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7 Prediction of Palm Based Biodiesel Properties Through the Preparation of Empirical Equation

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ABSTRACT

Biodiesel is one of plant-based fuel that has been made mandatory by the Indonesian government for using in transportation, industries, and power plants. This obligation is gradually applied as stipulated in the regulation of the Ministry of Energy and Mineral Resources 12/2015 regarding to supply, use and plant-based fuel trade as alternative fuel. The percentage of biodiesel use was increasing from 20 % in 2016 to 30 % in 2030. The quality of biodiesel has to be continuously improved to support the regulation. Biodiesel with high performance has excellent flame characteristics, high oxidation stability and easy to flow at low temperature. These characteristics are highly determined by the composition of fatty acid methyl esters (FAME). Empirical equations were formulated based on primary data to predict the essential parameters of biodiesel, consisting of oxidation stability, cloud point, Iodine value, viscosity and density. The primary data were obtained by analyzing three biodiesel samples from domestic producers and two samples of biodiesel blend between palm stearin and soybean biodiesel. Identification results showed that all three samples have different FAME profiles which consist of palm oil biodiesel, palm stearin biodiesel and palm olein biodiesel. The empirical equations were formulated using statistical software and validated by comparing the calculated results with actual biodiesel parameters. This method reduced the testing time up to 13 working days and reduced testing cost up to 67% or IDR 1 190 000 per sample. The empirical equations formulated in this study were able to predict the essential parameters of biodiesel with the error was.

Keywords: essential parameter, FAME, palm oil, palm olein, palm stearin

INTRODUCTION

Biofuel can be Indonesia's flagship energy product that has strategic value because it is a renewable energy source that can produce liquid fuels (Joelianingsih *et al.* 2018). The biofuel that has been developed can be mixed into its equivalent fuel and does not require new commercial

infrastructure because it can use existing infrastructures such as depots, tankers, piping systems, tankers and gas stations. Biodiesel is one type of biofuel that has been produced and required for its use in Indonesia for the transportation, industrial and power generation sectors.

The mandatory program on the use of biodiesel in Indonesia has two benefits.

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First, supporting new and renewable energy policies through an energy mix that encourages the creation of national energy security, and second, encouraging stabilization of crude palm oil (CPO) prices through the influence of controlling the demand and supply of palm oil and derivative products.

There are several types of palm-based biodiesel raw materials based on their fatty acid composition, such as CPO Refined bleached deodorized palm (RBDP) oil, RBDP olein, RPDP stearin and super olein. While the types of palm oil based on its quality can be divided into, Refined palm oil/olein/stearin, high fatty acid crude palm oil (HFCPO), palm fatty acid distillate (PFAD), palm sludge oil (PSO)/CPO trench and used cooking oil (Che Man *et al.* 1999).

The characteristics and parameters of each biodiesel are different, depending on the raw material. There are several types of the parameter used to identify the characteristics of biodiesel, namely, parameters that are affected by the biodiesel production process, parameters that are influenced by the level or composition of fatty acid methyl ester (FAME), as well as parameters that are affected by both. Parameters that are influenced by the level or composition of FAME are viscosity, density, cloud point, iodine value, cetane number and oxidation stability (Ramos *et al.* 2009; Hong *et al.* 2014; Joelianingsih *et al.* 2015). While the biodiesel production process influences other parameters such as free glycerol levels, total glycerol, acid numbers, and monoglyceride levels, both at the raw materials preparation stage, the reaction process and at the biodiesel purification stage. If biodiesel has met the FAME level (minimum 96.5 wt. %), then the characteristics of biodiesel derived from the same raw material will have same characteristics of viscosity,

density, cloud point, iodine value, cetane number and oxidation stability. Among these parameter parameters, the effect of the composition of FAME on viscosity and density is minimal. Contrary, the effect of the FAME composition is very significant on the cloud point, cetane number, iodine value and oxidation stability.

Determination and process sequence to obtain characteristics of biodiesel is not a short process because various types of tests are needed to verify its quality. Several tests were carried out mainly for biodiesel producers to obtain biodiesel characteristic data to meet applicable standards and regulations in their country. Fulfillment of standards is undoubtedly essential because it is closely related to whether a product is to be marketed. The costs required for testing are prohibitive considering the complexity of the tests performed. Therefore, a new method is needed to minimize testing costs. One of the breakthroughs is to know the relationship between the characteristics of biodiesel one with the other characteristics in the form of empirical formulations or equations. The empirical equation of the relationship between biodiesel characteristics requires only a few data to obtain the overall characteristics of a biodiesel sample. The developed equation was validated by comparing predictions of correlation to experimental data in the literature.

