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The Effect of Outdoor Temperature Conditions and Monoglyceride Content on the Precipitate Formation of Biodiesel-Petrodiesel Blended Fuel (BXX)

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This study examines the effect of monoglyceride content in biodiesel on the precipitate formation of BXX fuel at three different outdoor temperatures in three locations: Serpong in Banten Province, Lembang in West Java Province and Batu in East Java Province. Each location has its height of 100, 1,500 and 2,000 meters above sea-level (asl), respectively. A modified CSFT method of ASTM D 7501 was applied. Biodiesel was varied for monoglyceride contents to approximately 0.4%, 0.6% and 0.8% and mixed with petrodiesel fuel to produce B10, B20 and B30. Each sample was placed outdoors and soaked for 21 days in a 100 mL - closed separating funnel. After 21 days, each sample was then vacuum-filtered and the precipitate retained on the filter paper was then washed with petro-ether prior to drying under vacuum condition. Then, its constant weight was measured. The results showed that the amount of precipitate formed was influenced by the soaking outdoor temperature, biodiesel percentage in BXX fuel and monoglyceride content. The proposed standard (SNI 7182:2015) for monoglyceride content parameter in B100 for producing the quality of B30 equivalent to that of B20 was found to be 0.6%-mass.

Keywords: biodiesel, B20, precipitate, monoglyceride, filter clogging.

1. Introduction

Renewable and non-conventional energy has already become important options for energy security and sustainability of the countries, like in Japan¹. Meeting energy demand has always become a challenge for every country. The choice of energy utilization in Indonesia, the share of new and renewable energy (NRE) in the national energy mix has been targeted to be set to 23% by 2025. The government issued the regulation to set up the executive team for the development of biofuel in Indonesia². Since then, the studies and activities towards the implementation of biofuel have been intensively done. Bioethanol, biodiesel and pure plant oil have been the types of biofuels that were developed, tested and implemented. Although a study has proved that bioethanol was said to be economically produced in Indonesia³, however, only biodiesel has been mandated to be implemented since 2006 and increased its volume ratio in the blended fuel up to 20% since January 2016². Pure plant oil has been used only for petrodiesel fuel

substitution in the power plant.

Biodiesel, also known as fatty acid methyl ester (FAME), is a renewable fuel and considered as an attractive substitute for conventional diesel fuels. The advantages of biodiesel over petroleum diesel fuel are a higher flash point, improved lubricity, lower toxicity, lower sulfur content and biodegradability^{4,5,6}. Although biodiesel has been widely used as blended fuel with petroleum diesel fuel, however, biodiesel still has some technical problems such as oxidative stability, low-temperature performance, nitrogen oxides (NOx) emissions^{7,8} and insoluble impurities causing fuel filter clogging^{9,10,11}. Such problems are related to the fatty acid compositions present in the feedstocks.

Biodiesel is produced by esterification or transesterification reactions of fatty acids (plant or animal oil). In a transesterification reaction, which is a reversible reaction, of biodiesel production, one mole of triglyceride reacts with three moles of alcohol (molar ratio of methanol to vegetable oil of 3:1) to form one mole of glycerol and three moles of the respective fatty

acid alkyl esters⁴). Transesterification of triglycerides with alcohol produces alkyl esters (biodiesel) and glycerol. This reaction happens through step-wise transformations from triacylglycerol (triglycerides) to diacylglycerol (diglycerides), and eventually monoacylglycerol (monoglycerides) to become glycerin^{12,14}. In each step, fatty acid methyl ester is made. In normal optimum condition, the yield of conversion is considerably high, so that the trace of unreacted glycerides is very low¹⁵. However, the glyceride impurities, especially monoglycerides, even in very low amount can affect the biodiesel properties significantly¹³. Biodiesel production process can produce impurities such as saturated monoglycerides (SMG) which can precipitate at low temperature condition. This is due to high final melting temperature (FMT) or melting point of SMG and will form solid deposits above cloud point (CP). The precipitation occurs due to the big difference between cloud point and final melting point, and this happens on high saturated monoglycerides contain between 0.2-0.3% wt^{16,17}. The implementation of biodiesel blended with petroleum diesel will cause precipitation at low temperatures and may cause clogging of fuel filters in a vehicle¹⁴.

