Density and Viscosity Measurements for Olive Oil Biodiesel, Diesel Fuel and n-Butyl Alcohol Ternary Blends

Mert Gülüm and Atilla Bilgin

***Abstract*—In this study, olive oil biodiesel was produced, the ternary blends including commercially available diesel fuel, produced olive oil biodiesel and n-butyl alcohol were prepared, and densities and kinematic viscosities of the blends were measured at 15°C and 40°C according to ISO 4787 and DIN 53015 standards, respectively. Experimental results show that viscosities non-linearly and densities linearly as expected decrease with increasing n-butyl alcohol content in blend. For the variation of kinematic viscosities of ternary blends with respect to alcohol content in the blend, the four-term exponential model was derived using the least squares regression method.**

***Index Terms*—Olive oil biodiesel, density, viscosity, fuel properties, ternary blends, n-butyl alcohol, four-term exponential model.**

I. Introduction

The rapid consumption of fossil fuels and rising their prices have boosted research efforts on investigating alternative fuels [1], [2]. Alternative fuels should be easily available at low cost, renewable, environment friendly and techno-economically competitive [3]. In this context, vegetable oils have become more attractive recently as an alternative fuel for diesel engine. In fact, using vegetable oils as fuel is not new and dates back to the end of the 19th century with the inventor of the diesel engine [4]. In 1900, at the Universal Exhibition in Paris, the OTTO company exhibited a small engine which ran exclusively on groundnut oil [5], [6]. The engine, which had initially been designed to run on diesel oils, was worked with vegetable oil without any modification. Many researchers from different countries still have studied on using of different types of vegetable oils [7]-[11], however, a number of problems including clogged filters, deposits in the combustion chamber and injector tip, injector cocking, injector pump failure and etc. are being encountered in their use especially for long-term operation due to the low cetane number and high viscosity of vegetable oils [12], [13]. Four methods to reduce high viscosity to enable their use in common diesel engines without these problems have been investigated: blending with diesel fuel, pyrolysis, micro-emulsification (co-solvent blending) and transesterification [14]. The pyrolysis and the emulsification produce heavy carbon deposits, incomplete combustion, an increase of lubricating oil viscosity and undesirable side products such as alkanes, alkenes, alkadienes, aromatic compounds and carboxylic acids [15]. Thus, transesterification (or alcoholysis) is the most common method and only it leads to the product commonly known as biodiesel, i.e., alkyl esters of oils and fats [14]. The transesterification reaction represented by the general equation shown in Fig. l(a) consists of a number of consecutive and reversible reactions as shown in Fig. l(b) [16].

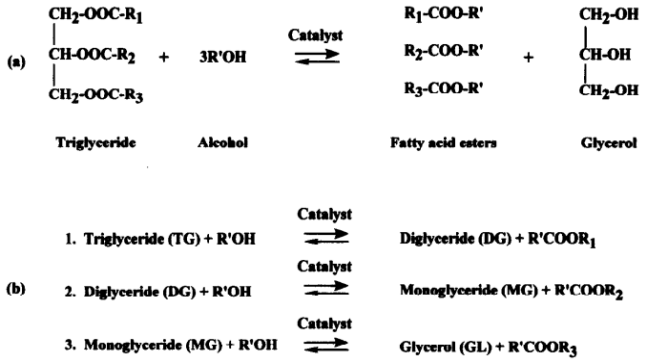

Fig. 1. Transesterification of triglyceride with alcohol. (a) General equation; (b) three consecutive and reversible reactions. R_1, R_2, R_3 and R' represent alkyl groups [16].

The first step is the conversion of triglyceride to diglyceride, which is followed by the conversion of diglyceride to monoglyceride and of monoglyceride to glycerol, yielding one methyl ester molecule from each glyceride at each step [16]. In this process, methanol is the commonly used as alcohol due to its low cost. Ethanol is also a preferred alcohol because it is derived from agricultural products and is renewable and biologically less objectionable in the environment [17]. Transesterification reaction can be occurred either with or without a catalyst, homogeneous or heterogeneous. The homogeneous catalysts can be acid, basic or enzymes. Their inconvenience is the need to purify the products (esters and glycerol) in order to remove catalyst residues, making it impossible its reuse after the reaction. Heterogeneous catalysts can be recovered after the reaction and are easily removed, making purification simple. Basic catalysts are the most used in large scale because they are less corrosive for industrial equipment and give a higher yield when compared to acid catalysts [18].

