

INTERNSHIP REPORT
THE DEVELOPMENT OF BIODIESEL IN PT. SINARMAS BIODIESEL ENERGY


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Biotechnology

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2022

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ABSTRACT

Global warming is being caused by humanity's insatiable use of fossil fuels as energy, which artificially amplifies the natural greenhouse effect and global warming. This sparked a desire in the scientific community to look into more environmentally acceptable alternative fuels. One of the many options being taken into consideration right now is biodiesel. This research is aimed to develop a biodiesel using a transesterification method in accordance with the specification which validates the quality of the biodiesel by adding the right amount of chemicals. This development used RBDPO and methanol with sodium methoxide as their catalyst to create the biodiesel. After the results are obtained. This trial already shows a good quality of biodiesel which already fits the parameter and gives a high yield percentage. In this internship, we managed to produce biodiesel from Refined Bleached Deodorized Palm Oil (RBDPO). By applying different dosing of methanol and sodium methoxide continuously, the results of the end product demonstrated a good quality of biodiesel which fulfilled the parameter specification criteria and resulted in high purity levels.

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Thanking you, Ryan Matthew

A handwritten signature in black ink, appearing to read 'Ryan Matthew', with a long, sweeping flourish extending to the right.

22-12-2022

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LIST OF ABBREVIATIONS

ASTM	= American Society for Testing Materials
AV	= Acid Value
BPO	= Bleached Palm Oil
CIE	= International Commission of Illumination
CPO	= Crude Palm Oil
CV	= Curriculum Vitae
DIRJEN EBTKE	= Direktorat Jenderal Energi Baru Terbarukan dan Konservasi Energi Kementrian ESDM
EN	= European Standards
FAME	= Fatty Acid Methyl Ester
FG	= Free glyceride
ISO	= Sistem Informasi Standar Nasional Indonesia
IV	= Iodine Value
MG	= Monoglyceride
MSTFA	= N-methyl-N-trimethyl silytrifluora acetamide
OSI	= Oxidation Stability Index
PD	= Product Development
QA	= Quality Assurance
QC	= Quality Control
QFS	= Quality Food Safety
RBDPO	= Refined Bleached Deodorized Palm Oil
RGB	= Red Green Blue
SBE	= Sinarmas Biodiesel Energy
TG	= Total glyceride
USC	= University of Southern California

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CHAPTER 1. INTRODUCTION

1.1. A brief history

Sinarmas Biodiesel Energy (SBE) is one of the world's leading agribusiness companies with business activity from seed stage to final product in seed-to-shelf. This company has a cooperation with PT. SMART Tbk to supply the palm oil for the creation of biodiesel in PT. SBE for current and future needs, with sustainability at the core of its activities. Large-scale, vertically integrated operations support the team in ensuring efficiency, quality, and sustainability throughout the supply chain. The activities are innovation-driven and guided by sustainability, to ensure SBE remains a leader in the world's sustainable biodiesel production and is always a partner of choice.

Thanks to its focus and investment in R&D (research and development), the company is at the forefront of agronomy and technology. In the downstream business, the processing facilities are capable of producing high-quality products to supply international markets for agronomy, foodstuffs, oleochemicals, and bio-energy. The most sought-after products include biodiesel and the byproduct of the biodiesel manufacturing industry is glycerin. The biodiesel has been sold to many industries such as Pertamina and Shell. Meanwhile, there are three types of glycerin that are sold to other industries: Crude glycerin, Yellow glycerin, and Refined glycerin. To get good glycerin to be sold, the crude glycerin needs to be dry by doing combustion, which then is in distillation to get the yellow glycerin. Thus, with the bleaching of the yellow glycerin, it would become a refined glycerin.

1.2. Vision and mission (SBE)

The vision of Sinarmas Biodiesel Energy is to become the best integrated global agribusiness and consumer products company, as well as to be the partner of choice. The mission is to efficiently provide high-quality and sustainable agribusiness and consumer products, solutions, and services, to create added value for stakeholders.

1.3. The main activity

There are two types of workers in Quality Control (QC) that are regular-hour workers and quality control officers with shift-hour work. The quality control officer checks all the parameters needed to be checked for every chemical needed such as methanol, bleaching earth, sodium methoxide, et cetera. The parameters depend on the chemical itself. For example, the Fatty Acid Methyl Esters (FAME) as a main ingredient have several parameters such as soap content, acid value, ester content, methanol content, moisture content, flash point, and more. Therefore, after the sample has been obtained from the plant or seller, QC needs to check the parameter to ensure the quality of the chemical is top-notch and ready to be used for creating

biodiesel or glycerin. The regular-hour workers, meanwhile, have their own individual assignments. There is the QC biodiesel analyst who inputs the sample of Biodiesel or incoming chemical validation, and admin who contacts the buyer/seller, Quality assurance section head who has the job to validate the COA from another incoming chemical, etc.

1.4. Organizational structure

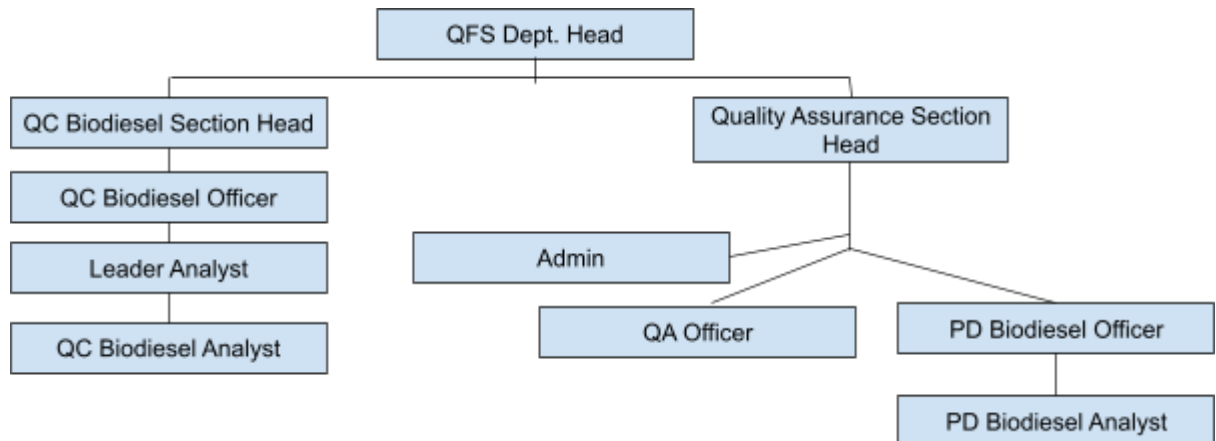


Figure 1. Organizational Structure of QC Laboratory.

