

MONITORING MICROWAVE DIELECTRIC PROPERTIES DURING TRANSESTERIFICATION REACTION FOR PALM BIODIESEL PRODUCTION

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ABSTRACT

Microwave energy offers a fast method to produce biodiesel with advantages of enhancing the reaction rate and improving the reaction separation. The dielectric properties at frequency range from 0.2 GHz to 20 GHz of a mixture during transesterification reaction which are used for producing palm biodiesel were presented. The monitoring was done by placing a dielectric probe at the upper level and lower level of the mixture to measure the dielectric properties during the formation of biodiesel and glycerin respectively. In the transesterification reaction, the mixture consisting of vegetable oil, methanol and sodium hydroxide was heated at a constant temperature of about 45-50°C and stirred for homogeneity. Results of the measurement over the frequency range showed drastic changes on the dielectric properties in the first 3 minutes of the reaction and after 3 minutes, the dielectric properties slowly decreased and approached the dielectric properties of biodiesel or glycerin. This study gives valuable information on the optimum mixing time for microwave transesterification reaction. These properties could be used to estimate absorbed power by the mixture for application in microwave transesterification process as dielectric loss of the mixture changed from 20 to about 0.5 as the transesterification reaction was completed.

INTRODUCTION

Palm biodiesel is an alternative fuel derived from palm oil and can be used in compression ignition engines, i.e. diesel engines without any modifications. Biodiesel is renewable, biodegradable, non-toxic and safe to handle and most importantly, free from sulfur. It refers to methyl esters derived from palm oil through a process known as 'transesterification'. The process involves reacting vegetable oil with methanol (alcohol) in the presence of a catalyst (sodium hydroxide) to produce biodiesel and glycerol [6].

In this paper we propose a microwave measurement technique for monitoring the dielectric properties of the reactants during transesterification reactions until the formation of biodiesel and glycerol. The application of microwave irradiation in the

transesterification process greatly reduces the time required to achieve maximum conversion [1][7][8][10]. It was claimed that the use of microwave-assisted transesterification process dramatically reduces the reaction time from 75 min to 4 min (at 60°C) thus involving a dramatic save in time [8]. The power absorbed per unit volume by the material under the microwave radiation can be expressed as

$$P = 2\pi f \epsilon_0 \epsilon''_{eff} |E_{in}|^2 \dots\dots\dots (1)$$

where E_{in} is the magnitude of the internal electric field, ϵ''_{eff} is the relative effective loss factor, ϵ_0 is the permittivity of free space and f is the operating microwave frequency. The knowledge about dielectric properties of raw material used in the transesterification process, reactants during transesterification reactions and the final products are useful for the prediction of microwave power absorption during the microwave-assisted transesterification reaction which is normally done at frequency 2.45 GHz.

The objective of the work is to utilize microwave applications for the production of biodiesel from palm oil. The measurement of the dielectric properties with time was implemented for the frequency range from 0.2 GHz to 20 GHz. The results of the dielectric properties at 2.45 GHz were presented as it is useful for microwave transesterification application.

EXPERIMENTAL DETAILS

Materials

In this experiment, a feedstock, alcohol and catalyst were used to produce palm biodiesel as described below;

Feedstock

Natural vegetable oils are extracted or pressed to obtain crude oil. These usually contain free fatty acids, phospholipids, sterols, water, odorants and other impurities. For the purpose of this study, palm oil was purchased from grocery in Serdang, Selangor.

Alcohol

Alcohols are primary and secondary monohydric aliphatic alcohols having 1-8 carbon atoms. Among the alcohols that can be used in the transesterification process are methanol, ethanol, propanol, butanol and amyl alcohol. Methanol and ethanol are used most frequently, especially methanol because of its low cost and its physical and chemical advantages (polar and shortest chain alcohol). In this study, methanol was the alcohol of choice. [Methanol (Analytical) HmbG Chemicals Array 99.8%].