Modeling in an empirical equation requires limited and constant effectiveness, even if used repeatedly. The empirical equation is expected to have a small deviation value at the level of percent error and specific to each character to be determined. Some characteristic data is needed to get the overall characteristics of biodiesel. So, the results obtained reflect laboratory test characteristics. The ease of use is also a standard in the sustainability of biodiesel application in the

industry. Not only to minimize costs and time in use, but accuracy is also expected.

Several researchers have reported several empirical equations study. Ramos *et al.* (2009) compiled empirical equations to predict the value of cetane number, iodine value, oxidation stability and cold filter plugging point (CFPP) as a function of FAME composition of 10 types of vegetable oil as raw material for biodiesel. García-Martín *et al.* (2019) have developed empirical equations to predict the value of cetane number as a function of the composition of FAME derived from olive and sunflower oil. Based on the literature above, the empirical equation for palm-based biodiesel has not been reported previously.

Based on the characteristics described above, several characteristics will be examined in this study, namely the influence of the composition of FAME on oxidation stability, cloud points and iodine numbers. The empirical equation for these five parameters for palm-based biodiesel never been studied before, and it become our novelty for this work. The reason for choosing these characteristics is because the value of FAME on biodiesel can be readily determined from the value of oxidation stability, cloud point and iodine number. Three types of palm-based biodiesel were used in this work, which consists of palm oil biodiesel (PB), palm stearin biodiesel (PSB) and palm olein biodiesel (POB), and they randomly sell in the biodiesel market in Indonesia. Finally, empirical equation for these five properties become the target outputs.

MATERIALS AND METHODS

Materials

Three random samples of pure biodiesels used in this study were obtained from

three different Indonesian biodiesel producers whose product has been certified to meet the SNI 7182:2015 standard. Soybean Biodiesel (SB) (Agnique® ME18S-U Methyl Soyate) was a commercial product which was purchased from BASF (Mississauga, Canada). The internal standard solution used for characterizing the ester which was produced by SIGMA (St. Louis, Missouri, USA).

FAME Analysis

Composition and content of FAME in biodiesel were analyzed using gas chromatography (GC) Shimadzu GC-2010 (Kyoto, Japan). All samples were analyzed in the Chemical Engineering Laboratory, Institut Teknologi Indonesia. The GC analysis method for determining the methyl ester contents in biodiesel complies with EN14103 (2011) method where this method is suitable for FAME which contains methyl esters between C₆ and C₂₄ (EN 14103 2011; Garcia-Martin *et al.* 2019). The substrate that was used for FAME analysis was split into an analytical column Rtx®-WAX, RESTEK (Pennsylvania, USA) capillary column (30m×0.32mm internal diameter×1µm film thickness) was used. The column temperature was set to 185 °C, and then the temperature program ramped from this temperature to 240 °C at 5 °C min⁻¹. The injection was operated in a splitless mode, the injector and detector temperatures being 250 °C. The substrate that was used for FAME analysis was split into a capillary column with a polar stationary phase and flame ionization detector (FID). In this study, the GC was calibrated using nonadecanoic acid methyl esters (FAME C₁₉) as the FAME standard. The ester C content, expressed as a mass percentage, was calculated using equation.

$$C = \frac{\sum A_{EI}}{A_{EI}} \times \frac{W_{EI}}{W} \times 100$$

where ΣA is the total peak area of the methyl ester in $C_{6:0}$ to that in $C_{24:1}$; AEI is the peak area corresponding to nonadecanoic acid methyl ester; WEI is the weight, in milligrams, of the nonadecanoic acid methyl ester being used as the internal standard; W is the weight, in milligrams, of the sample.

Physicochemical Characterization

Several physicochemical parameters of the biodiesel sample such as kinematic viscosity, density, cloud point, iodine value, and oxidation stability were analyzed following AOCS Cd 1-25 (1993), EN 15751 (2014), ASTM D5773 (2017), ASTM D445 (2018) and ASTM D4052 (2018), respectively. All samples were analyzed in the LEMIGAS Research and Development Centre for Oil and Gas Technology, Jakarta.

