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Stability of Pure Biodiesel (B100), Biodiesel Mixture (B40), and Petroleum Diesel (B0) Due to Storage

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ABSTRACT

Biodiesel is produced from synthetic ester compounds of vegetable and animal oils through a process of refining, bleaching, degumming, transesterification, and esterification. The quality and characteristics of the fuel are greatly influenced by the storage. The purpose of this research is to determine and analyze the effect of storage conditions on the physical and chemical changes of biodiesel (B100), biodiesel blend (B40), and petro diesel (B0). Experiment was conducted by storage fuels using glass bottle for 35 days arranged into four conditions based on cap and wrapping using aluminum foil, namely P1 (with cap, with wrapping), P2 (with cap, no wrapping), P3 (no cap, with wrapping), and P4 (no cap, no wrapping). Results showed that fuel quality decreases due to storage indicated by an increase in water content, density, acid number, and color parameters, as well as a decrease in the specific calorific value of all fuels. The condition of the storage container affected the changes the quality parameters. Based on its impact on fuel quality decline, the order of storage container conditions from the best is P1 (with cap, with wrap) > P2 (with cap, no wrap) > P3 (no cap, with wrap) > P4 (no cap, no wrap).

1. INTRODUCTION

Population growth coupled with rapid economic growth and increasingly advanced technological developments has resulted in increasingly high energy use. This also happens in Indonesia that placing this country as one of the high energy user. Primary energy consumption in Indonesia in 2021 reached 1,185 million barrels of oil equivalent (mboe) with fossil fuels contribution of 54% constituted of coal of 299.19 mboe (25.2%), natural gas of 74.58 mboe (6.3%) and mineral oil of 264.56 mboe (53.8%) (MEMR, 2022). This has made Indonesia as the country with the largest level of energy consumption in the Asia Pacific region after China, India, Japan and South Korea (Afriyanti *et al*., 2018). On the other side, the availability of petroleum oil in Indonesia is increasingly running low. Indonesia's petroleum oil reserves in 2021 are 4.17 million barrels, while the daily petroleum production level is 1.007 million barrels. Therefore, our oil reserves will only last for the next 9.5 years (Dirjen Migas, 2021).

Biodiesel is a liquid fuel with characteristics similar to petroleum diesel fuel so it can be developed as a transportation fuel for diesel engines. Biodiesel is produced from ester compounds synthesized from vegetable oils through transesterification (Haryanto *et al*., 2015; 2017; 2020; 2021). Therefore, it not only is considered as renewable fuel, but also biodegradable. The application of biodiesel is performed by blending it into petroleum diesel fuel to produce a mixed fuel which is marketed under the name “biosolar” BXX, where B stands for biodiesel and XX represents the percentage. For example, B30 means blended fuel with a composition of biodiesel 30% and petroleum diesel 70%. Mixing biodiesel into petroleum diesel fuel will increase the cetane number of the fuel that burnt more completely and reduce petroleum diesel consumption. Apart from that, compared to petroleum diesel, biodiesel blend fuel produces lower and non-toxic emissions for carbon monoxide (CO), hydrocarbons (HC), sulfur oxides (SO_x), smoke density, aromatic compounds, and particulate matter (PM) (Shirneshan, 2013; Darmawan, 2013; Cappenberg, 2017; Rajendran *et al*., 2023; Leung *et al*., 2006). In the power generation sector, the use of biodiesel is reported to save more fuel and reduce the CO_2 emissions, as well as decrease the cost (Zakaria *et al*., 2014).

Currently, Indonesia has implemented a mixture of biodiesel and petroleum diesel as a transportation fuel. Over a period of eight years, the biodiesel blending level has continued to be increased from 15% (B15) in 2015, 20% (B20) in 2016, and 30% (B30) in 2020. (Shahara *et al*., 2022) conducted a simulation to evaluate the economic impact of B30 implementation and revealed that execution of the B30 increases the performance of real gross domestic product (GDP) by 0.058% and results in a trade surplus of 0.023%. As a concrete manifestation of the government's commitment to accelerating the energy transition sustainable, starting on February 1 (2023) the mandatory blending level of biodiesel has been increased to 35% (B35) (Kementerian ESDM, 2023). This has positioned Indonesia as the leading country in the world in term of biodiesel utilization (Wirawan *et al*., 2024). The B35 policy is claimed to be able to absorb around 13.15 million kL of biodiesel for domestic industries. This policy is also estimated to save foreign exchange of USD 10.75 billion (equivalent to around IDR 161.25 trillion) and increase the added value of downstream industries by IDR 16.76 trillion. The B35 policy is also projected to reduce greenhouse gas emissions by 34.9 million tons of CO_2 (Hardiyanto & Prawoto, 2023).

The government does not stop here, efforts are continuing to increase biodiesel in the fuel mix. Currently, the government is developing a program to mix biodiesel with petroleum diesel fuel into B40 to further reduce domestic oil consumption. This is an effort to reduce petroleum imports in the midst of a global crisis situation that is dependent on diesel fuel. Another goal is to increase the supply of energy supplies in a sustainable manner. The high demand for diesel fuel and increasingly limited supplies have resulted in the intensive development of biodiesel made from palm oil as raw material. The mixing composition of 40% biodiesel and 60% diesel oil produce biosolar B40 (Ramadhani *et al*., 2017). Biosolar B40 have characteristics that are not much different so they can be used as fuel for diesel engine. However, fuel oil with a high biodiesel content has a higher cetane number, burns more completely, and is biodegradable. According to the findings of the road test, cars using B40 fuel could successfully travel 50,000 km without experiencing any technical problems. Inspection of engine components and analysis of used engine oil verified that B40 met the requirements of engine manufacturer. PERTAMINA (2023) reported that utilization B40 fuel in test vehicles did not result in significant changes in power, fuel consumption and emissions. Apart from that, there was no significant impact on the physicochemical properties of the lubricant used. However, there was an increase in the rate of B40 water content of 1.1 ppm/day, slightly higher than B30 which was 1 ppm/day. In conclusion, B40 demonstrated their suitability for operation in Indonesia, thereby validating their viability for 40% biodiesel to be implemented in the country (Mokhtar *et al*., 2023).