Based on **Fig 1.**, the Quality Food Safety (QFS) department is led by the QFS department head. Meanwhile, there are two branches that oversee the head of the department. The first one is the QC biodiesel section head who is accompanied by the QC biodiesel officer, lead analyst, and QC biodiesel analyst. On the other hand, the Quality Assurance (QA) section head is accompanied by the admin, QA officer, Product Development (PD) biodiesel officer, and PD biodiesel analyst.

1.5. The student's unit or department

The student is assigned as a QC intern for SBE. This department is responsible to understand the parameters that are needed to create high-quality biodiesel and glycerin. There are several working routines that have to be checked daily such as moisture, acid value, iodine value, density and kinematic viscosity, flash point, oxidation stabilization index, pH meter, color, cleaning the glasses after used, resupplying the WYPALL, and many more.

1.6. Interesting fact of the company

Sinarmas Biodiesel Energy is already part of Direktorat Jenderal Energi Baru Terbarukan dan Konservasi Energi Kementerian ESDM (Dirjen EBTKE) or Sistem Informasi Standar Nasional Indonesia (ISO) 7182:2015 which concludes that this company cares for and creates a quality excellent product. There are labels of chemicals created to ease the understanding of what chemical is checked in the

laboratory. The components checked in the laboratory are not only the final product. Several byproducts or products which are part of the process are also checked. There are also additional parameters that are used for an imported product such as EN14214.

Dirjen EBTKE or ISO 7182:2015 has been started since January 2016. It is controlled by Peraturan Menteri Negeri dan Sumber Daya Mineral No. 12 Tahun 2015 about the third rule of Peraturan Menteri ESDM No. 32 Tahun 2008 about Provision, Utilization and Trading of Biofuels as Other Fuels. By using this program several functions are increasing the added value of the economy downstream of the palm oil industry, creating job opportunities, reducing greenhouse gas emissions and improving environmental quality, and improving national energy security through energy diversification by giving local energy potential priority.

CHAPTER 2. INTERNSHIP ACTIVITIES

2.1. Working conditions

Time: Monday - Friday

08.00 - 12.00 Working Hour (Checking samples with several instruments)

12.00 - 13.00 Lunch Break

13.00 - 17.00 Working Hour (Checking samples with several instruments)

2.2. Internship tasks and experience gained

The intern is assigned several tasks in the laboratory. Checking the samples coming from the product and by-products with several instruments is done. One of them is a moisture test in which a sample is injected into the vessel of the KF coulometric apparatus, which then the iodine will be generated at the anode (Sugiarto et al., 2021). When all the water has been titrated, an electrometric end point detector detects excess iodine, and the titration is stopped. According to Faraday's Law, 1 mol of iodine reacts with 1 mol of water based on the stoichiometry of the reaction, and as a result, the amount of water is proportionate to the total integrated current.

Another test is the Density and Kinematic Viscosity test. It works by placing the test object in the measurement cells at a precisely controlled and predetermined temperature (Lapuerta & Canoira, 2016). The measuring cells are made up of a U-tube that oscillates and a pair of spinning concentric cylinders. The equilibrium rotational speed of the test specimen's inner cylinder under the effect of the shear stress and an eddy current brake, along with adjustment information, is used to calculate the dynamic viscosity (Spetuch et al., 2015). The U-oscillation tube's frequency and adjustment information work together to determine the density. By dividing the dynamic viscosity by density, the kinematic viscosity is determined.

Titration cover many parameters such as acid value (1 g of sample requires mg of potassium hydroxide to neutralize the free acids) (Zhang et al., 2015), purity test (mass of pure compound in the impure sample) (Lau(deceased), 2001), soap content (Percentage of saponification happened in the biodiesel creation) (Chanakaewsomboon et al., 2020), salt content (limited to five parts per million of the amount of sodium in biodiesel) (Barker et al., 2013), iodine value (determining amount of iodine (grams) could be added into 100 g of biodiesel), and fatty acid content (Pinzi et al., 2013). The Oxidation Stability Index (OSI) is used to find the stability of biodiesel and to know how long the biodiesel is working with good performance and quality (Kumar & Sharma, 2015). A steady 110°C airflow is used to expose the sample. The airflow transports highly volatile, secondary oxidation products (mostly formic acid and acetic acid) into the measuring vessel, where they are absorbed in the measuring solution (distilled water). Here, the conductivity is

recorded continually. Thus, an increase in conductivity can be used to identify the organic acids. The induction time of the induction period, which is a beneficial property for oxidation stability, is the length of time up until these secondary reaction products appear.

Gas chromatography is also used for finding the ester content, free glycerin, monoglyceride, total glycerin, and methanol content. The analyst will prepare the sample by silylating it with N-methyl-N-trimethyl silyltri-fluoroacetamide (MSTFA). With the methods prepared, the glycerin content is measured by comparing the monoolein, diolein, and triolein standards respectively (Tonini et al., 2012). Cloud point with a cold filter plugging point is checked daily for cloud point, the sample is cooled under the specified temperature to check at which temperature the sample will freeze (Anup Pradhan & Dev Sagar Shrestha, 2007). Whereas the cold filter plugging point, the sample is drawn into a pipetted into a controlled vacuum through a standardized wire mesh filter. The test will be stopped after the wax crystals that build up are sufficient to stop or slow down the flow of sample movement and fail to return completely to the available jar in under 60 seconds (Lapuerta et al., 2018).

Flashpoint is also measured for biodiesel by filling the brass test cup with a cover of specified dimensions which is heated and the specimen stirred at specified rates. With the ignition source directed into the sample, when a flash is detected, flashpoints are determined (Ha et al, 2006). The color test is also measured for Glycerin and biodiesel, this is measured automatically using the coulometer (Nedambale et al, 2014). The pH value is also prepared since the final product should be around neutral pH. A filter test is also used to check if a black spot is present in the final product.

There is a new experience that allows the intern to understand how to create biodiesel, how to maintain the quality of biodiesel, how to create a positive environment in the working laboratory, how to have a great connection with the staff and leaders, and have a great laboratory experience.