Preparation and Validation Empirical Equation

An equation to explain the relationship between the composition of FAME as an independent variable, and the critical parameters of biodiesel as a dependent variable, compiled from the results of data preparation using the Microsoft Excel application. The equation obtained is used to obtain a predictive value where the predictive value is compared with the measured value to obtain the average deviation rate (% error). The complete schematic of this research steps can be shown in Figure 1.

RESULTS AND DISCUSSION

Fatty Acid Composition from Pure Biodiesel

The results of the FAME composition from three palm-based biodiesel are presented in Table 1. The first three biodiesel (sample A, B and C) are pure biodiesel with different types, following by other three

biodiesel, namely CPO, POB, and PSB as comparative data (Salunkhe *et al.* 1992; Basiron 2005). The FAME composition in the form of methyl palmitate and methyl oleate can be used as a determinant of the type of biodiesel material. Both have specific values that make them different from other types of raw materials affected by the distribution of carbon amount from fatty acids (Bamgboye & Hansen 2008). PSB is a heavy fraction produced by biodiesel palm oil fractionation which has a higher amount of methyl palmitate composition than the composition of biodiesel palm oil (Cardoso *et al.* 2014). Its methyl palmitate composition also higher than the composition of methyl oleate but has a lower methyl oleate composition. Palm olein biodiesel, which is a mild fraction from the fractionation process of the palm oil biodiesel, has an equal value of methyl palmitate and oleate composition or tends to approach the same value (Basiron 2005). The composition of the biodiesel samples was then compared with the FAME composition of comparative data from the literature.

From that table, it can be seen that sample A is included in the type of PSB. Biodiesel made from palm stearin has unique characteristics, where the percent composition of palmitic acid is higher compared to CPO and POB. This type was indicated by the value of the palmitic acid composition in sample A which in the range of palm stearin range, 47.2-73.8% $m\ m^{-1}$, where its composition results were 56.26% $m\ m^{-1}$. Unlike the case with other biodiesel samples, sample B have different characteristics which can be seen in the percent composition of oleic acid. The oleic acid is in the range of POB composition, i.e., 40.7-43.9% mm with the oleic acid composition of 42.62% $m\ m^{-1}$. Whereas for Sample C having characteristics that resemble CPO biodiesel. It can be seen from the percent composition of

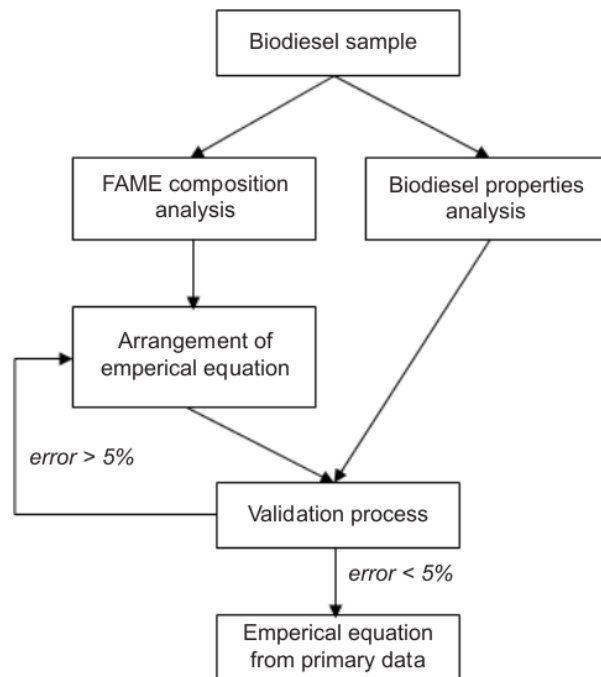


Figure 1 Schematic of research steps.

