Development of Fast Check Test Kit for Biodiesel Quality Monitoring

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***Abstract*—Determination of fatty acid methyl esters (FAME) content in petroleum diesel blends and monitoring of unreacted glycerides in biodiesel after transesterification are important aspect of blending process and production as well as quality control of distribution operations. In this study, fast check analysis method with special measuring device and chemical solvent mixture were developed to provide on-site analysis check for biodiesel quality monitoring. Biodiesel Test Kit have shown comparable result with ASTM D7371 with correlation coefficient (R^2 > 0.99) for biodiesel-diesel blends B5, B10 and B20. Result has indicated biodiesel sample from various feedstock such as Palm, Jatropha and Soybean are not affected the accuracy of biodiesel test kit. The use of Triglycerides Test Kit in determination of triglyceride content in biodiesel from various feedstock have shown good correlation coefficient (R^2 > 0.99) for triglycerides 3, 4 and 5%(v/v). Again, biodiesel derived from Palm, Jatropha and Soybean do not affect the accuracy of triglycerides test kit. The developed methods are providing fast, simple and affordable on-site fast check analysis in measuring FAME content in diesel and triglycerides content in final FAME product economically especially in rural area.**

***Index Terms*—Biodiesel, FAME, biodiesel test kit, triglycerides test kit.**

I. Introduction

Due to the increasing pressure for fuel energy security, to reduce global emissions of greenhouse gases and of the finite nature of fossil fuels, alternative transport fuels such as biodiesel are being increasingly sought [1]. Biodiesel is receiving increasing attention as an alternative, non-toxic, biodegradable, and renewable diesel fuel [2]. Biodiesel is a mixture of mono alkyl esters of long-chain [C16-C18] fatty acids, usually methyl or ethyl esters, obtained by transesterification of the triglycerides contained in vegetable oils and animals fats [3].

Biodiesel can be used pure or mixed with petroleum distillates to attain blends defined with the abbreviation BX, where X stands for the biodiesel percentage (v/v) [4]. The designation of pure biodiesel is B100 and the mixtures are named by the BXX abbreviation, which indicates the B100 volume (in percent) in the mixture with diesel. Consequently, B2 is constituted by 2% of B100, and 98% of diesel; B5 by 5% of B100 and 95% of diesel [5]. The most recent study of B20 blend quality was completed prior to the adoption of ASTM D7467-08, the quality specification for B6–B20 [6]. The determination of FAME concentrations in diesel fuel blends is an important aspect of production and blending process as well as quality control of distribution operations [7]. Several spectroscopic and chromatographic techniques have been employed for the analysis of biodiesel blends, including IR [8], [9], HPLC [10], H-NMR [11] and comprehensive two dimensional gas chromatography [12].

Apart from that, glycerides content is the key parameter to define biodiesel quality [13]. A high content of glycerides, especially triglycerides (unreacted plant oil in transesterification), may cause the formation of deposits at the injection nozzles and in the valves [14]. Several methods for determination of glycerides content in biodiesel, most of them based on gas-chromatography techniques [15].

However, the problem and limitation of current analysis technology are cost and the availability of advanced facilities which is not available on engine operation site. The analysis methods and analysis devices utilizing gas chromatography and FTIR require expertise and specialized skills. This makes it difficult to carry out on-site analysis especially in rural area. The present invention was aim to solve all above problems with providing fast, simple and affordable on-site fast check analysis in measuring FAME content in diesel and triglycerides content in final FAME product economically.

II. Experimental

A. Materials

Soybean methyl ester (SME) was acquired from Marathon Biodiesel Co. Ltd (USA), Palm methyl ester (PME) was purchased from Carotech Sdn Bhd (Malaysia) and Jatropha methyl ester was prepared from crude jatropha oil (CJO) which supplied by Bioenergy Co. Ltd (Thailand) using base catalyst (NaOH) transesterification method [16]. A petroleum diesel samples were acquired from Petronas petrol stations. Soybean cooking oil (*Western Family*) and palm cooking oil (*Vesawit*) were purchased from local hypermarket. Alcohol chemical solvents (purity 99.9%) were purchased from *MERCK* Malaysia.

