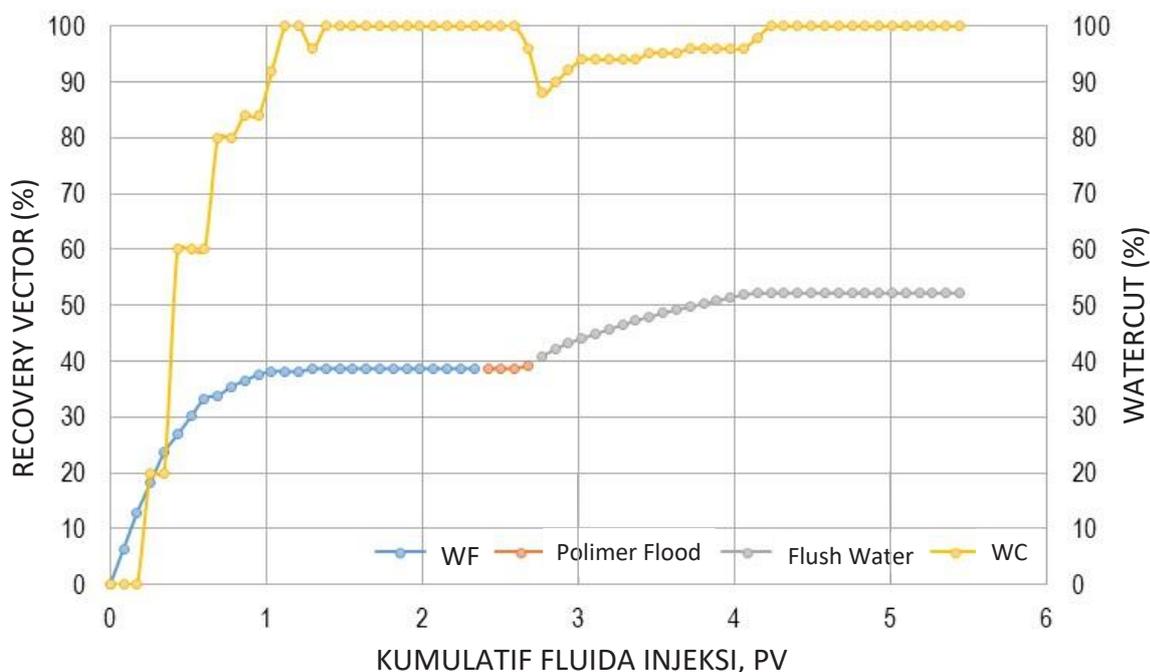


Figure 6
Coreflooding test results

The injectivity tests were carried out using a step-up rate of 0.3, 0.6, and 1 cc/min for every concentration of polymer solution. The core used in the injectivity and coreflood test could be seen in Table 3. The results of the polymer injectivity can be seen in Figure 5. From the test results, the polymer has a Residual Resistance Factor (RRF) value near 1 (one) which means that the permeability of the core plugs after polymer injection were not changing as much and also indicates plugging did not happen after the polymer injection.

From coreflooding test result, recovery factor (RF) after waterflood is at 7.2 cc or 38.7 % (OOIP), and Sor after waterflood is at 11.4 cc (61.3% OOIP). With injecting 0.35 PV polymer, recovery of oil is at 2.4 cc or 13% OOIP. This result shows that by injecting polymer after waterflood, additional oil recovery can be gained at about 13.5% OOIP or about 22% ROIP (see Figure 6).

CONCLUSIONS

The test results for all the parameters already meet the criteria for polymer screening as chemical EOR. Based on the polymer screening test and polymer performance test that have been done, a polymer concentration of 1000 ppm is suitable for polymer injection with a rule of thumb that polymer viscosity should be four times higher than oil viscosity (6.988 cSt) which gives about 26 cP and from coreflood result which gives about 22% ROIP. From these results, this polymer has the potential to be implemented on the pilot scale in a light oil reservoir.

ACKNOWLEDGMENTS

Thank you to the Exploitation Department in R&D Center for Oil and Gas Technology “LEMI-GAS” for the technical and non-technical support for this research.

GLOSSARY OF TERMS

Symbol	Definiton	Unit
API	: American Petroleum Institute	
EOR	: Enhanced Oil Recovery	
FR	: Filtration ratio	

HPAM	: Hydrolyzed Poliacrilamide
OOIP	: Original Oil in Place
RF	: Recovery factor (%)
RF	: Resistance Factor
RRF	: Residual Resistance Factor
mD	: mili Darcy
RPM	: Revolutions per minute
PV	: Pore Volume
ROIP	: Residual Oil in Place
PPM	: part per million
cSt	: centi Stoke
cP	: centi Poise

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The Effect of Methanol-Gasoline (M20) and Ethanol-Gasoline (E20) Blends on Material Compatibility

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ABSTRACT - Alcohol has the potential to be used as an alternative to fossil fuels to reduce total emissions from spark-ignition (SI) engines. The impact of a mixture of 20% methanol and ethanol in gasoline on the compatibility of Ethylene Propylene Diene Monomer (EPDM) and polyamide materials, which are used as fuel hoses in SI vehicles, is presented in this study. The immersion test methodology was employed to study the influence of both types of alcohol on gasoline blend to compatibility properties i.e., hardness and weight change. Based on the result, EPDM and polyamide materials have different characteristics of material compatibility with E20 and M20. Tests on M20 and E20 fuel samples on EPDM material show a higher effect on hardness by 5-9% than pristine gasoline. Additionally, there was no change in the weight of the polyamide material in the RON 90, E20, and M20 test samples. However, there was a change in the hardness of the polyamide material by 6-11% in RON 90, E20, and M20 fuels. Moreover, there was no change in the FTIR spectrum, indicating that there was no dissolution of the EPDM and polyamide materials into the test fuel for 6 weeks of immersion.

Keywords: Ethanol, methanol, compatibility, EPDM, polyamide.

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INTRODUCTION

Oxygenated fuel is one of the promising alternative fuels to lower exhaust emissions. One of the oxygenate compounds that have been studied and have the potential to be widely applied in alcohol (Canakci et al., 2013; Masum et al., 2015a, 2015b). Alcohol has a high octane number (*Research Octane Number*, RON), high oxygen content, sulfur-free, and low-carbon. Based on these characteristics, there are currently many studies exploring methanol and ethanol as substitute fuels for gasoline (Awad et al., 2018; Yusri et al., 2017). Methanol has a RON value of 129-134, Oxygen content of 49.9%-mass, a vapor pressure of 32.2 kPa, and a Lower Heating Value

(LHV) of 26,1 MJ/kg. On the other side, ethanol has a RON of 109, an Oxygen content of 34.7 %-mass, a Vapor pressure of 17.2 kPa, and a Lower Heating Value (LHV) by 27.0 MJ/kg (Aghahosseini Shirazi et al., 2019; Calvin et al., 2022). With a high RON value, the addition of methanol and ethanol can also be used as mixed components to increase the RON of gasoline (Octane Booster). In addition, the oxygen content in methanol and ethanol also has the potential to produce perfect combustion resulting in reduced emissions of carbon monoxide and unburned hydrocarbons (UHC). But, the percentage of mixing methanol and ethanol as gasoline blended fuels is still being researched and explored due to a concern about

the resulting characteristics of having to adapt to the vehicle engine technology currently on the market (Sugiarto et al., 2019, 2020).

In general, adding methanol and ethanol results in a linear increase in octane numbers and oxygen content (Calvin et al., 2022). The use of ethanol-gasoline and methanol-gasoline fuel blends causes to decrease in CO and unburned HC emissions significantly, this is due to improving combustion process as a result of oxygen content in ethanol and methanol (Canakci et al., 2013). However, some studies show that methanol and ethanol experience an azeotropic effect (mixing is not ideal) with a gasoline vapor pressure at low concentrations. Methanol and ethanol can affect vapor pressure and gasoline distillation curves, thus forming a non-ideal curve due to polarization in the equilibrium of fuels. The highest vapor pressure is produced in the 10-20% methanol/ethanol concentration range in gasoline (Abdurrojaq et al., 2021; Aghahosseini Shirazi et al., 2019). Therefore, although methanol and pure ethanol have a lower vapor pressure, mixing them with gasoline increases steam pressure compared to pristine gasoline. Therefore, the percentage of methanol and ethanol used in gasoline must produce fuel characteristics that can produce optimal performance and not negatively affect metal and non-metal materials in engine component systems (Durbin et al., 2016; Vyas et al., 2013).

Some research shows that the use of methanol and ethanol as a mixture of gasoline in the automotive sector requires the selection of metal and non-metal materials whose durability is appropriate. Methanol and ethanol can interact, thus severing the bond between the resin and fiberglass from the fuel tank, causing the leak, resulting in the resin material attached to the valve and other engine parts due to deposits and blockages in the entrance valve. Another problem that may arise is the corrosive nature of metal fuel tanks due to the very high affinity of methanol and ethanol to water. Ethanol mixture 20% (E20) causes many problems in various plastics, rubbers, and metal components. Some polymer materials such as Nylon-6, Nylon-66, PET, and Polyetherimide (PEI) showed the same with 3 fuel mixtures including 50/50 toluene-isooctane, E10, and E20 (Durbin et al., 2016; Vyas et al., 2013). Other materials compatible with the E10 fuel mixture include Delrin, which should not be used Neoprene Polymers, Nitrile, or HNBR that are not compatible with E10 (Turner et al., 2013; Wouters et al., 2020).

Currently, research on the effect of methanol and ethanol mixtures on the compatibility of non-metallic materials of Ethylene Propylene Diene Monomer (EPDM) and polyamide is still limited. Identification of the material of the parts was done by FTIR and DSC for non-metal parts and by XRD and XRF for metal parts (Anggarani Riesta, Cahyo S. Wibowo, 2015). As one of the building materials of fuel lines in vehicles, the effect of methanol and ethanol on the compatibility of EPDM and polyamide materials is significant to be explored. Therefore, this study presents the effect of a mixture of 20% ethanol (E20) and 20% methanol (M20) on gasoline on the compatibility of EPDM and polyamide materials. The test refers to SAE J1748 2018-08 method for compatibility of polymer material properties exposed to fuel mixtures/oxygenate substitutes with additives.

METHODOLOGY

Preparation Fuel Test

The gasoline used is a type of commercial gasoline with RON 90. Methanol and ethanol are used as a type of fuel grade that comes from one of the producers in Indonesia. Fuels of 20% methanol (M20) and 20% ethanol (E20) in RON 90 gasoline is carried out on a volume basis and in cold conditions to minimize evaporation. After mixing and then homogenizing, the fuel is stored in the refrigerator at a temperature of 0-4 °C.

Preparation of Non-Metal Materials

Two types of non-metal materials as fuel lines in vehicle engine combustion systems, EPDM and polyamide are used in this study. The two types of fuel lines are obtained from one of the vehicle manufacturers in Indonesia. Both materials were identified with FTIR (Fourier Transform Infra-Red

Spectroscopy) with ASTM E1252-13 test method and Transition Temperature Analysis with DSC (Differential Scanning Calorimetry) with ASTM D3418-15 test method. Hose material is cut precisely by adjusting the area of the immersion container. Hoses that are already in small size are then dried and weighed to gain the initial weight of the specimen (the test is carried out outside the LEMIGAS).

Immersion Test

Material compatibility testing is conducted for 6 weeks or ± 1000 hours of immersion with a change of test fuel every 1-week interval. Immersion is

conducted constantly at $55 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ in dry baths. Measurement of weight and hardness after drying the material for 60 minutes at a temperature of 40-50°C. Analysis of material changes is done by testing the hardness and weighing of material weights each week. This compatibility test is also calculated with the F test (statistical math formula) to see the movement of material changes each week with the hypothesis that if F calculates lower than the F table then the material change has reached equilibrium compared to the measurement in the previous week. The immersion test refers to the SAE J1748 2018-08 method “Methods for Determining Physical Properties of Polymeric Materials Exposed to Hydrocarbon Fuels or Their Surrogates and Their Blends with Oxygenated Additives”.

RESULTS AND DISCUSSION

The characteristics of methanol and ethanol gasoline in Table 1 have differences in the research octane number, density, boiling point, vapor pressure, and oxygen content. Methanol has the highest octane number when compared to gasoline and ethanol. Contrarily, gasoline has the lowest density compared to ethanol and methanol. The characteristic of vapor pressure in gasoline is the lowest, the addition of alcohol to gasoline mixtures increases the vapor pressure by 19 kPa for methanol by 20% and 6 kPa for ethanol by 20%. While M20 and E20 have oxygen content obtained from methanol and ethanol so it is believed to improve combustion and also make emissions cleaner compared to gasoline. (Abdurrojaq et al., 2021).

Identification of material hose with FTIR in Table 2 shows that hose has EPDM material composition (Ethylene Propylene Diene Rubber) with six spectrums of wavelength compared to reference data, and the other one hose has a polyamide material composition of fourteen wavelength spectrum. Transition Temperature Analysis with DSC to measure enthalpy changes due to changes in the physical

Table 1
Fuel characteristics gasoline 90, M20 and E20

No	Test Parameter	Method Unit	Test ASTM	Fuel Characteristic		
				Gasoline 90	M20	E20
1	Octane Number	RON	D2699	90,1	98,9	97,5
2	Oxygen Content	% m/m	D6730	0,0	9,8	6,9
3	Vapor Pressure	kPa	D5191	59,0	79,7	65,0

4	Density (T: 15°C)	kg/m ³	D4052	725,4	737,4	738,5
5	Copper Strip Corrosion	Merit	D130	1a	1b	1a
6	Mercaptan Sulphur	%-mass	D3227	<0,0003	<0,0003	<0,0003
7	Methanol Content	%-mass	D5845	0	20,1	0
8	Ethanol Content	%-mass	D5845	0	0	19,9
9	Water Content	ppm	D6304	39	81	194

and chemical properties of a material as a function of temperature or time and can identify and determine the characteristics of matter.

The first material with the composition of the material is EPDM and the second material is Polyamide with each tested with 3 fuels and measured every week to see the rate of Change of the material. The rate of change in the EPDM material hose is shown in Figure 1. Changes in EPDM material weight indicate a considerable decrease for the M20, E20, and RON 90. The result of material immersion in

Table 2
Analysis wavelength spektrum FTIR and transition temperature

No	Sample	Results	Wavelength (cm ⁻¹)	Reference	Transition Temperature
1	H1	EPDM	2954; 2924; 2853; 1462; 1377; 721	ISO 465 : 2010 (E) Table B.6 Figure B.10	T _{glass} = -55,17°C
2	H2	Polyamide	3307; 2920; 2852; 1638; 1540; 1465; 1420; 1374; 1262; 1190; 1158; 1055; 938; 723	Hummel 2189	T _{melting} =182,70°C

E20 is a change in weight and hardness that is higher than M20 and RON 90. EPDM material undergoes a 29% weight change in E20 fuel. While the immersion in M20 and RON 90 fuels causes weight changes of 20% and 18%. The results showed that the use of 20% ethanol can cause an 11% increase in weight change when compared to pure gasoline. Conversely, on the M20 fuel, there is a no different change compared with pure gasoline.

In general, EPDM materials experienced increased hardness in each test fuel. This increase occurs on each measurement week as can see in Figure 1. In the 6th week of measurements obtained adjacent results from all three fuels. In contrast to the results of weight measurements, the M20 fuel-soaking fuel hardness measurement produces the highest change with a value of 30%. Meanwhile, E20 and RON 90 fuels caused a change in hardness in EPDM materials

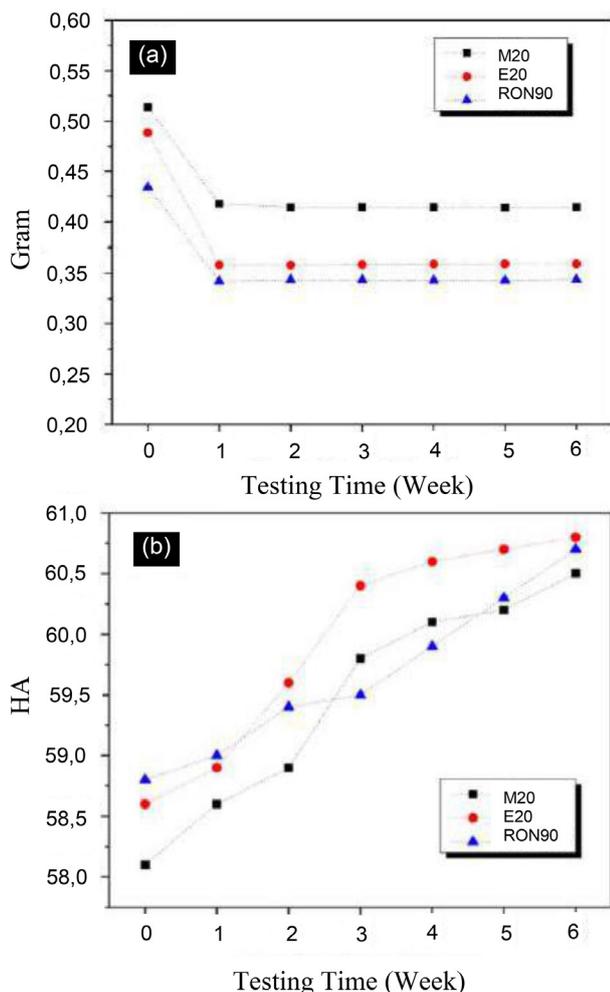


Figure 1
 (a) Weight change graphic - EPDM,
 (b) Hardness change graphic - EPDM

with values of 26% and 21%. The results showed that the use of 20% methanol and ethanol had a change in hardness of 5-9% higher than that of pure gasoline. EPDM rubber absorbs the fuel alcohol-gasoline mixture through the pores, then the drying process evaporates the remaining fuel in the pores which keep repeating every week, causing the material to swell and shrink during the testing process.