The use of biodiesel as a fuel has achieved promising potential worldwide because of its several benefits such as: (i) it is plant sourced and therefore its combustion does not contribute to current net atmospheric CO_2 levels; (ii) it has higher cetane number, which shortens ignition delay and provides smoother engine operation; (iii) it is biodegradable; (iv) it is non-toxic and non-aromatic, and has a low emission profile; (v) it has well lubrication properties. The use of biodiesel can easily increase the lubricity of the diesel with a low sulfuric content, enhancing the operation of the moving parts of the engine and the fuel pump; (vi) it has higher flash

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point temperature, making it safer to handle and store; (vii) it can be produced using domestic feedstocks, reducing the countries' dependency on fossil fuels [16]-[21]. Table I list potential biodiesel feedstocks in different regions and countries.

TABLE I: POTENTIAL BIODIESEL FEEDSTOCKS IN DIFFERENT REGIONS AND COUNTRIES [22]

Region	Country	Feedstocks
Asia	China	Jatropha/Waste cooking oil
	Indonesia	Palm oil/Jatropha/Coconut
	India	Jatropha/Pongamia Pinnata
	Japan	(Karanja)/Soybean/Rapeseed
	Malaysia	Waste cooking oil
	Philippines	Palm oil
	Bangladesh	Coconut/Jatropha
	Pakistan	Rubber seed/Pongamia
	Thailand	Palm/Jatropha/Coconut
	Iran	Palm/Jatropha/Castor/Algae
	Singapore	Palm oil
Africa	Ghana	Palm
	Zimbabwe	Jatropha
	Kenya	Castor
	Mali	Jatropha curcas
Europe	Norway	Animal fats
	Sweden	Rapeseed
	France	Rapeseed/Sunflower
	Germany	Rapeseed
	Greece	Cottonseed
	Spain	Linseed oil/Sunflower
	Italy	Rapeseed/Sunflower
	Turkey	Sunflower/Rapeseed
	UK	Rapeseed/Waste cooking oil
North America	Ireland	Frying oil/Animal fats
	Canada	Rapeseed/Soybeans
	Mexico	Animal fat/Waste oil
	USA	Soybeans/Waste oil/Peanut
	Cuba	Jatropha curcas/Moringa
South America	Argentina	Soybeans
	Brazil	Soybeans/Palm oil/Castor
	Peru	Palm/Jatropha
Australia and Oceania	Australia	Beauty leaf, Jatropha curcas
	New Zealand	Waste cooking oil/Tallow

However, biodiesel has also some drawbacks: (i) it has higher cost than diesel fuel mainly due to the cost of virgin vegetable oils [23]; (ii) it has poor low-temperature properties and its chemical nature makes it more susceptible to oxidation during long-term storage in comparison to diesel fuel [24]; (iii) the heating value of biodiesel is approximately 8% lower than that of diesel fuel [25], which increases brake specific fuel consumption and decreases engine torque; (iv) biodiesel has higher values of viscosity, density, speed of sound and bulk modulus that may cause injection system and combustion anomalies. The faster propagation of pressure waves caused by biodiesel's higher speed of sound and the more rapid pressure rise resulting from biodiesel's greater bulk modulus may shift the injection timing settings from their optimized factory settings, leading to earlier combustion. This can result in higher combustion temperatures and pressures causing higher NO_x emission in the exhaust of a biodiesel-fueled diesel engine [26].

Density is an important fuel property because it influences production, transportation and distribution processes, and all processes that take place in the internal combustion engine [27]. Density data are required to be known to properly design reactors, distillation units, separation process, storage tanks and process piping [27], [28].

Viscosity represents flow characteristics and tendency of fluids to deform with stress [29]. It is one of the most important fuel properties, because it affects the operating conditions of injection systems, especially at low temperatures when fuel fluidity is reduced. Viscosity influences fuel lubricating capacity [30], and affects the atomization of a fuel upon injection into the combustion chamber and eventually the formation of soot and engine deposits [31].