2.3. Compare between theory and practice

At the university, the basic theory has been given for the laboratory skills that are applied during the internship, such as properly using gas chromatography, titration, pH meter, spectrophotometer, and many more. The methods are provided theoretically beforehand, but there are differences between theory and practice. In practice, only the method of how to use the instrument is required to be understood without understanding why the component is used, why the button needs to be pressed, and why calibration is used. Because everything is already prepared by the company for doing the QC, the analyst only needs to perform the analysis based on

the instructions and guidance given. But, to become a better analyst, both theory and practice have to be understood.

There are also courses at the university about safety, security, and quality control in the bioindustry. This explained the theoretical principle of biosafety and biosecurity, hazards that could happen in the laboratory, as well as risk assessment. But in real-life practice, some of the analysts are careless about the standard of practice in the laboratory. Risk assessment is already prepared beforehand and the analyst is required to understand and perform as instructed. With the prior preparation of the theoretical knowledge and guideline, the intern is expected to understand what is needed to be done should there be an occurrence/occurrences of risk.

2.4. Difficulties encountered and how overcome them

There are difficulties during the beginning of the internship program as the intern still has minimal knowledge about the company and the scope of work. The intern is still in the process of adapting when working with the samples in the library and occasionally forgot the method of checking the samples. Several samples were accidentally spilled on the laboratory floor. But the analysts continued reminding and guiding on how to fully understand the work. Eventually, the intern is starting to get accustomed to the working environment. The sense of responsibility allowed the intern to overcome this difficulty.

Several issues were encountered during the trial. The results of the first trial was shown in **Appendix 2**. In the first trial, sodium methoxide, and methanol were out of specification (passed the storage time) before the dosing began, but the trial still continued. Thus, the used component is replaced several times and resulting in a lower biodiesel quality. Another problem is a prolonged time interval for the first biodiesel (7 days), which is contradictory with the second trial as it was conducted for only 2 days and resulted in high-quality biodiesel. Another issue arose during the separation process; a poor separation performance would mean the washing process has to also be properly done to obtain high purity and low moisture. That is why to overcome it, we need to be precise with our work, especially in a laboratory precision is a key point to becoming great laboratory workers. Thus, the second trial did accurately and efficiently resulting in great results.

CHAPTER 3. PROJECT DESCRIPTION

3.1. Project background

Global warming is being caused by humanity's insatiable use of fossil fuels as energy, which artificially amplifies the natural greenhouse effect (Shaheen & Lipman, 2007). Global warming, which is mostly brought on by the greenhouse gas emissions from the combustion of fossil fuels, is to blame for this increase in the depletion of fossil fuels and the temperatures of the Earth's atmosphere. This sparked a desire in the scientific community to look into more environmentally acceptable alternative fuels. One of the many options being taken into consideration right now is biodiesel. There are other term for biodiesel that is FAME. Biodiesel has gained attraction as a potential replacement for fossil fuels in diesel engines (Huang et al., 2012). Biodiesel is made from oil and fats which use chemicals and catalysts. Biodiesel could be an alternative for reducing fossil fuel depletion and greenhouse gas emissions.

Oil crops, such as soybean, rapeseed, and others; oil trees, such as Chinese pistachio and palm oil; and other animal fat, and waste oil food are the three main categories of feedstocks for biodiesel ("Biodiesel," 2008). In this trial development, Crude Palm Oil (CPO) was the raw material due to its abundance in Indonesia. It is found that Indonesia is the biggest producer of palm oil in the world (Shigetomi et al., 2020). However, it needs to be refined, therefore from CPO, it is bleached to create bleached palm oil (BPO) which is then refined and deodorized into refined bleached deodorized palm oil (RBDPO) (Mata, 2010). biodiesel is particularly significant because of its exceptional environmental and renewable characteristics. The fact that this fuel is produced with natural and renewable raw materials is one of its benefits, given the negative effects of petroleum-fueled diesel engines on the environment and petroleum supplies (Huang et al., 2012). This research is aimed to develop a biodiesel in accordance with the specification which validates the quality of the biodiesel by adding the right amount of chemicals.

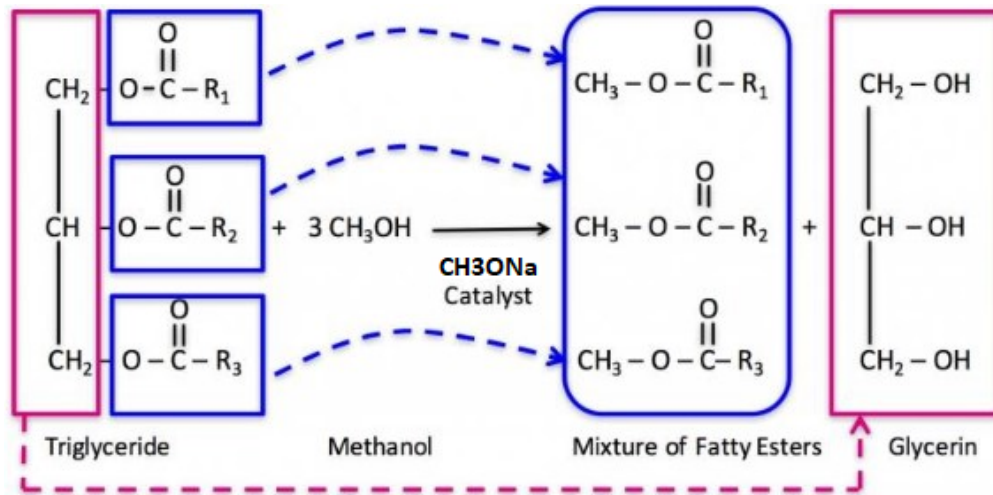


Figure 2. The chemical reaction of biodiesel production. This was processed using RBDPO (Triglyceride) and Methanol that goes through the Trans-Esterification method using sodium methoxide as the catalyst. Hence the process results in biodiesel and glycerin (Najafpour et al., 2008).

There are several methods that can be used to create biodiesel: esterification, electrolysis, and trans-esterification. Esterification has been used to create biodiesel due to an effective technique and might prevent several problems such as fuel filter plugging, injector cooking, etc (Dulawat et al., 2020). However, esterification is not preferred due to lower yield and the need for a longer time to create biodiesel (Vieira et al., 2013). Electrolysis is one of the newest methods used for biodiesel production. It shows that it could give a high yield of biodiesel (90-98%) (Rachman et al., 2018). But it is still not preferred for the production of biodiesel on a large scale due to its novelty. Trans-esterification is used for big-scale production since it gives a good yield of product in a shorter amount of time rather than esterification (Duran et al., 2014).