Polyamide materials have different changes to EPDM. In Figure 2 it is seen that there is no change in the weight of the polyamide material in the ron 90, E20, and M20 fuels. Meanwhile, there was a change in the level of polyamide material hardness during the 6-week immersion time. The highest hardness change was produced by polyamide material soaked with M20 fuel, at 11.5%. E20 fuel causes a 6% violent change in polyamide material. While, on RON 90 fuel, as a control fuel, there was a 10% change in hardness in polyamide material. Figure 2

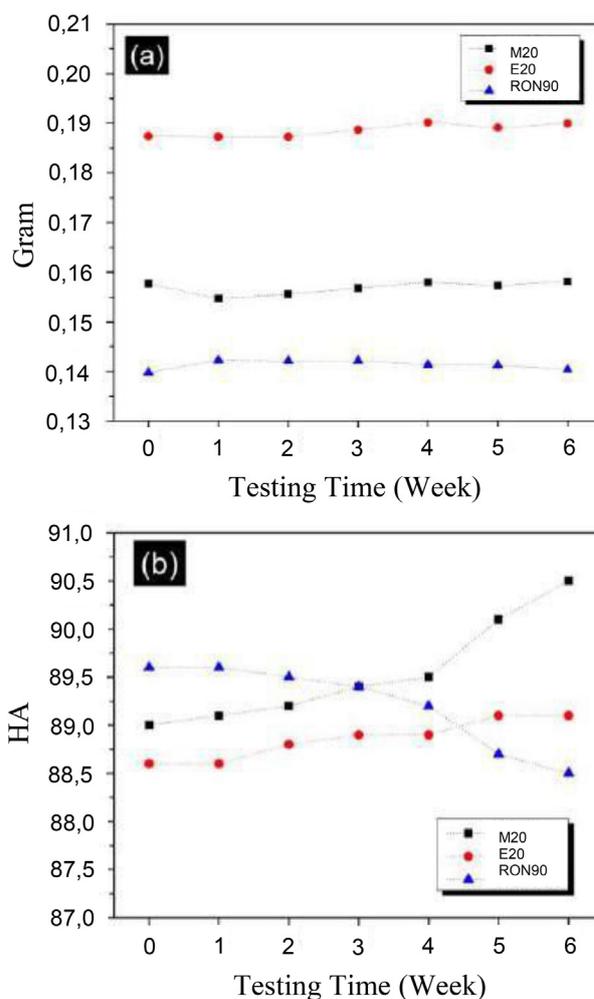


Figure 2
 (a) Weight change graphic - polyamide,
 (b) Hardness change graphic - polyamide

These results showed the polyamide material was compatible with RON 90, E20, and M20 fuels on the weight parameter, but there was a change in the hardness parameter.

The graphic in Figure 3. indicates a percent value change from the beginning of the measurement to the end of the sixth week of measurement. EPDM materials have a change effect with the immersion method much greater than polyamides. EPDM material undergoes stated degradation with a decrease in weight and a 20-30% increase in hardness. Polyamide materials with E20 immersion fuel have the smallest changes between M20 and RON 90 fuels. Figure 4 shows the FTIR spectrum of pure RON 90 fuel compared to the fuel used to soak EPDM and polyamide. Test results showed the transmission produced by the fuel before and after testing was still identical, clustered, and did not undergo any shift or expansion of the spectrum. These results showed no

The Effect Of Methanol-Gasoline (M20) And Ethanol-Gasoline (E20) Blends On Material Compatibility
(Nurmajid Abdurrojaq, et al.)

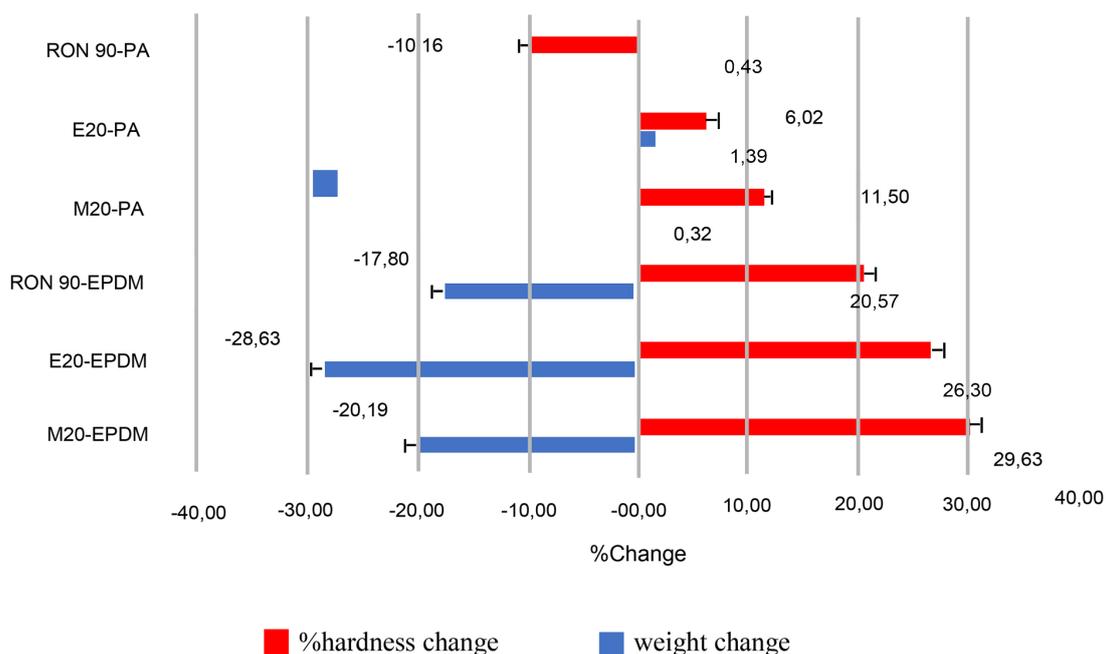


Figure 3
Change graphic of materials non-metal

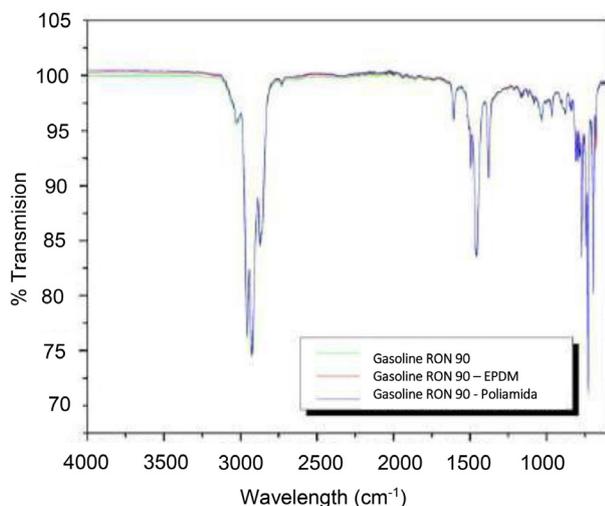


Figure 4
Spectrum changes in FTIR transmission
fuels gasoline RON 90

solvent detected in EPDM and polyamide materials during 1000-hour immersion testing on the fuel. The composition of the fuel function group was also not changed by FTIR detection. Spectrum graph FTIR of M20 fuel in Figure 5. Early fuel shows have an OH group bond with a single bond visible from the curves at wavelengths 3200-3600 cm^{-1} . Fuel after immersion indicates the dilation of the spectrum curves at wavelengths 3200-3600 cm^{-1} indicates that

a single bond OH group increases which indicate that water vapor joining the alcohol compounds in the fuel mixture increases due to the appearance of hygroscopic properties of the alcohol. This also happens with the E20 fuel seen in Figure 5. The spectrum at wavelengths 3200-3600 cm^{-1} appears deeper curves wave indicating the presence of OH groups and in fuel after immersion of the spectrum the presence of OH groups is increased because the hygroscopic nature of alcohol affects E20 fuel.

In immersion testing for 6 weeks or 1000 hours is calculated with a Test F for each material and fuel. To monitor the significance of test F is required to compare it with Table F which refers to the SAE J1748 2018-08 Methods for Determining Physical Properties of Polymeric Materials Exposed to Hydrocarbon Fuels or Their Surrogates and Their Blends with Oxygenated Additives. In this study, 3 test specimens (in immersion containers) were conducted for every 1 test (1 fuel for 1 type of material) and 3 measurements were taken for each specimen. Then the value of F used in Table F is 5.14.

This test obtained F calculate for each test (1 fuel for 1 type of material) can be seen in Table 3. Values smaller than 5.14 are EPDM materials on all types of M20, E20, and RON 90 test fuels. That states that equilibrium has been achieved and the process

of material change has tended to be the same. In contrast, polyamide material has not been declared equilibrium in each specimen because the value of F calculate is still greater than F Table.

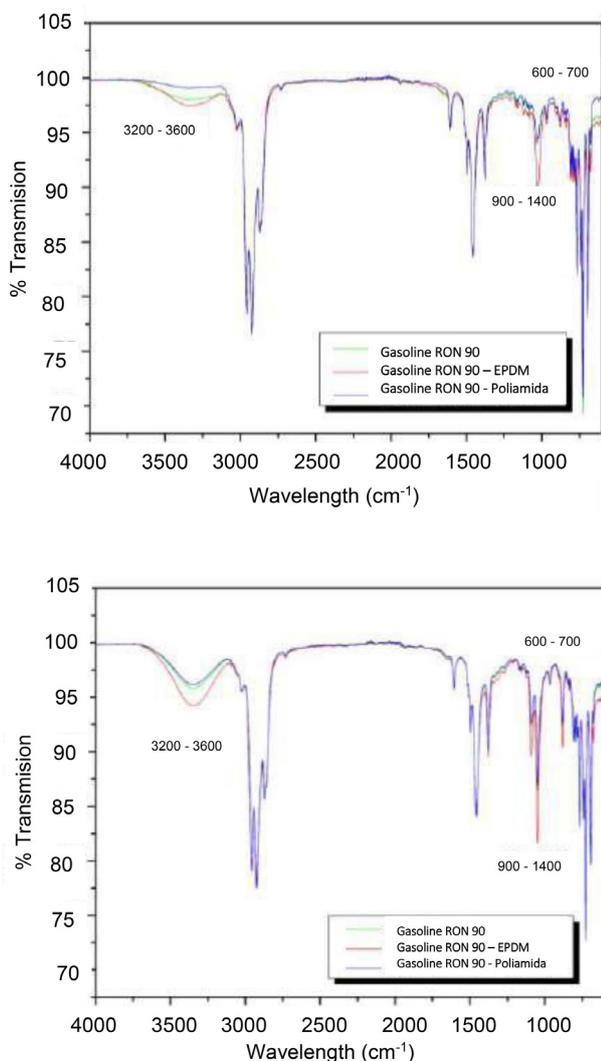


Figure 5
Spectrum changes in FTIR transmission mixed gasoline fuel with 20% ethanol and 20% methanol

CONCLUSIONS

In summary, the effect of 20% methanol (M20) and ethanol (E20) in gasoline on the compatibility of EPDM and polyamide materials have successfully been carried out in this study. Here, both test sample materials, EPDM and polyamide, have different characteristics of material compatibility with E20 and M20. Based on the results, M20 and E20 fuel test samples show that

Table 3
F table value

F Table	F Calculate					
	M20 (EPDM)	E20 (EPDM)	RON 90 (EPDM)	M20 (Polyamide)	E20 (Polyamide)	RON 90 (Polyamide)
5,14	0,027097	0,025608	0,272253	12,0792816	11,94391842	12,7760935
Hasil	< F Tabel	< F Tabel	< F Tabel	> F Tabel	> F Tabel	> F Tabel

EPDM material has a higher effect on hardness changes by 5-9% compared to pristine gasoline. However, there was no change in the weight of the polyamide material in the RON 90, E20, and M20 fuel tests. Furthermore, there is a change in the hardness of the polyamide material by 6-11% in the RON 90, E20, and M20 fuel tests. It was clear that polyamide material is more compatible with exposure to E20 and m20 fuels compared to EPDM. Moreover, there was no change in the FTIR spectrum, indicating that there was no dissolution of the EPDM and polyamide materials into the test fuel for 6 weeks of immersion.

EPDM hose undergoes physical changes over a long period when exposed to a mixture of gasoline and alcohol, then the use of EPDM hose can be placed on the part that is slightly in contact with the fuel blends. Polyamide is one of the materials that can be prioritized if the fuel used is a mixture of gasoline and alcohol because its durability can be very long.

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GLOSSARY OF TERMS

Symbol	Definition	Unit
ASTM	American Standard Testing and Material	
DSC	Differential Scanning Calorimetry	°C
EPDM	Ethylene Propylene Diene Rubber	
E20	Mixed fuel gasoline with 20% ethanol	%v/v

FTIR	Fourier Transform Infra-Red Spectroscopy	cm ⁻¹
HNBR	Hydrogenated Nitrile Butadiene Rubber	
M20	Mixed fuel gasoline with 20% methanol	%v/v
PA	Polyamide	
RON	Research Octane Number	
RON 90	Gasoline fuel with content the octane number value of 90	
SAE	Society of Automotive Engineers	

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Parameter Analysis of Polymer on Sandstone Reservoir in Indonesia: An Experimental Laboratory Study

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ABSTRACT - Polymers are often used to increase oil recovery by improving sweeping efficiency. The screening was carried out as a first step in evaluating the test parameters of several polymers of the Hydrolyzed Polyacrylamide (HPAM) type in fluid and sandstone reservoir rocks. The test was carried out using a reservoir fluid classified as light oil (35°API) and at a reservoir temperature (60°C). The HPAM polymers used are A1, F1, F2, F3, and P1 polymers. The test parameters carried out on these 5 types of polymer (A1, F1, F2, F3 dan P1) include a compatibility test for formation water. The rheology polymer test includes concentration vs Tres, and shear rate vs viscosity which aims to determine the type of polymer solution being tested is a non-Newtonian or pseudoplastic fluid group. Thermal stability test of polymer for 60 days to determine the stability of the polymer solution and whether it is degraded or stable. Filtration testing with criteria FR value < 1.2, screen factor test, and adsorption testing using the static method with a standard limit of adsorption value < 400 µg/gr and polymer injectivity test. From these tests, scoring (range 0-100) was carried out to determine polymer candidates in polymer flooding testing. The F1 polymer candidate for the sandstone reservoir was obtained with a score of 82.25. From the scoring results, the selected F1 polymer candidate has a concentration value of 2000 ppm. For thermal degradation, the polymer F1 2000 ppm experienced degradation of 15.5%. The results of the F1 2000 ppm polymer static adsorption test were 54.8 µg/gr. With the RRF = 1 value indicating rock permeability after injection of polymer F1 2000 ppm, it tends not to experience plugging due to injection of polymer solution.

Keywords: Polymer, sandstone, rheology, injectivity.

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INTRODUCTION

The use of polymer solutions in the application of chemical EOR injection technology has a role in increasing oil recovery efforts by improving oil mobility in porous media. The addition of the polymer solution is expected to increase the viscosity value of the displacement fluid so that it can form a "piston-like" effect to increase the volumetric sweep efficiency of the light oil reservoir. (Sheng, 2010; Seright et al, 2008; Shah and Schechter, 1977). The polymer screening and performance testing must be done before pilot scale implementation in the

oil fields. The partially hydrolyzed polyacrylamide (HPAM) polymers were used in this study. Several tests have to be passed to make sure the HPAM polymer performance in reservoir conditions meets the criteria which will improve the oil recovery of the mature fields. Several tests which must be done were screening tests or rheology evaluations such as compatibility tests, viscosity vs. shear rate tests, thermal stability tests, filtration tests, and static adsorption tests. The injectivity tests and core flooding tests were also carried out to know the performance of the polymer injection into the native and/or syn-

thetic core. The purpose of this study is to investigate the performance of the HPAM polymer injection in increasing oil recovery in light oil reservoirs.

METHODOLOGY

This research study begins to understand the rheological properties of the polymeric material and provides more insight into the adequacy of polymer HPAM from its behavior through porous media (reservoirs). HPAM concentrations that were tested were at 500 – 3000 ppm. The material of HPAM was mixed in injection water (see Table 1 for injection water composition). The experiment consists of several tests conducted experimentally, starting by testing compatibility, shear test, screen factor, filtration, adsorption, thermal degradation, scoring, and injectivity (Poettman and Hause, 1978; Lemigas, 2008). All the tests were carried out at 60°C as the reservoir temperature.