Table 1 FAME composition from three different samples of palm-based biodiesel

FAME	Formula	Sample A	Sample B	Sample C	CPO [12]	POB [13]	PSB [12]
Methyl caprate	C10:0	0.00	0.00	0.10	n/a	n/a	n/a
Methyl laurate	C12:0	0.13	0.16	0.10	< 1.2	0.1 – 0.5	0.1 – 0.6
Methyl miristate	C14:0	1.11	0.71	0.89	0.5 – 5.9	0.9 – 1.4	1.1 – 1.9
Methyl palmitate	C16:0	56.26	41.72	44.04	32 – 59	37.9 – 41.7	47.2 – 73.8
Methyl palmitoleate	C16:1	0.21	0.11	0.17	< 0.6	0.1 – 0.4	0.05 – 0.02
Methyl stearate	C18:0	5.15	5.92	4.71	1.5 – 8	4.0 – 4.8	4.4 – 5.6
Methyl oleate	C18:1	29.93	42.62	39.75	27 – 52	40.7 – 43.9	15.6 – 37.0
Methyl linoleate	C18:2	6.38	8.21	9.93	5 – 14	10.4 – 13.4	3.2 – 9.8
Methyl linolenate	C18:3	0.83	0.54	0.32	< 1.5	0.1 – 0.6	0.1 – 0.6

n/a: not available

palmitic acid which is in the range 32-59% m m⁻¹ with the palmitic acid composition value of 44.04% m m⁻¹ (Salunkhe et al. 1992).

Fatty Acid Composition from Blend Biodiesel

Besides of pure biodiesel, blend biodiesel was also prepared to be analyzed its FAME composition. PSB was cho-

sen to be blended with soybean oil (SB) because of three factors; its price is the cheapest than POB or CPO, has high oxidation stability, and has a low cloud point (Chetpattananondh & Tongurai 2008; Pattamaprom et al. 2012). For that, PSB was blended with SB in several mass ratios; 95:5 and 90:10 and named as Sample D and E, respectively. The mixing process was performed in a beaker glass for

15 minutes with 500 rpm of agitation. The FAME composition from SB and blend biodiesel samples (Sample D and Sample E) are shown in Table 2. Theoretical results obtained by multiplying mass fraction and composition from their pure form (PSB and SB) and then sum it up, while actual result obtained from GC analysis. From the results, there are two things which can be noticed. First, blend biodiesel shows that the composition value is not much different from the theoretical results. It can be proved by % error value which shows the percentage deviation between the theoretical FAME composition the value and the actual was low, and it was in the tolerance range. Second, the FAME composition of Sample D and E were more or less similar to PSE since PSE is the dominant fraction in these blend biodiesel.

The Physical Characteristic of Biodiesel

The Oxidation Stability (OS), Cloud Point (CP), Iodine Value (IV), viscosity (η)

and density (ρ) test results of the five biodiesel samples are presented in Table 3. Oxidation stability is one of the essential criteria for biodiesel quality. It depends on the number of bonds included in biodiesel. The oxidation of biodiesel can produce compounds from acid decomposition, aldehydes, esters, ketones, peroxides, alcohols, which can affect the characteristics of biodiesel and combustion activities in the engine (Chuck *et al.* 2012). Oxidative degradation during long-termed storage can be occurred due to air, heat, light and pro-oxidant substances (Knothe 2007). In Table 3, oxidation stability in Samples A, B and C (pure biodiesel) has a value range of 16-18 hours whereas the blended biodiesel has a lower oxidation value of 9.95 and 8.53 hours, respectively. Reduce the oxidation mixture of the biodiesel mixture containing SB which has unstable oxidation stability compared to PSB. However, all samples have a value higher than the global standard set, for example, minimum 3 hours according to ASTM D6751-12

Table 2 FAME composition from SB and blend biodiesel

FAME type	SB	Actual	Sample D theoretical	% error	Actual	Sample E theoretical	% error
C10:0	n/a	0.00	0.00	0.00	0.06	0.00	100
C12:0	n/a	0.13	0.12	8.33	0.13	0.11	18.18
C14:0	0.07	1.00	1.06	5.67	1.16	1.01	14.85
C16:0	10.83	55.33	53.99	2.48	51.41	51.72	0.60
C16:1	0.21	0.33	0.21	57.14	0.40	0.21	90.48
C18:0	4.18	4.97	5.11	2.74	5.27	5.06	4.15
C18:1	22.73	29.65	29.57	0.27	28.54	29.21	2.29
C18:2	53.17	7.86	8.72	9.86	11.86	11.06	7.23
C18:3	8.81	0.86	1.23	30.08	1.17	1.62	27.78

Table 3 Test results of key parameters of biodiesel

Sample	Density (kg m ⁻³)	Viscosity (mm ² s ⁻¹)	Cloud Point (°C)	Iodine Value g (I ₂ per 100 g biodiesel)	Oxidation Stability (h)
A	865.60	4.74	17.4	44.75	17.00
B	857.00	4.44	13.2	53.28	16.00
C	857.50	4.59	13.3	52.11	18.00
D	857.80	4.58	17.2	50.63	9.95
E	857.10	4.50	16.8	55.20	8.53

(ASTM D 6751 2012) and a minimum of 8 hours according to EN 14214:2012 (EN 14214 2014).