Currently, there are available tests for assessing the precipitation of biodiesel-petroleum diesel blended fuel and its clogging effect on fuel filter, namely ASTM D 7501 for Cold Soaking Filtration Test (CSFT)^{18,19} and ASTM D 2068 for Filter Blocking Test (FBT)^{20,21}. CSFT combines steps of cold soaking (in terms of soaking

temperatures and time) and filterability for determining fuel filter clogging tendency. FBT is a method for determination of the fuel filter blocking tendency and filterability of middle distillate fuel oils and non-petroleum liquid fuels such as biodiesel, where the fuel users demand good cleanliness of the fuel. Previous studies were conducted for Cold Soaking Filtration Test of BXX for varied time of soaking and type of monoglyceride^{22, 23}.

2. Materials and Method

2.1. Fuel Preparation

Biodiesel (B100) sample was made of palm oil and supplied by PT Wilmar Nabati Indonesia in Gresik, East Java. Petroleum diesel fuel (B0) used in the study was from PT Pertamina. Biodiesel was analyzed for several quality parameters according to SNI 7182:2015²⁴, as shown in Table 1, whereas the result for petrodiesel fuel was shown in Table 2. Increased monoglyceride (MG) content in B100 (for testing) was made by adding monopalmitin to the biodiesel sample so that its MG content reached at 0.6 and 0.8 wt %, respectively, prior to blending. MG contents in biodiesel were determined by ASTM D 6584²⁵ with GC-FID²⁶. Monopalmitin was obtained from Tokyo Chemical Industry (TCI) Japan, with specifications of purity >95% (GC), melting point 73.0 to 77.0°C. Monopalmitin was used to increase monoglycerides content in biodiesel for the test purpose.

Table 1. Biodiesel Fuel (B100) Specification and Quality

No	Parameter	Unit	Result	Limit SNI 7182-2015		Methods
				B100 Sample	Min	Max
1	Density at 40°C	kg/m ³	855.3	850	890	SNI 7182-2015/ ASTM D 4052 Calculated
2	Ester content	% mass	98.7	96.5		
3	Free glycerol	% mass	0.006		0.02	
4	Total glycerol	% mass	0.128		0.34	EN 14105
5	Mono-glyceride:	% mass	0.369		0.8	
	a. Monopalmitin	% mass	0.204			
	b. Monoolein	% mass	0.136			
	c. Monostearin	% mass	0.029			

Table 2. Petrodiesel Fuel (B0) Specification and Quality

No	Parameter	Unit	Result	Standard B0 (Solar 48)		Methods
				Min	Max	
1	Density at 15°C	kg/m ³	843.8	815	860	ASTM D4052
2	Kinematic Viscosity at 40°C	mm ² /s	2.6	2	4.5	ASTM D445
3	Cloud Point	°C	9.7	-	18	ASTM D5773
4	Sulfur content	% mass	0.106		0.25	ASTM D4294

Table 3. BXX Fuel (B10, B20 and B30) Specification and Quality

No	Parameter	Unit	Result			Limit	Methods
			B10	B20	B30		
1	Density at 15°C	kg/m ³	845.5	848.3	851.0	815-860	ASTM D4052

2	Kinematic Viscosity at 40°C	mm ² /s	2.87	2.98	3.14	2.0-4.5	ASTM D445
3	Cloud Point	°C	9.5	9.7	9.5	18 max	ASTM D5773
4	Water content	%-vol	183.27	249.87	302.33	500 max	ASTM D6304
5	Sediment content	%mass	None	None	None	0.01 max	ASTM D473
6	FAME content	%mass	10.1	20.1	29.9	-	ASTM D7806
7	Total Acid Number	mg KOH/g	0.061	0.089	0.110	0.06	ASTM D664
8	Oxidation Stability - Rancimat Method	hours	42.32	36.21	36.11	35	EN 15751

BXX samples were prepared by blending B0 and B100 by %-volume to produce B10, B20 and B30. BXX samples were analyzed for several quality parameters²⁷⁾, as shown in Table 3. Before blending, B0 fuel was filtered to remove the residue so that only impurities from B100 could be considered to influence the filter clogging in the BXX precipitation test. For comparison, the B20 existed in the market, known as Biosolar, was also included in the test.

2.2. Precipitation Test

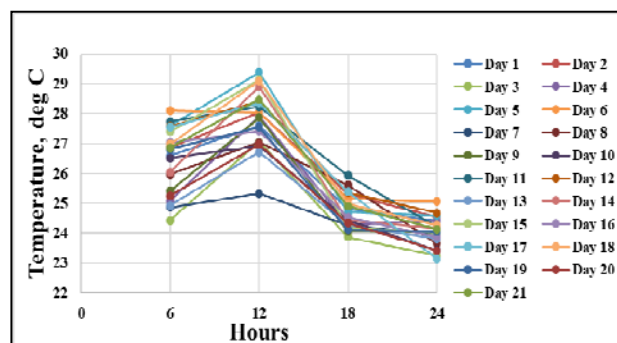
The precipitation test adopted modified ASTM D 7501 for Cold Soaking Filtration Test (CSFT). This test was performed by placing 100 mL of each B-XX sample in a 100 mL-separating funnel. Blank test using B0 was also conducted by filtering B0 through same size filter paper without soaking. The samples were then placed outdoors for 21 days in three different locations: Serpong in Banten Province, Lembang in West Java Province and Batu in East Java Province, where each having its height above sea-level (asl) of 100, 1,500 and 2,000 meters, respectively. The last two places were in highlands and have low temperature conditions.