Reliable regression models as a function of temperature or volume percent of blend component are of great important to practically predict binary or ternary blends' densities and viscosities. There are several such models for different binary and ternary blends of diesel, biodiesel and alcohol in literature. Laza and Bereczky [32] investigated densities, kinematic viscosities, heating values, flash point temperatures, cetane numbers and evaporation characteristics of rapeseed oil-alcohol blends including 1-propanol, 2-propanol, izobütanol, 1-butanol and 2-butanol. Alptekin and Canakci [23] determined densities and kinematic viscosities of biodiesel-diesel fuel blends including two commercially available diesel fuel (shell extra diesel and normal diesel) and six biodiesels produced from sunflower, canola, soybean, cottonseed, corn oil, waste palm oils. Moreover, linear, quadratic and Arrhenius type equations were proposed to predict their density and kinematic viscosity. Lapuerta *et al.* [33] tested stabilities, viscosities, lubricities and cold filter plugging point temperatures of diesel fuel-alcohol blends. In the study performed by Chen *et al*., [34], oxidation stabilities, densities, kinematic viscosities and cold filter plugging point temperatures of microalgae biodiesel-diesel fuel blends were determined, and linear correlation depending on biodiesel content was established to describe density and viscosity variations. Verduzco *et al.* [35] researched density and kinematic viscosity for diesel-biodiesel fuel mixtures in the range of 293.15 K – 373.15 K. Empirical correlations for density and viscosity variations were suggested. Verduzco *et al.* [36] investigated variations of cetane number, density, viscosity, heating value with respect to molecular structures (i.e. molecular weight and degree of unsaturation) for two different biodiesels produced from beef tallow and soybean oil. Yasin *et al.* [37] measured flash point temperatures, densities, viscosities, acid values and cetane numbers of mineral diesel fuel, palm oil biodiesel, biodiesel-diesel fuel blends and biodiesel-diesel-alcohol blends. Some regression equations were proposed. Yuan *et al.* [38] measured kinematic viscosities of four biodiesels and their blends with No. 2 diesel fuel in the temperature range from 20 to 100 ℃. Vogel equation and a weighted semilog blending equation were used for viscosity-temperature and viscosity-blending ratio relationships, respectively. In the study performed by Alptekin and Canakci [39], sunflower, soybean, canola, corn and cottonseed oil biodiesels were produced and blended with two different diesel fuels at the volume basis of 2%, 5%, 10%, 20%, 50% and 75% to measure density, viscosity, pour point, distillation temperature and flash point of them. It was reported that the fuel properties were very close to those of diesel fuels at low concentrations up to 20% of biodiesel content in the blend. In the study carried out by Nogueira *et al.*

[40], binary and ternary blends containing soybean oil, soybean biodiesel and diesel fuel were prepared. Densities and viscosities were measured at 293.15 K, 313.15 K, 333.15 K, 353.15 K and 373.15 K at atmospheric pressure. The experimental data were correlated to predictive models based on the group contribution method. Wang *et al*. [41] reported density and viscosity of binary mixture for n-hexadecane with ethyl caprylate, ethyl caprate and ethyl laurate esters at the temperatures from 298.15 K to 323.15 K and at atmospheric pressure (0.1 MPa). Viscosities of binary blends were correlated, and excess molar volumes and viscosity deviations were obtained.

However, in literature, there are few studies about deriving new regression models for olive oil biodiesel-diesel fuel-n-butyl alcohol blends' densities and viscosities. Therefore, the aims of this study are (1) to determine densities and viscosities of the ternary blends including different n-butyl alcohol content, and (2) to derive new regression model for estimating kinematic viscosities of the ternary blends. In this context, first, olive oil biodiesel was produced and blended with commercially available diesel fuel at a volume ratio of 20%. The biodiesel-diesel fuel blend was mixed with 2, 4, 6, 8, 10, 15 and 20% volume ratios of n-butyl alcohol. The blends were prepared at room temperature. Then, densities and kinematic viscosities of the prepared diesel-biodiesel-n-butyl alcohol ternary blends were measured at 15°C and 40°C according to international standards, respectively. Finally, using the measurement data, the new four-term exponential model was proposed through the least squares method.