Catalyst

Among the most commonly used alkaline catalysts in the biodiesel industry is sodium hydroxide (NaOH) and alkali-catalyzed transesterification is much faster [6]. In this study NaOH pellets were used about 0.5-1.0% by weight of oil. The catalyst was measured only when ready to immediate use to prevent hydration.

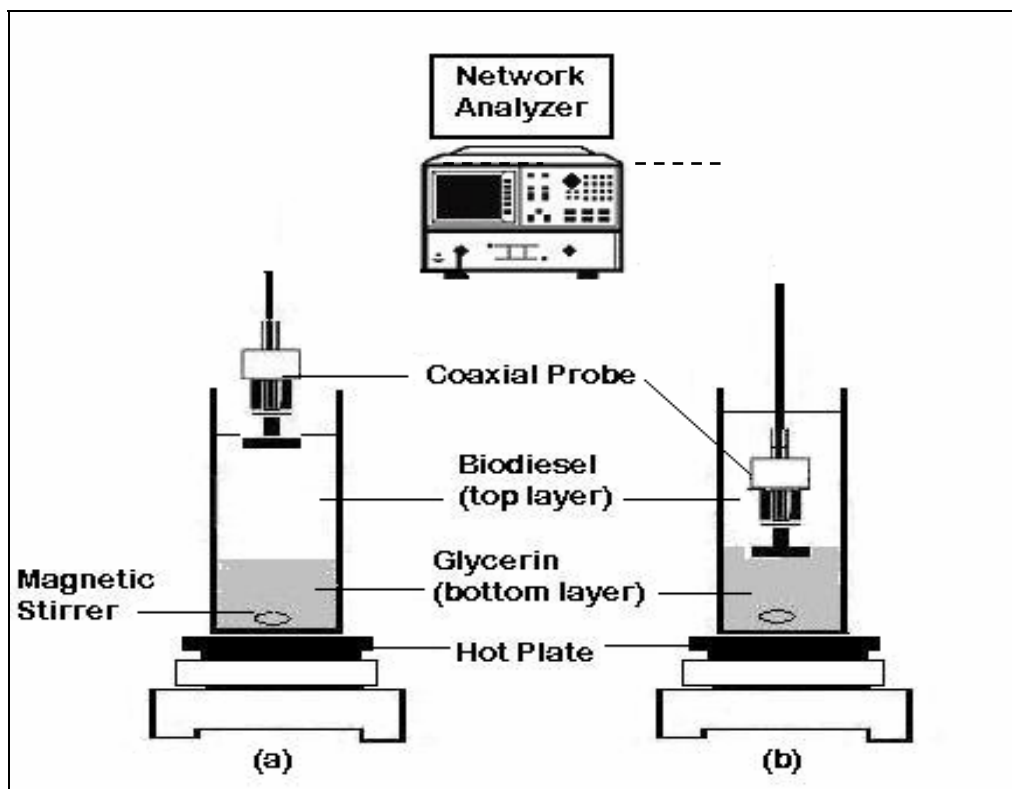


Figure 1: Measurement set-up for monitoring microwave dielectric properties of the mixture during transesterification reaction (a) biodiesel detection (b) glycerin detection.

Measurement of dielectric properties was done with a HP Network Analyser 8270B using the open-ended coaxial probe method (Gregory et al. 2006) with the frequency ranging from 0.2 GHz to 20 GHz. The network analysers was controlled by Hewlett-Packard 85070B dielectric kit software and calibrated using air, short-circuit and deionised water at 25°C. All measurements were implemented at room temperature 25°C - 27°C. In each measurement the sample was placed in the cylindrical container and the sensor was placed facing downward inside the sample at about 3 mm from the surface. The thickness of the sample was about 3 cm. For monitoring dielectric properties of the mixture during transesterification reactions two sets of mixture were prepared. The first set for detection of the formation of glycerin at the lower layer (see Fig. 1b) and second set of reaction for detection of biodiesel (see Fig. 1a). Both mixture were generally heated at constant temperature 45-50°C for several minutes until a clear separation between biodiesel and glycerin (as by-product) can be observed. The dielectric properties of the reaction were monitored for every minute until complete formation of biodiesel and glycerin.