In addition to compatibility with the existing engines, using a high mixture ratio of biodiesel offers several benefits (Reksowardojo *et al*., 2023). Biodiesel has better lubricity as compared to petroleum diesel such that reduces friction and wear for sliding components and extends the lifetime of the engine. Fazal *et al*. (2013) reported that under rotating speed of 1500 rpm the wear of steel ball decrease by 10% using biodesel blend B20 as compared to pure petroleum diesel and that the addition of biodiesel effectively reduce the friction and wear of the sliding components and thus prolong the engine lifetime. However, there are some concerns need to be addressed. The performance and lifespan of engine components are significantly influenced by the properties of biodiesel, including its sources and production process. Furthermore, biodiesel is a fuel that is hygroscopic (easily absorbs water vapor) and easily oxidized (Agumsah *et al*., 2023), therefore fuel stability has been a main concern (Komariah *et al*., 2017). During storage, some of the biodiesel characteristics generally change so that it requires good handling and storage methods. Biodiesel can degrade due to oxidation, contact with water, sunlight, temperature elevation, and/or microbial activity (Komariah *et al*., 2017).

Matheofani *et al*. (2021) asserted that the quality and characteristics of fuel will be greatly influenced by the storage period. The longer the storage process, the more degradation occurs due to contact with air and water vapor, resulting in an increase in acid number, water content and color changes (Amelia *et al*., 2017). Owing to the presence of dissolved oxygen, the physical and chemical quality of fuel oil will change, such as the formation of aldehyde compounds, saturated acids and polymer compounds (Musadhaz *et al*., 2012). A high amount of free fatty acids will increase the acid number. This will affect the fuel lines which corrode easily. The water content that is hydrolyzed during storage has an impact on increasing free fatty acids which will cause corrosion in the engine and encourage microbial growth that inhibits fuel flow. The permitted water content of biodiesel and biodiesel mixtures is 0.05%. Changes in density, acid number, water content, color and calorific value of fuel are influenced by the condition of the container, temperature and duration of storage (Harahap & Abrasyi, 2021). Research on the stability of the characteristics of biodiesel samples and biodiesel mixtures that are easily oxidized is important to determine how much the characteristics of biodiesel change during storage. This is related to the quality of biodiesel including density, acid number, water content, color and calorific value. Maintaining fuel quality is an important aspect to ensure its quality meets applicable standards. Therefore, in this research the effect of storing biodiesel samples and biodiesel mixtures was studied to see the stability of their characteristics during storage.

2. MATERIAL AND METHODS

2.1. Materials and Tools

Research was carried out for ±100 days in the laboratory of a biodiesel industry in Lampung. Biodiesel samples (B100) were obtained from a biodiesel factory in Lampung, while diesel fuel (B0) was obtained from diesel vehicle fuel tanks. Biodiesel mixture (B40) was composed of 40% biodiesel and 60% diesel fuel.

The equipment used included a 25 ml pycnometer (BORO 3.3) to measure the density of fuel oil, a moisture meter (CA-200 Mitsubishi) to measure the water content of fuel according to the ASTM D6304/EN ISO 12937 method, a bomb calorimeter (Parr 6400 automatic) to measure the calorific value of fuel oil according to the ASTM D 204 method, and Lovibond EC 3000-ASTM to measure the color of fuel oil with a wavelength of 517.3 nm according to the ASTM D-1500 method. The tools for analyzing acid number of fuel oil through the titration process (AOCS Cd3d-63 method) included a 100 ml clear glass bottle, rubber cap, aluminum foil, 300 ml Erlenmeyer, 3 ml syringe, 100 ml beaker, 1000 ml measuring cup, hot plate, analytical balance, cuvette, wick string, neutral IPA solution, potassium hydroxide (KOH) pH above 10, and clear phenolphthalein indicator with pH below 8.3

2.2. Experimental Design

The experimental was constructed according to completely randomized design (CRD) with two main factors. The first factor was the type of fuel consisting of pure biodiesel (B100), biodiesel mixture (B40), and diesel fuel (B0). Another factor was storage conditions that influence biodiesel stability. In this study fuels were stored within clear glass container divided into four conditions based on cap and wrapping using aluminum foil, namely P1 (with cap, with wrapping), P2 (with cap, no wrapping), P3 (no cap, with wrapping), and P4 (no cap, no wrapping). Storage was carried out in a laboratory room exposure to light where the samples were placed on a table and arranged randomly. The samples were stored for 35 days, and measurement of stability parameters on was carried out at the initial and the end (day 35).

2.3. Water Content

The water content of the oil was measured using a moisture meter (CA-200 Mitsubishi) by activating the power button. Next, press the titration button on the moisture meter and wait until the status is ready. Take 3 ml of fuel sample into the syringe. It is important to ensure that there are no air bubbles in the injection syringe by removing part of the sample from the syringe. Put silicon rubber on the needle to avoid leaks. Next, weigh and record the weight of the sample in the syringe as the initial weight before pressing the start button. Then, inject the sample into the Karl Fisher beaker according to the water content range that corresponds to the type of fuel oil. After that, weigh and record the weight of the remaining sample in the syringe as the final weight. Press the sample button and enter the initial weight and final weighing weight. Finally, wait until the tool displays water content of the fuel and recorded the results.