II. Materials and Methods

A. Materials

To produce biodiesel by means of basic-catalyzed transesterification, methanol, potassium hydroxide and anhydrous sodium sulfate (Merck, 99.6% purity) were used.

B. Biodiesel Production

Transesterification reaction parameters were taken as follows: 0.6250% catalyst concentration, 70°C reaction temperature, 90 minutes reaction time and 2.5 oil/alcohol volume ratio [42]. Anhydrous sodium sulfate was used to remove moisture from washed biodiesel. Transesterification reaction was carried out in a 1 L flat-bottomed reaction flask, equipped with a magnetic stirrer heater, thermometer and spiral reflux condenser. Before starting the reaction, potassium hydroxide was dissolved in methanol by stirring in a small flask, and this alcohol/catalyst mixture was added into the oil that was formerly warmed. Then, the final mixture was mixed with stirring by means of the magnetic stirrer heater. After the transesterification, lower phase (glycerol) was removed by a separating funnel, while upper one (biodiesel) was washed with warm distilled water at about 60 ℃. This process was repeated several times until the pH is neutral. The washed biodiesel was distilled under vacuum distillation to remove water and methanol. Then, it was dried over anhydrous sodium sulfate (left over night) and finally filtered using qualitative filter papers.

C. Density Measurement

The densities were determined at 15°C by means of pycnometer in accordance with ISO 4787 standard. Details of the measurements were given in [43]-[45].

D. Dynamic Viscosity Measurement

The dynamic viscosities were determined at 40°C in accordance with DIN 53015 standard. Measurements were made by universal Haake Falling Ball Viscometer, Haake Water Bath and stopwatch. Details of the measurements were given in [43]-[45].

The kinematic viscosities were determined by dividing dynamic viscosity to the density at the same temperature. The measurements were conducted three times for each sample and the results were averaged.

E. Uncertainty Analysis

Measured physical quantities are used to get targeted results in experimental studies. Uncertainties of measuring devices naturally cause uncertainties in the computed quantities, too. Uncertainty analysis provides us for determining uncertainties in the targeted results to be aware of the reliability of them. A method of Kline and McClintock [46] was used in this study to compute uncertainties of targeted results such as density and viscosity. According to this method, if the result R is a given function of the independent variables x_1, x_2, x_3, … x_n and w_1, w_2, w_3, … w_n are the uncertainties of each independent variables, then the uncertainty of the result w_R is calculated by using the equation:

$$w_R = \left[\left(\frac{\partial R}{\partial x_1}.w_1 \right)^2 + \left(\frac{\partial R}{\partial x_2}.w_2 \right)^2 + \left(\frac{\partial R}{\partial x_3}.w_3 \right)^2 + \ldots + \left(\frac{\partial R}{\partial x_n}.w_n \right)^2 \right]^{1/2} \quad (1)$$

III. Results and Discussion

Fig. 1 represents density values measured at 15°C for olive oil biodiesel-diesel fuel-butyl alcohol ternary blends. As shown in this figure, densities vary between 831.67 kg/m^3 and 837.99 kg/m^3, and they linearly decrease with increasing alcohol content in the blend, as expected, due to the lower density value of n-butyl alcohol compared to the biodiesel and diesel fuel. Therefore, the linear equation was fitted to the experimental values:

$$\rho = \rho(X) = a + bX \quad (2)$$

where ρ is density (kg/m^3), a and b are regression constants, and X is volumetric alcohol content in blend in %.

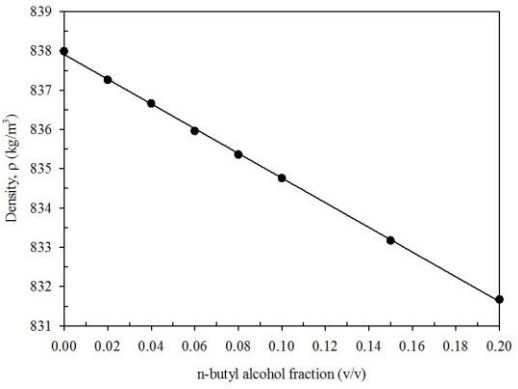

Fig. 1. Density variation with respect to n-butyl alcohol fraction.