Table 1
Brine/water injection composition

Parameter	Value	Unit
ANION		
Chloride	8748	mg/L
Bicarbonate	1970	mg/L
Sulfate	0.16	mg/L
Carbonate	98	mg/L
Hydroxide	0	mg/L
CATION		
Sodium	6150	mg/L
Calcium	80.2	mg/L
Magnesium	134.2	mg/L
Ferum	0	mg/L
Barium	0	mg/L
Total Dissolve Solid	17550	mg/L

- **Compatibility test**
The solubility of HPAM with various concentrations was visually observed at both room and 60°C temperature to investigate the phase solution, color changing, and precipitation.
- **Shear test**
All the rheological experiments were performed on Brookfield DVIII with a UL adaptor. For each test (viscosity vs concentration and viscosity vs shear rate), the polymer solutions were prepared

with varying concentrations. Concentrations ranged from 500 – 3000 ppm of polymeric material in brine. The viscosity vs concentration of HPAM was measured using a shear rate of 7 rpm and the viscosity vs shear rate was measured using a shear rate from 50 rpm to 250 rpm.

- **Screen Factor**
Tests were carried out to determine the qualitative size of the polymer and to determine the viscoelastic behavior of the polymer solution (Sorbie, 1991). Viscoelastic is a characteristic that is viscous and elastic when it is deformed (Jouenne, S and Heurteux, 2017).
- **Filtration test**
A filtration test was conducted to evaluate whether the polymer solution has free of aggregates which could lead to formation plugging. The measurement of the filter test is pumped through a 3 µm membrane with a differential pressure of 2 bars.
- **Adsorption test**
The polymer solutions were prepared to conduct a static adsorption test according to Recommended Practice (RP 63) using native core
- **Thermal degradation test**
Tests were performed for 3 months at 60°C temperature under anaerobic conditions in sealed glass ampoules.
- **Injectivity test**
HPAM injectivity test was run at the selected concentration (based on the compatibility test parameter, $M < 1$, adsorption value $< 400 \mu\text{g/g}$) temperature of 60°C, and slow injection rates of 0.3 cc/min, 0.6 cc/min, and 1 cc/min.

RESULTS AND DISCUSSION

Polymer screening on a laboratory scale is carried out to determine the characteristics of polymers in a reservoir. Recent popular material of polymer that is assured to accommodate oil fields is HPAM. HPAM has most often been used to achieve a more favorable mobility ratio and improve macroscopic sweep in chemical EOR by increasing the viscosity of the water. When dissolved in fluid, the polymer solutions have a viscosity that depends on many aspects: concentration, molecular weight, temperature, and salinity (Lemigas, 2008). In this study, the investigation of polymer flood has been performed

using a sandstone reservoir. The polymer compatibility test for injection water was carried out at room temperature and reservoir temperature of 60°C. The result of that test is shown in Table 2 which shows the good polymer solutions with clear, no sediment for each of the polymer concentrations.

Table 2
Aqueous stability polymer

Polymer	Aqueous Stability
F1	Clear, no sediment
F2	Clear, no sediment
F3	Clear, no sediment
A1	Clear, no sediment
P1	Clear, no sediment

The rheological properties of the HPAM polymer solution were evaluated by measuring the apparent viscosity vs concentration and viscosity vs shear rate. This experiment is one of the most prominent screenings of an injected chasing fluid during the chemical flooding process. Figure 1 demonstrates variation shear rate from 7 rpm to 330 rpm was conducted on the viscosity of 1 concentration of each polymer at 60 °C temperature. This result presents that HPAM is generally classified as a non-Newtonian fluid because the viscosity changes when the shear rate was applied. Thus, the type of fluid rheology is pseudo-plastic fluid. In this desired condition, pseudo-plastic fluid was known as shear thinning, in which viscosity decreases as the shear rate increases.

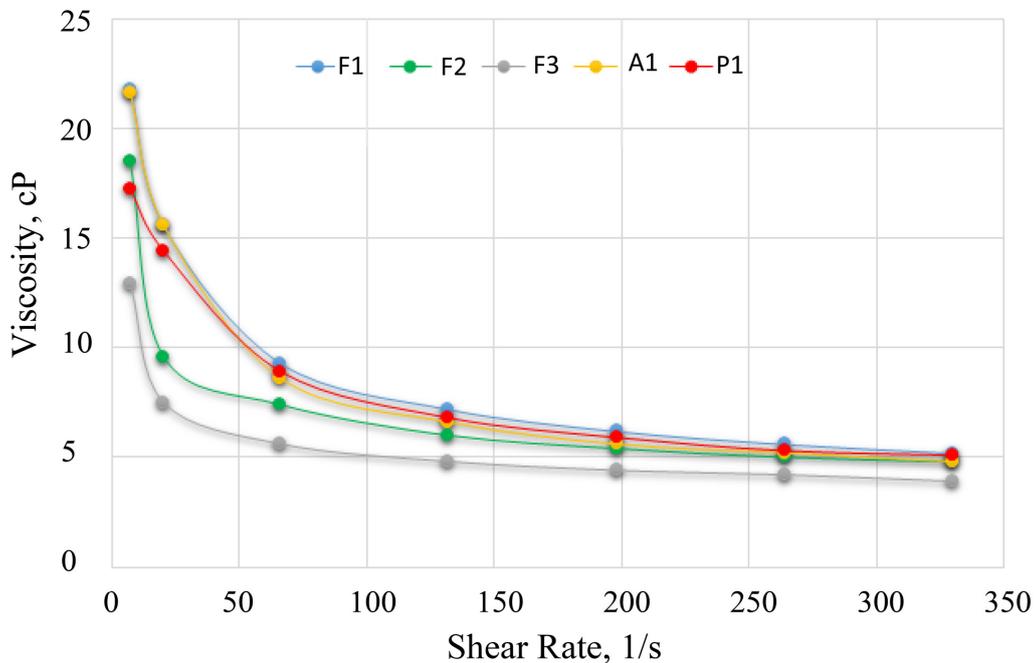


Figure 1
Effect of polymer on shear rate test results

The experiment result of viscosity vs concentration demonstrated the viscosity increasing steadily with increasing polymer concentration at 60 °C temperature. This behavior greatly contributes to the shear thickening of the HPAM solution when the polymer flows at a high shear rate in porous media. A screen factor test was carried out to determine the quality of the polymer solution. Based on the results of the screen factor test shown in Figure 2 concluded that a higher polymer concentration indicates the polymer solution was long to flow. The equation used to determine the screen factor (Sorbie, 1991) is:

$$SF = \frac{t(\text{solution}) (\text{second})}{t(\text{solvent}) (\text{second})}$$

The filtration test was performed to determine whether the polymer can flow through the rock pores and to evaluate the effect of debris. Figure 3 informs a volume plot graph against the time of the polymer through the filter paper. Each concentration solution ensured that polymer hydration had been achieved. The value of the FR for F1 2000 ppm is 1, F2 2000 ppm is 1.2, F3 2500 ppm is 1.1, A1 1500 ppm is 1.02, and P1 2000 ppm is 1.29.

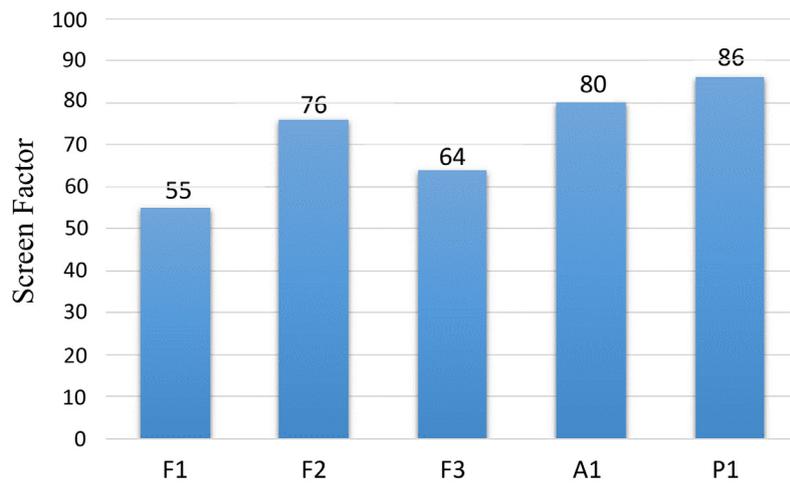


Figure 2
Screen factor test results

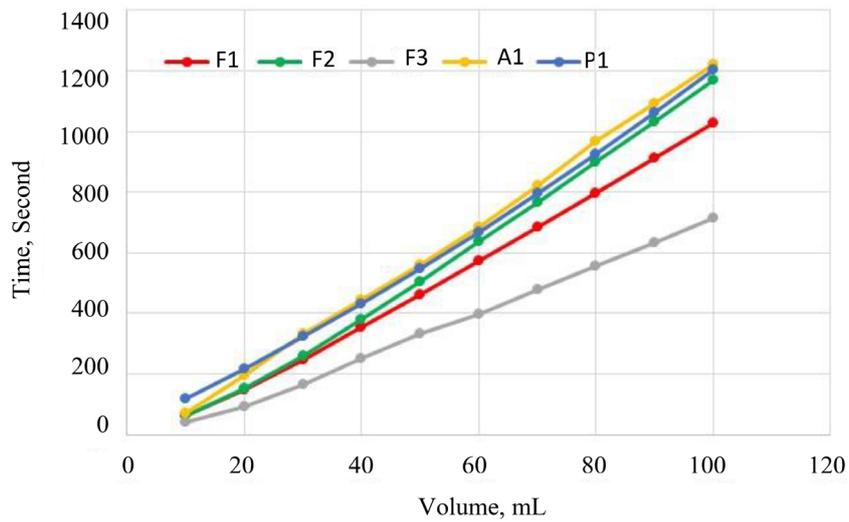


Figure 3
Filter test using 3 μ m membrane results

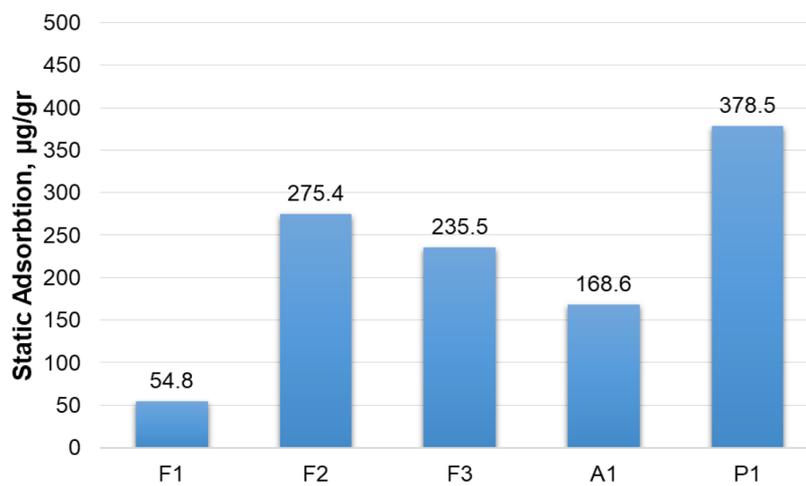


Figure 4
Adsorption static on native core results

This result rapidly indicates that F1, F2, F3, and A1 are acceptable as it does not tend to plug porous media in the reservoir because the requirement of the filtration ratio (FR) value was below 1.2. If the FR value > 1.2 indicates an indication of polymer causing plugging in rock pores. A static adsorption test

is carried out using the native core. Based on each polymer solution, the F1 polymer solution obtained has an adsorption value of 54.8 g/gr and can be seen in the distribution of adsorption on each polymer in Figure 4.

Table 3
Scoring parameter of the polymer

PARAMETER	CATEGORY	SCORE	PERCENTAGE	F1		F2		F3		A1		P1	
				Test Result	Score								
Price, US\$/kg	< 3		20										
	3 - 4		15	3	15	3	15	3	15	4	10	4	10
	4 - 5	20	10										
	> 5		5										
PV Injected, PV	≤ 0.3		10										
	0.3 - 0.5		8	0.3	10	0.3	10	0.3	10	0.3	10	0.3	10
	0.5 - 0.8	10	6										
	> 0.8		4										
Concentration, %	< 0.15		15										
	0.15 - 2.0		12	0.20	12	0.25	9	0.20	12	0.15	15	0.20	12
	0.2 - 0.3	15	9										
	0.3 - 0.5		6										
	> 0.5		3										
Screen Factor	≤ 50		10										
	50 - 60		7.5	55	7.5	76	2.5	64	5	80	2.5	86	2.5
	60 - 75	10	5										
	75 - 100		2.5										
Filtration Ratio	< 1		10										
	1.1 - 1.2		7.5	1.00	10	1.20	5	1.10	7.5	1.02	7.5	1.29	5
	1.2 - 1.3	10	5										
	> 1.3		2.5										
Static Adsorption, µg/g	< 100		10										
	100 - 200		8	54.8	10	275.4	6	235.3	6	167.0	8	378.5	4
	200 - 300	10	6										
	300 - 400		4										
	> 400		0										
Molecular Weight, million Dalton	< 10		10										
	10 - 15		8										
	15 - 20		6	8	10	12	8	20	4	15	6	15	6
	20 - 25	10	4										
	> 25		2										
Thermal Degradation (%)	0 - 10		15										
	10 - 20		12										
	20 - 40		9	15.5	12	14.05	12	16	12	38.6	9	35.6	9
	40 - 50	15	6										
	> 50		3										
		100		86.5		67.5		71.5		68.0		58.5	

The laboratory thermal degradation test was conducted to investigate the remaining viscosity after aging as the primary criterion for chemical EOR. F1 presented a good polymer candidate as it performed the remarkable viscosity decay during an aging period at 60°C temperature (see Figure 5). The viscosity of F1 2000 ppm maintains a constant value in the last 30 days, and the rest after 3 months of the aging period, increased slowly from 22 cP to 27 cP in the first 20 days. The viscosity of F2 2000 ppm maintains a constant value in the last 30 days, and the rest after 3 months of the aging period, decreases slowly from 20 cP to 14 cP with a viscosity retention

percentage of 20%. Thus, more effective preparations should be developed to improve their thermal degradation. Based on the results of the thermal stability test, scoring is carried out on several test parameters and characteristics of the polymer. Table 3 shows the F1 polymer has a score of 86.5 assuming the F1 polymer price is 3\$/kg, the pore volume injected into the reservoir is 0.3. To understand the performance of the polymer on the rocks, injectivity tests were carried out using polymer F1 2000 ppm. The characteristic of the rocks that were used is sandstone native core plugs with a permeability range of 1500 to 2500 mD and an average porosity of 0.26.