After 21 days of soaking, each sample was then vacuum-filtered through a 0.8 μ m filter paper. Cellulose acetate membrane filters used to filter BXX was SartoriusTM with specification of diameter 47 mm and particle retention 0.8 μ m. Precipitate retained on filter paper was then washed with petro-ether and dried with vacuum condition, and then its weight was measured. Petroleum ether from Merck[®] was used to wash precipitate, so that only monoglyceride in the precipitate remained on filter.

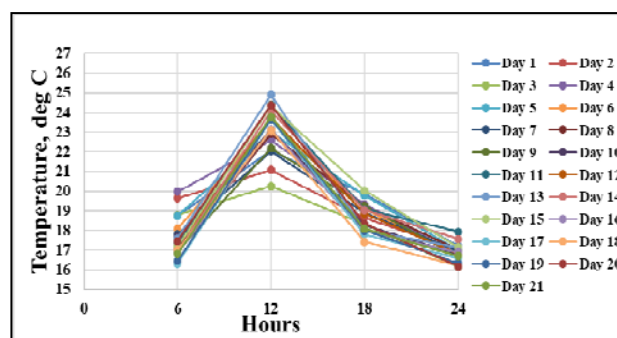
3. Results and Discussion

The temperature data recorded during the precipitation test for each location (Serpong, Lembang and Batu) were shown in Figure 1 (a), (b) and (c), respectively. For the same recorded time everyday in 21 days, the outdoor temperature in Serpong (100 meters asl) was higher than those in higher lands, i.e. Lembang (1,500 meters asl) and Batu (2,000 meters asl). For each day in every location, the highest temperature was recorded at noon, and dropped to lowest temperature at midnight. The maximum higher and lower temperature difference was

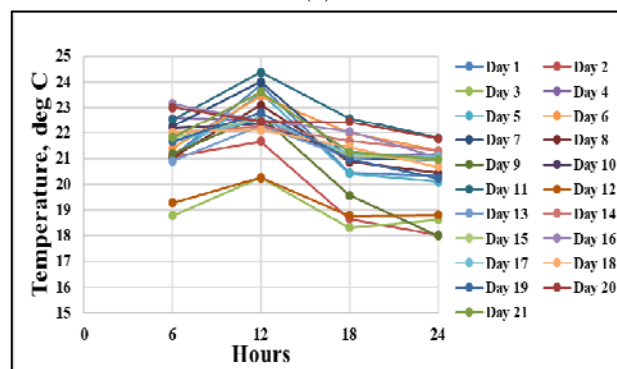
9°C. This difference is considered not so significant to affect the precipitation.



(a)



(b)

