An identification and characterization of biodiesel fatty acid based by using dielectric sensor

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Abstract. The fatty acids composition is identified by a gas chromatography mass spectrometer (GC-MS), then it is characterized in saturated and unsaturated components. This paper investigates biodiesel fatty acid by using dielectric constant measurements and focuses on dielectric sensor based on biodiesel chemical properties characterization. The objectives of this paper are identification fatty acids and determination of correlation dielectric properties and biodiesel fatty acid characterization. The proposed method is dielectric constant by using capacitance sensor are applied to determine the response dielectric sensor from the fatty acid composition. Sixteen fatty acid methyl esters were identified and two characterizations the amount of saturated and unsaturated fatty ester fractions. The model parameter was determined by regression analysis for estimating the relationships among fatty acid content and dielectric properties. The results show that measurements of electrical properties, successfully used for the characterization of fatty acids. The dielectric constant of biodiesel was found increasing as the saturated decreases. This relationship becomes calibration for the assessment of the quality of biodiesel based on the dielectric sensor. The model reveals that the fatty acid composition affects the value of the biodiesel dielectric and show that dielectric sensor potentially to handle for characterization of biodiesel fatty acid content.

1. Introduction

Biodiesel is a fuel based ester (Fatty Acid Methyl Ester: FAME) must meet the requirements of the specification to be used in diesel engines. A mixture of different fatty acid methyl esters is commonly referred to as biodiesel, which is a renewable alternative fuel. Triacylglycerol (oil or fat) transformed to form esters and glycerol esters are produced are called biodiesel [1,2,3]. ASTM International (American Society for Testing and Materials) defines biodiesel as a mixture of long-chain monoalkylic esters from fatty acids. The fatty acids identification was needed for production to determine the biodiesel quality. The composition of fatty acid content, structure feature and the length of carbon atoms determine the properties of physics and chemistry biodiesel. However, the physicochemical analysis requires high costs and time. This study presents the use of a dielectric
sensor and a quick and inexpensive method to correlate the physicochemical variables to the dielectric constant of the fatty acids biodiesel characterization. The change in dielectric constant of FAME from different biodiesel production will be presented. This research wants to apply the application of dielectric measurements to identified saturated degree of biodiesel fatty acid content, thus, the relation between the electrical properties and the degree of saturated were analyzed. The proposed method allows reducing costs in the characterization of fatty acids and the reduction in analysis time. In addition, the method allows an assessment of the quality of biodiesel based on fatty acids composition. As a commercial fuel, biodiesel quality is analyzed using sophisticated equipment to make sure it meets the requirements of international standards and to be stored in the long term. The quality of biodiesel based on physicochemical properties depending on the distribution of fatty acid [4], [5]. Generally, gas chromatography (GC) has been the most widely used method for the analysis of biodiesel owing to its generally higher accuracy in quantifying minor components but not typically used for online measurement [6]. However, the accuracy of GC analyses can be influenced by factors such as baseline drift, overlapping signals, derivative samples and aging of standards and samples. Furthermore, gas chromatography is a measurement that is not real-time and requires expensive equipment costs that are not easily applied to process monitoring and control in small and medium scale industries [7].

Most of the important parameters of the end product biodiesel is a fatty characterization of mono-alkyl esters, fatty acids, glycerol, and derivatives. Feature structure, the length of the carbon atom, and the degree of saturation of fatty acids determines the physical properties of biodiesel such as viscosity, flash point (FP), cetane number, density, high heating values [5], [8]–[12]. It takes the alternative technologies that are a simple, non-invasive and low cost to evaluate and monitor the production process and the quality of biodiesel. Romano and Sorichetti 2011, the parameters that determine the quality of biodiesel can be classified into two major groups, the first includes public properties (density, viscosity, flash point, cloud point, pour point, cetane number, numbers neutralization), the second describes the chemical composition and purity of a mixture of fatty acid esters (alcohol content, ester content, the content of mono, di and triglycerides, total quantity of glycerol, iodine value).