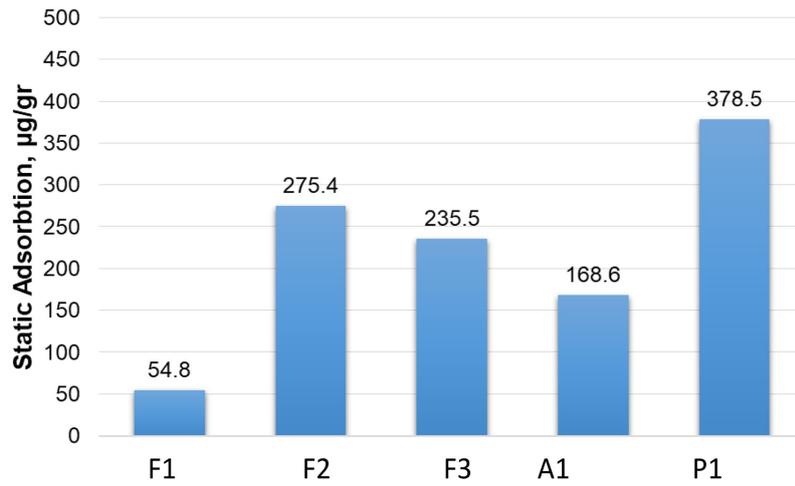


Figure 5
Thermal degradation test results

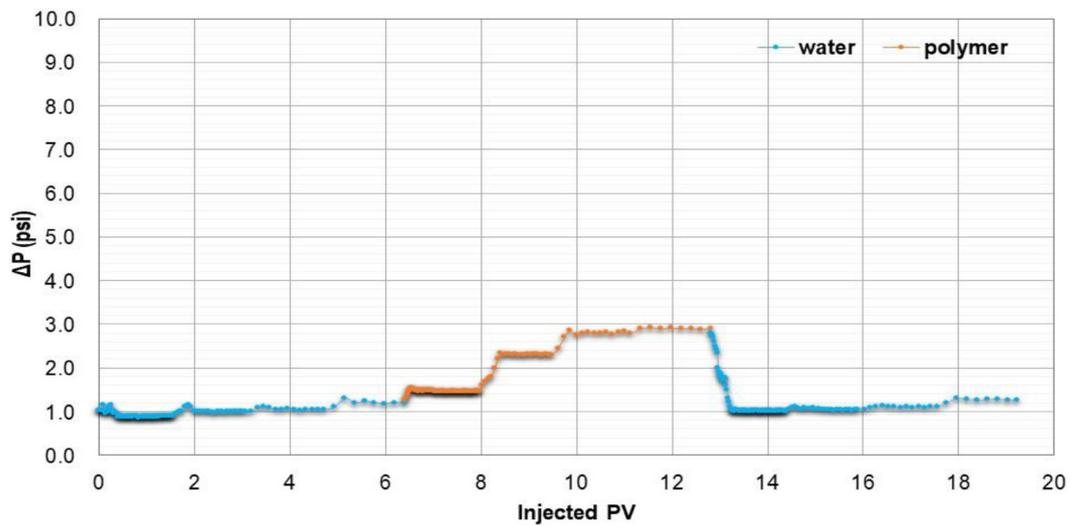


Figure 6
ΔP Distribution vs injected pore volume

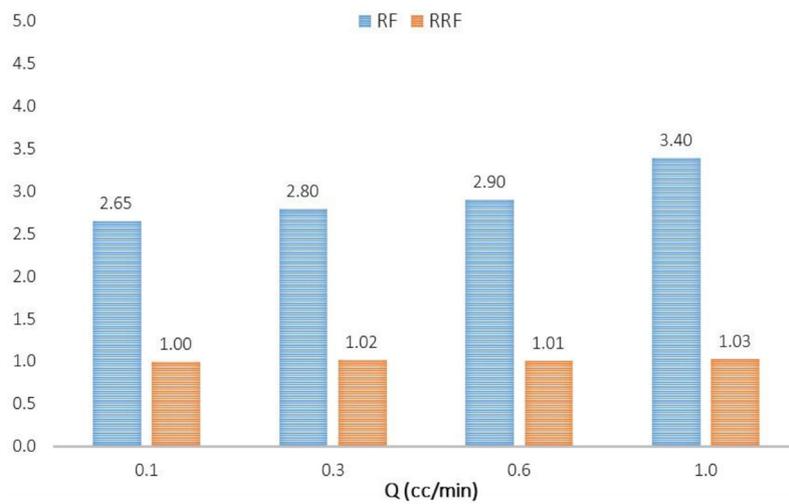


Figure 7
Injectivity test result (RF & RRF)

The injectivity tests were carried out using the step-up rate of 0.1, 0.3, 0.6, and 1 cc/min. The results of the polymer injectivity can be seen in Figure 7. Based on the test results, the polymer has a Residual Resistance Factor (RRF) average value of 1 (one) which means that the permeability of the core plugs after polymer injection were not changing as much after the polymer injection.

Table 4
Native core characteristic

Core	D (inch)	L (inch)	W (gr)	O	Ka (mD)
1	1.5	6.0	316.0	0.28	764.0

CONCLUSIONS

The test results for all of the parameters already meet the criteria for polymer screening as chemical EOR. Based on the polymer screening test and polymer performance test that have been done, polymer concentration (F1) of 2000 ppm is suitable for polymer injection with a rule of thumb that polymer viscosity should be four times higher than oil viscosity (6.988 cSt) which gives about 22.71 cP. Accordingly, then the conclusion obtained from this study is that F1 2000 ppm was selected. F1 with a concentration of 2000 ppm was resistant in reservoir conditions, it is shown in the thermal stability test (Figure 5). Also, the polymer concentration (F1) had the best score of the scoring parameters shown in Table 3 which gives about 86.5 out of 100. The results of the injectivity test indicated the rate of injectivity affected the RRF value. Based on these results, this polymer has the potential to be implemented on the pilot scale in the light oil reservoir.

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GLOSSARY OF TERMS

Symbol	Definition	Unit
API	American Petroleum Institute	°
cP	Centi Poise	

cSt	Centi Stoke
EOR	Enhanced Oil Recovery
FR	Filtration Ratio
HPAM	Hydrolized Polyacrylaamide
M	Mobility Ratio
mD	mili darcy
ppm	parts per million
rpm	rotate per minutes
RP 63	Recommended Practice 63
RRF	Residual Resistance Factor
SF	Screen Factor

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Converting Catalytic Palm Oil (MEPO) to Produce Biogasoline Using Zeolite Faujasite Catalyst From Fly Ash with Nickel Impregnation (Ni)

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ABSTRACT - The process hydrocracking methyl ester of palm oil into fractions biogasoline by faujasite catalyst of fly ash impregnated with nickel have been made. Preparation for faujasite synthesis of fly ash can be done by removing organic compound and refluxing HCl. Synthesis do by melting the fly ash which has been prepared with NaOH 1: 1.2 and in aging for 8 hours and in the hydrothermal autoclave for 24 hours. The character faujasite using XRD and Si / Al ratio produces crystallinity main peak of 67% and Si / Al ratio of 1.65. Hydrocracking process using a variety of 4 catalyst used fly ash leaching results, faujasite, Ni-Faujasite 2%, and Ni-Faujasite 4%. Test the activity and selectivity of the catalyst to produce liquid product analyzed by GC-MS with the best catalyst was Ni-Faujasite 4% to yield 42.34% of the activity and selectivity of biogasoline fraction of 7.12%. The impregnation of the nickel catalyst is made by soaking in salt of nickel and then oxidation using O₂ gas and reduction using H₂ gas. The impregnation of nickel will affect the character of the catalyst so that the activity and selectivity of the catalyst is changed. The impregnation of nickel 4% on faujasite successfully done with nickel content of 3.71%, increasing Si / Al ratio of 2.27 and an acidity of 0.0035 mol/g.

Keywords: faujasite, impregnation, nickel, hydrocracking, biogasoline.

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INTRODUCTION

Human needs in the field of fuel oil are increasing. Efforts to find alternatives to obtain renewable fuel sources must be carried out. One of the renewable energy sources is the production of biofuels obtained from palm oil. The composition of palm oil consists of long hydrocarbon chains with oleic acid and palmitic acid content exceeding 80% which is a great potential for palm oil as a raw material (Farouq et al, 2009). Palm oil conversion process can be done in various ways. Yusman (2012) carried out catalytic cracking using methyl ester palm oil with a modified natural zeolite (modernite) catalyst impregnated with 3% nickel which was car-

ried out with a weight ratio of 2:1 oil/catalyst for 30 minutes at 450 oC to produce a biogasoline fraction of 12.06 %. The hydrocracking process of palm oil methyl esters using a modified natural zeolite (modernite) catalyst impregnated with 2% nickel carried out with an oil/catalyst ratio of 2:1 for 30 minutes at a temperature of 450 oC has been carried out by Junaidi (2011) by producing a biogasoline fraction of 19 ,62%.

The hydrocracking process produces a larger biogasoline fraction, so this research uses the hydrocracking process. The hydrocracking process in this study uses a faujasite zeolite synthesized from fly ash. Natural zeolites (modernites) are usually found

in sediments as a result of alteration of volcanic ash. The sedimentation process is also mixed with other minerals such as feldspar and quartz to form a non-uniform and irregular structure. Irregularity is what makes mineralogy researchers make zeolite with zeolite stability with other minerals (Las, 2004).

Synthetic zeolites can be made under hydrothermal conditions. Hydrothermal system is a closed system by using water as a solvent to produce water vapor pressure on the system (Rodhie, 2006). The zeolite that will be synthesized this time is the faujasite zeolite. Faujasite zeolite is a synthetic zeolite from alumina and silica sources in the environment of sodium hydroxide solution at 100 °C (Tovina, 2009). This zeolite has a 3-dimensional axial structure with pores perpendicular to each other and is constructed from construction units 4-6 and 6-6 with a Si/Al ratio of faujasite of 1-3 (Rodhie, 2006).

Previous researchers have synthesized faujasite from fly ash. Sutarno (2004) used fly ash from PLTU Suralaya with a Si/Al ratio of 2.14 to produce faujasite which had a Si/Al ratio of 2.79 with the crystallinity of XRD results showing 70% (main peak) of the standard faujasite. Faujasite synthesis was also carried out by Somerset (2004) using fly ash from South Africa with a Si/Al ratio of 1.95 but zeolite A and sodalite were also found. The synthesis of faujasite zeolite in this study came from fly ash of the Paiton Probolinggo PLTU with the largest content of SiO₂, which is 30.25% and Al₂O₃ which is 14.52% (PJB Paiton, 2002). This content has the potential to be used as a faujasite zeolite.

Hydrocracking in this study used a faujasite catalyst synthesized from fly ash impregnated with Ni metal to produce Ni-Faujasite. Nickel metal impregnated onto a zeolite carrier will increase the acidity and specific surface area of the catalyst if the Ni metal is evenly spread on the surface of the zeolite (Wulansari, 2004). Hayat (2007) explained that the distribution of Ni²⁺ metal is better than that of Co²⁺ which is based on a smaller Ni metal radius, thereby increasing its distribution inside and outside the pore cavity. Yusro (2012) used natural zeolite (modernite) impregnated with 3% nickel with a hydrocracking process at a temperature of 450 oC for 30 minutes in methyl palmitate to produce a biogasoline fraction of 12.01%. Sutarno (2007) impregnated Ni metal at a weight ratio of 4% of the faujasite catalyst synthesized from fly ash of the Suralaya PLTU with hydrocracking in petroleum distillate, the ratio of oil:catalyst weight was 2:1 with a temperature condi-

tion of 320 oC for 45 minutes producing a gasoline fraction of 8 %.

The use of a catalyst from fly ash that has been impregnated by Ni has the potential to be developed as an alternative to biogasoline production from palm oil using the hydrocracking method. This research uses a faujasite zeolite catalyst from fly ash PLTU Paiton Probolinggo. Faujasite zeolite catalyst from fly ash was then impregnated with active metal Ni. Sutarno (2007) revealed that changes in the catalytic properties of a metal can occur with variations in the composition of the carrier from the carrier preparation, therefore, in this study, variations in the levels of Ni metal were carried out on the catalyst. The hydrocracking results will be characterized using GC-MS.

METHODOLOGY

A. Fly ash preparation

50 grams of fly ash from PLTU Paiton-Probolinggo was soaked and stirred in water at 100oC for 2 hours to extract water-soluble organic matter.

B. Leaching Fly Ash

Reflux 10 g of fly ash (procedure A) and 100 mL of 5 M HCl solution. Reflux was carried out at 90 oC for 1 hour. The refluxed fly ash was then washed with distilled water until the pH of the washing filtrate was neutral and the solids were dried in an oven. Smelting was carried out on fly ash resulting from reflux of 5 M HCl with solid NaOH (weight ratio of NaOH: fly ash = 1,2) at a temperature of 550 oC for 1 hour.

C. Hydrothermal

The smelting result (Procedure B) was then dissolved in distilled water, stirred and allowed to ferment for 8 hours. The results of dissolving and aging are then reacted hydrothermally in an autoclave for 24 hours at a temperature of 100 oC. The results of the hydrothermal reaction were then washed with distilled water until neutral and in an oven at 100 oC (Somerset, 2004).

D. Ni Impregnation

The impregnation of Ni on faujasite (Procedure C) was carried out by the wet impregnation method. Ni(SO₄).6H₂O salt with variations of 2% and 4% was dissolved into 100 mL of distilled water while stirring until homogeneous. The result of the dissolution is added with 10 g of the extraction result then

heated and evaporated at a temperature of 80 oC to 90 oC while stirring until the water evaporates. The result of evaporation is continued by oxidation process with oxygen gas 20 mL/minute at a temperature of 500 oC for 2 hours and reduction at a temperature of 500 oC with hydrogen gas 20 mL/minute for 2 hours (Handoko, 2001).

E. Faujasite Crystallinity

The obtained solid (Procedure C) was then characterized by X-ray diffraction method. X-ray diffraction measurements were carried out using an Xpert MPD X-ray diffractometer. The measurement conditions are, target = Cu, voltage 40 kV, current 30 mA, differegen slit = 0.25 degrees, and receiving slit = 12.7500. The crystallinity of the synthesized Faujasite was determined from the ratio of the height or intensity of the 8 main peaks of the synthesized Faujasite with standard Faujasite.

F. Determination of Total Acid

The total acidity of the catalyst can be determined gravimetrically on the basis of the adsorption of ammonia gas on the catalyst surface. The porcelain dish was then heated in an oven at a temperature of 120 oC for 2 hours, after which it was cooled and the mass was weighed. The acidity of the catalyst represents the number of moles of NH₃ adsorbed to the catalyst per gram by weight. The measurement of zeolite acidity was carried out by the method of adsorption of ammonia base on the surface of the catalyst. Ammonia will be adsorbed on the surface of the Bronsted acid site and the Lewis acid site (Handoko et al, 2009).

G. MEPO Hydrocracking Process

Palm oil methyl ester (MEPO) as much as 10 grams was placed in the feed tank and 3 grams of catalyst from each catalyst variation. The heat of the reactor system is set at 450oC and H₂ gas with a gas flow rate of 20 mL/min in Figure 1. The reaction product in the form of gas is passed into a condenser (coolant) until it turns into a liquid. Hydrocracking of palm oil methyl ester (MEPO) was carried out for 60 minutes. The reaction products from each catalyst variation were analyzed using GC-MS (Haliq, 2012).

H. Calculation of Activity and Selectivity

The cracking product of palm oil methyl ester was analyzed using GC-MS. The data from the GC-MS analysis was used to test the activity and selectivity of the catalyst. The sample was inserted into

the GC-MS tool set with an FID detector. Column temperature at 60 oC. Flow rate 0.51 mL/min. The detector temperature is 280 oC. Injector temperature 310 oC. Analysis time 21 minutes. Injection Volume 0.20 mL. %Selectivity=Amount of % area of hydrocarbon compounds (Haliq, 2012)

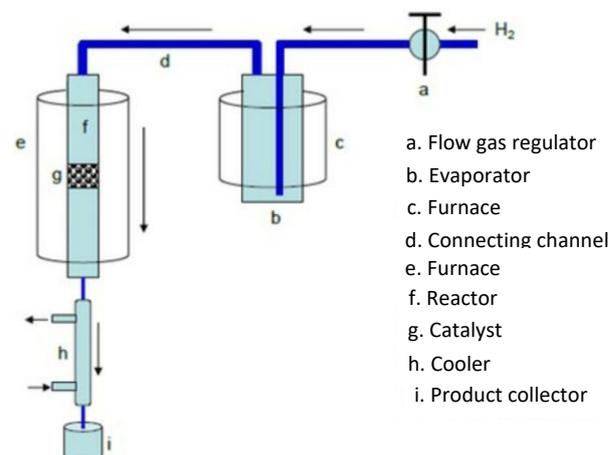


Figure 1
Fixed bed reactor (Handoko, 2009)

RESULTS AND DISCUSSION

A. Synthesis of Faujasite Zeolite From Fly Ash

• Fly Ash Preparation Results

Faujasite synthesis begins with the preparation of fly ash from the Paiton-Probolinggo PLTU. The result of the preparation has a grayish color due to the loss of impurities. The carbon in fly ash is separated based on differences in density. The nature of carbon with a large surface area and high absorption will absorb alkaline solutions so that it can reduce its concentration (Fansuri, 2015). Reducing the concentration of the alkaline solution will reduce the ability to dissolve Si and Al in the next faujasite synthesis process. The results of the fly ash immersion were then refluxed using 5M HCl solution for 1 hour. The result of the reflux process is that fly ash changes color from gray to brownish black. The reflux process ended by neutralizing the fly ash with distilled water to remove excess acid species. Reflux with HCl increases the ratio of Si/Al because the dissolving process of Al species is easier than Si species. Reflux can also remove metal impurities such as iron and calcium. Calcium ions in the hydrothermal solution system result in the deposition and stabilization of silicate species so that they do not form a Faujasite framework structure (Sutarno, 2004).

• **Hydrothermal**

Hydrothermal in the synthesis of faujasite is done by melting the prepared fly ash with NaOH. Hydrothermal is a heating process at a certain temperature with the balance of steam and water maintained. The presence of water here is needed because it is to determine the crystallinity (of the zeolite). The result of hydrothermal and aging is the formation of faujasite with a bright white color and solid form as shown in Figure 2.