2.4. Density

Fuel density was measured according to the ASTM D 4052 method using a 25-ml pycnometer. The oil samples was previously heated to a temperature 40 °C. The measurement was carried out using an analytical balance by weighing the empty 25-ml pycnometer (M_1), and the pycnometer filled with 25 ml of sample (M_2). The density (ρ, g/ml) of the fuel oil was calculated according to the following equation:

$$\text{Density } (\rho) = \frac{M_2 - M_1}{25} \tag{1}$$

2.5. Acid Number

The acid number was measured by preparing a 300 ml Erlenmeyer glass and fuel oil. Then, the Erlenmeyer flask is placed on an analytical balance and the balance is zeroed. A sample of 20 ml was slowly put into the Erlenmeyer flask. Next, 50 ml of neutral IPA solution was added and three drops of phenolphthalein indicator were added. Titration of the sample was then carried out using 0.1 KOH solution slowly while shaking the solution until the color changed gradually to pink over 30 s. The results of using KOH for titration will appear on the instrument monitor, and these results are recorded in the calculation data. The value of acid number (in g KOH/g oil) was determined using Equation (2):

$$\text{Acid Number} = \frac{V_{KOH} \text{ x } N_{KOH} \text{ X } 56.1}{M} \tag{2}$$

where V_{KOH} is the volume of KOH solution used for titration (ml), N_{KOH} is the normality of KOH solution (mol/ml), 56.1 is the molecular weight of KOH (g/mol), and M is sample (oil) weight (g).

2.6. Color

Color measurements can be carried out qualitatively by looking visually, or quantitatively using a color meter (Lovibond EC 3000-ASTM) with a wavelength of 517.3 nm. Color measurement was carried out by cleaning the cuvette with hexane solution until there is no fuel oil. Next, the fuel oil sample was put into a 25 ml cuvette until it was full. The color meter was turned on and the zero button is pressed, then the cuvette was inserted into the measuring instrument until it is ready to check the color of the sample. The results of the color measurement determine the concentration level of the fuel. The higher the color value, the darker the color and the lower the quality.

2.7. Calorific Value

The tool to measure calorific value was a calorimeter (Parr 6400). Calorific value was measured by weighing a fuel sample of about one gram in a sample cup using an analytical balance and recording it. The thread for ignition was prepared by tying it to the ignition wire (electrode). The cup containing sample was placed at the end of the igniter rod, and make sure the thread has touched the fuel. The prepared material was put into the vessel by turning the cover firmly. The calorimeter was closed, and the burning process of the oil sample was started. After waiting for some minutes the bomb calorimeter has burned all the samples, the final results (fuel heating value) was displayed and then recorded.

Table 1. Initial data for fuel mixture before storage along with SNI standard for B0, B35, and B100

Parameter of quality	Unit	B0	SNI B0	B40	SNI B35	B100	SNI B100
Density (ρ)	g/L	841.0	815-870	858.1	815-880	862.0	850-890.0
Acid number (max)	mg-KOH/g	0.150	0.6	0.100	0.6	0.067	0.400
Water content (max)	ppm	260	500	247	400	93	350
Color (max)	--	1.5	3.0	1.6	3.0	0.7	3.0
Heating value	cal/g	10,487	--	10,093	--	9,888	9,132

3. RESULTS AND DISCUSSION

Table 1 shows the initial conditions of the fuel before storage. As compared to diesel fuel, pure biodiesel (B100) has lower values of heating value, color, water content, and acid number. It, however has higher density value. The quality of biodiesel used in this experiment meets national standards in terms of density, acid number, water content, color, and heating value. Except for color, the biodiesel mixture (B40) shows reasonable characteristics in between diesel fuel (B0) and pure biodiesel (B100). Biosolar B40 marketed domestically must meet quality standards of B35 which include, among other things, mass density of 815-880 g/L (at 15 °C), water content of 400 mg/kg or 400 ppm, maximum total acid number of 0.6 mg KOH/ g, and maximum color of 3 (Kementerian ESDM, 2023). Quality standards for pure petroleum diesel fuel (B0) and pure biodiesel (B0) have slightly different values as listed in Table 1. Based on the values of our measurement, it can be concluded that fuels used in this experiment have quality fulfills the established standards for each fuel type. The exception is for B40 which has calorific value 10,093 cal/g, which is lower than the standard value for B35 (10,516 cal/g).

3.1. Moisture Content

Moisture in biodiesel fuel can have several negative effects, which can affect fuel quality and, in turn, engine performance (He *et al*., 2007; Barabás & Todoruţ, 2011; Fregolente *et al*., 2012; Sarin, 2012; Agumsah *et al*., 2023). Water in the biodiesel may cause corrosion of engine components in the fuel system, such as tanks, pipes, and injectors. This corrosion can damage components and reduce engine life. Moisture in the fuels can be a medium for microorganism growth such as bacteria and fungi. These microorganisms may clog fuel filters and cause decreased engine performance. Water can also cause the breakdown of esters in biodiesel through the process of hydrolysis, which produces free fatty acids. These free fatty acids can increase the acidity of the fuel, which in turn can cause corrosion and damage to the engine. In addition, water can mix with biodiesel and form an emulsion which cause blockages in filters and fuel injection systems, which can interfere with fuel flow to the engine. Furthermore, the presence of water in biodiesel can reduce the calorific value of the fuel, as well as cause incomplete combustion, potentially producing more smoke and harmful emissions, and reducing engine performance. The presence of water in biodiesel can come from an imperfect purification process during the biodiesel production process, as well as free water that increases due to suboptimal handling and storage procedures (Matheofani *et al*., 2021).