Table I lists densities values, regression constants, R value and % relative errors between measured and calculated values from the linear equation. The R value and max. relative error were determined as 0.9999 and 0.0107%, respectively.

Fig. 2 illustrates kinematic viscosity change of the ternary blends versus alcohol content. Viscosities measured at 40°C vary between 2.557 mm^2/s and 3.094 mm^2/s, and they non-linearly decrease with increasing alcohol content. The four-term exponential model was proposed for the change of kinematic viscosity with respect to n-butyl alcohol fraction:

$$v = v(X) = ae^{-bX} + ce^{-dX} \quad (3)$$

where v is kinematic viscosity (mm^2/s), a, b, c and d are regression constants and X is volumetric alcohol content in blend in %.

TABLE I: MEASURED DENSITIES, % RELATIVE ERRORS AND REGRESSION CONSTANTS AND CORRELATION COEFFICIENT (R) FOR THE LINEAR EQUATION

Alcohol fraction X (%)	Measured density ρ (kg/m^3)	Regression constants		R	% relative errors
		a	b		
0	837.99	837.9	-31.45	0.9999	0.0107
2	837.26				0.0013
4	836.66				0.0022
6	835.96				0.0063
8	835.36				0.0029
10	834.76				0.0060
15	833.17				0.0015
20	831.67				0.0072

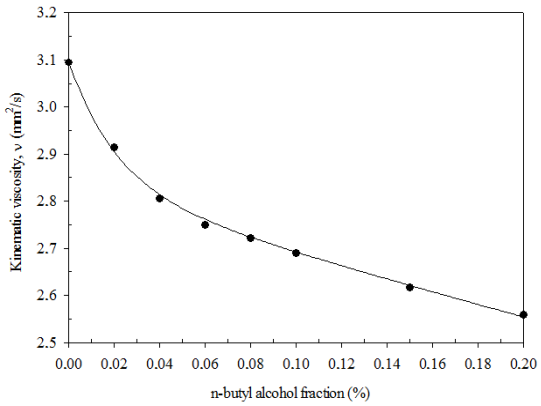

Fig. 2. Kinematic viscosity variation with respect to n-butyl alcohol fraction.

TABLE II: MEASURED KINEMATIC VISCOSITIES, % RELATIVE ERRORS AND REGRESSION CONSTANTS AND R VALUE FOR THE FOUR-TERM EXPONENTIAL MODEL

Alcohol fraction X (%)	Measured viscosity ν (mm^2/s)	Regression constants				R	% relative errors
		a	b	c	d		
0	3.094	0.2863	41.39	2.809	0.4705	0.9999	0.0420
2	2.914						0.2128
4	2.805						0.2246
6	2.749						0.2073
8	2.721						0.1948
10	2.689						0.1673
15	2.615						0.1223
20	2.557						0.0078

Table II lists measured kinematic viscosity values at 40°C, regression constants and R value for the four-term exponential model, and relative errors. As shown in this table, R value and max. relative errors were computed as 0.9999 and 0.2246%, respectively. According to Fig. 2 and the results given in Table II, the proposed four-term exponential model is quite suitable to (1) describe the variation of kinematic viscosity with respect to alcohol content and to (2) estimate kinematic viscosities of diesel-biodiesel-alcohol ternary blends.

IV. CONCLUSION

In this study, the basic fuel properties such as density and kinematic viscosity for olive oil biodiesel-diesel fuel-n-butyl alcohol ternary blends were measured according to the related international standards. Moreover, the four-term exponential model was proposed to estimate kinematic viscosities of the ternary blends. The following conclusions can be drawn from this study:

- Densities linearly and kinematic viscosities non-linearly decrease with increasing n-butyl alcohol content, respectively.
- The maximum relative error and correlation coefficient (R) for linear density-alcohol fraction variation were determined as 0.0107% and 0.9999, respectively.
- The proposed four-term exponential model with 0.2246% of maximum relative error and 0.9999 of the correlation coefficient is well suitable to describe the variation of kinematic viscosity-alcohol content in blend for the ternary blends.

Compared to the binary blends, decreasing of viscosities with using n-butyl alcohol can be considered to be positive effects on spray development and atomization hence engine performance and exhaust emissions while the use of much alcohol can also lead to poorer ignition quality i.e. diesel knock in a diesel engine.

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