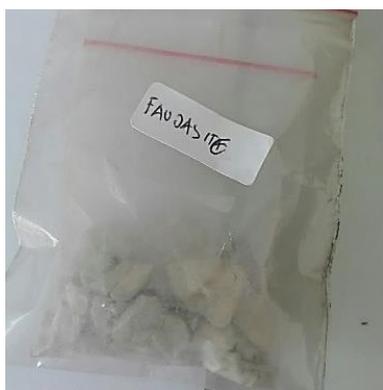
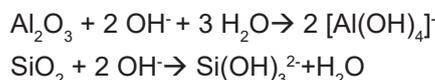


Figure 2
Faujasite crystal

The prepared fly ash was added with alkaline NaOH so that it was able to attract protons and resulted in the deposition of Al³⁺ cations to produce aluminate metal deposits. Alkali metal Na also ac-

celerates the nucleation process because it reduces the effect of protonic acid sites (Bronsted acid) and Na is smaller in size so that it is easier to exchange (Ulfah, 2006). Sodium aluminate and sodium silicate are formed by the reaction:

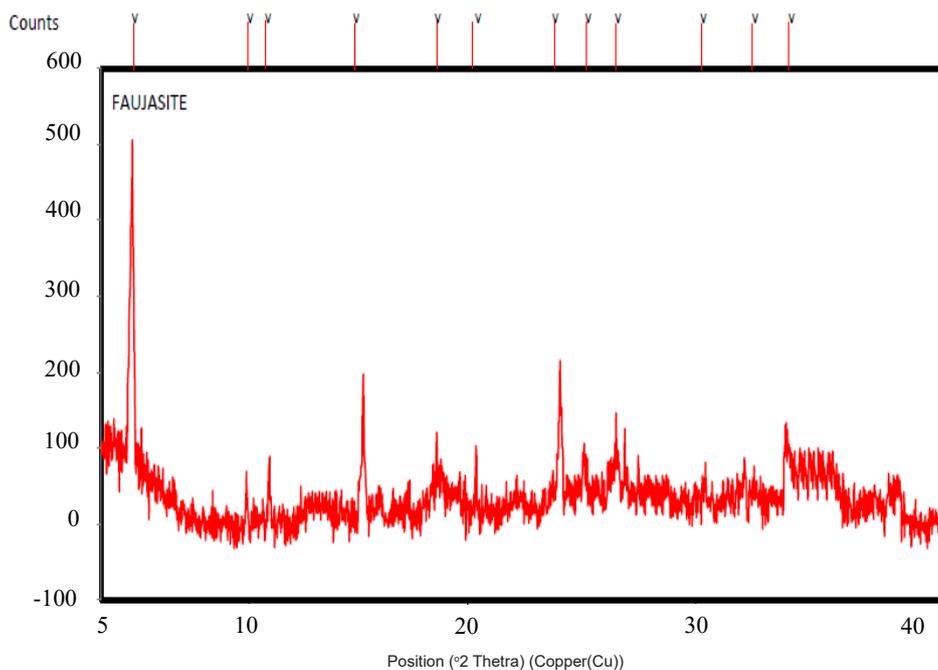


B. Characteristics of Faujasite Zeolite

• **Characteristics of XRD Faujasite**

The resulting Faujasite was analyzed using XRD. Comparison of the 2 theta angle from the synthetic faujasite from fly ash was matched with the standard faujasite from Nino (2013). The XRD results on the x axis indicate the 2 theta angle while on the y axis it is the intensity. Figure 4.3 shows the XRD diffractogram of synthetic faujasite and standard faujasite from Nino (2013).

XRD results show a similar pattern between synthetic faujasite and standard faujasite (Nino, 2013) in other words, the synthesis of faujasite from Paiton PLTU fly ash this time has been successfully carried out. The characteristic peak at 2 theta angle of the faujasite is 8 then matched with the synthesized faujasite listed in Table 1.



(a)

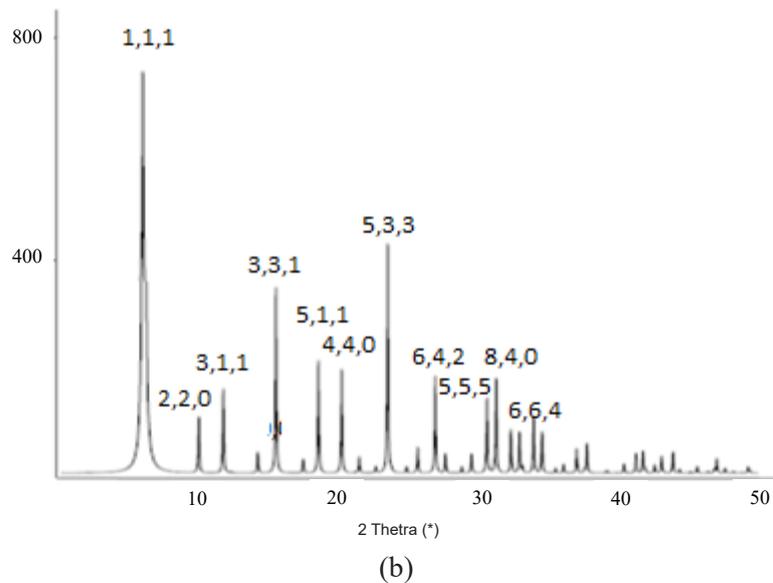


Figure 3
(a) Synthesis faujasite XRD diffractogram, (b) standard faujasite (Nino, 2013)

Table 1
Comparison of peak characteristics of 2 theta

No.	2 Theta (degrees) Standard Faujasite (Nino, 2013)	2 Theta (degrees) Faujasite Synthesis
1.	6.19	6.2
2.	10.11	10.9
3.	15.61	15.8
4.	20.30	20.4
5.	23.58	23.9
6.	26.97	27.1
7.	30.66	30.2
8.	33.99	33.2

The main peak of the faujasite is at 2 theta angle of 6.2°. The main peak crystallinity of synthetic faujasite was 67%. The appearance of another peak is Hydroxysodalite. The Si/Al ratio of leached fly ash is only 1.28 causing a very high amount of dissolved alumina causing the crystal growth rate to be fast so that zeolite structures that are easier to form are Hydroxysodalite (Sutarno, 2004). The time required for hydrothermal and aging also affects the crystallinity as done by Sutarno (2004) has a main peak crystallinity of 70%, but when compared to synthetic faujasite from Paiton PLTU fly ash this time it has a slightly lower crystallinity but the method used is quite efficient because shorter synthesis time.

• Characteristics of Faujasite Si/Al Ratio

Faujasite synthesized by Paiton fly ash was analyzed using AAS to determine the ratio of Si to Al. The results of the analysis showed that the Si/Al ratio of faujasite was 1.65. Faujasite generally has a Si/Al ratio between 1-3. The results of this synthesis have entered the range. Faujasite synthesized this time has similarities with the ratio of Si/Al from faujasite Y. Faujasite type Y is a zeolite with more silicon content than aluminum (Rodhie, 2006). Y type Faujasite has better thermal stability and crystallinity.

The synthesized Faujasite was then compared with Type X and Y type. Table 2 shows the crystallographic data of X and Y faujasite.

Table 2
Differences between faujasite X and faujasite Y
(Baerlocher, 2007)

Differentiator	Faujasite X	Faujasite Y
Unit Cell	Cube	Cube
Unit Cell Constant	25,009	24,2576
Framework	D6R	D6R
Si/Al . Ratio	1-1,5	1,5-3

Faujasite synthesized this time when viewed from the XRD data between type X and type Y, this time the faujasite has a tendency similar to that of type Y with a unit cell constant of 24,503. A review of the Si/Al ratio also shows that the faujasite synthesized this time is similar to type Y faujasite. From

the data above, it can be concluded that the faujasite synthesized from fly ash this time is more similar to type Y faujasite zeolite.

• Hydrocracking Activity and Selectivity Catalyst Activity

Catalyst activity is the ability of a catalyst to produce new compounds. The new compound is a conversion of 2 dominant products from MEPO, namely methyl palmitate and methyl oleate by producing a mixture of new compounds between types of paraffins, olefins, naphthenes, carboxylic acids, aromatics, methyl esters, aldehydes, and ketones. The activity value can be seen from the concentration of the number of new products produced after the hydrocracking process with each catalyst divided by the total concentration of MEPO before hydrocracking shown in Figure 4.

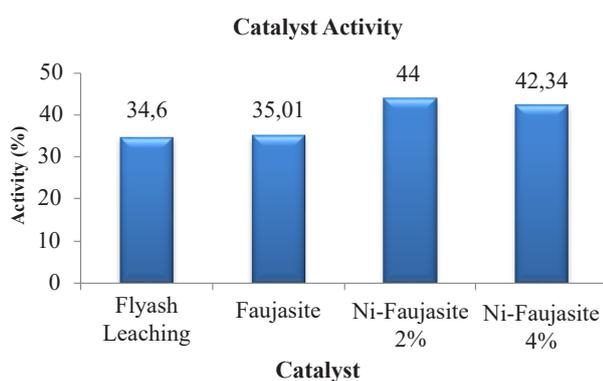


Figure 4
Catalyst activity

The activity of the catalyst increases with each catalyst used. Leached fly ash resulted in 34% activity. The ability of leached fly ash for hydrocracking is still not good because the acidity and Si/Al ratio of the leached fly ash catalyst is still low. Faujasite catalyst that has not been impregnated experienced a slight increase in activity from the leached fly ash catalyst. The acid site formed caused the hydrocracking activity of the faujasite catalyst to increase slightly to 35.01%. The slight increase was caused by the not yet active faujasite zeolite as a catalyst. Catalysts that have not been activated so that they do not have an active site to activate them are given acid treatment (HF, HCl, and NH₄Cl) to open the pores on the surface of the zeolite (Satterfield, 1980). The impregnation process on the catalyst showed that the nickel content increased the activity of the catalyst. Faujasite catalyst impregnated with nickel as much as

2% had the largest activity of 44% and then decreased to 42.34 on catalyst impregnated with 4% Ni.

I. Catalyst Selectivity

Catalyst selectivity is the ability of a catalyst to produce certain products. The selectivity of the catalyst was grouped into 3 groups, namely C₆-C₁₂, namely the biogasoline fraction, C₁₃-C₁₈, the biodiesel fraction, and C₁₉-C₂₄, the biokerosene fraction. The percentage of selectivity can be determined by the total % area of the GC-MS results obtained and then presented in Table 3.

Table 3
Catalyst selectivity of various fractions

Fraction	Fly ash Leaching	Faujasite	Ni-Faujasite 2%	Ni-Faujasite 4%
C ₆ -C ₁₂	0,27	1,23	3,68	7,12
C ₁₃ -C ₁₈	7,57	6,84	11,0	2,25
C ₁₉ -C ₂₄	0	1,7	1,04	0

The catalyst selectivity process for each catalyst was dominated by products with medium fraction liquid hydrocarbons (C₁₃-C₁₈). Hydrocracking using 4% Ni-Faujasite catalyst has the highest selectivity value in cracking producing short fractional liquid hydrocarbon compounds (C₆-C₁₂) which is 7.21%. The higher nickel content increases the selectivity of the catalyst due to the increasing number of Lewis acid sites. The hydrocracking results for the short fraction (C₆-C₁₂) consisted of a mixture of 1-hexene, 5 methyl hexene, methyl cyclopentene, benzene, 1-heptene, n-heptane, toluene, 1-octene, 2,4-dimethyl hexane, 1-nonene, 1-decena, 1-undecana, 1-dodecena.

Determination of the activity and selectivity of the catalyst for the formation of short fraction liquid hydrocarbons cannot be separated from the characteristics of the catalyst, such as acidity, Si/Al ratio and the amount of nickel metal impregnated on the catalyst from the results of research conducted by Yusman (2012). The results of the characterization of the 4% faujasite catalyst have higher acidity, Si/Al ratio, and impregnated nickel than other catalysts. This research contributes to the conversion process from palm oil methyl esters to short fraction liquid hydrocarbons with activity of 42.34% and selectivity of 7.12%.

- **The Effect of Nickel Impregnation Variations on Catalyst Selectivity and Activity**

The impregnation in this study used Ni metal. Impregnation was carried out with variations of 2% and 4% of the weight of the faujasite. Comparative data is shown from the percentage of nickel content of leached fly ash and synthetic faujasite. Leached fly ash has a nickel content of 0.09%. Fly ash from Paiton-Probolinggo does contain nickel but in low levels. Faujasite decreased nickel content to 0.083%. The existence of a hydrothermal process

which is a process of forming faujasite crystals so that there may be a decrease in nickel content but not significantly in the formed faujasite crystals. The addition of 2% and 4% salt showed an increase in nickel content to 2.05% and 3.7147%, respectively. The relationship between the amount of impregnated nickel with the activity and selectivity of the catalyst has a different trend. The graph in Figure 5 shows that the total nickel content for the cracking process with optimum activity is 2% nickel impregnated catalyst. Ni-Faujasite 4% catalyst decreased the activity of the catalyst against the hydrocracking process.

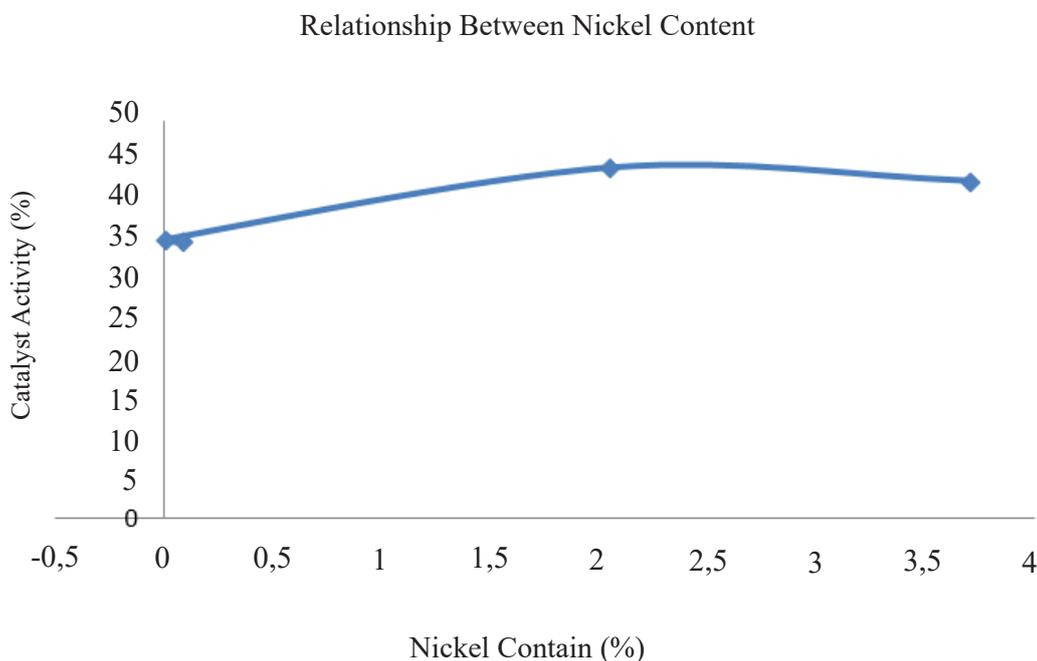


Figure 5
The relationship between nickel levels with catalyst activity

The increasing concentration of active metal impregnated onto the surface of the carrier may result in a decrease in the specific surface area of the catalyst. This phenomenon can be explained that the impregnated active metal is distributed unevenly or accumulates at the pore mouth (sintering) and closes the pore mouth (Figure 6). This situation occurs because the cohesive interaction between the active metal and the active metal is stronger than the adhesive interaction between the metal and the zeolite carrier. This situation should be analyzed using the BET method to provide information on the decrease in the specific surface area of the zeolite (catalyst) even though the impregnated metal showed an increase (Bartholhomew, 2006).

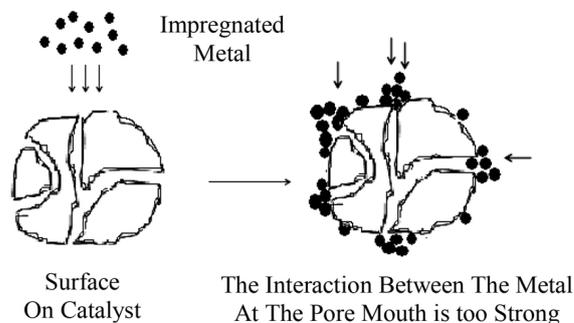


Figure 6
Accumulation of impregnated active metals in the pore mouth which causes a decrease in surface area (Bartholhomew, 2006).

The relationship between nickel content and catalyst selectivity can be seen in the graph in Figure 7 that the value of catalyst selectivity is increasing. The selectivity of using leaching and faujasite fly ash catalysts has a small value because nickel has not had a significant effect on nickel impregnated catalysts.

The use of nickel during impregnation showed an increase in both the 2% Ni-Faujasite and 4% Ni-Faujasite catalysts. The highest nickel content was in the 4% Ni-Faujasite catalyst which produced the maximum biogasoline product (Figure 7).