In this internship, we used the trans-esterification to produce biodiesel (Figure 2). From **Fig 2**. It shows how the biodiesel or fatty acid methyl ester is created using RBDPO (Triglyceride), methanol, and sodium methoxide as the catalyst (Zahan & Kano, 2018). One mole of triglyceride reacts with three moles of methanol to produce three moles of mono-alkyl ester and one mole of glycerol (Stamenković et al., 2008). To increase the rate of reaction, a catalyst was used. This was to shift the equilibrium side to the product because the reaction is reversible (Gashaw et al., 2015). Several types of catalysts include sodium hydroxide, potassium hydroxide, and sulfuric acid (Parkar et al., 2012), in this project, sodium methoxide was used. Instead of using caustic soda (sodium hydroxide) and caustic potash, 70% of biodiesel producers now use sodium methoxide (KoochiKamali et al., 2012). This is because the manufacturer switched to a better water-free process that generates high-value and high-quality end products, cheaper purification costs, and more consistent biodiesel quality by lowering the unwanted product (Hsiao et al., 2018).

The fatty esters then will undergo a washing and drying process to give good purity of biodiesel. The advantages of trans-esterification are high conversion at a reasonable price, benign reaction conditions, product characteristics that are more akin to petro-diesel, and suitability for industrial-scale production (Najafpour et al, 2008).

Table 1. The parameter to create good quality biodiesel is based on several test methods such as ASTM, SNI, and Sinarmas Evaluation. Requirements of the minimum/maximum included in the figure.

Number	Test Parameter	Test Method	Requirements	Units (Min/Max)
1	Moisture	ASTM D-6304	350	ppm, max
2	Acid Value	SNI 7182:2015	0,4	mg-KOH/g, max
3	Soap Content	ASTM D6751-03	5	mg-KOH/g, max
4	Ester Content	SNI 7182:2015	96,5	%-mass, max
5	Free Glyceride Content	SNI 7182:2015	0,02	%-mass, max
6	Monoglyceride	SNI 7182:2015	0,55	%-mass, max
7	Total Glyceride	SNI 7182:2015	0,24	%-mass, max

Sinarmas has already used SNI 7182:2015, ASTM, and as their standard for biodiesel mentioned in **Table 1**. To validate it, several parameters have been checked to obtain standardized biodiesel. These are the parameters:

- **Acid value**

High acidity is correlated with the corrosion of metals and the formation of deposits in engines and can damage elastomers in fuel system lines. For producers, too high of an acid number for biodiesel products indicates an unfavorable production process. (Zhang et al., 2015). Hence, the higher acid value lowers the yield of ester content and increases the sodium hydroxide (NaOH) consumption for neutralization. And also by increasing the acid number causing fuel degradation or hydrolysis of the FFA (Mahajan et al., 2006).

- **Free glycerol, Monoglyceride, Total glyceride**

Free glycerol is how much glycerol that is not processed into a biodiesel after the reaction. It reflects the quality of biodiesel. An excessive amount of free glycerin could lead to issues with storage or the fuel system (Bondioli & Della Bella, 2005). Too much free glycerol would mean that the washing process is not well-performed, and vice versa (Wenxuan He, 2011). The glyceride content (MG, TG) is measured to check whether the washing process is effective and does not leave too many glycerides that should be separated. (Wenxuan He, 2011). High levels of total glycerin can cause deposits to accumulate at injection nozzles, pistons, and valves as well as cause injector fouling (Thoai et al., 2017).

- **Ester methyl content**

Fatty acid methyl esters, often known as ester content, serve as a gauge for the biodiesel's purity. The number of double bonds and the length of the fatty acid chains in the fatty acid methyl esters that are created when fats, oils, or fatty acids react with methanol vary (Baptista et al., 2008). Ester methyl content needs to be high since the main component is biodiesel. Lower ester methyl content correlated to a lower quality of the biodiesel (Sajjaanantakul et al., 1989).

- **Moisture (water content)**

The quality control of the feedstock and the finished product is significantly impacted by the water contamination of biodiesel. Despite being regarded as hydrophobic, biodiesel can have up to 1,500 ppm of dissolved water, omitting suspended water droplets (Mirghani et al., 2011). Water diminishes the calorific value of biofuels, speeds up corrosion, encourages the growth of microbes, and raises the likelihood that oxidation products will accumulate during long-term storage (Abduh et al., 2019). The presence of water will cause a decrease in the heat of combustion, and foam, and is corrosive. It indicates that the quality of biodiesel is improving if the water content in the fuel is less than the 0.05% national standard for Indonesia. Moisture content is another indicator of biodiesel quality (Freedman et al., 1984). When the temperature is cold, the water can crystallize and clog the fuel lines (Sugiarto et al., 2021).

- **Soap Content**

The soap content is also important to be checked due to saponification might occur in the development of biodiesel. As can be seen from **Fig 3**, whereas RBDPO could go to saponification (Ortiz Lechuga et al., 2020). Several problems could occur if the soap content is high such as clogging the fuel filters, leaving ash in the engine, and having a bad quality biodiesel (Nurul et al., 2021).

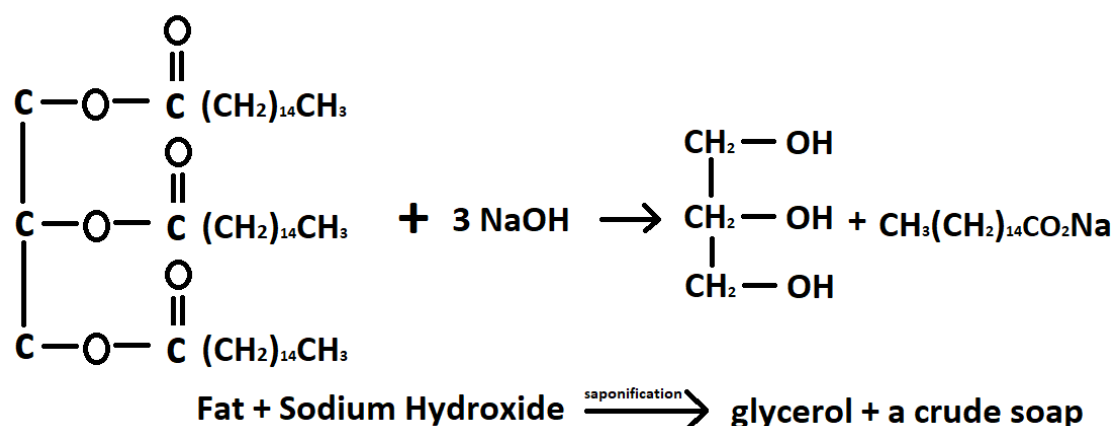


Figure 3. Saponification process

There are other parameters that have not been checked which on **Appendix 1**, together with the explanation.