Some measurement techniques such as the implementation of electrochemical impedance spectroscopy [14], dielectric spectroscopy [15], have managed to characterize the physical properties of biodiesel based on the dielectric constant, resistivity and relaxation time or frequency. Dielectric spectroscopy has been successfully used for monitoring the production and characterization of biodiesel, these measurements give a quantitative indication of the advance of the purification process and the conversion to biodiesel [13]. Permittivity and Conductivity are used to characterize feedstocks from different origins and biodiesel content estimation of biodiesel blend [7], [16], to characterize the different stages of the production process during the purification process and in the final product [17], [18], and also for the characterization of FAME [19]. Although in the literature there are references on the dielectric of biodiesel properties there is comparatively less information on correlation dielectric sensor with fatty acid in the final product. The physical and chemical fuel properties of biodiesel fundamentally depend on the fatty acids distribution of the triglyceride obtained from the raw material used for biodiesel production [5], [8]. Fatty acid content such as higher degree of unsaturation of biodiesel fuels led to a longer ignition delay and, consequently, a more retarded start of combustion [20]. Dielectric measurements are relevant for the production and characterization of biodiesel since the refractive index of FAME depends essentially on the electronic polarization of the molecules of fatty acid esters. The reduction of the dielectric losses of treated oil is affected due to the elimination presence of contaminants [16], [17]. The differences in dielectric properties among tested oils generally correspond to the degree of unsaturation as evidenced by iodine values (IV), increase with the degree of unsaturation or the number increasing of double bonds of unsaturated fatty acids in oils [21]–[23]. Since dielectric properties of materials are closely correlated with saturated/unsaturated fatty
acids in oils and therefore, researchers properly designed and calibrated electrical instruments, which can be directly used to determine the saturated/unsaturated fatty acids in oils accurately. The technique is reliable enough for predicting fatty acid in biodiesel.

In this paper, we report results from the investigation of fatty acid content in biodiesel production used the dielectric sensor. Quantitative characterization and quality biodiesel based on fatty acid composition were analyzed using Gas Chromatography, which the amount and type of fatty acids determine the physical properties of biodiesel [24]. Dielectric sensors use capacitance to measure the dielectric constant of a surrounding medium. The sensitivity of the capacitive sensor is affected by the dielectric constant of the target material being detected. The permittivity of materials is most commonly expressed in relation to the permittivity of free space and therefore named relative permittivity (dielectric constant). The same dielectric models applied to biodiesel with the content and composition of fatty acids that are different. The dielectric sensor is currently in widespread use in biodiesel production since it makes possible the characterization of the sample and the detection of moisture and other contaminants that impact on the quality of the feedstocks and the final product. In this paper, biodiesel classification is determined by dielectric constant measurements in the range from 100 Hz to 100K Hz, for samples of biodiesel at room temperatures. As an alternative biodiesel quality testing, dielectric properties require little independent evaluation.

The electrical properties are capable of measurements in both laboratory and industrial environments, so very potential applied to the automated process control from online measurement systems [19]. The objectives of this paper are identification fatty acids by Gas Chromatography and determination of correlation dielectric properties and characterization of biodiesel fatty acid. This relationship becomes calibration for the assessment of the quality of biodiesel based on the dielectric sensor. The results presented in this work show that dielectric sensor is potentially useful for these purposes, and this technique is fast, non-destructive, real time and reasonable cost.

2. Materials and methods

2.1. Materials

Samples of biodiesel used are biodiesel feedstock RBDPO and used cooking oil. Testing was performed using three samples in different production systems. The percentage composition of fatty acids in the biodiesel was determined by the gas chromatography-mass spectrometer (GCMS), and the dielectric properties were determined by LCR meter.

2.2. Biodiesel Characterization

Biodiesel Characterization by using GC-MS analysis showed that major fatty acid components in all esters were saturated and unsaturated fatty acid. The retention time and peak height/area were used in qualitative and quantitative determination of the biodiesel components. The presence of saturated fatty acid in the obtained biodiesel leads to high viscosity, high cetane number and better biodiesel stability [25]. The dielectric constant values of biodiesels were mainly affected by unsaturated fatty acids. Further- more, the dielectric constant of biodiesels increased with increasing the degree of unsaturation or the number increasing of double bonds of unsaturated fatty acids in biodiesel [23]. The higher level of unsaturated fatty acid reduces fuel quality, because of its easy oxidation [26] and saturated fatty acids such as 16:0 or 18:0 are more stable than unsaturated ones like 18:1, 18:2 and 18:3, respectively, which decreases the fuel quality [27].