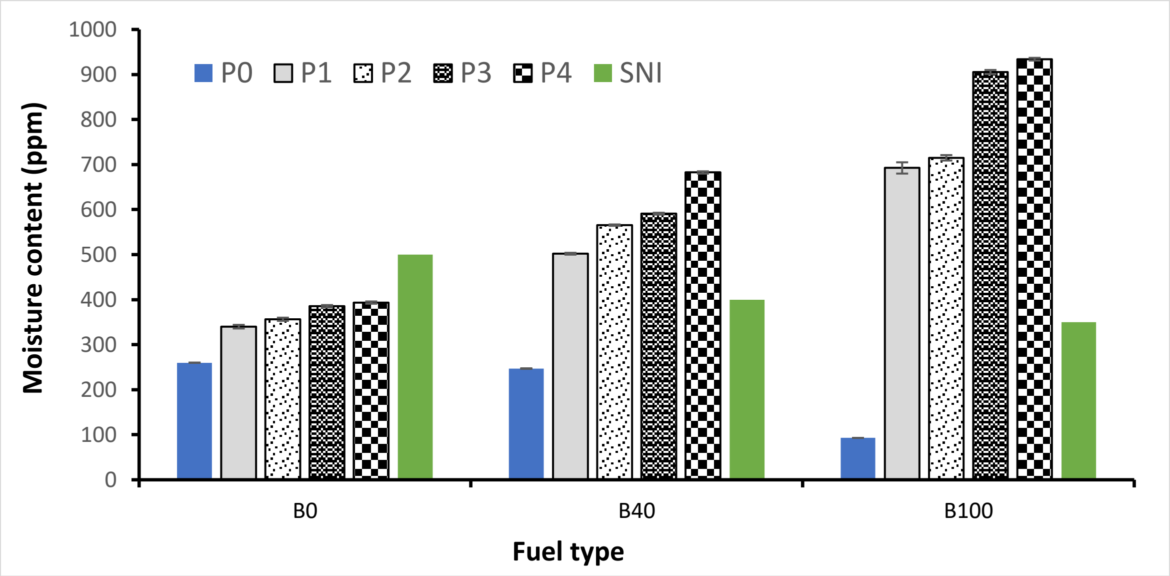

Figure 1. Effect of storage conditions on the water content of fuels after 35 days storage (P0 is initial value at day 0).

Our research results show that the water content in the fuel increased after 35 days of storage (Figure 1). ANOVA analysis showed that the type of fuel and storage conditions significantly affected the increase in water content. Further DMRT tests concluded that the effect of the type of fuel from the highest was B100 > B40 > B0. Diesel fuel (B0) produced the lowest increase in water content, which was between 80-134 ppm and an average of 2.3-3.8 ppm/day. However, the final water content of diesel fuel was still within the permitted limits based on the SNI standard of 500 ppm. Biodiesel B100 experienced the highest increase in water content, which was 600-841 ppm or an average of 7.3-12.4 ppm/day or 6.7 times B0. B40 fuel experienced an increase in water content of 255-436 ppm or 7.3-12.4 ppm/day or 3.1 times B0. The final water content of pure biodiesel fuel (B100) and its mixture (B40) has exceeded the maximum limit according to the SNI standard, which is 350 ppm for B100 and 400 ppm for B35. Further DMRT tests also concluded that the P4 (no cap, no wrap) treatment produced the highest water content increase effect for all types of fuel. The effect of storage conditions on the increase in fuel water content in order from the highest is P4 (no cap, no wrap) > P3 (no cap with wrap) > P2 (with cap, no wrap) > P1 (with cap, with wrap). Several studies have reported a similar phenomenon where biodiesel contains more water than diesel after storage. Matheofani *et al.* (2021) reported that biodiesel stored in closed, opaque jerry cans recorded an increase in water content per 2 weeks of 15 ppm for B100 biodiesel, followed by 8 ppm for B40, and 5.5 – 6.2 ppm for B30. Meanwhile, He *et al.* (2007) reported that the water absorption rate by biodiesel was 9 times that of diesel fuel (B0).

The increase in water content can occur because the fuel absorbs water from the atmospheric air during storage. This is very clearly seen from the treatment of storage without a lid (P3 and P4) which resulted in a higher increase in content compared to storage with a lid (P1 and P2). He *et al.* (2007) explained that biodiesel has a unique chemical structure where the carboxyl group (-COOH) has a stronger tendency to absorb moisture than diesel fuel. Aquino *et al.* (2012) stated that the increase in water content is not only caused by the hygroscopic nature of biodiesel, light and temperature conditions during storage also trigger biodiesel oxidation. Fuel stored in a closed container also experiences an increase in water content. This can also be seen from the results of our study where the P2 treatment, even though the container was closed during storage, the fuel experienced an increase in water content of up to 37% for B0, 129% for B40 and 645% for B100. This is in line with research from Khalid *et al.* (2015) which concluded that biodiesel stored in glass containers that were protected from light exposure recorded the best results compared to fuel stored in plastic containers exposed to external light. Alleman & McCormick (2016), explained that oxidized biodiesel can cause high acid numbers, high viscosity, and the formation of gum and sediment that clogs the filter. Tsuchiya *et al.* (2006) added that the biodiesel oxidation process will also produce free water. The degree of physical and chemical damage to the biodiesel produced is quite significant when the fuel is exposed to sunlight and air (Wu *et al.*, 2011).

Other factors that affect fuel damage include storage temperature. High storage temperatures will provide higher acid values compared to low temperatures (Hanis *et al.*, 2014). Zakaria *et al.* (2014) reported that the rate of increase in water content of biodiesel stored at low temperatures reached 56% for 12 weeks, and reached 139% when stored at room temperature. Leung *et al.* (2006) also reported that of 12 biodiesel samples stored at different temperatures and environments for one year, biodiesel stored at low temperatures (4 and 20 °C) experienced damage of less than 10%, while samples stored at 40 °C experienced damage of up to 40%.