3.2. Scopes of project

- Understanding the production process of biodiesel
- Obtained a high quality result of biodiesel
- Understanding the principle of instrument used

3.3. Objectives

The objectives of this project are to produce high quality biodiesel that follows the standard from Dirjen EBTKE or SNI 7182:2015 by measuring the suitable dosing percentage and analyzing the quality of biodiesel through several parameters.

3.4 Materials and methods.

3.4.1. Materials

The project internship trial uses several materials such as RBDPO as the raw material that is produced by PT. Sinarmas Biodiesel Energy by refining the BPO that is obtained from PT. SMART Tbk. Other chemicals used are sodium methoxide, methanol, and phosphoric acid which are obtained from another supplier and are not informed due to the confidentiality of the company.

3.4.2. Methods

The procedure overview of Trial biodiesel is displayed in **Fig 4**.

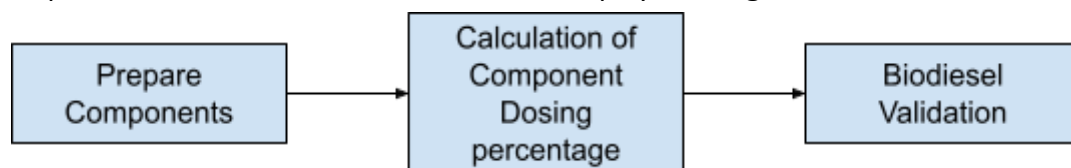


Figure 4. Flowchart of biodiesel development.

3.4.2.1. Prepare Components

Every component has its own specification to create a good quality biodiesel. Therefore, for RBDPO, the moisture content, Free Fatty Acid (FFA), Iodine Value (IV), and color were checked. The moisture content and purity of methanol, sodium methoxide, and phosphoric acid are checked using the weighing scale and KF coulometric titration.

$$\frac{V \text{ Titrate} \times M \text{ NaCl/HCL} \times MW \text{ Component}}{m \text{ Component}}$$

Equation 1. Titration formula for FFA, IV, AV, and purity. V titrate is titration volume, M is molarity of the sodium chloride or hydrochloric acid, MW is the molecular weight of the sample m is the weight of the component.

The raw material, the color, Iodine Value (IV), and Free Fatty Acid (FFA) are also measured. The Color is checked manually using a standard light source with a colored glass disk ranging from 0.5 to 8.0 according to the RGB (Red, Green, Blue) USC (University of Southern California) system as shown in **Table. 2**. The IV and FFA are also checked by titration mentioned in **Eq.1**. From the analysis of the raw material and other components, the results are in the specification which is allowed to be used for the biodiesel trial.

Table 2. Glass color standards by the American Society for Testing Materials (ASTM) together with the University of Southern California (USC) for the chromaticity coordinates with the International Commission of Illumination (CIE) for the Luminous Transmittance with the unit of T_w for parameters on SNI:7182:2015

Glass Color Standards				
Chromaticity Coordinates (RGB USC system)				
ASTM Color	Red	Green	Blue	Luminous Transmittance (CIE Standard Source C) T_w
0.5	0.462	0.473	0.065	0.86 ± 0.06
1	0.489	0.475	0.036	0.77 ± 0.06
1.5	0.521	0.464	0.015	0.67 ± 0.06
2	0.552	0.442	0.006	0.55 ± 0.06
2.5	0.582	0.416	0.002	0.44 ± 0.04
3	0.611	0.388	0.001	0.31 ± 0.04
3.5	0.64	0.359	0.001	0.22 ± 0.04
4	0.671	0.328	0.001	0.152 ± 0.022
4.5	0.703	0.296	0	0.109 ± 0.016
5	0.736	0.264	0	0.081 ± 0.012
5.5	0.77	0.23	0	0.058 ± 0.010
6	0.805	0.195	0	0.04 ± 0.008
6.5	0.841	0.159	0	0.026 ± 0.006
7	0.877	0.123	0	0.016 ± 0.004
7.5	0.915	0.085	0	0.0081 ± 0.0016
8	0.956	0.044	0	0.0025 ± 0.0006

With these components prepared, the results are obtained which can be seen on **Table 3**. The RBDPO has a 300 ppm moisture with 0,08% FFA, 51,52 I2/100 g, 4,3 color. Whereas the sodium methoxide has a 1264 ppm with 31,47% purity. This is due to sodium methoxide usually being sold at 30% weight percent. For methanol, it has 625,5 ppm, and phosphoric acid has 85,47% purity. From this result, every component has met the standard for the biodiesel trial to begin.

Table 3. The results of the components parameter

29/08/2022	TRIAL BIODIESEL			
	Components			
	RBDO	NaOCH ₃	MeOH	Phosphoric Acid
Quantity	500,09 gr			
Moist	300 ppm	1264 ppm	625,5 ppm	-
FFA	0,08%	-	-	-
IV	51,52 I ₂ /100 g	-	-	-
R	4,3	-	-	-
Purity	-	31,47%	-	85,47%

3.4.2.2. Calculation of Component Dosing Percentage

The dosing of components is important to ensure the high quality of biodiesel that meets the specification fiction (Table 1). For the dosing in internship we vary the percentage of methanol, the catalyst and the phosphoric acid. To obtain the biodiesel in accordance with the correct parameters, the dosing percentage is determined, calculated, and prepared to be mixed. Two components are involved in determining the dosing percentage, whereby the first percentage of each dosing represents methanol and the second percentage of each dose represents sodium methoxide.

**Figure 5. Flowchart of biodiesel production process**

In 500 gr of RBDO, we used 50 gr of methanol (10%) and 5,25 gr of sodium methoxide (1.05%) for the first dosing (Table 4). The dosing percentage of each component is displayed on **Table 4**. where the first dosing can be written as 10% for and 1,05% for methanol, and NaOH₃, respectively. For the second dosing as 1,5% and 0,45%, for methanol, and NaOH₃, respectively. For the third dosing has the dosing percentage as the first dosing. The washing process used hot water for dosage 1 & 2 while dosage 3 involves the addition of 0,05% phosphoric acid as it stops the reaction to do the neutralization of the methoxide ion (Vávra et al., 2018). Thus, helping with the removal of soap. It was calculated by determining how many grams

of raw material (RBDPO) were used and the other component will be determined by weighting the weight/volume from the percentage dosing prepared.