Cloud point is the temperature at which crystals first begin to form in fuel, usually at low temperatures. When at low temperatures, the potential for the precipitation of wax crystals is quite significant (Bhale *et al.* 2009). The decrease in temperature causes the growth of these wax crystals. These crystals may clog filters on storage tanks or combustion chambers. From Table 3, the CP was in the range 13.2-17.4 °C. According to literature, biodiesel commonly has a cloud point in range 13-16 °C (Barabás & Todorut 2011)

Iodine value has a close connection with the oxidation stability of biodiesel. IV is the total unsaturated fatty acid measured in iodine per 100 g of the biodiesel sample. The IV value is very dependent on the nature and composition of FAME from the biodiesel feedstock. Biodiesel which has a high IV is easily oxidized when exposed to water and tends to polymerize and precipitate the shape of the injector nozzles and combustion chamber [24]. The biodiesel IV from all sample was in the range between 44.75-55.20 g I₂ per 100 g biodiesel. It is lower than the global standard of biodiesel IV according to EN 14214:2012, with a value of 120 g I₂ per 100 g biodiesel (EN 14214 2014), means the quality of all samples are good because difficult to be oxidized.

Viscosity is an important parameter because it affects the size of droplets and penetration and the quality of atomization (Park *et al.* 2007). High viscosity causes poor atomization of fuel because it makes larger droplets, thus increasing the engine work which causes the pump and injector energy increased. Increased in viscosity can be measured directly from the number of carbon atoms in FAME caused by the van der Waals force in the non-polar

part (Ramírez-Verduzco *et al.* 2012). Saturated FAME has a higher viscosity than unsaturated FAME for the same number of carbon atoms (Yuan *et al.* 2009). From Table 3, the viscosity of all samples has a range between 4.44-4.74 mm² s⁻¹. This value meets with biodiesel standard based on ASTM D6751-12 (ASTM D6751, 2012).

The mass of fuel injected into the fuel chamber and fuel-ratio are two things that are affected by fuel density (Alptekin & Canakci 2008). That is because the fuel pump injection meter is calculated based on volume, not mass. High-density biodiesel contains higher mass in the same volume. Increased density of biodiesel is accompanied by a decrease in molecular weight and an increase in FAME unsaturation level and it is always contrariwise proportional to the number of carbon atoms (Rocabruno-Valdés *et al.* 2015). The density of all samples has a range between 857.10-0.865.60 kg m³·⁻¹. This value meets with biodiesel standard based on EN 14214 (2014).

Empirical Equation Calculation

The FAME results from GC analysis was used in this study to make an empirical equation. Based on the test results obtained the composition of FAME and the value of essential parameters as presented in Table 4. Based on the FAME composition data from five parameters, an equation was obtained using the Microsoft Excel application, summarized in Table 5. From that table, the thing can be noticed that C14, C16:0, C18:1 and C18:2 are the methyl ester which affects to physical properties of biodiesel and it is compatible with the literature (Mohammadi & Najafi 2015). Moreover, the error percentage of empirical equations are low with a range from 0 to 0.52%, means these empirical equations are robust enough.

Table 4 The FAME composition from all samples

Sample	C10 (X ₁)	C12 (X ₂)	C14 (X ₃)	C16:0 (X ₄)	C16:1 (X ₅)	C18:0 (X ₆)	C18:1 (X ₇)	C18:2 (X ₈)	C18:3 (X ₉)
A	0.00	0.13	1.11	56.26	0.21	5.15	29.93	6.38	0.83
B	0.00	0.16	0.71	41.72	0.11	5.92	42.62	8.21	0.54
C	0.10	0.10	0.89	44.04	0.17	4.71	39.75	9.93	0.32
D	0.00	0.13	0.99	55.33	0.33	4.97	29.65	7.86	0.86
E	0.06	0.13	1.16	51.41	0.40	5.27	28.54	11.86	1.17