RESULTS AND DISCUSSION

Fig. 2 shows the spectrum of the dielectric properties of the vegetable oil (palm oil). By observing the dielectric loss spectrum, generally the dielectric loss for vegetable oils is in the lower region because there is no contribution from conductive loss and dipole orientation.

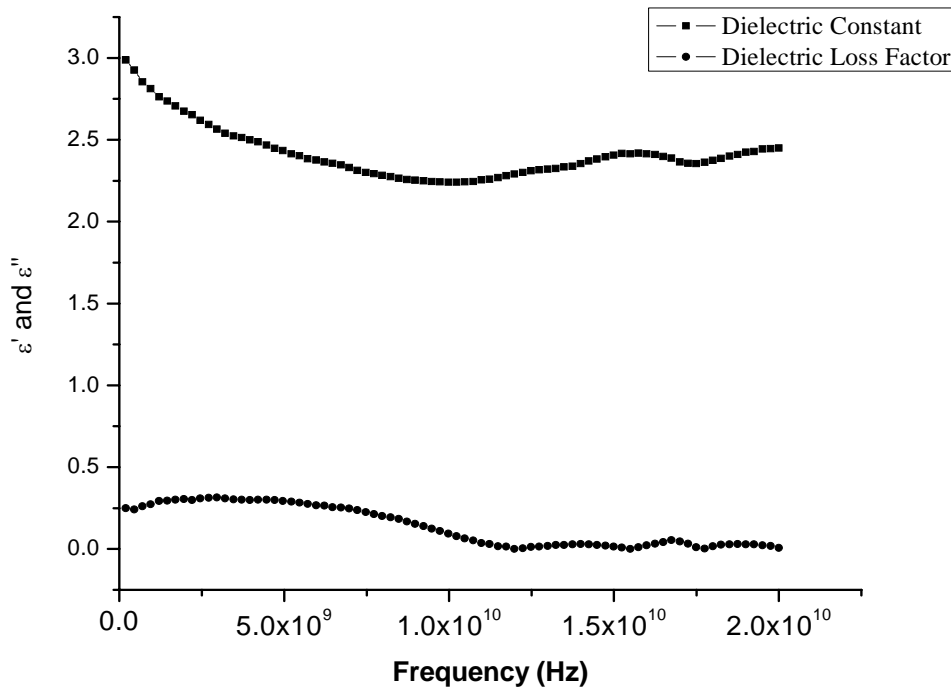


Figure 2: Dielectric properties of palm oil

Dielectric properties of methanol is quite high at frequency around 2.45 GHz which is due to dipole orientation as can see in Fig. 3.

As methanol is added with catalyst, the dielectric loss is much higher which results from the contribution of the ionic conductivity of the NaOH. It is clearly seen that at 2.45 GHz, the dielectric loss of NaOH at 10% concentration can reach the value of dielectric loss of about 21.