Table 4. The dosing percentage of each batch

	MeOH	NaOCH ₃	Washing
Dosage 1	10%	1,05%	Hot water
Dosage 2	1,50%	0,45%	Hot water
Dosage 3	10%	1,05%	Hot water + Phosphoric Acid 0,05%

The dosing process involves the mixture of RBDPO with methanol and sodium methoxide as can be seen from **Table 4**. Every dosing is processed for around 2 hours. After the dosing is done, it is separated using a separating funnel as can be seen on **Fig 6**. until all the water is flushed out of the funnel. Dosing 2 and 3 are then continued for the sample of Dosing 1. After the third dosing, the reaction is stopped using phosphoric acid. Then the washing process is started using hot water input into the biodiesel inside the separating funnel to wash out the water which increases the purity of biodiesel. To completely remove the water content, the biodiesel is dried by stirring at a high temperature (130°C) and then checked periodically until the moisture content matches the parameter.



Figure 6. The biodiesel in a separating funnel to separate the biodiesel from glycerin

3.4.2.3 Biodiesel Validation

After the final product is done, several parameters such as moisture, acid value, soap content, ester content, free glyceride, monoglyceride, and total glyceride are checked. The null hypothesis is that the trial biodiesel developed will go in accordance with the specification, whereas the alternative hypothesis was that the trial biodiesel developed will not go in accordance with the specification.

To check the moisture, a sample is injected into the vessel of the KF coulometric apparatus, which then the iodine will be generated at the anode (Sugiarto et al., 2021). When all the water has been titrated, an electrometric end point detector detects excess iodine, and the titration is stopped. According to Faraday's Law, 1 mol of iodine reacts with 1 mol of water based on the stoichiometry of the reaction, and as a result, the amount of water is proportionate to the total integrated current.

For acid value, it works by doing a titration. First, the sample was weighed and put into a flask mixed with 100 mL of solvent and 1 mL of the indicator. Thus, titrate until the appearance of the first permanent pink color. The soap content principle is similar to acid value by titrating the sample that is mixed with solvent and indicator which then titrate until the color is permanently yellow.

To check the FG, MG, and TG. Gas chromatography is also used. This works by mixing the sample, internal standard, and MSTFA (N-Methyl-N-trimethylsilyltrifluoroacetamide) N-heptane which then inject into the injection port and start the analysis, the glycerin content is measured by comparing the monoolein, diolein, and triolein standards respectively (Tonini et al., 2012).

CHAPTER 4. RESULTS AND DISCUSSION

After the preparation, dosing, washing, and drying are done, the product obtained (**Fig 7.**), then was checked for its quality parameter. However, in this test result, not all of the parameters from SNI 7182:2015 were checked due to limited equipment. Hence, moisture, acid value, soap content, free glyceride, monoglyceride, and total glyceride could be obtained. The results that were found are shown in **Table 5.**

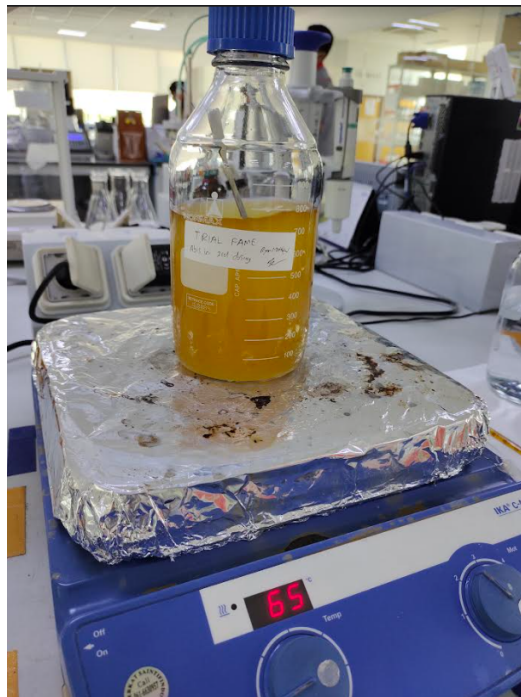


Figure 7. The biodiesel final product

Table 5. Trial biodiesel result. It shows the moisture, acid value, soap content, ester content, free glyceride, monoglyceride, and total glyceride.

Product Quality		Standard Quality	Status Product
Moisture, ppm	279	< 350	OK
Acid value (AV), mg KOH/g	0,07	< 0,4	OK
Soap, ppm	0	< 5	OK
Ester, %	99,05%	> 96,5	OK
Free Glyceride (FG)%	0,001	< 0,02	OK
Monoglyceride (MG) %	0,291	< 0,55	OK
Total Glyceride (TG)%	0,085	< 0,24	OK

For the moisture content, it resulted in 279 ppm which fits the parameter. This moisture could be maintained by several factors such as the containers that

need to be under an air-tight container to reduce the flow of air, which could increase the moisture content and lower the quality of the biodiesel (Abduh et al., 2019). The longer the drying process, the lower the moisture content, and the higher the purity. However, it ultimately leads to a lower biodiesel yield (Sugiarto et al., 2021).

In the trials, the acid value showed an excellent result that is 0,07 mg KOH/g which is way below the parameter that is below 0,4. This value needs to be maintained as the higher acid value may cause corrosiveness when biodiesel is used in the machine. The acid value will affect the soap content which has the opposite result as the acid value. The lower acid value results in high soap content in the biodiesel. However, in this trial, the soap content is measured to be 0 as when titration occurs, no foam is present visually, resulting in soap content below 5, which fits the parameter of below 30. The soap content needs to be checked because a high soap content would mean that the saponification reaction in the biodiesel process is happening instead of transesterification (Edmonds & Mattikow, 1958). This resulted in the production of soap instead of biodiesel.

The residual oil content also has a correlation with the ester content. A higher ester content means a lower residual oil content. This trial showed a high ester content of 99,05% (>96,5%). Hence, the biodiesel production resulted in high quality. The same case is true with the residual oil (MG, TG) where the results showed an MG value of 0,291, and a TG value of 0,085. This fitted the parameter from SNI 7182:2015 that MG is below 0,55 and TG is below 0,24. And for the FG, the result is 0,001 which fits the parameter (<0,2). A high FG could be caused by the washing process. This is due to the final product being biodiesel and glycerin which has undergone a separation process.