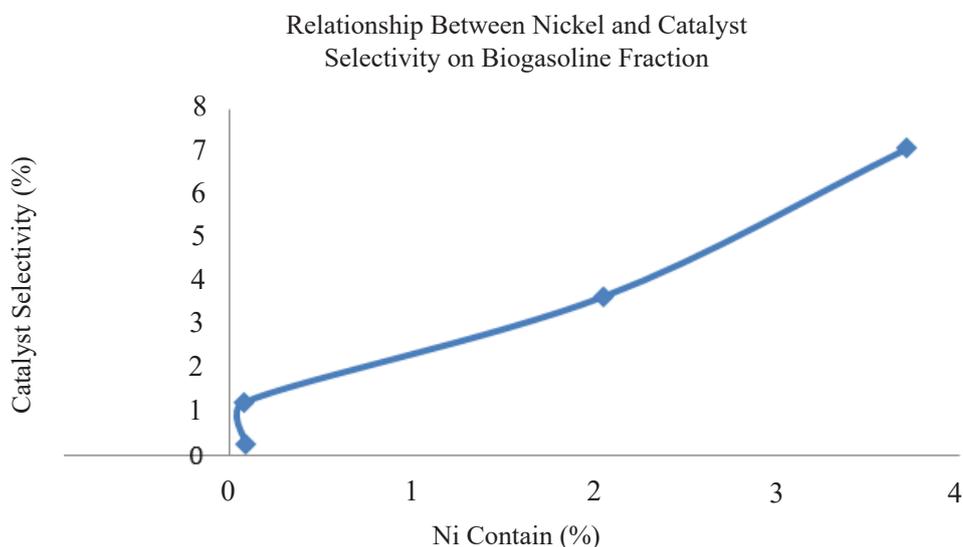


Figure 7
Correlation of nickel levels with catalyst selectivity to biogasoline fraction (C6-C12)

The nickel content is very influential on the selectivity of the catalyst to obtain short carbon fractions (C6-C12). Haliq (2011) has conducted research using nickel metal for the catalytic cracking process, so that alkane and alkene products have a fairly large concentration compared to other products produced. The development of Ni metal causes an increase in the number of products, especially in the form of paraffins and olefins. This study also showed that the highest use of nickel metal was in Ni-Faujasite 4% with a nickel content of 3.7147% resulting in a selectivity of the biogasoline fraction of 7.12% (Figure 7).

II. Relationship Between Si/Al Ratio With Catalyst Activity And Selectivity

The measurement results with AAS show that from various processes the trend is increasing. Leached fly ash has a low Si/Al ratio of 1.27. Faujasite has increased the ratio to 1.64. Faujasite has increased the ratio of Si/Al due to the hydrothermal process (Hamdan, 1992). This stage is a crystal growth stage, so it is possible that under these conditions there will be more silica reacting than alumina, causing the Si/Al ratio of the faujasite to increase.

Faujasite impregnated with 2% and 4% nickel had Si/Al ratios of 2.080 and 2.272, respectively. Impregnation of the faujasite catalyst increases the Si/Al ratio. The increase in the Si/Al ratio is due to dealumination events during oxidation and reduction at high temperatures (500 oC) (Handoko, 2009).

The relationship between the Si/Al ratio with activity and selectivity has a different trend as well. Similarly, the trend of nickel content with catalyst activity with optimum activity on 2% Ni-Faujasite catalyst. The graph in Figure 8 shows the relationship between the Si/Al ratio and the catalyst activity.

The biggest catalyst activity using 2% Ni-Faujasite catalyst. Faujasite zeolite synthesized this time has similarities with type Y faujasite. Type Y faujasite has a higher Si/Al ratio than type X faujasite. The large Si/Al ratio makes this faujasite has smaller pores so that the structure is more stable for catalyst activity. . The characteristics of the catalyst in terms of nickel content also affect the effect of the Si/Al ratio with catalyst activity as previously discussed, that as much as 2% nickel content allows a more even distribution (Bartholomew, 2006) so that the optimum catalyst activity is 2% Ni-Faujasite catalyst. It is the same as the trend of the relationship between

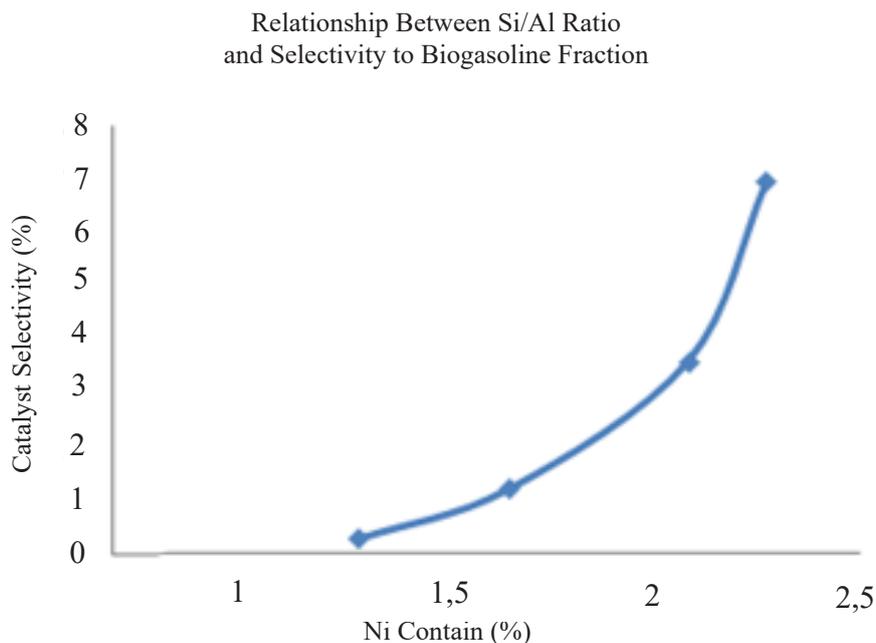


Figure 8
The relationship between Si/Al ratio and catalyst activity

nickel content and the selectivity of the catalyst with the trend of the relationship between the ratio of Si/Al to the selectivity of the catalyst. The graph of the relationship between the Si/Al ratio and the selectivity of the catalyst is shown in Figure 8.

The trend of the relationship between the Si/Al ratio on the selectivity of the catalyst shows that the higher the Si/Al ratio, the greater the selectivity of the catalyst. Catalysts with a high Si/Al ratio will have high thermal stability (Hamdan, 1992) and are effective in cracking non-polar compounds (Sutarti and Rahmawati, 1994). Catalysts containing more silicon than the affinity for non-polar molecules will increase because the Si-O (silicate) group is neutral. Neutral silicate zeolite will have minimal polarity so that it gives preference to the adsorption of non-polar reactants, namely non-polar compounds (Sutarti and Rahmawati, 1994). 4% Ni-Faujasite catalyst with a Si/Al ratio of 2.22 resulted in an optimum catalyst selectivity of 7.12%.

III. The Relationship Between Acidity With Catalyst Activity And Selectivity

The acidity of the leached fly ash showed the smallest value, which was 0.00012 mol/g. Fly ash from leaching has not yet formed a zeolite framework so that the Bronsted acid site has the smallest value for the acidity of this catalyst. The increase in acidity was seen in the faujasite of 0.000353 mol/g.

The increase is caused by the hydrothermal process that forms the zeolite framework, the framework can cause the appearance of Bronsted acid sites. Nickel impregnation also increases the acidity of the catalyst. The more nickel contained in the catalyst, the greater the acidity value of the catalyst. The acidity of 2% and 4% nickel impregnation were 0.003071 mol/g and 0.003494 mol/g, respectively.

The increase in acidity of the nickel impregnated faujasite has a significant increase. This phenomenon occurs because the metal Ni distributed on the catalyst as a carrier matrix has d orbitals that are not fully filled so that it can be used by NH_3 molecules to form complexes with NH_3 as ligands. In this case, Ni metal acts as a Lewis acid site contributor and contributes to the increase in the acidity of the catalyst in the 2% Ni-Faujasite and 4% Ni-Faujasite catalysts. The more Ni metal is deposited, the more Lewis acid is formed and the more NH_3 gas that can be bound by a nickel impregnated catalyst (Satterfield, 1980). Faujasite catalyst impregnated with nickel as much as 2% and 4% did not experience an increase in acidity so rapidly.

The more active metal impregnated onto the surface of the zeolite, the smaller the amount of impregnated Ni metal but relatively allows an increase in the specific surface area of the zeolite solid, although the increase in surface area is not too large. This phenomenon can be explained that the higher the amount of active metal impregnated

on the catalyst, the competition between the active metals occurs so that nickel will block each other. Furthermore, accumulation occurs in one place even though it is not at the pore mouth, so that it is not evenly dispersed onto the surface of the zeolite. The possibility that occurs in this situation is a multilayer layer of impregnated active metal, so that the presence of active metal in the second layer and so on will be easily released again when thermal treatment (Bartholomew, 2006). This situation may cause the Lewis acid site of 2% Ni-Faujasite and 4% Ni-Faujasite not to be significantly different.

The acidity of the catalyst is strongly influenced by the acid site of the catalyst. The ability of the catalyst is seen from the ability to absorb NH_3 base. Satterfield (1980) stated that the acidity of a catalyst is defined as the ability of the catalyst to adsorb ammonia bases due to the presence of both Bronsted acid sites and Lewis acid sites on the catalyst surface. The reaction that occurs is shown in Figure 9 by chemisorption. The more acid sites on the catalyst allow the more NH_3 to absorb, which means the acidity value of the catalyst is getting bigger. The increase in the acidity of the catalyst will be proportional to the amount of NH_3 gas bound to the catalyst. The relationship between acidity with activity and with catalyst selectivity is also different from other

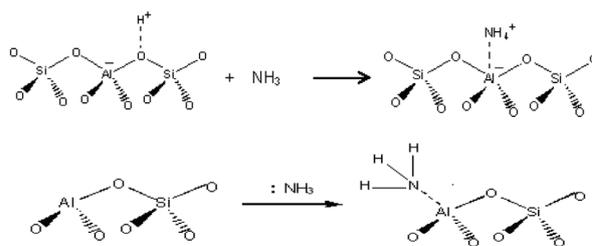


Figure 9
Chemisorption of ammonia on zeolite surfaces in determining catalyst acidity in bronsted acid (a) Lewis Acid (b) (Satterfield, 1980).

catalyst characteristics. The catalyst activity has the optimum condition on 2% Ni-Faujasite catalyst, while the selectivity of the catalyst has a selectivity for the biogasoline fraction using 4% Ni-Faujasite catalyst with the highest acidity of 0.0035 mol/g. The graph in Figure 10 shows the relationship between catalyst acidity and catalyst activity.

The trend between catalyst acidity and optimum activity indicates the use of 2% Ni-Faujasite catalyst. Ni-Faujasite 4% catalyst does have a higher acidity but its activity decreases. The higher the acidity value of the catalyst, the more acid sites available. The available acid sites are expected to make the catalyst more active in the cracking process. The

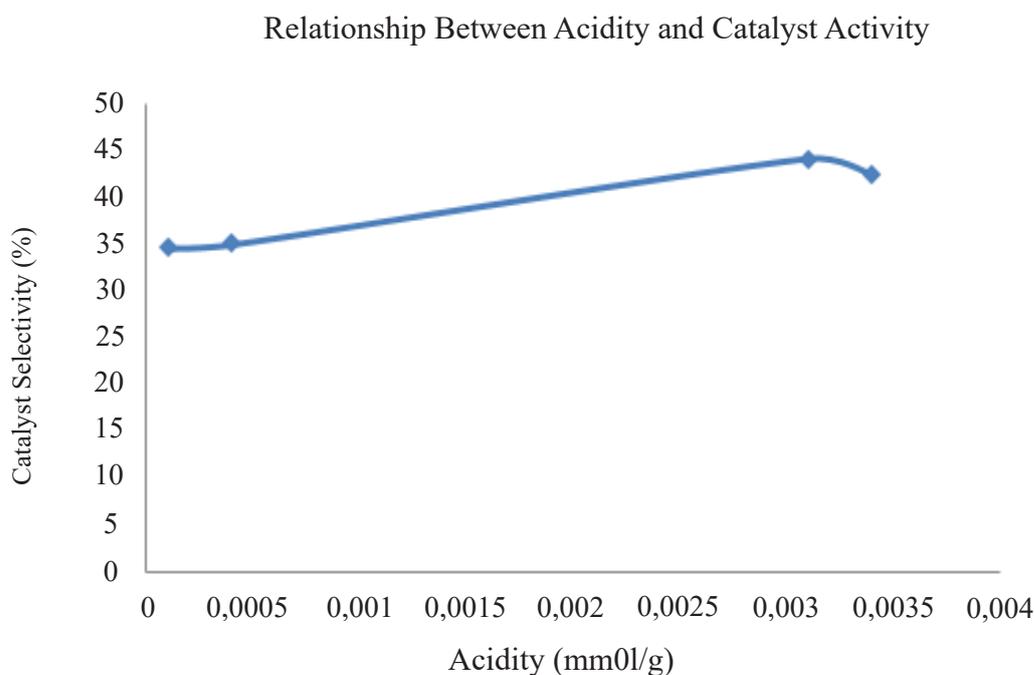


Figure 10
The relationship between acidity and catalyst activity

acidity characteristic of the catalyst is still related to the nickel content which may be distributed less evenly (Bartholomew, 2006) causing its activity to decrease compared to the 2% Ni-Faujasite catalyst, although the acidity is lower but not significantly different from the 4% Ni-Faujasite catalyst.

The trend of the relationship between acidity and selectivity of the catalyst to the biogasoline fraction shows that the higher the acidity of the catalyst, the greater the selectivity to the biogasoline fraction. Figure 13 shows the relationship between acidity and catalyst selectivity for the biogasoline fraction.

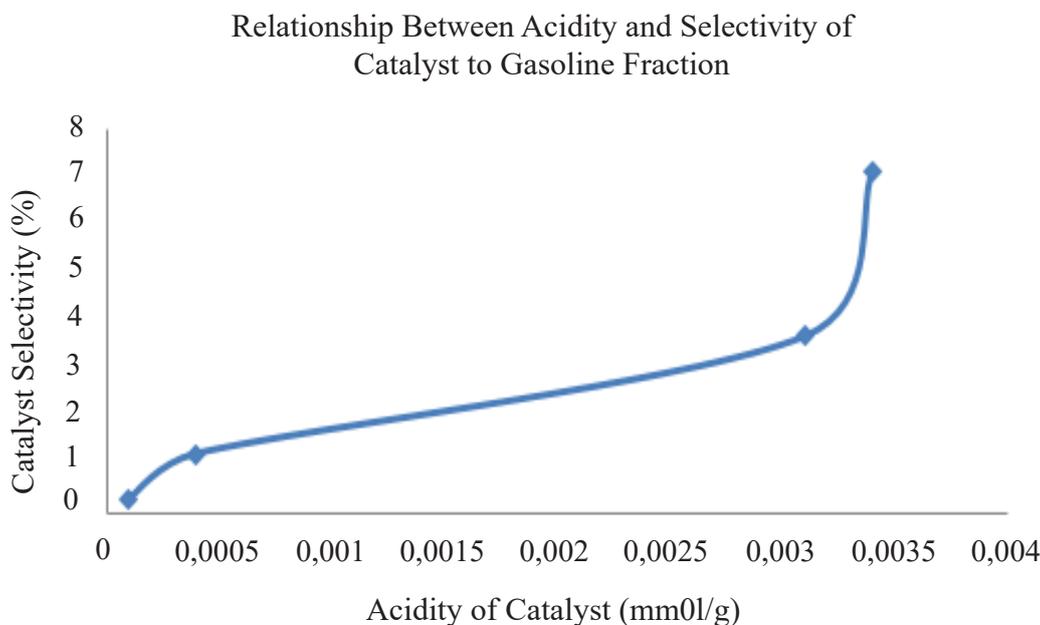


Figure 11
Relationship between acidity and catalyst selectivity to biogasoline fraction.

Catalysts with high acidity have more acid sites. The many acid sites will later affect the catalytic cracking process in producing the product. The increase in selectivity starting from the leached fly ash catalyst with the lowest acidity has the lowest selectivity as well. The graph in Figure 11 shows that the higher the acidity of the catalyst, the greater the selectivity for the biogasoline fraction. In this study, the highest acidity was 0.0035 mol/g on 4% Ni-Faujasite catalyst which was able to convert into liquid hydrocarbon products with the largest short fraction of 7.12%.

CONCLUSIONS

The conclusion in this study is that the characteristics of faujasite synthesis from fly ash are the main peak crystallinity of 67% and the Si/Al ratio of 1.64. Impregnation of Ni increases the ability of the catalyst in hydrocracking methyl ester palm oil to produce biogasoline fraction with activity yield

of 42.34%. and a selectivity of 7.12% using a 4% Ni-Faujasite catalyst.