Generally, the main components of each biodiesel were five FAMEs methyl palmitate (hexadecanoic acid, methyl ester; CAS No. 112-39-0), methyl stearate (octadecanoic acid, methyl ester; CAS No. 112-61-8), methyl oleate (octadecenoic acid, methyl ester; CAS No. 112-62-9), methyl linoleate (octadecadienoic acid, methyl ester; CAS No. 112-63-0), and methyl linolenate (octadecatrienoic acid, methyl ester; CAS No. 301-00-8). Three other FAMEs were present in minor amounts methyl
myristate (tetradecanoic acid, methyl ester; CAS No. 124-10-7), methyl arachidate (eicosanoic acid, methyl ester; CAS No. 1120-28-1), and methyl behenate (docosanoic acid, methyl ester; CAS No. 929-77-1) [25], [28]. The composition of biodiesel fatty acids is varying from source to source and usually contains methyl esters of C-12 to C-24 fatty acids having quite appreciable quantities of unsaturated (C18:1 and C18:2) fatty acids [29].

Quantitative analysis is used to identify the amount of fatty acid in biodiesel because biodiesel quality is reflected in the composition of fatty acids [30]. Identification of FAME components of biodiesel was analyzed by using GC-MS (ASTM E202). Gas chromatography to analyze the content of fatty acid using Agilent 6890 GC Method. The oven program was given as follows initial temperature 60 °C post temperature 80 °C maximum temperature 325 °C. The previous studies have done calculations fatty acid composition of biodiesel based on measurements GCMS [29], [31].

2.3. Dielectric Properties Measurement

The testing dielectric of biodiesel using a sensor with two parallel electrodes is done by fixing in PCB plate. Dielectric measurements on a sample of biodiesel using LCR meter Lutron 8014. The tests were carried out and verified with the various reference fuel in 3 sample reference biodiesels from RBDPO and cooking oil. Some previous researchers showed that measurements of electrical properties, successfully used for the characterization of fatty acid methyl ester (FAME) based on mixtures of fatty acid methyl esters from different vegetable oils [19], [32], [33].

A capacitor is a device that stores electric charge. The simplest example of a capacitor consists of two conducting plates of area A (A = 4 cm x 4 cm), which are parallel to each other, and separated by a distance d (d = 2.5 mm) as shown in Figure 1. Electrical properties of the samples were determined from dielectric constant in the frequency 100 Hz and at room temperature.

The capacitance C (farads) of a parallel plate capacitor of area A (m²), the separation between the plates separated by empty space (or air) is:

\[
C = \varepsilon_0 \frac{A}{d}
\]  

(1)

where \( \varepsilon_0 \) is the permittivity of free space (8.85 x 10^{-12} F/m, farads/meters), d is the separation between the plates (meter). If the empty space is replaced by a dielectric, capacitance increases [34], [35]. The dielectric constant of material is complex and proportional to the material electrical permittivity and free space permittivity.

\[
K = \frac{\varepsilon}{\varepsilon_0}
\]  

(2)

\[
K = \varepsilon_r + i\varepsilon_i
\]  

(3)

where K is the complex dielectric constant, \( \varepsilon \) is the electrical permittivity, \( \varepsilon_0 \) is the permittivity of free space, \( \varepsilon_r \) is the real part of the complex dielectric constant will be referred to as the dielectric constant of material and \( \varepsilon_i \) is the imaginary part of the complex dielectric constant can be called loss factor, dissipation factor or dielectric loss [36]–[40]. The permittivity of materials is most commonly expressed in relation to the permittivity of free space and therefore named relative permittivity (dielectric constant).

**Figure 1.** A parallel-plate capacitor
3. Results and Discussion

In order to understand the fatty acid content and dielectric constant of biodiesel. The contents of fatty acids were analyzed using GCMS, identify the fatty acid content consisting of saturated and unsaturated. Furthermore, the correlation analysis of dielectric properties and chemical composition based on the amount of saturated and unsaturated.