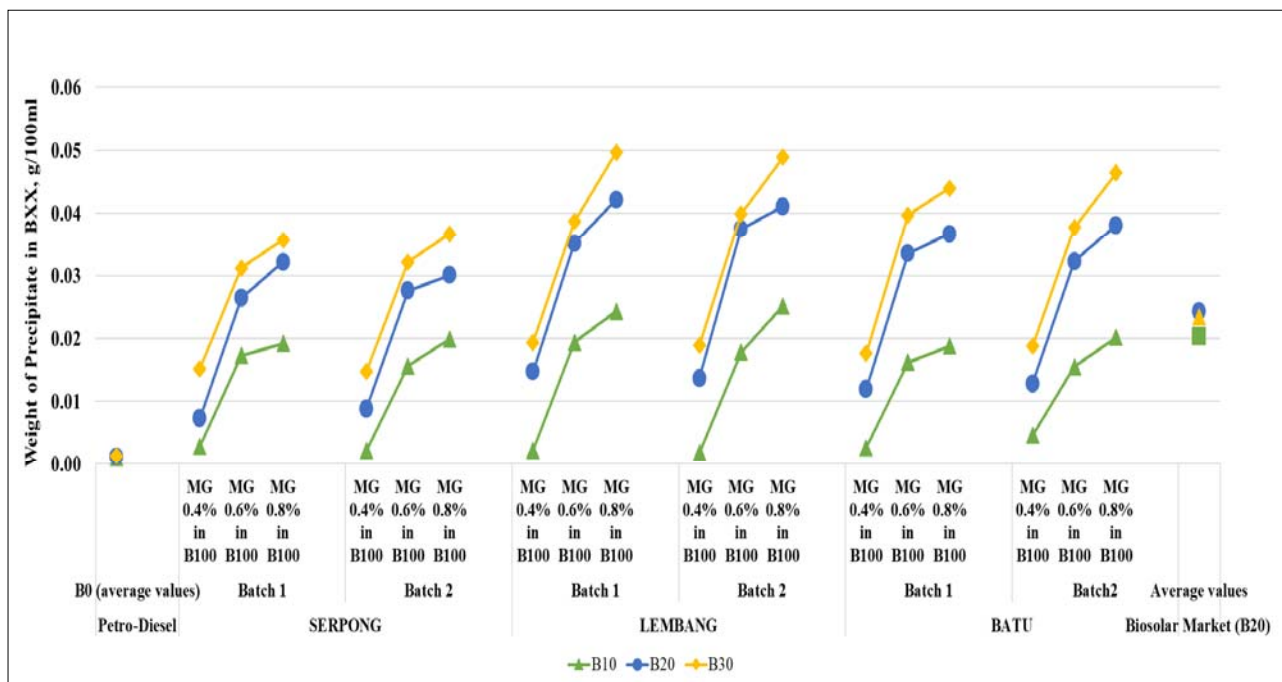


(c)

Fig. 1: Temperature Data Recorded during Precipitation tests. Test locations: (a) Serpong, Banten Province; (b) Lembang, West Java Province and (c) Batu, East Java Province

Table 4. Results of Precipitation Tests

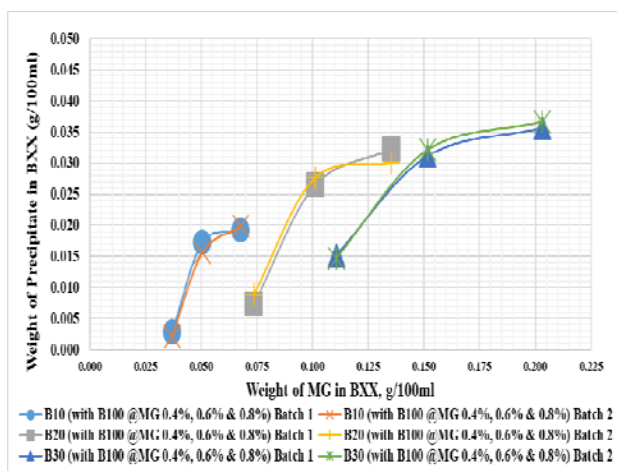
Sampel	Weight of MG in BXX, g/100 ml	Weight of Precipitate in BXX, g/100 ml					
		Serpong		Lembang		Batu	
B10		B10 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 1	B10 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 2	B10 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 1	B10 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 2	B10 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 1	B10 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 2
B10-with B100 0.4%MG	0.0369	0.0028	0.0021	0.0022	0.0018	0.0026	0.0046
B10-with B100 0.6%MG	0.0506	0.0173	0.0156	0.0194	0.0178	0.0162	0.0155
B10-with B100 0.8%MG	0.0677	0.0192	0.0199	0.0243	0.0252	0.0188	0.0202
B20		B20 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 1	B20 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 2	B20 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 1	B20 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 2	B20 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 1	B20 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 2
B20-with B100 0.4%MG	0.0737	0.0073	0.0088	0.0147	0.0137	0.0119	0.0128
B20-with B100 0.6%MG	0.1011	0.0264	0.0276	0.0352	0.0375	0.0335	0.0323
B20-with B100 0.8%MG	0.1354	0.0322	0.0301	0.0422	0.0411	0.0367	0.0381
B30		B30 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 1	B30 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 2	B30 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 1	B30 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 2	B30 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 1	B30 (with B100 @MG 0.4%, 0.6% & 0.8%) Batch 2
B30-with B100 0.4%MG	0.1106	0.0152	0.0147	0.0194	0.0189	0.0176	0.0188
B30-with B100 0.6%MG	0.1517	0.0312	0.0322	0.0387	0.0399	0.0397	0.0377
B30-with B100 0.8%MG	0.2031	0.0357	0.0367	0.0497	0.0489	0.0440	0.0464
B0		0.0009	0.0011	0.0012	0.0011	0.0012	0.0014
B20 Market		0.0196	0.0209	0.0258	0.0229	0.0246	0.0222