3.2. Density

Density of material is one of the important quality parameters of liquid fuel. This is because density will determine the volume of fuel storage and transportation. Fuel with low density requires large containers in transportation. Density also determines the specific calorific value of the fuel: the higher the density, the lower the specific energy value. Figure 2 shows the value of fuel density after 35 days of storage. For comparison, the initial fuel density value before storage (P0) is also shown. Initially, diesel fuel (B0) and pure biodiesel (B100) have densities of 841 and 862 g/L, respectively. The density of 40% biodiesel mixture (B40) is between the two, which is 858.1 g/L. National Standard of Indonesia SNI 7182-2015 requires the density of B100 biodiesel to be between 850 - 890 g/L (BSN, 2015). Meanwhile, the Decree of the Director General of New, Renewable Energy and Energy Conservation No. 195.K/EK.05/DJE/2022 concerning Standards and Quality (Specification) of Biodiesel Fuel requires the density of B35 to be between 850 - 880 g/L (Kementerian ESDM, 2023). This means that the biodiesel used in this study has a density within the national standard range.

After 35 days of storage, the density of the three fuels increased. The density of diesel oil (B0) increased slightly to between 843 g/L and 845.1 g/L. This means an increase of 2.0-4.1 g/L or 0.24-0.49‰ compared to the initial density value. Pure biodiesel increased its density to between 863.9 g/L and 865.8 g/L, or an increase of 0.22-0.44‰. Biodiesel mixture (B40) increased its density to between 859.7 g/L and 861.2 g/L, or an increase of 0.19-0.36‰. Results from the ANOVA analysis showed that storage container conditions were not significant on the density of fuels with a significance value of 0.246 ($p>0.05$). Treatment P4 (no cap, no wrap) has resulted the highest density for all fuels, namely 865.8; 861.2; and 845.1 g/L, respectively for B100, B40 and B0 fuel. Meanwhile, the lowest density values on day 35 occurred in treatment P1 (wit cap, with wrap) with values of 859.7 g/L for B40, 863.9 g/L for B100, and 843 g/L for B0. This means that after storage for 35 days, all fuels still have density values within the range of national standard, namely 815-

870 g/L for B0, 815-880 g/L for B40, and 850-890 for B100. However, this results suggest that the fuels must be stored in a closed container and protected from exposure to light and ambient air so that damage to the biodiesel during storage can be minimized.

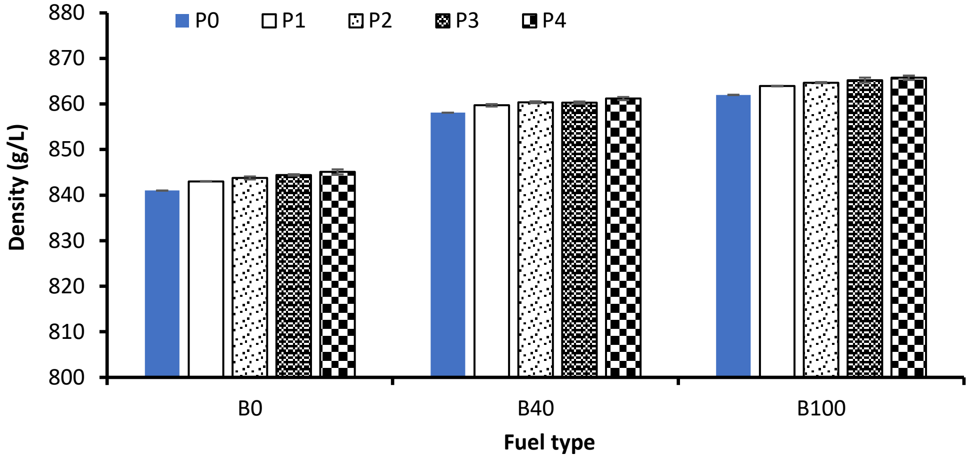

Figure 2. Effect of storage conditions on the density of fuels (B0, B40, B100) after 35 days (P0 is initial value at day zero).

Several previous studies have also reported a small increase in fuel density during storage. For example, the density of biodiesel mixture B40 stored at temperature 30 °C in transparent glass bottles increased from around 863 g/L to 869 g/L or an increment of 6 g/L for 9 weeks of storage period (Khalid *et al*., 2013). Ashraful *et al*. (2014) also reported that palm biodiesel mixture B40 had a density of 793.9 g/L in the first week of storage, and increased slightly to 797.3 g/L (an increase of 3.4 g/L) after 12 weeks of storage. Another study reported a maximum increase of 25 g/L in the density of some biodiesel blends after storage for 12 weeks at high temperature (40-50 °C) (Zakaria *et al*., 2014). Many other studies reported similar thing about little increase of biodiesel density due to storage including Wu *et al*. (2011), Fathurrahman *et al*. (2024), Ndana *et al*. (2012), Jose & Anand (2016), Kassem *et al*. (2018), Mazumdar *et al*. (2013), and Komariah *et al*. (2017).

The increase in biodiesel density during storage can be caused by several factors. Biodiesel can undergo oxidation when exposed to oxygen from the air. This process produces compounds such as peroxides, fatty acids, and aldehydes, which have a higher density, thus increasing the density of biodiesel. During storage, the water content of biodiesel increases due to the hygroscopic nature of biodiesel (absorbing water from the surrounding environment) and because the degradation of biodiesel also produces water. Water has a higher density than biodiesel and when mixed with biodiesel adds mass without significantly increasing volume, thereby increasing density.