3.1. Chemical Composition of Biodiesel Using GC-MS

GC-MS analysis was used the chemical composition of the three biodiesel fuel samples for profiling of various fatty acid methyl esters. Twelve peaks were observed in the area picture; each peak corresponds to a fatty acid methyl ester and was identified from the library match software (Willey09TH). The fatty acid chromatogram showing the different components present in each biodiesel sample.

Each FAME was present in different proportions in the three samples. The physicochemical properties of biodiesel are influenced by the structural features of fatty acid, chain length, the degree of unsaturation, and branching of the chain [8]. Table 1 gives the comparative analysis of saturated and unsaturated properties of fatty acids. The result shows that the biodiesel contains saturated fatty acid 63.97%, 61.93%, 55.23%, and unsaturated fatty acid 36.03%, 38.075%, 45.062% respectively for sample 1 (S1), sample 2 (S2) and sample 3 (S3). The characteristics of these biodiesel samples fall within a narrow band. Methyl ester contains a higher percentage of saturated fatty acid, with a lower percentage of unsaturated from GC analysis showed which clearly proved that the prepared esters have a very good fuel property [25]. The influence of these proportions on the dielectric properties will be discussed in the sections that follow.

3.2. Dielectric constant of Biodiesel

Dielectric properties of biodiesel samples were measured at three different total amount of saturated and unsaturated fatty acid. In the dielectric characterization of materials, simple measurement is capacitance and then converted to the dielectric constant. Capacitance measurement of materials and air using LCR meter, in which the tube is filled to biodiesels and measurement of air in the empty tube. Calibration of measuring instruments in the short and open circuit. Based on the results of measurements of air capacitance, constant dielectric obtained on the empty cell is 1,005. Constant dielectric was calculated by the formula 1 and 2. The dependence of the dielectric constant on the frequency and saturated fatty acid content in this work are illustrated in Figure 2. The dielectric constant of all samples decreased with increasing frequency at individual saturated fatty acid. Generally, the dependence of the dielectric constant of biodiesels on frequency as well as overall values, are similar to those obtained for other agricultural commodities including those used in other biofuel studies was also reported [39], [41]–[44].

Prediction content of saturated FAME is possible using the measurement of dielectric because it has different properties in each polar fatty acid component. This observation is in close agreement with earlier results, i.e., [25] observed comparative analysis of saturated properties with unsaturated properties of fatty acids and [23] reported the effect of oil composition on dielectric properties. Saturated FAME of biodiesel FAME composition for predicting physical properties of biodiesel from its FAME composition using regression equation. Dielectric measurements can be used to predict the FAME content of saturated fatty acids in biodiesel because have different polar properties. Using the regression equation by previous researchers that the physical properties of biodiesel and biodiesel fatty acid composition have an excellent correlation [45], [46].

Figure 3 shows the dependence of dielectric constant on saturated fatty acid content at 100 Hz, 120 Hz, 1K Hz, 10 K Hz and 100 K Hz. This observation is appropriate with earlier results [23,37] reported the differences in dielectric properties among tested oils generally correspond to the degree of
unsaturation. Fatty acid composition of biodiesels is another determining factor of dielectric constant. Dielectric constant increases with the decreases in the degree of saturation for all frequencies, which increases in the degree of saturation as the electron rich double bond has a negative charge and tends to oscillates to a certain extent, with the changing electric field.

The saturated fatty acids contain single bonds between carbon atoms of parent chain of molecule. Each carbon atom connected with four other atoms in molecule. Since all are single covalent bonds which are sigma bonds, C-C bonds are very strong and difficult to cleave under normal conditions. The unsaturated fatty acids have multiple covalent bonds like double or triple covalent bonds between carbon atoms of parent chain which have pi-bonds. Pi-bonds are weaker than sigma bonds so pi-bonds can easily cleaved. The ability for the π electron to move freely between carbon atoms gives rise to a high electrical conductivity [33], [47]. The permittivity depends on dielectric constant \( \varepsilon' \), which is related to capacitance of a substance and its ability to store electrical energy.