Several published studies related to the development of biodiesel were also found. In Brazil, the researchers optimized the removal of moisture using hydrogel from 2160 ppm to 240 ppm (Fregolente et al., 2015). Another research checked the acid value using non-aqueous coulometric titration, with potassium hydroxide in isopropanol as a titrant and p-naphtholbenzein as an indicator. With an accuracy of 3.3%, the results ranged from 0.198 to 1.17 (Mahajan et al., 2006). Another research conducted by Wall et al. (2011) used ion exchange resins and solid adsorbents for the soap content. This resulted in the soap content to reduce from 1200 ppm to 50 ppm for about 550-bed volumes of processed biodiesel. For the optimization of FG, MG, and TG using ultrafiltration polymeric membrane, however, there was a permeate flux which is limited by international standards (Tajziehchi & Sadrameli, 2021).

This trial demonstrated good quality of biodiesel which fulfilled the parameter specification criteria and resulted in high purity levels (**Table 5.**). However, this result could not be applied on an industrial scale, as washing is only done once on the mentioned scale. This ensures the production cost is reduced while still maintaining a good final product. This circumstance explained the reason why the

soap content and acid value resulted in a desirable outcome; due to multiple washing.

CHAPTER 5. CONCLUSION & RECOMMENDATION

In this internship, we managed to produce biodiesel from Refined Bleached Deodorized Palm Oil (RBDPO). From the results we found that the raw material/chemical used is important to ensure the quality of biodiesel, because if the raw material is out of specification (components purity and moisture do not fit the parameter), the biodiesel produced will not meet the quality standard parameter. By applying different dosing of methanol and sodium methoxide continuously, the results of the end product demonstrated a good quality of biodiesel which fulfilled the parameter specification criteria and resulted in high purity levels. Every inspected parameter displayed results which are in accordance with the standard of Dirjen EBTKE: SNI 7182:2015 and ASTM. Although there are other comparative studies between several researchers, this result already fits the parameter from the standards given.

In this internship, we only checked moisture content, acid value, soap content, ester content, free glycerides, monoglyceride, and total glyceride. However, there are more parameters that determine the quality of biodiesel that could be checked based on the Dirjen EBTKE: SNI 7182:2015. Thus, the researcher needs to make sure that the chemicals used are in specification so a high-quality biodiesel can be created.

CHAPTER 6. SELF REFLECTION

6.1. Identification of strength and weakness during internship

Several strengths and weaknesses have been identified during the internship program. One of my strengths is persistence, which is shown by how I want to understand the material fully. Social skills are also one of my strengths which is shown by how I am able to socialize with other workers, giving joy and a conducive workplace environment. I am also agile in finishing daily routine work at the pace of the other workers.

However, there are several weaknesses, which is that I am sometimes not aware of laboratory practice, causing an accident to happen during work (spilling the sample onto the floor). Another weakness is I may have made a mistake in doing measurements for testing.

6.2. Benefits of internship placement

This internship placement has brought many improvements for me. Become more persistent in finding the solution to the problem, learning to be punctual, and using my time as efficiently as possible. Learning to be responsible is one of the most important values prepared from this internship. The company gives a lot of detailed explanations on how to keep the product in a good condition. And by giving a trial and internship in PT. Sinarmas Biodiesel Energy, my lab skill has been improved by helping other workers to check the samples.

6.3 To what extent i3L and its values (Grit, Role-Model, and Integrity) contributed to the benefits

i3L has taught me at the very beginning about teamwork and integrity skills which prepare us to go to an internship at a higher skill level. One of the important points is market readiness which gives well-rounded graduates through unique learning ambience and industry exposure. Another value is striving to leverage national resources to accelerate Indonesia's competitiveness in the global landscape. Knowledge-wise i3L has prepared the theory and practical instruction on how to do research, scientific writing, and how to work in laboratory neatly and efficiently.

6.4 Contribution and relevance of subjects at i3L to the internship success

i3L has prepared many materials which are practically used in laboratories. One of the relevant materials in this company is by using titration for finding the

moisture content of the sample. Also, gas chromatography is used to find how much ester content and total glycerin content is in biodiesel. And there are major skills that are pH test, salt content, and using a lot of laboratory equipment (burette, pipette, micropipette). Also from i3L, the students should understand the basics of what to do and not do in a laboratory.

6.5 Contribution of BRIGHT Sessions for his/her soft skills development

There are many BRIGHT Sessions that improve soft skills development. One example is the BRIGHT session in collaboration with Kalbio Global Medika which talks about life after graduation, and where the students want to continue. Is it internship, work, magister, or even Doctorate? This gives an insight to each student on what they want to do after college. It also mentioned several factors in which soft skills are needed in the future such as time management and communication.

Another important session prepared by Pri Notowidigdo who is the IC-Suite Headhunter & Executive Coach, at PRI for People Matters explains what leadership skills are needed in work industries. And another session prepared by Kalbe explains how to be accepted at an interview by preparing a good curriculum vitae (CV) and a great interview (good listener & talker).

6.6 Impact of the student on the workplace

I understand the work of each member of QC laboratories. This includes testing each sample from incoming chemicals, biodiesel, and glycerin. There are many variables that are tested for each sample such as moisture, density, viscosity, flash point, cloud point, et cetera. My presence gives an enjoyable experience and a conducive work environment.

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APPENDICES

Appendix 1. SNI Parameters that has not been checked on trial biodiesel

Table A1. The parameters from DIRJEN EBTKE or SNI 7182:2015

Number	Test Parameter	Test Method	Requirements	Units (Min/Max)
1	Density	SNI 7182:2015	850-950	kg/m ³
2	Kinematic Viscosity at 40°C	SNI 7182:2015	2,3-6,0	mm ² /s (centistokes (cSt))
3	Cetane Number	SNI 7182:2015	51	Min
4	Flash Point	SNI 7182:2015	130	°C, min
5	Steel Plate Corrosion (3 hours at 50°C)	SNI 7182:2015	number 1	
6	Distillation Temperature 90°C	SNI 7182:2015	360	°C, max
7	Carbon Residue	SNI 7182:2015	0,05	%-mass, max
8	Sulfated Ash	SNI 7182:2015	0,02	%-mass, max
9	Sulfur Content	SNI 7182:2015	10	mg/kg, max
10	Phosphor Content	SNI 7182:2015	4	mg/kg, max
11	Iodine Value	SNI 7182:2015	115	%-mass (g-I ₂ / 100 g), max
12	Oxidation Stabilization Index	SNI 7182:2015	600	minute
13	Color	ASTM D-1500	3	max
14	CFPP (Cold Filter Plugging Point)	ASTM D-6371	15	°C, max
15	Metal I (Na+K)	EN 14108/14109, EN 14538	5	mg/kg, max
16	Metal II (Ca+Mg)	EN 14538	5	mg/kg, max
17	Total Contaminant	2276, ASTM D 5452, ASTM	20	mg/liter, max

Other Parameters that are listed in DIRJEN EBTKE or SNI 7182:2015

- Density and kinematic viscosity

This parameter correlates with the purity of the biodiesel in a directly proportional manner. In other words, a low density and viscosity would result in a low purity, and vice versa. This would ultimately lead to a poor flow of biodiesel in the engine (Lapuerta & Canoira, 2016).