3.3. Acid Number

The acid number of fuel can be used as an indicator to determine the level of fuel degradation. Figure 3 shows changes in the acid value of the fuel after storage for 35 days. It is worth noting that the acid value of B100 is lower than those of other fuels (B0 and B40). Biodiesel generally has a higher acid number as compared to petro diesel. The acid number is a measure of the amount of free fatty acids or other acidic substances present in the fuel. In biodiesel, the acid number can be higher due to the presence of residual free fatty acids from the feedstock oil or fat used in its production. This is occurred especially for biodiesel made from low-quality feedstocks or if it has been stored for a long time, which can lead to the formation of additional acids through hydrolysis or oxidation. Petro diesel, being a refined product from crude oil, typically has a lower acid number since it undergoes extensive processing to remove impurities, including acidic components. High acid numbers in fuels can be problematic as they may cause corrosion and other issues in engines and fuel systems. Ashraful *et al*. (2014) reported a similar thing where the acid number value of B100 was lower than B40 which contains 60% petro diesel.

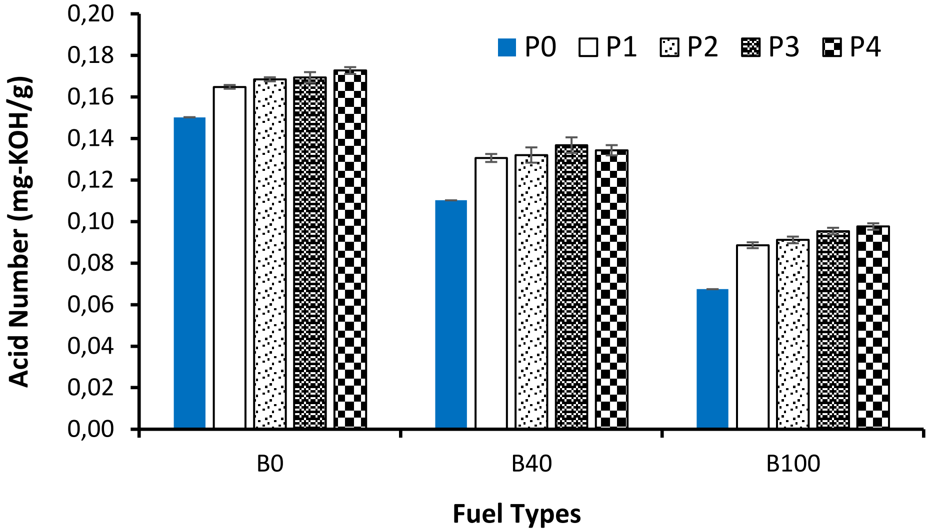

Figure 3. Effect of storage conditions on the acid number of B0, B40, and B100 after 35 days storage (P0 is initial density value (day zero) and standard deviation of three replications are presented by error bars).

All fuels showed an increase in acid number values after storage. However, the final acid number is still lower than the limit of the SNI standard (0.6 mg KOH/g) for petro diesel (B0) and biodiesel blend (B35), and 0.4 mg KOH/g for biodiesel B100. ANOVA analysis showed that storage conditions significantly affected the acid number with a significance value of 0.041 ($p<0.05$). Storage condition P4 (no cap, no wrap) produced the highest acid number value, namely 0.172 mg-KOH/g, 0.134 mg-KOH/g, 0.098 mg-KOH/g, respectively for B0, B40, and B100. Treatment P1 (with cap, with wrap) produced the lowest increase in acid number for all fuels, namely 0.164 mg-KOH/g (B0), 0.131 mg-KOH/g (B40), and 0.089 mg-KOH/g (B100). Similar things were reported by Leung *et al.* (2006) where the acid number value of biodiesel stored in conditions exposed to air and water experienced a much higher increase compared to sealed storage. The increase in acid number was also reported by several other researchers. Ashraful *et al.* (2014) reported an increase in acid number due to storage for three types of biodiesel and its mixtures, including jatropha, palm oil, and coconut oil biodiesel. Palm oil biodiesel (B100) increased its acid number from 1.03 mg KOH/g (week 1) to 3.01 mg KOH/g (week 12). In the same period, its blend (B40) increased its acid number from 3.17 to 5.13 mg KOH/g, while B0 diesel oil increased from 0.59 to 1.93 mg KOH/g. Bouaid *et al.* (2007) also reported an increase in acid number that exceed the standard at week 12 for 5 biodiesel samples stored under exposed to light, while in dark storage only 1 biodiesel sample with an acid number exceeding the standard after 12 weeks of storage.

The increase in acid number is an indication that the fuel has degraded during storage. According to Ashraful *et al.* (2014), the increase in acid number is triggered by the increase in hydroperoxides oxidized to fatty acids. Biodiesel will be oxidized when exposed to air. Acids can also be formed when esters are hydrolyzed by water into glycerol and acids (Bouaid *et al.*, 2007; Zakaria *et al.*, 2014). Meanwhile, Wu *et al.* (2011) showed that high temperatures is important factor that contributes to the increase in acid number during storage. Storage time is another factor that increase acid number: the longer, the higher. Ashraful *et al.* (2014) revealed a linear relation between acid number and storage time.

3.5. Color

Color can be used as an indicator of the quality of a fuel. The standard used to measure fuel color is ASTM D1500, which provides a color scale ranging from 0.5 to 8.0 based on measurements using the coulometry method. Fuel with a low color value (0.5 - 3.0) has a clear or slightly yellow visual appearance indicating that the fuel has a high level of purity and is slightly contaminated by substances such as sulfur or oxidation that can cause dark colors. The higher the color value (3.0 - 5.0) of the fuel indicates the presence of contaminants. Darker fuel colors (values 5.0 - 8.0) can be caused by oxidation, the presence of high sulfur, or the presence of other impurities.

Figure 4. The visual color of fuels, before (top) and after (bottom) storage. (From left: biodiesel mixture B40, neat biodiesel B100, petroleum diesel B0).