B. Blends

Biodiesel blends were prepared by adding biodiesel (SME/JME/PME) to diesel at 5, 10 and 20wt% with 3 replicates each. Both biodiesel blends were previously analyzed by FTIR using ASTM D7371 for verification. For triglycerides blends, cooking oil (soybean/palm) and crude jatropha oil were mixed to biodiesel (SME/PME/JME) respectively at 1, 2, 3, 4 and 5wt% to represent triglycerides content in biodiesel with 3 replicates each. Both mixtures of biodiesel and cooking oil samples were previously analyzed by GC using EN14105 for verification.

C. Solubility Test

Solubility test of Malaysia diesel, Palm FAME and Palm

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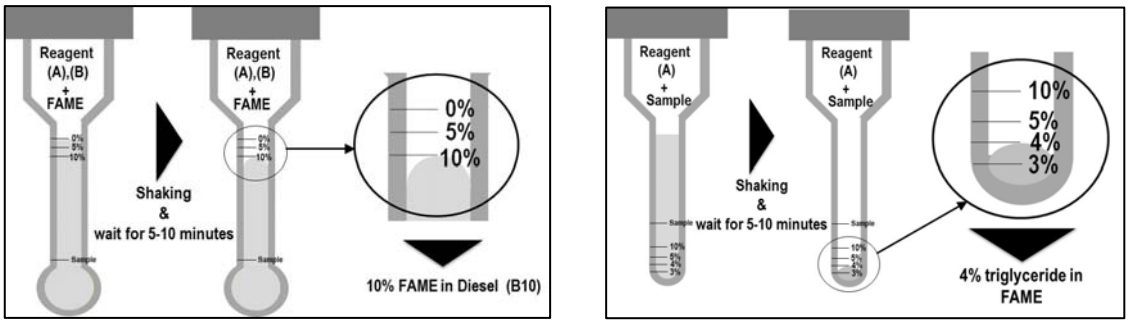
triglycerides in various chemical solvent (alcohols, acetone and Solubility test of Malaysia diesel, Palm FAME and Palm triglycerides in various chemical solvent (alcohols, acetone and toluene) were performed at room temperature 20-23°C using mixing ratio 1:1, 5:1 and 10:1. Observation results were recorded as shown in Table I.

D. Fast Check Test Kit

Measurement of the FAME percentage in diesel were described in FIG. 1a by using special analysis vessel as analysis test kit in the measuring method and for the measuring device for measuring FAME content in diesel (Patent pending). Reagents that are used include a reagent (A) methanol (for FAME content) and reagent (B) white oil (as additive). First, a sample is added into the measuring vessel until sample line, indicating a specified amount. Next, reagent (B) is pour into the measuring vessel follow by reagent (A). The measuring vessel is shaken moderately with lid on and waits for 5 to 10 minutes. As shown in FIGURE 1(a), the FAME percentage is read from the surface boundary on the FAME scale. Thus the FAME % in diesel oil is obtained.

After that, vessel is washed with isopropanol to clean up chemical fuel sample.

Measurement of the triglycerides percentage in biodiesel were described in Fig. 1(b) by using special analysis vessel as analysis test kit in the measuring method and for the measuring device for measuring triglyceride content in biodiesel (Patent pending). Reagents are reagent (A) mixture of methanol and ethanol (for triglyceride content). First, a sample is added into the measuring vessel until sample line, indicating a specified amount. Next, reagent (A) is pour into the measuring vessel. The measuring vessel is shaken moderately with lid on and waits for 5 to 10 minutes. As shown in Fig. 1(b), the triglycerides percentage is read from the surface boundary on the triglycerides scale. Thus the triglycerides % in biodiesel oil is obtained. After that, vessel is washed with isopropanol to clean up chemical fuel sample.