**Fig. 2:** Results of Precipitation Tests in Serpong, Lembang and Batu.

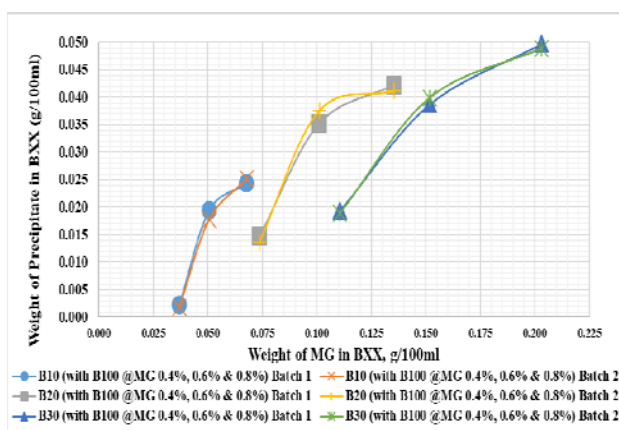
The results of the precipitation test showed that precipitation of B10, B20 and B30 occurred in three locations, as shown in Table 4 and Figure 2. The results showed that the amount of precipitate formed in BXX fuel was influenced by soaking temperature condition, biodiesel percentage in BXX fuel and monoglyceride content in biodiesel. The lower the soaking temperature

condition was applied so that the higher amount of precipitate was formed. The precipitates formed in Lembang and Batu were higher than that in Serpong. It was also found that biodiesel concentration in BXX and monoglyceride content in biodiesel could affect the formation of precipitate. Increasing biodiesel concentration in BXX could increase the amount of

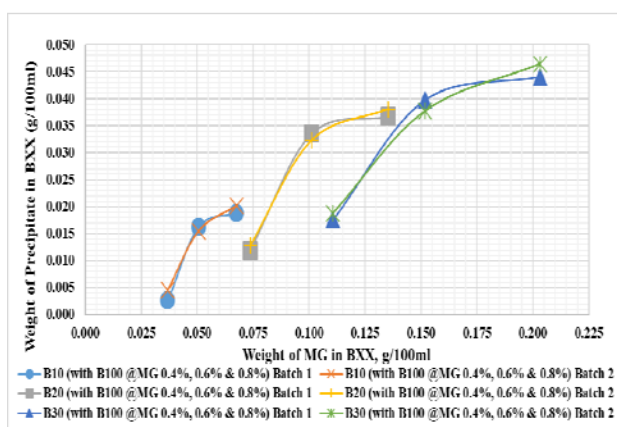
precipitate. This was due to the increased content of impurities in BXX, which were mainly from monoglycerides in biodiesel



(a)



(b)



(c)

Fig. 3: Results of Precipitation Tests in: (a) Serpong, (b) Lembang and (c) Batu

Up to now, biodiesel has been mandated to be blended up to 20%-volume (B20) in Indonesia since January 2016, known as Biosolar, having precipitate of just above 0.02 g/100 ml (Figure 2). The percentage of biodiesel are planned to be increased to 30% by volume (B30) in 2020.

In order to get a new standard limit of monoglyceride content parameter in B100 as stated in SNI 7182:2015, for producing quality of B30 equivalent to that of B20, therefore, the results were then extended for further assessment, to determine the relationship between the amount of precipitate formed in B10, B20 and B30 fuels, and the monoglyceride content in corresponding BXX fuel (referred as the weight of MG in BXX, calculated in g/100ml, shown in Table 4). Figure 3 illustrated the correlation between amount of precipitate in B10, B20 and B30 fuels (with variation of monoglyceride content of 0.4, 0.6 and 0.8%-mass in biodiesel) for each outdoor temperature condition.

4. Conclusion

From the precipitation test conducted in three locations, i.e. Serpong, Lembang and Batu, each with different height above sea-level and outdoor temperature conditions, it can be concluded that the amount of precipitate formed in BXX fuel was influenced by soaking outdoor temperature conditions, biodiesel percentage in BXX fuel and monoglyceride content in biodiesel. The lower the soaking temperature condition was applied; the higher amount of precipitate was formed. Biodiesel concentration in BXX and monoglyceride content in biodiesel could affect the formation of precipitate. Increasing biodiesel concentration in BXX and higher monoglyceride content in B100 could make the amount of precipitate to increase. It could be suggested that the monoglyceride content parameter for B100 standard for producing quality of B30 equivalent to that of B20 (given for B20 that the upper limit value of monoglyceride content in B100 of 0.8%), was proposed to be 0.6%-mass, based on the similar amount of precipitate resulted in B20 and B30.

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