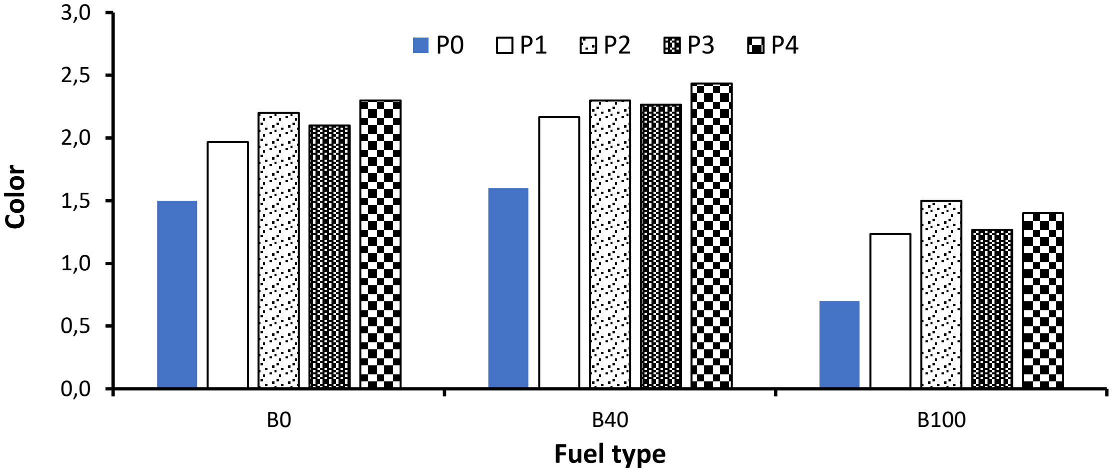

Figure 5. Effect of storage in open air and light conditions on the color values of B40, B100 and B0

Figure 4 depicts the visual of color change of the fuels after 35 days stored in different conditions. As detailed in Figure 5, initially all fuels were clear with low color values (<3.0). Pure biodiesel fuel (B100) reveals the clearest one with lowest color value of 0.7 and significantly lower than that of pure diesel fuel (B0) with color value of 1.5. Biodiesel mixture B40 had a color value similar to B0, namely 1.6. These results support the research reported by Effriandi *et al.* (2019) where biodiesel B100 had color value of 0.5 and biodiesel mixture B20 had a color value of 1.5. Figure 4 also shows that after 35 days of storage, the color values of all fuels increased by about 0.5 points from their initial values. This means that the clarity of the fuel color has decreased, indicating changes that lead to degradation and reduce its quality. According to Effriandi (2019), the color of biodiesel stored for a certain period of time will change due to oxidation. Biodiesel is more susceptible to oxidation biodegradation than diesel oil. This is due to the high content of unsaturated ester compounds containing many double bonds that are susceptible to oxidation. During the storage process, the biodiesel degradation process occurs, which is characterized by a change in color and a higher color value.

The results of the ANOVA analysis showed that the storage condition treatment factor had no significant effect on the color of the fuel after 35 days of storage. The results of the study also concluded that the color values of all fuels increased, but was still in the clear range with a color value of less than 3.0.

3.4. Heating Value

Heating value is the total amount of energy released from a fuel through a complete combustion reaction. Figure 6 demonstrates calorific value of the three fuels (B0, B40, and B100) before and after storage under different container conditions. Results from ANOVA analysis reveals that fuel types as well as storage container conditions are significant on the calorific value. The type of fuel determines its calorific value, where petroleum diesel fuel (B0) has the highest calorific value, which is 10,487 cal/g. While pure biodiesel fuel B100 has the lowest calorific value (9,888 cal/g). The calorific value of the biodiesel used in our study was slightly higher than some types of biodiesel reported by Oliveira & da Silva (2013), including soybean biodiesel (9,436 cal/g), jatropha (9,430 cal/g), rapeseed (9,431 cal/g), and crambe (9,695 cal/g). Chemical composition of the fuels is responsible for the lower calorific value of the biodiesel fuels. Biodiesel is primarily composed of fatty acid methyl esters (FAME), which have different chemical structures compared to the hydrocarbons in petroleum diesel. The carbon chains in biodiesel molecules are typically longer and contain more oxygen atoms, whereas petroleum diesel consists mostly of hydrocarbons with little or no oxygen. The presence of oxygen in biodiesel reduces the amount of energy that can be released during combustion because oxygenated compounds have lower energy densities.

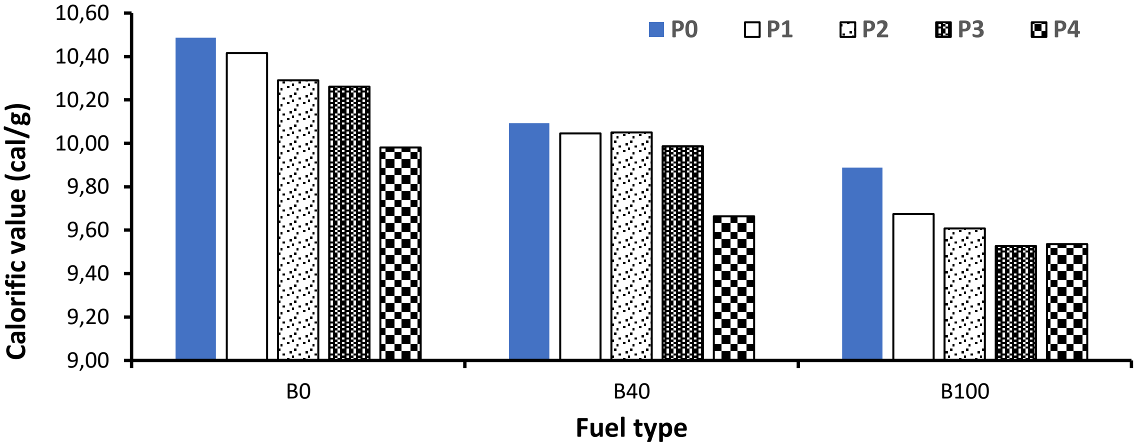

Figure 6. Effect of storage conditions on the calorific value of B0, B40 and B100 fuels after storage of 35 day