Table 5 Empirical equations of biodiesel characteristic

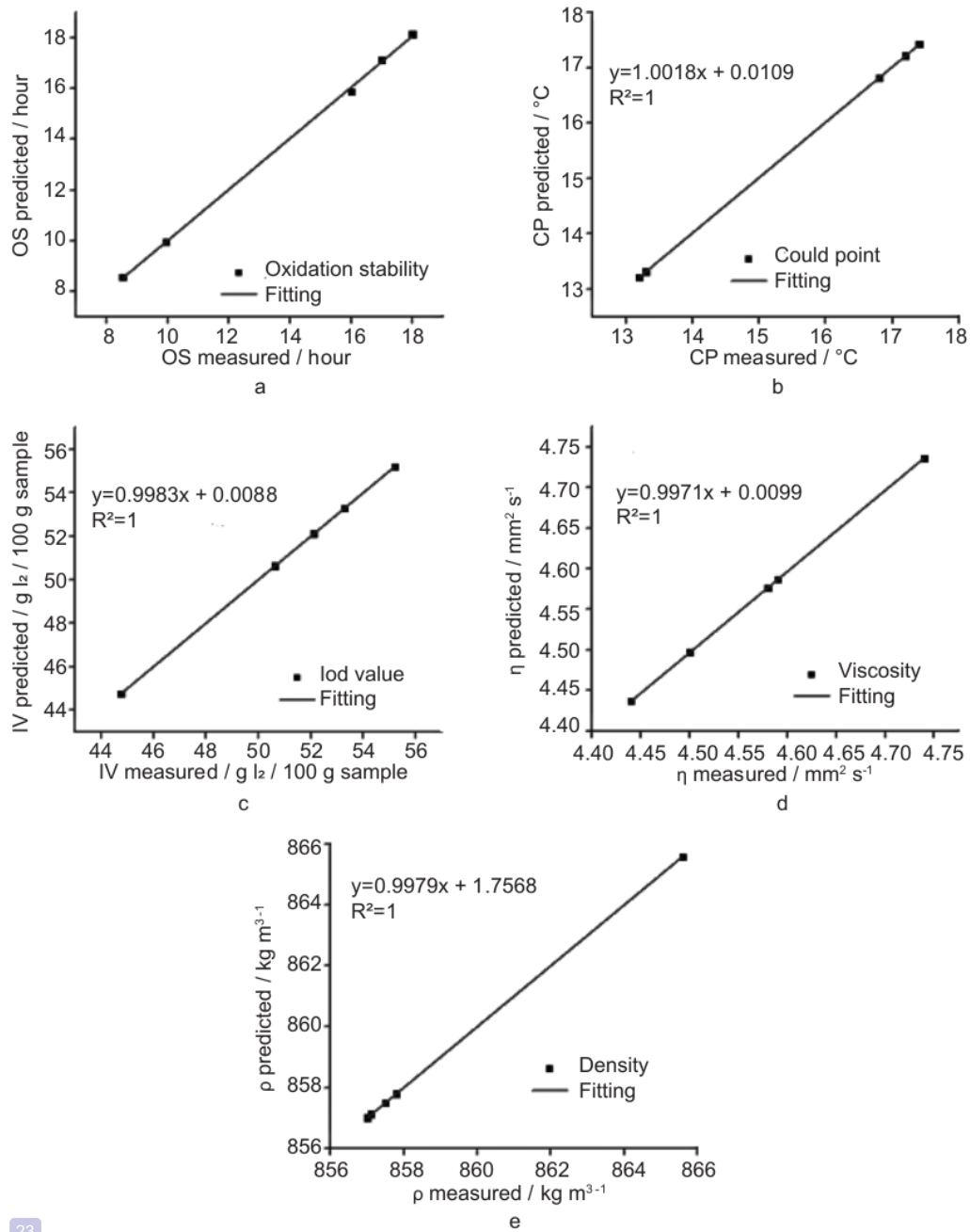
Parameters	Equations	% error
Oxidation stability (OSI)	$OSI = -162.471 + 42.293X_3 + 1.062 X_4 + 2.5024 X_7 - 0.334 X_8$	0.52
Cloud point (CP)	$CP = 53.716 - 0.892 X_3 - 0.26X_4 - 0.588X_7 - 0.482 X_8$	0.11
Iod value (IV)	$IV = 149.694 - 26.749X_3 - 0.7975 X_4 - 1.2462 X_7 + 1.09876 X_8$	0.01
Viscosity (η)	$V = -2.769 + 0.9866X_3 + 0.0676 X_4 + 0.0809 X_7 - 0.0286 X_8$	0.07
Density (ρ)	$\rho = 897.317 + 39.4679 X_3 - 0.96185 X_4 - 0.13386 X_7 - 2.75065 X_8$	0.00