![Figure 2](image2.png) **Figure 2.** The dielectric constant as function of frequency for different saturated fatty acid contents

![Figure 3](image3.png) **Figure 3.** The dielectric constant as a function of saturated fatty acid
Table 1. Measured values of fatty acid in biodiesel

| Type       | Fatty acid      | Peak area (%) |
|------------|-----------------|---------------|
|            |                 | S1  | S2  | S3  |
| Unsaturated| 1. Oleic        | 35.466 | 34.698 | 41.701 |
|            | 2. Palmitoleic  | 3.021  |        |      |
|            | 3. Eicosenoic   | 0.564  | 0.356  | 1.854 |
|            | 4. Linoleic     |        |        | 0.719 |
|            | 5. Octadecadienal|     |        | 0.467 |
|            | 6. Eicosapentaenoic |    |        | 0.321 |
| Total unsaturated |      | 36.030 | 38.075 | 45.062 |
| Saturated  | 1. Caprylic     | 0.215  |       | 0.383 |
|            | 2. Lauric       | 0.567  | 1.247  | 1.041 |
|            | 3. Palmitic     | 45.84  | 45.521 | 40.210 |
|            | 4. Stearic      | 10.037 | 8.806  | 6.756 |
|            | 5. Arachidic    | 1.674  | 0.427  | 1.860 |
|            | 6. Myristic     | 4.734  | 4.553  | 4.332 |
|            | 7. Margaric     |        | 1.156  |      |
|            | 8. Hexadecanoic | 0.489  |        |      |
|            | 9. Docosanoic   | 0.328  |        | 0.358 |
|            | 10. Lignoceric  | 0.299  |        | 0.290 |
| Total Saturated |       | 63.970 | 61.925 | 55.230 |

The dielectric properties of biodiesels were correlated with their different compositions using a statistical model. The best subset regression was used to determine which parameters affect the dielectric properties of biodiesels such as dielectric constant. The parameters include percent of fatty acids which are the main components in biodiesel. For saturated FAME, there is a nonlinear decrease in dielectric properties with increasing saturated fatty acid content. The plot of the predicted saturated fatty acid versus the actual output saturated fatty acid are depicted in Figure 4. The saturated fatty acid of FAME was given as the dependent variable Y, while the dielectric properties (capacitance and dielectric loss) were given as variable x. Regression diagnostic performs a multilinear regression of the response in Y on the predictors in x described as the following multiple regression equations:

$$Y=77.86+0.05 \times_1 - 90.38 \times_2$$  \hspace{1cm} (4)

where Y is the predicted saturated fatty acid (%) of biodiesel, $x_1$ is capacitance and $x_2$ is a dielectric loss of dielectric properties of biodiesel. A high correlation coefficient ($R^2=0.981$) indicates the excellent correlation between the saturated fatty acid and the dielectric properties of biodiesel.

![Figure 4. The saturated fatty acid prediction base on dielectric properties](image-url)
Several kinds of biodiesel derived from RBDPO and cooking oil were selected to verify the practicability of the dielectric measurement. Their typical FAME compositions are shown in Table 1 base on GC-MS. The predicted saturated fatty acid of these biodiesels is also plotted in Fig. 4. The desired result is observed namely, that all of the predicted values are in the range of the experimental values. The results are consistent with earlier results, it is reported that dielectric constant for vegetable oil, soybean oil is higher than palm olein suggesting that dielectric increase with degree of unsaturation [23], [48]. It seems that the technique is reliable not only in preliminary tests for specific biodiesel origin. This indicates that the present correlative model is a simple and practical method to give a satisfactory predicted saturated fatty acid of FAME composition of a biodiesel from its dielectric properties without arduous and expensive experimental determination.

The measurements of dielectric properties, successfully used for the characterization of fatty acids and the dielectric sensor potentially to handle for characterization of biodiesel fatty acid content. In practice, in the case of an important number of potential effects, this could be achieved from the computational viewpoint by training. Need more observation by using another parameter such as temperature to develop a handy measurement for fatty acid identification.

4. Conclusion
Fatty acid characterization using dielectric sensor showed that the increasing dielectric constant of biodiesel was found as the saturated decreases. The results show that measurements of electrical properties, successfully used for the characterization of fatty acids. For future works, it is required to embed this approach to a handy sensor that integrates dielectric probe with identification tool.

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