The storage condition treatment factor also has a significant effect on the calorific value. Storage in an open glass container and without an aluminum foil blanket (P4) has produced the lowest calorific value for all types of fuel. Under storage condition of P4 (no cap, no wrap), the fuel experienced a decrease in calorific value of 4.83% for petro diesel (B0), 4.25% for B40, and 3.56% for B100. This decrease in calorific value is related to changes in the values of the quality parameters that have been discussed previously. The increased water content of the fuel is directly related to the decrease in calorific value because some of the chemical energy of the fuel is used to evaporate water during combustion. The increase in density causes the fuel to become heavier, so that the specific calorific value of the fuel (cal/g) becomes lower. Increased acid number is related to the presence of free fatty acids (FFAs), compounds that are partially oxidized and contain oxygen. Since energy is released during combustion primarily from the oxidation of carbon and hydrogen, the presence of oxygen in FFAs means that these molecules have less potential energy compared to hydrocarbons that are not oxidized. Ultimately, this reduction in quality results in an accumulative effect in the form of a decrease in the specific calorific value of the fuels.

The national standard required that neat biodiesel (B100) should have a minimum calorific value of 9123 cal/g. After storage for 35 days, all fuels demonstrated a decrease in calorific value but did not exceed the SNI standard. This means that even though degradation and quality reduction occur due to storage, biodiesel fuel still has a calorific value that meets SNI standards. The decrease in fuel calorific values were also reported by other researchers, including Wu *et al.* (2011), Silviana & Buchori (2015), Ndana *et al.* (2012), and Jose & Anand (2016). Table 1 summarizes the effect of storage on the calorific value of fuels along with storage condition. Our results were in the range of the reported values.

Table 2. Comparison of fuel calorific values before and after storage

Biodiesel type	Storage condition	Calorific value (cal/g)		Decrease		References
		Initial	Final	(cal/g)	(%)	
Palm oil (B100)	Clear glass, no cap, room temperature, 7 weeks	9,888	9,536	352	3.56	This work
Palm oil (B40)		10,093	9,664	430	4.25	This work
Petro diesel (B0)		10,487	9,980	507	4.83	This work
Sun flower (B100)*	Polypropylene bottle, 25 C, 32 weeks	9,468	9,457.5	11	0.11	Wu *et al.* (2011)
Sun flower (B100)*		9,468	8,979.3	489	5.16	Wu *et al.* (2011)
Soybean (B100)*		9,465	9,430.8	34	0.36	Wu *et al.* (2011)
Soybean (B100)*		9,465	9,339.4	126	1.33	Wu *et al.* (2011)
Sun flower (B100)*	Polypropylene bottle, 40C, 32 weeks	9,463	9,444.1	19	0.20	Wu *et al.* (2011)
Sun flower (B100)*		9,463	8,849.5	614	6.48	Wu *et al.* (2011)
Soybean (B100)*		9,463	9,424.0	39	0.41	Wu *et al.* (2011)
Soybean (B100)*		9,463	9,261.5	202	2.13	Wu *et al.* (2011)
Palm oil (B100)	PVC (open), 2 month	9,528.84	9,482.85	45.99	0.48	Silviana & Buchori (2015)
Palm oil (B100)	PVC (closed), 2 month	9,528.84	9,462.69	66.15	0.69	Silviana & Buchori (2015)
Palm oil (B100)	Galvanic (open), 2 month	9,528.84	9,478.61	50.23	0.53	Silviana & Buchori (2015)
Palm oil (B100)	Galvanic (closed), 2 month	9,528.84	9,514.59	14.25	0.15	Silviana & Buchori (2015)
Castor oil*	Plastic rubber container, 10 weeks	9,505	8,652	853	8.97	Ndana *et al.* (2012)
Jatropha oil*		9,737	9,372	365	3.75	Ndana *et al.* (2012)
Neem oil*		9,609	9,362	247	2.57	Ndana *et al.* (2012)
Soybean oil*		9,535	8,805	730	7.66	Ndana *et al.* (2012)
Rubber seed oil*		9,446	8,785	661	7.00	Ndana *et al.* (2012)
Cotton seed oil*		10,003	9,382	621	6.21	Ndana *et al.* (2012)
Coconut (B100)	Glass container, contact to air and light, 1 year	9,479	8,365	1,114	11.75	Jose & Anand (2016)
Karanja (B100)		9,097	8,748	349	3.84	Jose & Anand (2016)

*) The cited references provided calorific value in form of graphs. The values listed in this table are best prediction extracted from the graphs using WebPlotDigitizer application from https://automeris.io/wpd/

4. CONCLUSIONS

Based on the research that has been done, it can be concluded that fuel quality decreases due to storage. This is indicated by an increase in the value of water content, density, acid number, and color parameters, as well as a decrease in the specific calorific value of biodiesel and its mixtures. The condition of the storage container affects the changes in these quality parameters. Based on its impact on fuel quality decline, the order of storage container conditions from the best is P1 (with cap, with wrap) > P2 (with cap, no wrap) > P3 (no cap, with wrap) > P4 (no cap, no wrap). During 35 days of storage, it can be seen that petro diesel fuel (B0) has better stability compared to pure biodiesel (B100) and its mixture (B40). Storage treatment with closed bottles and without exposure to light can reduce the rate of fuel damage.

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