

**2012 International Conference on Advanced
Material and Manufacturing Science
(ICAMMS 2012)**

December 20-21, 2012

Edited by

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Trans Tech Publications

2012.12

Preface

We cordially invite you to attend the 2012 International Conference on Advanced Material and Manufacturing Science (ICAMMS 2012) & 2012 International Conference on Frontiers of Mechanical Engineering, Materials and Energy (ICFMEME 2012), which will be held in Beijing, China during December 20-21, 2012. The mission is to bring together experts in the field of Material, Manufacturing Science, Mechanical Engineering and Energy, and to discuss emerging issues in the knowledge innovation era.

Since June 2012, the Organizing Committees have received more than 950 manuscript papers, and the papers cover all the aspects in Material, Manufacturing Science, Mechanical Engineering and Energy. Finally, after review, about 420 papers were included to the proceedings of ICAMMS 2012 from more than 950 submissions. The conferences have been supported by many universities and research institutes, such as Beijing University of Posts and Telecommunications, North University of China, Beijing Jiaotong University, Science Technology Press. Many Professors of these universities and research institutes play an important role for the successful holding of the conference, so we would like to take this opportunity to express our sincere gratitude and highest respects to them.

At the same time, we will also express our sincere thanks for the understanding and support of every author. Owing to time constraints, imperfection is inevitable, and any constructive criticism is welcome.

With our warmest regards,
Conference Organizing Committees
December 20-21, 2012
Beijing, China

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Feasibility of using high-intensity ultrasound assisted biodiesel production from mixed crude palm oil in two-step process

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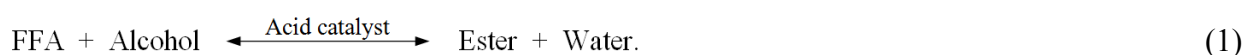
Keywords: transesterification; ultrasound; biodiesel; mixed crude palm oil

Abstract. Biodiesel from a high free fatty acid (FFA) mixed crude palm oil (MCPO) can be produced to high fatty acid methyl ester (FAME) conversion by a two-step process. The first process is acid-catalyzed esterification to reduce FFA in oil followed by a base-catalyzed transesterification process to produce biodiesel from esterified oil. In this study, transesterification of esterified oil with methanol (MeOH) in the presence of KOH as base-catalyst was performed in a 1,000 W ultrasonic homogenizer at a low frequency of 18 kHz. The use of high-intensity ultrasound to accelerate the reaction, the high surface power density of 1.62 W/mm² and the volumetric acoustic energy of 20 W/mL were fixed. The objective of this study was to determine the various parameters (KOH concentration, MeOH concentration, and initial temperature of oil) to produce the FAME conversion. The results showed that over 98 wt.% of FAME conversion could be achieved with 5 g KOH/liter of oil, 15 vol.% of MeOH, the total residence time of 20 seconds, and temperature of 30 °C. Moreover, the triglyceride (TG) was rapidly converted to FAME within reaction time of 10 seconds when using the base-catalyst of 10 g KOH/liter of oil and 20 vol.% of MeOH. Consequently, the use of high-intensity ultrasonic irradiation can shorten the reaction time, reduce the chemical and electricity costs.

Introduction

Biodiesel is a renewable alternative fuel that can be produced from vegetable oils or animal fats and used in any compression ignition engine without major modifications [1,2]. Furthermore, the biodiesel fuel has many advantages over petroleum diesel fuel, as it produces less smoke and airborne particles. It is nontoxic together with having lower carbon monoxide levels and low hydrocarbon emissions and being a biodegradable, renewable fuel [3,4]. Biodiesel is defined as a fatty acid methyl ester (FAME) or fatty acid ethyl ester (FAEE). It can be produced by transesterification of TG with mono-alcohol in the presence of either an alkaline or acidic catalyst. The TG is converted in three steps to diglyceride (DG), monoglyceride (MG), and glycerol (GL) in the production of three moles of esters and one mole of glycerol [5].

The major problem in biodiesel production from mixed crude palm oil (MCPO) arises from the free fatty acid (FFA) content. The FFA content in the raw material using a transesterification reaction should not exceed 1 wt.% since it will react with alkalis to produce soap (the saponification process). As a result, ester conversion was decreased by the formation of soap [4,6]. In this study, the two-step biodiesel production was used to study the FAME from MCPO using high-intensity ultrasonic irradiation assisted by low-frequency ultrasonic irradiation of 18 kHz. The esterified oil was produced by continuous acid-catalyzed esterification of the first-step process. the FFA was converted to esters by direct esterification with the acid-catalyst. Esterification reaction was shown in Eq. 1. Subsequently, the esterified oil was used as a feedstock in the base-catalyzed transesterification to produce the biodiesel. Transesterification reaction was shown in Eq. 2.