(a) Biodiesel test kit (b) Triglycerides test kit

Fig. 1. Diagram illustrated states before and after the measuring method for measuring the FAME % in diesel or triglycerides in biodiesel according to the embodiment of the present invention.

TABLE I: SOLUBILITY OF DIESEL, FAME AND TRIGLYCERIDES IN VARIOUS ALCOHOLS

Alcohols	FAME (Palm)			Triglyceride (Palm Cooking Oil)			Diesel (Malaysia Petronas)		
	Ratio – Alcohol: FAME, Triglycerides, Diesel								
	1:1	5:1	10:1	1:1	5:1	10:1	1:1	5:1	10:1
Methanol	○	○	○	●	●	●	●	●	▲
Ethanol	○	○	○	●	●	●	▲	◆	○
Propanol	○	○	○	○	○	○	○	○	○
Isopropanol	○	○	○	○	○	○	○	○	○
Butanol	○	○	○	○	○	○	○	○	○
Decanol	○	○	○	○	○	○	○	○	○
Acetone	○	○	○	○	○	○	○	○	○
Toluene	○	○	○	○	○	○	○	○	○

○: Complete dissolution ▲: 0~20% dissolution ◆: 20~80% dissolution ●: No dissolution

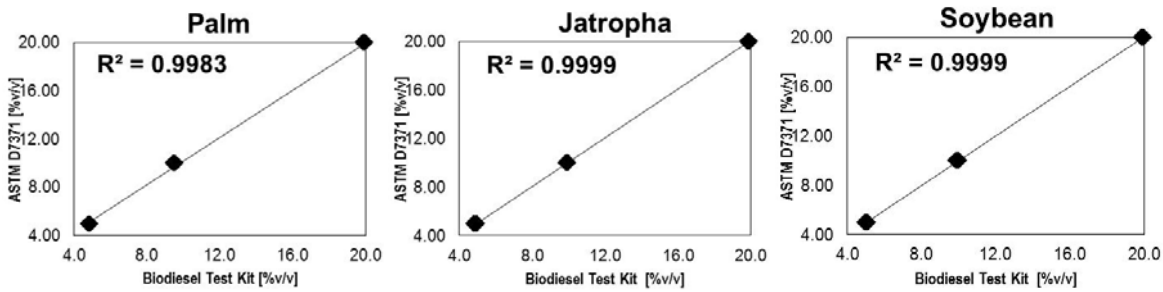


Fig. 2. Biodiesel test kit analysis result for b5, b10 and b20 of palm/jatropha/soybean biodiesel with malaysia diesel

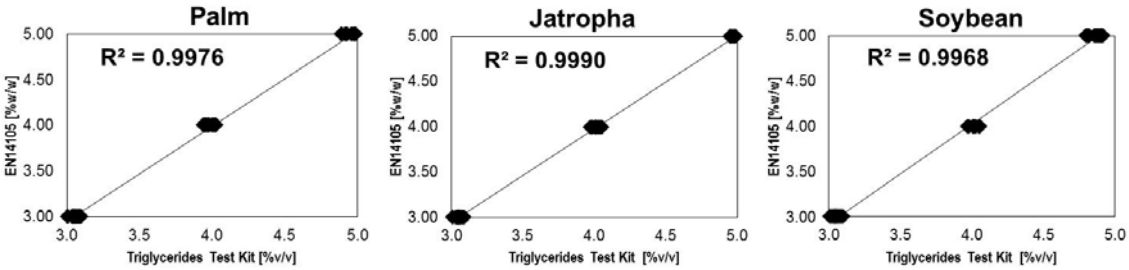


Fig. 3. Triglycerides test kit analysis result for tg3, tg4 and tg10 of cooking oil with palm/jatropha/soybean biodiesel