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GOSSARY OF TERMS

Symbol	Definition	Unit
XRD	X Ray Difraction	
GC-MS	Gas Chromatography- Mass Spectroscopy	
MEPO	Methyl Ester Palm Oil	
AAS	Atomic Absorption Spectroscopy	
PLTU	Pusat Listrik Tenaga Uap	

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Laboratory Studies for The Development of a Demulsifier in Handling Production Fluid Emulsions in The “SRG” Field

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ABSTRACT - The “SRG” Oil Field is located in the South Sumatra basin, and the oil produced is classified as heavy oil and generally water-oil emulsion occurs. As a result of the formation of this emulsion which will cause corrosion of equipment in the field. The samples that have been taken in the field are then investigated in the laboratory of PT Farca Risa Sejahtera. First, perform BS&W testing on GS-belimbing and GS-11 oil samples to determine the water content and deposits present in the oil. The second is to determine the ratio of the amount of oil and formation water to be used in subsequent tests. The third selection of demulsifiers for formulation materials is based on the ability of water drop, clear water and interface. The four demulsifier formulations combine the demulsifiers that pass the selection into 5 formulas with the hope of uniting the advantages and covering each other’s shortcomings of each demulsifier that passes the selection. The fifth test is overtreated to determine the appropriate dose for the use of a predetermined demulsifier formula. Emulsion sample testing was also carried out on CGS oil samples (GS-belimbing oil and GS-11) plus the oil present in the pits. The six BS&W tests after using the new formula. GS-belimbing has a production rate of $\pm 22,000$ BFPD with a water cut value obtained from the separator test in the field and validated by the BS&W test in the laboratory of $\pm 92\%$, the value of oil production in GS Belimbing is ± 1760 BOPD. While the GS-11 has a production rate of $\pm 33,000$ BFPD with a water cut value of $\pm 91\%$, the value of oil production in GS 11 is ± 2970 BOPD. While the CGS has a fluid production rate of $\pm 58,000$ BFPD with a water cut of $\pm 90\%$, the value of oil production at the CGS is ± 5800 BOPD. Formula code H5 with a composition of 10% (F-13; water drop) plus 10% (1030; interface) and 80% (F-16; clear water) which was selected for GS-belimbing. The formula with code A1 which has a composition of 80% F-8 plus 10% 1030 and 10% F-16 was chosen for the GS-11. For the CGS, the S5 formula is 10% (F-16 clear water) plus 10% (1030; interface) and 80% (F-8; water drop). The results of the BS&W test after the new formula showed that there was no water in the oil in the centrifuge tube and it was stated that the BS&W value was close to 0%. There are 3 demulsifier products from the formulation, namely HAS-1 for GS-belimbing, HAS-2 for GS-11, and HAS-3 for CGS plus pit. The amount of HAS-3 demulsifier that needs to be injected into the CGS is 7.31 gallons per day (GPD). The number of HAS-1 demulsifier injected into GS Belimbing was 2.22 GPD, while the number of HAS-2 demulsifier injected into GS-11 was 3.74 GPD.

Keywords: Crude oil, Demulsifiers, Bottle test, Formulation, Dose, Overtreat

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INTRODUCTION

Water is typically produced in the oil field at the same time as the oil. There are two forms of water in oil: free water and water emulsion. Free water does not pose a significant threat to the production process,

but the existence of emulsified water poses a threat and necessitates careful treatment moving forward. Emulsions in pipes or reservoirs in the petroleum industry can lower output (Subiatmono et al., 2007). Mechanical, thermal, electrical, and chemical meth-

ods can be used to separate water from a crude oil emulsion (Grace, 1992). The use of a demulsifier in a chemical separation process is the most effective of the four approaches. By integrating several methods, such as chemical separation methods combined with thermal methods, can increase the efficiency of water separation in crude oil. Demulsifier and reverse demulsifier chemicals are classified as surface active agents (surfactants) which function to break and separate water and oil emulsions.

The function of the demulsifier is to overcome emulsion problems that occur in an oil field, the results of which are indispensable as a reference in overcoming further emulsion problems (Yodi, 2018). The method used to determine the performance of the demulsifier is the bottle test. With the bottle test, observations will be made about the demulsifier being tested from the fastest water drop value, namely the speed at which water and oil separate, water clarity or separate water clarity, and a good interface, which is a straight line that separates oil and water, then adjusted to the conditions at field and the optimal price will be obtained.

This demulsifier was created over the course of seven stages. Taking samples of crude oil and formation water in the field for later testing in a lab constitutes the first stage. Next, evaluate the crude oil samples using the BS&W (basic sediment and water) method at PT. Farca Risa Sejahtera. The ratio of crude oil to formation water utilized in the bottle test is then determined in the third stage. Choose a demulsifier that received high marks from the SP-starfruit, SP-11, and SPU samples together with pit oil as your fourth option. Fifth, based on the decision that was taken in the previous stage, formulate the demulsifier that has been chosen. To identify the ideal dose to employ in the new formula, the sixth performed an overtreat test. On the sample that has been injected with the new formula, the seventh step is to perform the BS&W test once more.

METHODOLOGY

An emulsion is defined as a system consisting of two immiscible liquid phases stabilized by an emulsifier (surfactant), where one liquid phase is dispersed in the other liquid (Dian Nadia, 2018). In the presence of an emulsifying agent, water and oil can form an emulsion. Water in the reservoir can be bound as an emulsion or in the form of free water. Free water can be separated only by using a physical process, namely by the influence of gravity. Water

that forms an emulsion with petroleum will be difficult to separate.

Definition of an emulsion is a colloidal dispersion of one liquid (disperse phase) in another (continuous phase) (Tjuwati Makmur, 2010). He divided Type of emulsion can be divided into three parts:

- Oil in water.
- Water in oil.
- Complex/multiple emulsions.

There are several types of emulsions classified based on how the oil and water phases are present in the dispersion system (Henriquez, 2009). The words “oil” and “water” are used in a more polar sense, coming from two immiscible phases. Figure 1 gives various types of emulsions, such as Water in oil (W-O) emulsion gives water droplets dispersed in the oil phase, or oil in water (O-W) emulsion if what happens is that oil droplets are dispersed in the water phase, while two or more emulsions are denoted using W1-O-W2 or O1-W-O2. W1 (each O1) and W2 (each O2) provide the outermost and innermost phases. A biemulsion is an emulsion containing two droplets of different internal phases, with different sizes or properties.

A demulsifier is a surface active substance that has the function of reducing the surface tension between liquids by wetting, dispersing, and replacing the emulsifier layer, which causes water and oil droplets to separate. The most commonly used demulsifier is a nonionic surfactant. Nonionic surfactants also reduce electrostatic interactions with salts or the influence of pH. The requirements to be said to be a demulsifier are as follows:

- Has a high concentration which is diffused between phases.
- The adsorption rate is high between phases and creates interfacial tension
- Molecular partitions exist in the water and oil phases
- Dissolved in the organic phase.

The crude oil contains a number of components, which in nature have interfacial properties. These components especially are asphaltenes, resins and naphthenic acids. These components may accumulate at the water-oil interface and inhibit the oil droplets to a separate phase. Among these components, asphaltenes are the major material involved in emulsion

stabilization (R. Desrina, 2012). In the separation process, the demulsifier, which will break the emulsion, will go through 3 stages, namely flocculation, coalescence, and solid wetting. Flocculation is the process when colloids come out of suspension in the form of flocs or flakes, either spontaneously or due to the addition of a clarifying agent to an oil emulsion.

This clarifying agent is a demulsifier. At this stage, the flocs from the internal phase will be combined, and if the formation of the oil-water emulsion layer is weak, then these flocs which are incorporated will merge with the continuous phase. When the droplets of the dispersed phase combine after being churned to create larger droplets and merge with the continuous phase, the process is known

as coalescence, and the emulsion melts or cracks. The final step is solid wetting, which collects solid phases in the oil after the previous stage. Typically, crude oil contains particles including clay, paraffin, drilling mud, and iron sulfide. One of the most used techniques for the demulsification test is the vial test method.

This approach was chosen based on a number of field variables, including the demulsifier's chemical characteristics, its dosage, the testing period's duration, the temperature, and the amount of stirring. The theoretical surface area (grind out) of petroleum, formation water, water reduction, oil dryness, and interface quality are all measured using the bottle test method.

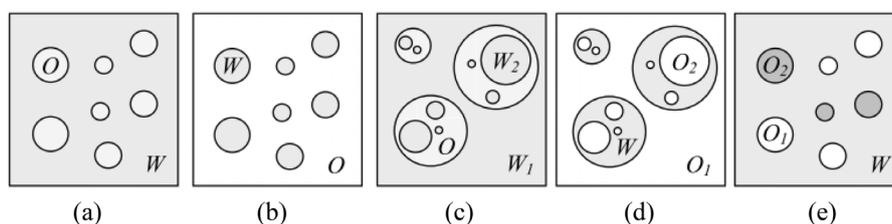


Figure 1
Various emulsions (Henríquez, 2009, p.12)

Data from bottle tests that measure the emulsion's strength on the basis of laboratory analysis can be attributed to the characteristics of crude oil. Another factor affecting the stability of an emulsion is how much water is still present in the oil. Several different parameters can be used to compute this remaining water. The emulsion state is still present for a portion of the leftover water in the oil phase.

Research Data

The value of oil output in GS Belimbing is around 1760 BOPD, with a production rate of 22,000 BFPD and a water cut value of 92% acquired from the separator test in the field and corroborated by the BS&W test in the laboratory. The value of oil output in the GS-11 is 2970 BOPD, despite having a production rate of 33,000 BFPD and a water cut value of 91%. The CGS produces fluids at a rate of 58,000 BFPD with a 90% water cut, however its oil production is worth 5800 BOPD. In order to prevent the oil in the tank from freezing and to ensure that the demulsifier functions well because it is more effective at high temperatures, the manufacturing facility's temperature is kept at 60 C. The number of doses needed to choose the demulsifier and formulation before figuring out the right formula dose is 50

ppm, or 0.005 ml per 100 ml sample. This value is decided based on the data already available or the demulsifier's prior use at CGS. If any chemical is injected into the wellhead, such as a scale inhibitor, it must be closed first to ensure that the fluid received is clean before sampling is done on a flowline close

Table 1
Types of demulsifier (Halwin Ariandi Siregar, 2022)

No	Types of Demulsifier	
	Name	Function
1	F-7	Water Drop
2	F-8	Water Drop
3	F-13	Water Drop
4	F-14	Water Drop
5	F-15	Water Drop
6	18406	Water Drop
7	F-9	Interface
8	F-10	Interface
9	F-12	Interface
10	F-25	Interface
11	1030	Interface
12	F-16	Clear Water
13	4114	Clear Water
14	9001	Clear Water

to the wellhead.

The fluid is then placed in a unique container after that. According to laboratory study, oil has an emulsion concentration of 7%, and 406 BEPD worth of emulsion is estimated to be present in CGS oil. While the rate of oil production in GS 11 is 2970 BOPD, the total fluid production rate is 33,000 BFPD with a water cut of 91%. The amount of emulsion in the research findings' oil was calculated to be 8%, or 238 BEPD, based on the amount of emulsion found in GS-11 oil. At GS Belimbing, the rate of total fluid production is 22,000 BFPD with a 92% water cut, and the rate of oil production is 1760 BOPD. According to laboratory study, oil has an emulsion percentage of 9%, and 158 BEPD of emulsion is estimated to be present in CGS oil. These are the many types of demulsifiers. Three demulsifier assessment criteria in breaking emulsions, namely the speed of separation (water drop), the clarity of the separated water (clear water), and the resulting interfacial tension, have been developed as a score system to aid in the selection process (interface). Demulsifiers are the most effective at resolving emulsion issues in the SRG field because they can at least achieve a total rating of 80.01 on the rating scale.

Table 2

Scale of rating for demulsifier based on interface criteria, water drop, and clear water, emulsion breaking capability (Source PT. Farca Risa Sejahtera)

Score	Rating Scale based on Ability			Total
	Interface	Water Drop	Clear Water	
Very good	33,3	33,3	33,3	99,9
Good	26,67	26,67	26,67	80,01
Moderate	20	20	20	60
Bad	13,3	13,3	13,3	39,9
Very bad	6,67	6,67	6,67	20,01
Does not work	0	0	0	0

RESULTS AND DISCUSSION

Basic Sediment and Water (BS&W) Test

In this experiment, GS-belimbing oil and GS-11 oil samples from two different 10 ml centrifuge tubes were combined to create two separate samples. The centrifuge tube was then heated for 10 minutes in a water bath at a temperature of 60°C.

The sample was then circulated in a centrifuge for 5 minutes, and observations were made on it. The results showed that the GS-belimbing oil sample had

9% emulsion, the GS-11 oil sample had 8%, and there was emulsion in the centrifuge tube. The test was then repeated using the same approach, but this time a demulsifier was injected before the centrifuge was used to rotate the sample. The demulsifier of choice is PT Farca Risa Sejahtera's F-46, which is the type being employed. 50 ppm F-46 was injected, spun for 5 minutes, and watched. Better separation and the absence of emulsion in the sample are the results.

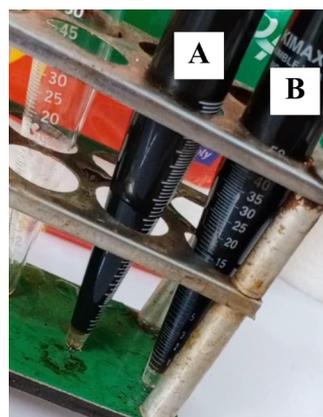


Figure 2
GS-belimbing (A) and GS-11 (B) Oil Sample Results from BS&W (Halwin Ariandi Siregar, 2022)

Determination of the Volume Ratio between Crude Oil and Water

Determining the ratio employed for this investigation is crucial before testing the demulsifier. The sample of oil used for this test comes from GS 11, which contributes the most oil to the main collection station (CGS). Four bottles of sani glass, each holding the following ratios of oil to water: 60 (oil): 40 (formation water), 65 (oil): 35 (formation water), 70 (oil): 30 (formation water), and 75 (oil): 25 were used in the test (formation water). The emulsion sample from these four samples that met the fewest requirements but wasn't saturated was chosen.



Figure 3
Comparison of the oil and water ratio of the formation. (Halwin Ariandi Siregar, 2022)

60:40 is the ratio According to experiments and observations, a ratio of 60:40 was selected with 60% crude oil and 40% formation water to be used for bottle testing demulsifier on GS and CGS. The separation process is not too fast and not too long for 15 minutes, and at a ratio of 60:40 the emulsion that occurs between oil and formation water can be seen.

Table 3
Capability value of formation oil and water comparison ratio (Halwin Ariandi Siregar, 2022)

Minyak:air	ml of water for 15 minutes	Ability Value Based on			Total
		Inter face	Water Drop	Clear Water	
60: 40	36	6.67	6.67	13.3	26.64
65: 35	34	6.67	6.67	6.67	20.01
70: 30	30	6.67	6.67	0	13.34
75: 25	0	0	0	0	0

GS-Belimbing Bottle test

This choice seeks to obtain a demulsifier that can function by fulfilling the GS assessment requirements. The parameters for belimbing crude oil are water clarity, interfacial tension, and speed of separation (water drop). The following 14 types of demulsifiers were used in the experiments: F-7, F-8, F-9, F-10, F-12, F-13, F-15, F-16, F-25, 1030, 4114, and 18406. The finest six demulsifiers will be chosen from among all demulsifiers based on the observations made. The demulsifier's capacity for the quickest water drop, clear water, and nice interface determines this best criterion. Six demulsifiers with the codes F-8, F-13, F-15, F-25, 1030, and F-16 were chosen in accordance with the observations and the findings.



Figure 4
Testing on 14 types of demulsifier for gs-belimbing (Halwin Ariandi Siregar, 2022).

The F-8 can quickly separate water from oil due to its clear clear water capabilities, straight interface, and water drop capacity. Excellent separation speed, acceptable interface lines, and well-separated formation water were all attained by F-13. The F-15 features a great interface, great clarity in the water, and great separation speed. Fast water drop from the F-25, decent water results during formation, and great interface lines. Demulsifier 1030 has good water clarity, nice interface, and outstanding separation ability. F-16 has exceptional water separation properties, and the resulting interface is superb.

Demulsifier Formulation for GS-belimbing

Following the demulsifier type selection experiment, a formulation employing the GS-belimbing crude oil technique was carried out. The crude oil was heated in a water bath and poured into 10 bottles with a ratio of the formation water and crude oil amounts that had been established in the earlier test. Then, depending on the dose of prior use, it is injected with a different demulsifier formula in each bottle up to 50 ppm or 0.005 ml per 100 ml sample. The formula for GS-belimbing is as follows:

- H1: 30% (F-15; water drop) + 60% (1030; interface) +10% (F-16; clear water)
- H2: 30% (F-13; water drop) + 50 % (1030; interface) +20% (F-16; clear water)
- H3: 10% (F-8; water drop) + 25% (F-15; water drop) +40% (1030; interface) +25% (F-16; clear water)
- H4: 25% (F-15; water drop) +20% (F-25; interface) +20% (1030; interface) +35% (F-16; clear water)
- H5: 10% (F-13; water drop) +10% (1030; interface) +80% (F-16; clear water)



Figure 5
Test the demulsifier formulas for GS-belimbing (Halwin Ariandi Siregar, 2022)

The formula with code H5 has the best water drop, interface, and clear water, and it is in compliance with GS-belimbing oil, according to testing, observation, and evaluation of all available samples. Additionally, this recipe serves as a suggestion that will be employed in GS-belimbing to create the demulsifier product HAS-1.