Oil is normally mixed with solution of alcohol and a catalyst in a constant stirred tank reactor. This is quite time and energy consuming. Using ultrasound instead of mechanical stirrer, the reaction time can be shortened. In sonochemistry, ultrasound can generate acoustic cavitations through high intensity acoustic fields in the medium phase until they reach a cavitation phenomenon that creates a bubble collapse. Before the bubbles collapse, the inside of the bubble may have as high pressure as 5000 atm that causes an instant temperature rise to at least 7200°C which in turn causes changes in the rapid physical and chemical reactions of the reaction mixture [7]. In addition, the ultrasonic field can increase the interface area between the immiscible fluids, resulting in rapid mixing in the liquids [8]. Most researchers believe that physical effect of ultrasound on the formation of the fine emulsion between immiscible fluids is responsible for accelerating the homogeneous transesterification reaction. However, few researchers have studied the biodiesel production from high FFA of oil by the ultrasound in two-step process. For instance, Deng et al. [9] studied the two-step process, this process was used to produce biodiesel from *Jatropha* oil having a high FFA content. All experiments were carried out in an 210 W ultrasonic reactor. Each mixture of *Jatropha* oil, methanol (MeOH), and catalyst (H₂SO₄ or NaOH) was filled in a 500 mL three-neck flask. The mixture was stirred at 600 rpm and was submerged in a water bath of ultrasonic cleaner under temperature of 60 °C. In first process, H₂SO₄ concentration of 1, 2, 3, 4, 5 and, 6 vol.%, MeOH concentration of 16, 24, 32, 40, and 48 vol.%, were used to investigate the reduction of the acid value. The results showed that after this pretreatment process, the acid value of *Jatropha* oil was reduced from 10.45 to 1.2 mgKOH.g⁻¹ with 40 vol.% of MeOH, 4 vol.% of H₂SO₄, and the reaction time of 1 hour. Subsequently, the esterified oil was used as the raw material of base-catalyzed transesterification to study the biodiesel yield. The results showed that a maximum purity of the methyl ester of 96.4% was achieved when 1.4 wt.% NaOH, 24 vol.% MeOH, and the reaction time of 30 min were used.

In current research, most of either transesterification or esterification reactions used low-intensity ultrasound for accelerating the reaction. As a result, the reaction time was typically long, whereas shorter reaction time should be attainable through a high-intensity ultrasound by increasing the acoustic energy density in a small ultrasonic reactor. Consequently, the objective of this present work is to study the base-catalyzed transesterification in batch reactor using high-intensity ultrasound. The esterified oil with methanol in the presence of KOH was carried out in the ultrasonic homogenizer at a low frequency of 18 kHz, methanol concentration, base-catalyst concentration, and initial temperature of esterified oil were investigated.

Materials and Methods

Materials. The FFA in mixed crude palm oil (MCPO) was reduced by the ultrasonic continuous acid-catalyzed esterification. The esterified oil from the esterification was used as feedstock in transesterification. The esterified oil containing 0.262 wt.% FFA, 12.099 wt.% ester, 77.367 wt.% TG, 8.673 wt.% DG, 1.599 wt.% MG, 1.16 wt.% MeOH, and 0.138 % water content. All chemicals, including 95% KOH and 98% MeOH, were commercial grade. A thin layer chromatograph with flame ionization detection (TLC/FID) (IATROSCAN MK-65; Mishubishi Kagaku Iatron Inc.; Tokyo, Japan) was used to analyze the conversion of FAME. Analysis used the following chemical standards: tripalmitin, palmitic acid, methyl palmitate (sourced from Nacala Tesque); 1,3-distearin; DL palmitin (mono palmitin) (sourced from Sigma Aldrich); and 1,2-di-stearin 99%, (sourced from Research Plus).

Methods. The FFA in MCPO was reduced from 12.785 wt.% to less than 1 wt.% with 18 vol.% of MeOH, 2.7 vol.% of H₂SO₄, and reaction temperature of 60 °C by the ultrasonic continuous esterification. In the second-step process, the esterified oil was used as a feedstock to produce biodiesel. The various variables (MeOH, KOH, and initial temperature of oil) were investigated.

Apparatus. Fig. 1 is a schematic diagram of the experiment setup. The 18 kHz, 1,000 W ultrasonic homogenizer (model: AKHGZ-50420K, ACME-KORN, Thailand) was used as the ultrasound source for producing the biodiesel from esterified oil.

Procedures. Initially, the esterified oil was preheated on a hot plate in order to lower its viscosity, and the temperature of the heated oil was maintained at 60 °C. When the MCPO was preheated to the desired reaction temperature and potassium methoxide at calculated amounts from the required ratios were added into the ultrasonic reactor. In all experiment, the 50 mL total volume of the esterified oil blended with potassium methoxide was fixed to control the volumetric acoustic energy of 20 W/mL and the high surface power density of 1.62 W/mm². The ultrasonic irradiation time was controlled by a digital timer which was installed in the ultrasonic generator and a DC cooling fan was used to cool the piezoelectric transducer.