TABLE II: RESULT SUMMARY OF BIODIESEL TEST KIT

Biodiesel Test Kit	Biodiesel content, %(v/v)	Average Test Kit Result, %(v/v)	Standard Deviation, SD
Palm/Jatropha/Soybean	B5	5.00	0.00
	B10	10.00	0.00
	B20	20.00	0.00

TABLE III: RESULT SUMMARY OF TRIGLYCERIDE TEST KIT

Triglyceride Test Kit	Triglyceride content, %(w/w)	Average Test Kit Result, %(v/v)	Standard Deviation, SD
Palm/Jatropha/Soybean	TG1	dissolve completely	-
	TG2	dissolve completely	-
	TG3	3.00	0.00
	TG4	4.00	0.00
	TG5	5.00	0.00

TABLE IV: ANALYSIS PRECISION AND DETECTION LIMIT OF FAST CHECK TEST KIT

Fast Check Test Kit	Feedstock	Correlation with Standard Method, R^2	Detection Limit, DL (based on test kit design)	
			Lower Limit	*Upper Limit*
Biodiesel	Palm	0.9983	5%(w/w)	20%(w/w)
	Jatropha	0.9999		
	Soybean	0.9999		
Triglycerides	Palm	0.9976	3%(w/w)	5%(w/w)
	Jatropha	0.9990		
	Soybean	0.9968		

III. RESULT AND DISCUSSION

A. *Solubility of Diesel, FAME and Triglycerides in Various Alcohols*

The solubility properties of diesel, FAME and triglycerides in various alcohols were shown in Table I. Methanol and ethanol have shown different solubility on FAME, triglycerides and petroleum diesel due to different polarity.

Petroleum diesel and triglycerides are non-polar, FAME is moderately polar, and alcohols have higher polarity among these compounds. FAME was dissolved completely in methanol and ethanol whereas triglycerides didn't dissolve methanol and ethanol and petroleum only dissolved partially in ethanol. An optimum solvent mixture ratio was figured out using methanol and ethanol as reagent in both fast check test kit (patent pending).

B. *Biodiesel Test Kit*

As shown in TABLE II and IV and Fig. 2, determination of FAME content in diesel using Biodiesel Test Kit were correlated well with FAME content determined by ASTM D7371 using FTIR method with correlation coefficient R^2 = 0.9983 (Palm), R^2 = 0.9999 (Jatropha) and R^2 = 0.9999 (Soybean) for B5, B10 and B20 biodiesel-diesel blends. Result also have shown biodiesel test kit with optimum chemical solvent (patent pending) able to determined Fame content from different feedstock (e.g. Palm, Jatropha and Soybean) in diesel. The detection limit of biodiesel test kit is 5wt% of FAME content according the test kit design.

C. *Triglycerides Test Kit*

As shown in Table III and IV and Fig. 3, determination of triglycerides content in biodiesel using triglycerides test kit were correlated well with triglycerides correlation coefficient R^2 = 0.9976 (Palm), R^2 = 0.9990 (Jatropha) and R^2 = 0.9968 (Soybean) for TG3, TG4 and TG5 triglyceride content in biodiesel. Result also have shown triglycerides test kit with optimum chemical solvent (patent pending) able to determined triglycerides content from different feedstock (e.g. Palm, Jatropha and Soybean) in biodiesel. The detection limit of triglycerides test kit is 3wt% of triglycerides content according the test kit design.

IV. CONCLUSION

This paper presented method and device based on screening analysis for determination of FAME content in diesel and triglycerides content in biodiesel by using special measurement device, namely Biodiesel Test Kit and Triglycerides Test Kit, respectively with optimum mixture

ratio of alcoholic chemical solvent and additives. The results obtained which correlated well with standard method analysis in this investigation suggest that the proposed method is promising on-site fast check analysis methods which provide fast, simple and affordable on-site fast check analysis in measuring FAME content in diesel and triglycerides content in final FAME product economically. Biodiesel Test Kit and Triglycerides Test Kit have great potential as on-site fast check analysis in rural area and country for biodiesel quality monitoring.

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