- Cetane number

The cetane number is the indicator of combustion speed and the compression needed for ignition. A high cetane number provides a rapid pressure rise and a low maximum pressure, thereby reducing the combustion sound. Hence, it would result

in an inefficient ignition due to slow transfer from biodiesel into the engine (Sivaramakrishnan & Ravikumar, 2012).

- **Flash point**

The Flashpoint is the temperature at which the biodiesel gives sufficient vapor to ignite in the air. If the flash point is too low, then the biodiesel is considered dangerous as it can burn the vehicle (Ha et al, 2006).

- **Copperplate corrosion**

Copperplate corrosion measures the quality of the copper plate to ensure that the copper used in the industry will not break due to corrosion (Andersen et al., 2003).

- **Carbon residue**

When a hydrocarbon stream is entirely distilled, the amount of coke residue left over is measured as carbon residue. The combustion is not perfect if the carbon residue is too high (Zerlia et al, 2000).

- **Distillation temperature**

The temperature of biodiesel is measured during distillation to check at what temperature the biodiesel evaporates (Yulistiani et al., 2020).

- **Sulfated ash**

Analyzing the amount of ash in food simply involves burning away organic material to reveal inorganic minerals. This aids in determining the quantity and kind of minerals present in food, which is crucial because the presence or absence of minerals can affect the physiochemical characteristics of food and the rate at which microbes develop (Hinz, 2007).

- **Sulfur content**

Sulfur content needs to be checked. If it is too high, it would create gum or sludge which breaks down the biodiesel (Jamal et al, 2010).

- **Phosphor content**

Heavy metal such as phosphor needs to be removed as they would damage the product. If there is high phosphorus content in biodiesel, then there is trouble with the washing process of biodiesel (Ryan et al., 1985).

- **Iodine value**

Iodine number is a measure of the number of unsaturated compounds contained in oil/fat, as well as compounds in the form of mono-, di-, and triglycerides, as well as polyunsaturated compounds. The presence of these unsaturated compounds is characterized by high levels of iodine. This can lead to the following problems:

- The occurrence of polymerization and the formation of deposits on the injector nozzle, piston ring, and piston ring thread, when in hot conditions.
- Lowers the oxidation stability of biodiesel, which in turn causes the formation of various degradation products that have a negative impact on engine operation.
- Decrease in the quality of fuel lubrication in the engine.

Iodine number is also correlated with viscosity and cetane number. If the measured viscosity and cetane number are low, this may indicate a high content of polyunsaturated in the biodiesel (Ebong, 2019).

- **Oxidation stability index**

Due to its chemical properties, biodiesel is more prone to oxidative degradation than diesel oil. This is related to the high content of polyunsaturated ester compounds which contain numerous double bonds and are vulnerable to oxidation. The low value of oxidation stability can cause problems with elastomers, especially in the fuel line system (Kumar & Sharma, 2015).

- **Colour**

The color of the biodiesel is measured to ensure that every biodiesel has the same appearance when sold to the consumer (Nedambale et al, 2014).

- **Cold flash plugging point (CFPP)**

CFPP is defined as the lowest temperature of biodiesel, at which biodiesel can still flow through a standardized filter in 60 seconds according to American Society for Testing Materials D 6371 (ASTM D 6371) (Lapuerta et al., 2018).

- **Cloud point**

An essential quality requirement for diesel fuels is cloud point. This is a sign of a fuel's propensity to crystallize wax at low temperatures. These crystals can clog the atomizer in a diesel engine's combustion chamber, which injects gasoline (Anup Pradhan & Dev Sagar Shrestha, 2007).

- **Sodium and potassium**

Excessive sodium and potassium metals could create ash in the machine (S. F. Alves et al., 2018).

- **Magnesium and Calcium**

Excessive amounts of magnesium and calcium could create a clog in the injection pump (S. F. Alves et al., 2018).

- **Total contaminant**

The total contaminant was defined as the amount of undissolved material remaining on the filter after the fuel sample passed through a 0.8 meter filter according to European Standard 12662 (EN 12662). In diesel oil, the total contaminants tend to come from the rest of the distillation process, which is inseparable. Whereas in biodiesel, not all transesterification processes take place in the distillation column. High contamination of biodiesel causes blockage of the fuel filter and injection pump (Komariah et al., 2022).

- **Methanol content**

Methanol content should be measured to ensure the methanol already becomes biodiesel.

Appendix 2. The result of the first trial biodiesel

Table A2. The result from the first trial biodiesel

Trial BIODIESEL #1			16/08/2022
Components			
Raw Material	RBDPO Ref 3	NaOCH3	
	1st // 2nd Batch	Moist	172,6 ppm
Quantity	250,3 // 139,36 gr	Purity	33,40%
Moist	22 ppm		
FFA	0,06	MeOH	1st // 2nd Batch
IV	51,99	Moist	533 // 233 ppm
R	4,6		
Phosphoric Acid			
Purity	82,25%		

Product Quality			
	First Batch	Second Batch	Third Batch
Moisture, ppm	316,8		
Av, mg KOH/g	0,215		
Soap, ppm	-		
Ester, %	99,35%		
Free Glyceride %	0	1,00%	0,200%
Monoglyceride, %	1,709	0,34	0,233
Diglyceride, %	1,907	0,09	0,048
Triglyceride, %	1,331	0,053	0
Total Glyceride, %	0,865	0,118	0,065

	MeOH	NaOCH3
Dosage 1	10%	1,05%
Dosage 2	1,50%	0,45%
Dosage 3	10%	1,05%
Dosage 4	1,50%	0,45%
Washing	Phosphoric Acid	
After dosing 2	0,05%	
After dosing 3	0,10%	
After dosing 4	0,05%	