Table 4
Demulsifier formula ability assessment
At the GS-belimbing (Halwin Ariandi Siregar, 2022)

Formula	Ability Value Based:			Total
	Water Drop	Interface	Clear Water	
H1	33.3	26.67	26.67	86.64
H2	26.67	33.3	33.3	93.27
H3	33.3	33.3	26.67	93.27
H4	26.67	33.3	26.67	86.64
H5	33.3	33.3	33.3	99.9

GS-11 Bottle test

There were 14 different types of demulsifiers employed in the GS-11 experiments, and the top 6 will be chosen. It was decided to use a demulsifier with the code F-8, F-14, F-10, F-25, 1030, F-16 based on the observations and the findings. The F-8 demulsifier code produced separation results quite quickly, had outstanding water clarity, and had a nice interface as well. F-10 has very good separation, nice clear water, and good interface line, while F-14 has a quick water drop, clear formation water, and outstanding interface. The F-25 features a superb interface, good clear water, and the ability to dump water quickly. The formation water produced at code 1030 is fairly good, the separation occurs quickly, and the interface is satisfactory. F-16 having the best capacity to clear water, good water drop, and good interface.



Figure 6
Testing of 14 types of demulsifier for GS-11
(Halwin Ariandi Siregar, 2022)

Demulsifier Formulation for GS-11

Following the demulsifier selection experiment, a formulation based on the six chosen demulsifiers was carried out. This is how the GS-11 demulsifier formula is put together:

- A1: 80% (F-8; water drop) +10% (1030; interface) +10% (F-16; clear water)
- A2: 80% (F-14; water drop) +10% (1030; interface) + 10% (F-16; clear water)
- A3: 10% (F-8; water drop) +30% (F-10; interface) +60% (F-16; clear water)
- A4: 60% (F-8; water drop) + 30% (1030; interface) +10% (F-16; clear water)
- A5: 50% (F-14; water drop) + 20 % (F-25; interface) +30% (F-16; clear water)

Using the bottle test method, the five formulae were injected into the samples. The five samples were then observed, and this was followed by formula development for GS-11. Based on the outcomes of examinations, observations, and evaluations performed on the GS-11 samples. Formula with code A1, which is consistent with the characteristics of GS-11 oil and has the best water drop, interface, and clear water. Additionally, this formula serves as a suggestion that will be incorporated into GS-11 to create the demulsifier product HAS-2.



Figure 7
Test the demulsifier formulas for GS-11 (Halwin Ariandi Siregar, 2022)

Table 5
Demulsifier formula ability assessment
at the GS-11 (Halwin Ariandi Siregar, 2022)

Formula	Ability Value Based on			Total
	Water Drop	Inter face	Clear Water	
A1	33.3	33.3	33.3	99.9
A2	33.3	33.3	26.67	93.27
A3	33.3	33.3	26.67	93.27
A4	33.3	26.67	26.67	86.64
A5	33.3	26.67	26.67	86.64

CGS Overtreat Test

The overtreat test was conducted to establish the ideal dosage and prevent demulsifier overuse. Based on the use of the demulsifier dose before the formulation was carried out, the 50ppm dose in the prior experiment was chosen. Oil from GS-11 and the oil that has been separated from the GS-belimbing will eventually be directed to the CGS due to field conditions. Following a meeting in a flowline, the oil and demulsifier from the two GSs will flow collectively to the washtank at the CGS.

Table 6
Demulsifier Dosage Data for CGS. Overtreat Test (Halwin Ariandi Siregar, 2022)

Sample	Injected Formula	
	GS-belimbing (H5)	GS-11 (A1)
A	20 ppm	30 ppm
B	30 ppm	20 ppm
C	30 ppm	30 ppm
D	40 ppm	40 ppm
E	50 ppm	50 ppm

When different demulsifiers are used with oil, an overdose may occur where the demulsifier is no longer effective. Both samples of GS-11 oil and GS-belimbing oil were combined for the test. The ratio of the oil mixture from GS-belimbing and GS-11 is changed based on the specific BOPD in order to approximate the field circumstances. According to the statistics provided, the proportion of GS-11 to GS-belimbing oil at CGS is 40:60. Before testing, combine GS-belimbing oil and GS-11 in a 1000 ml measuring cup according to the ratio and stir to combine. The oil mixture will then be applied to the sample in the sunlit glass later. Five samples will then be mixed after being injected with the GS-belimbing and GS-11 demulsifier formulations. The oil mixture will then be applied to the samples in a bright glass. The GS-belimbing and GS-11 formulas were injected into 5 samples in accordance with the doses listed in table 6.

The heated sample was added to four sani glass bottles, just like in the earlier testing, along with formation water in a 60:40 ratio. Next, the demulsifier formula discovered via the tests was injected into each bottle. prior testing using various doses in every container. If one of the examined samples is overtreated, the results of this test will establish the dosage limit. After the demulsifier was injected, the sample was shaken for 2 minutes while the water

and oil were separated, and after 1 minute, the water level of the separation was noted. The sample was then placed in a water bath for 2 minutes, after which it was removed to observe and record the water and oil separation. The separation process was observed every 2 minutes for 15 minutes.



Figure 8
CGS Overtreat test results (Halwin Ariandi Siregar, 2022)

Additionally, as stated in the table, a review of the water drop, interface, and clear water capabilities is conducted.

Table 7
Evaluating the CGS Overtreat Test Sample's Demulsifier Formula Ability (GS-belimbing + GS-11) (Halwin Ariandi Siregar, 2022)

Formula	Ability Value Based on			Total
	Water Drop	Interface	Clear Water	
A	33.3	26.67	26.67	86.64
B	33.3	26.67	26.67	86.64
C	33.3	33.3	33.3	99.9
D	33.3	26.67	26.67	86.64
E	33.3	26.67	26.67	86.64

Following a number of tests and evaluations, it was determined from the results that no sample had been overtreated. However, sample C, which has advantages over the other 4 samples in its ability to function in clear water and has a better interface, shown the best change. Therefore, 60 ppm with details of 30 ppm formula H5 (GS-belimbing) + 30 ppm formula A1 is the recommended dose for CGS without extra oil from the pit (GS-11).

CGS Plus Pit Oil Formulation

Additionally, the washtank will get the oil from the CGS pit. The oil will then mix with oil from GS-11 and GS-belimbing, thus a new demulsifier must be

created and injected before the oil reaches the wash tank. Because the oil in the CGS comes from GS in the SRG field and the BOPD GS 11 and GS Belimbing values are able to represent 50% of production in the SRG field and are assumed to be the majority, this formulation uses the best type of demulsifier that has been tested in the GS-belimbing and GS-11 bottle tests. Comes from the GS Belimbing and GS Belimbing regions. The demulsifier selection tests in GS Belimbing and GS 11 produce very identical results, and the demulsifier that passes is coded F-8, F-10, F-15, F-16, F-25, 1030. Five formulations using the following formula were created using the six different types of demulsifier:

- S1: 90% (F-16; *clear water*) + 10% (F-8; *water drop*)
- S2: 70% (F-16 *clear water*) +20% (F-10; *interface*) +10% (F-15; *water drop*)
- S3: 80% (F-16 *clear water*) + 15% (1030; *interface*) +5% (F-15; *water drop*)
- S4: 70% (F-16 *clear water*) +30% (1030; *interface*)
- S5: 10% (F-16 *clear water*) +10% (1030; *interface*) +80% (F-8; *water drop*)



Figure 9
Demulsifier formula test on CGS
(Halwin Ariandi Siregar, 2022)

Based on the ratio of the production percentages at CGS, the oil sample used in this test is a combination of GS Belimbing and GS 11 oils in a ratio of 40 (GS-Belimbing): 60 (GS-11). After being filled with liquid in the proportion of 60 (oil): 40 (formation water), the sample bottles were shaken for 2 minutes, then immediately submerged in a water bath. After 5 minutes, the sample was removed, added with oil from the pit in the amount of 20 ml, and then immediately injected with the new demulsifier formula. Additionally, a determination of the capability of the water drop, interface, and clear water is made.

Table 8
Demulsifier formula ability assessment in CGS formulation samples plus pit oil (Halwin Arindi Siregar, 2022)

Formula	Ability Value Based on			Total
	Water Drop	Interface	Clear Water	
S1	26.67	20	33.3	79.97
S2	33.3	20	26.67	79.97
S3	33.3	20	26.67	79.97
S4	33.3	20	33.3	86.6
S5	33.3	20	33.3	86.6



Figure 10
The results of the addition of F-46 for 2 hours
(Halwin Ariandi Siregar, 2022)

The temporary demulsifier formulations for CGS plus oil from the pit are S4 and S5, according to the experiments that have been conducted in accordance with the protocols and observations. changes in the formations' water volume S4 formula balances S5 formula in terms of water drop capabilities. The results for all samples' interface lines are not particularly good because the oil in the pits is made up of leftover oil from various cellar tanks, wells in the "SRG" field that enable rainwater or trash to enter, and any oil spills that may have occurred. In the event that the flowline pipe is repaired, it may also carry trash or other materials that may alter the properties of the oil in the pit.

According to observations, there are still lumps that resemble emulsions in each sample bottle. In an effort to solve this issue, the researchers applied the best demulsifier available in the PT Farca Risa Sejahtera laboratory F-46 to the chosen sample.

The results of this test show that the oil from the pit at the CGS is extremely damaging because it contains rainwater or garbage, and from an oil spill in the event of repair of the flowline pipe it can also

Diagram Ketinggian Air (ml) Uji Dosis SPU (SP Belimbing + SP 11) Ditambah Pit

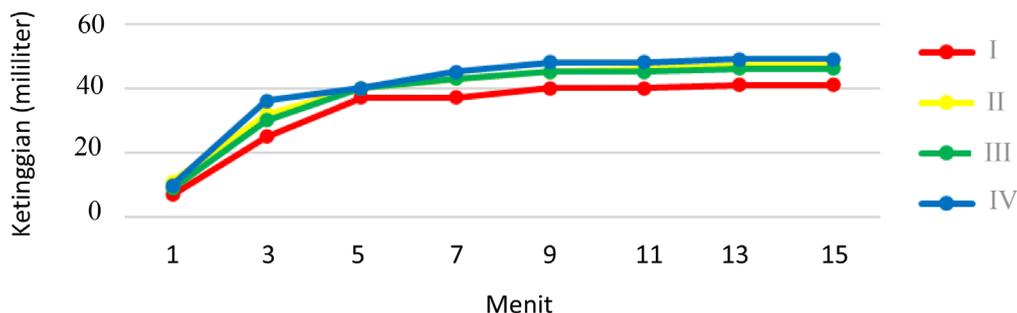


Figure 11 Graph of change in water level against time in the CGS overtreat test + pit oil

Table 9 Demulsifier dosage data for CGS overtreat test (Halwin Ariandi Siregar, 2022)

Sample	Injected Formula		Formula CGS+Pit	
	GS-belimbing (H5)	GS-11 (A1)	S4	S5
I	50 ppm	50 ppm	100 ppm	-
II	50 ppm	50 ppm	-	100 ppm
III	30 ppm	30 ppm	30 ppm	-
IV	30 ppm	30 ppm	-	30 ppm

carry garbage or dirt. The lumps that resemble emulsion on each sample bottle cannot disappear. It may change how the oil in the pit behaves.

Overtreat Test of CGS Plus Pit Oil

Due to the new oil formula in CGS (GS-belimbing and GS-11 + pit oil), an overtreat test for the chosen formula, specifically S4 and S5, is required. GS-Blimbing oil samples and GS-11 were combined in a ratio of 40 (GS-belimbing): 60 (GS-11), and the method was then heated in a water bath that had been preheated to 60 C. Similar to the procedure used in the previous test, the sample was heated and placed in 4 bottles. It was then mixed with formation water in a 60:40 ratio, shaken for 2 minutes, and then placed immediately in a water bath. After 5 minutes, the sample was removed and mixed with up to 20 ml of pit oil. It was then immediately injected with the CGS demulsifier formula plus pit with codes S4 and S5.

After being injected with demulsifiers S4 and S5, the sample was shaken for 2 minutes while be-

ing watched to see how the water and oil separated. After 1 minute, the water level of the separation was recorded, and after 2 minutes, the sample was

Table 10 Demulsifier formula ability assessment in the CGS overtreat test plus pit oil (Halwin Ariandi Siregar, 2022)

Sample	Ability Value Based on			Total
	Water Drop	Interface	Clear Water	
I	26.67	20	26.67	73.34
II	33.3	20	26.67	79.97
III	26.67	20	33.3	79.97
IV	33.3	20	33.3	86.6

removed to watch and record the water and oil separation. For 15 minutes, the separation process was recorded every 2 minutes. The GS-C formula sample overtreatment test results (CGS plus oil from the pit).

The capacities of the water drop, interface, and clear water are then evaluated. Because the previous test was the highest dose but there was no overtreatment, formula H5 and A1 were injected 50 ppm in samples I and II. A subsequent test was then conducted with a new formula added to test the overtreat. Testing and observation revealed that there was no overtreatment, although sample IV with the formulas H5 up to 30 ppm and A1 30 ppm with formula S5 30 ppm shown good changes and received high ratings for the fastest water drop, clear water, and good interface. As a result, the HAS-3 demulsifier product is recommended using the S5 formula and is aimed at CGS in the SRG field.

BS&W Test on Final Formula Sample

Three new demulsifier products HAS-1 from formula H5 for the Belimbing collection station, HAS-2 from formula A1 for the GS 11 collecting



Figure 12
Final BS&W test results on sample IV (Halwin Ariandi Siregar, 2022)

station, and HAS-3 from formula S5 for Pit oil from the main collection station were produced as a consequence of the study that was conducted. This experiment was conducted on sample IV, the results of testing the dosage of the CGS formula plus Pit oil on the CGS by taking only the top part, namely oil, because this sample is the assumption of the final product where all demulsifier formula products that have been made, namely HAS-1, HAS-2 and HAS-3, which have been injected into the emulsion sample with a predetermined dose of 30 ppm for each formula, are present. Figure 11 explains the outcomes of the centrifuge-based emulsion test on Sample IV. According to the BS&W test findings for sample IV, there was no water in the oil in the centrifuge tube, and the test's BS&W value was close to 0%. This test demonstrates the new formula's excellent performance and how well it addresses emulsion issues in the SRG field.

Application on SRG Field

Three demulsifier products are advised following a series of experiments and observations, as well as an evaluation of all samples that have been tested: the HAS-1 product with the H5 formula, which is composed of 10% (F-13; water drop) plus 10% (1030); interface) and 80% (F-16); clear water; and the HAS-2 product with the A1 formula, which is

composed of 80% (F-8; water drop) mixed with 10% (1030); interface) and 10% (F-8; water drop) (F-16 ; clear water). 10% (F-16 clear water) plus 10% (1030; interface) and 80% of the HAS-3 product with S5 formula for CGS (F-8; water drop). For each 100 ml of fluid, the three formulas' dose on the laboratory scale is 30 ppm.

The amount of oil produced may reach 5800 BOPD based on data from the main collecting station's predicted oil output. The amount of demulsifier that needs to be injected is 7.31 gallons per day in order to break the oil emulsion using the PPM calculation as in formula 3.2. (GPD). And for GS-belimbing and GS-11, based on BOPD data, the ratio of GS Belimbing and GS 11 crude oil sent to CGS is 60 (GS 11): 40 (GS Belimbing). Accordingly, the estimated BOPD at GS Belimbing is 1760 BOPD while the BOPD in GS 11 is 2970 BOPD, and the number of HAS-1 demulsifiers injected into GS Belimbing is 2.22 At GS Belimbing, GS 11, and CGS, the demulsifier formula was injected shortly before the fluid entered the wash tank, following the separator.

CONCLUSION

If a chemical, such as a scale inhibitor, is injected into the wellhead while sampling is being done on a flowline close to the wellhead, the wellhead must first be closed to ensure that the fluid collected is clean (not mixed with chemicals).

GS-belimbing has a production rate of 22,000 BFPD and an oil production value of 1760 BOPD. The water cut value was determined by the separator test in the field and validated by the BS&W test in the laboratory. The value of oil output in the GS-11 is 2970 BOPD, despite having a production rate of 33,000 BFPD and a water cut value of 91%. The CGS's fluid production rate is 58,000 BFPD with a 90% water cut, but its oil production is worth 5,800 BOPD.

Formula code H5, which is used for GS-belimbing, has 10% (F-13; water drop), 10% (1030; interface), and 80% (F-16; clear water). For the GS-11, the formula with code A1, which consists of 80% F-8 + 10% 1030 and 10% F-16, was chosen. The S5 formula for the CGS is composed of 80%,

GLOSSARY OF TERMS

Symbol	Definition	Unit
	Gathering Station	GS
	Central Gathering Station	CGS
	Barrel fluids per day	BFPD
	Barrel oil per day	BOPD
	Barrel emulsion per day	BEPD
	Gallons per day	GPD
%	Basic Sediment and Water	BS&W
Parts per million (ppm)	Injected rate	Dose

10% (1030; interface), and 10% (F-16 pure water) (F-8; water drop).

The BS&W test results revealed that the oil in the centrifuge tube was completely devoid of water and that the BS&W value was very near to zero.

The formulation contains three demulsifier products: HAS-1 for GS-belimbing, HAS-2 for GS-11, and HAS-3 for CGS plus pit. 7.31 gallons of HAS-3 demulsifier need to be injected into the CGS every day (GPD). HAS-1 demulsifier was injected into GS Belimbing at a rate of 2.22 GPD, whereas HAS-2 demulsifier was injected into GS-11 at a rate of 3.74 GPD.

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