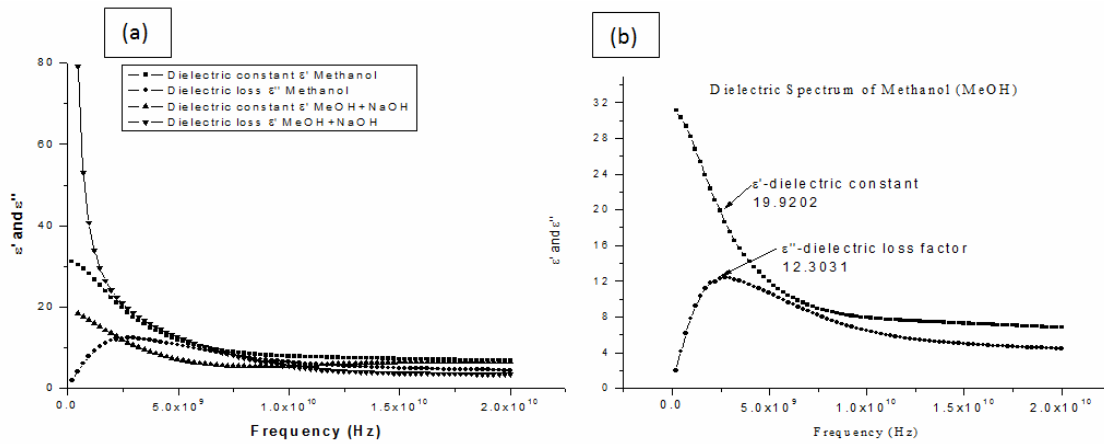


Figure 3: Dielectric properties of (a) the mixture of methanol and sodium hydroxide and (b) methanol.

Generally at the beginning of transesterification reaction, the dielectric loss of the mixture (methanol, vegetable oil and NaOH) at 2.45 GHz is about 18 which are quite high and suitable for microwave transesterification reaction.

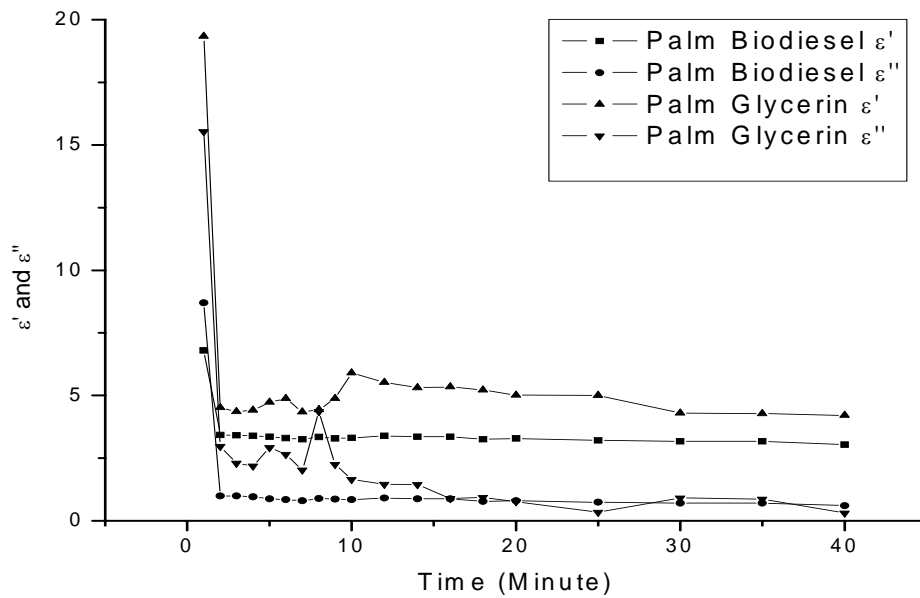


Figure 4: Variation of dielectric properties for palm oil mixture with time during transesterification reaction until the formation of biodiesel at top layer and glycerin at lower layer at 2.45 GHz.

The variation of the dielectric properties during transesterification reaction at 2.45 GHz in details is shown in Fig.4. It is clearly shown that within one to three minute palm oil shows a sudden drop in the dielectric properties of the mixture. For the dielectric properties of palm biodiesel the value at 2.45 GHz is about $3.3-j0.67$. In this conventional transesterification reaction, it normally takes about 20-30 minutes to complete the reaction, however from this observation, after 10 minutes the mixture already approached stability. Dielectric properties at the bottom layer are unsmooth as the mixture approached for the complete formation of glycerin. This effect may be due to the disturbance from the stirrer which is placed at the bottom of the container and non uniform temperature distribution in the mixture.

CONCLUSION

The results of the dielectric properties of the materials used in the transesterification process and kinetic dielectric properties during transesterification reaction in palm biodiesel especially at 2.45 GHz were presented. This information is useful for the optimization of reaction time, to estimate the power absorption during microwave transesterification process. It is clearly shown that the reaction time for formation of palm biodiesel is about 25 minutes.

ACKNOWLEDGEMENT

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