The obtained empirical equation can be used to predict the value of important palm-based biodiesel parameters, while the correlation between measured and predicted value shown in Figure 2a-e. Generally, all property data are accumulated near $y=x$ equation where the fitting line has near y -intercept and one slope. From those figures, all samples have coefficient determination (R^2) with a range between 0.9995 to 1. The higher R^2 shows high linearity of the model and high accuracy. In this case, the empirical equation model was made proven to be accurate in predicting the physical properties of palm-based biodiesel.

Testing Time and Testing Cost Reduction

The purpose of this study is to reduce the testing time and the cost of testing biodiesel properties. By using empirical equations, the cost and time of testing can be saved so that biodiesel testing with large samples can be done in a

short time. As explained in the previous section, GC analysis was used to identify the composition of the FAME, it has been carried out at the Institut Teknologi Indonesia, and tests for biodiesel properties using standard methods have been carried out at LEMIGAS. A comparison of the testing costs between the two methods is shown in Table 6. From that table, the total cost of using GC is IDR 600 000 and the total cost of biodiesel properties using the standard method is IDR 1 790 000. Means, determining the nature of biodiesel by using an empirical equation saves 67% or Rp1 190 000. In addition, GC analysis only needs one working day to find out the results of the composition of the FAME and then incorporate it into the empirical equation. However, it took longer about 14 working days to test the properties of biodiesel using standard methods. That is, the testing time can be saved around 13 working days. This is an advantage to test biodiesel applications on a mass scale.



23 Figure 2 The relationship between measured and predicted values of a Oxidation stability; b Cloud point; c Iodine value; d Viscosity; e Density.

Table 6 Cost and time testing for biodiesel

Parameters	Testing method	Cost per sample (IDR)	Time (day)
Oxidation stability (OSI)	EN 15751	920 000 ^a	
Cloud point (CP)	ASTM D5773	225 000 ^a	14 ^a
Iod value (IV)	AOCS Cd 12-5	300 000 ^a	
Viscosity (η)	ASTM D445	225 000 ^a	
Density (ρ)	ASTM D1298	130 000 ^a	
FAME composition	EN14103	600 000 ^b	1 ^b

^a Based on analysis price list in LEMIGAS

^b Based in analysis price list in Chemical Engineering Laboratory, Institut Teknologi Indonesia

CONCLUSIONS

In this study, the composition of FAME from pure palm based biodiesel was measured to predict the type of biodiesel and its characteristics. The palm-based biodiesel that has been successfully predicted has the type of Palm Oil Biodiesel, Palm Stearin Biodiesel, and Palm Olein Biodiesel. Besides, the FAME composition of various blend biodiesel consisting of Palm Stearin Biodiesel and Soybean Biodiesel was also successfully observed.

In addition to the composition of FAME, pure and blend palm based biodiesel has good characteristics because some essential parameters such as oxidation stability, cloud point, iodine number, viscosity and density meet international standards. The oxidation stability of the palm based biodiesel studied has a range of 16-18 hours, while the cloud point has a range of 13.2-17.4 °C. The iodine number has a range of 44.75-55.20 g I₂ per 100 g of biodiesel, the viscosity has a range of 4.44-4.74 mm² s⁻¹, and the density has a range of about 857.1-0.865.6 kg m³⁻¹.

Several empirical equations to predict some essential parameters of palm based biodiesel are obtained from the relationship between several FAME components and the essential parameters of the re-

sults of this study and have a % error between 0-0.52%. The empirical equation in the form of multiple linear regression is obtained from the comparison of actual parameters vs. predictive parameters, by generating R² values with a range of 0.9995 to 1, which shows that the empirical equation obtained is very accurate. Using this empirical equation reduced testing costs by 67% and testing time by up to 13 working days. For this reason, the FAME composition for biodiesel from palm oil biodiesel, palm stearin biodiesel, and palm olein biodiesel is recommended for analysis to determine its physical properties using this empirical equation to save costs and time. This is very useful to be applied in large sample sizes.

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