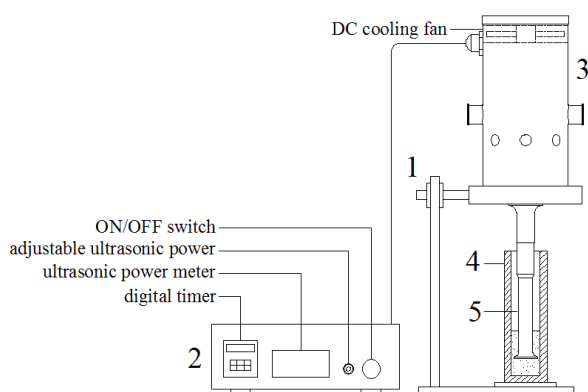


Fig. 1 Schematic diagram of the experiment setup. 1) Support stand with ring clamp, 2) Ultrasonic generator, 3) Ultrasonic homogenizer, 4) Ultrasonic reactor, 5) Ultrasonic circular horn

Results and Discussions

Effect of Base-Catalyst Concentration. The effect of base-catalyst on the FAME conversion at the esterified oil temperature of 60 °C and 20 vol.% of MeOH (Fig. 2) was investigated at various amounts of 0, 5 and 10 g KOH/liter of oil. The results indicated that the FAME conversion was sharply increased to over 98 wt.% after the reaction time of 10 and 20 seconds when 10 and 5 g KOH/liter of oil were used, respectively. On the other hand, when the non-catalyst was used, the conversion of TG to FAME was low. The authors recommend the 5 g KOH/liter of oil for producing the biodiesel because this condition can reduce the base-catalyst cost. Furthermore, the glycerides will react with base-catalyst to produce soap when using too much base-catalyst in transesterification.

Effect of Temperature. The effect of temperature on FAME conversion with ultrasonic irradiation in the presence of 5 g KOH/liter of oil and 20 vol.% of MeOH (Fig. 3) was investigated under 30 and 60 °C initial temperature of esterified oil to compare the FAME conversion. The results indicated that the FAME conversion was increased to over 98 wt.% after the reaction time of 20 and 40 seconds at the temperature of 60 °C and 30 °C, respectively. Therefore, initial temperature of esterified oil has significance with the rate of FAME conversion in base-catalyzed transesterification. However, the 30 °C was sufficient temperature to produce the FAME to over 98 wt.% and also to save the electricity cost for preheating the oil.

Effect of Methanol Concentration. The effect of MeOH on the FAME conversion at 5, 10, 15, and 20 vol.% of MeOH to oil at 30 °C and base-catalyst of 5 g KOH/liter of oil (Fig. 4) was investigated. The glycerides were converted rapidly to FAME because the oil to methanol ratio has a substantial effect on the reaction rate in transesterification. The results indicated that the methanol concentration is a significant variable affecting the FAME conversion, especially when the reaction in the ultrasonic batch reactor was carried out at the beginning. The glycerides can be converted to

FAME to over 98 wt.% when the reactant of mixture was accelerated via the ultrasound within 20 seconds of reaction time when using 15 vol.% of MeOH and 40 seconds of reaction time when using 10 and 20 vol.% of MeOH. However, the FAME conversion slightly increased because the dilution of some catalyst by the excess methanol when the 20 vol.% of MeOH was used [10].

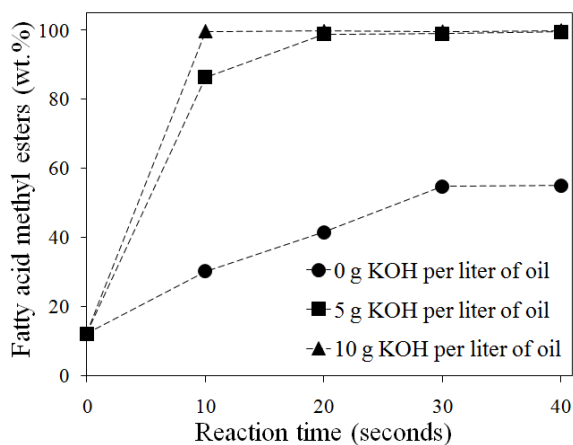


Fig. 2 Effect of base-catalyst on FAME conversion with ultrasonic irradiation in the presence of 20 vol.% of methanol and temperature of 60 °C

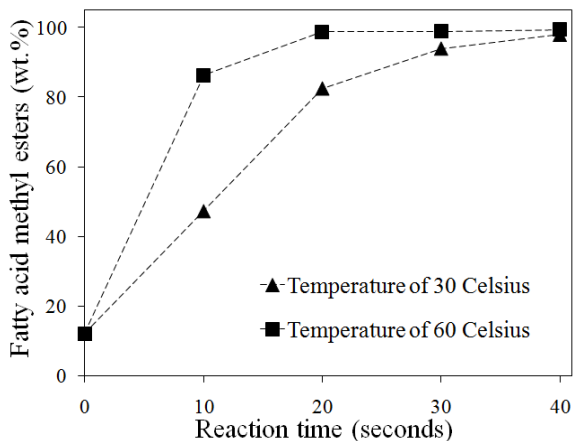


Fig. 3 Effect of temperature on FAME conversion with ultrasonic irradiation in the presence of 5 g KOH/liter of oil and 20 vol.% of methanol

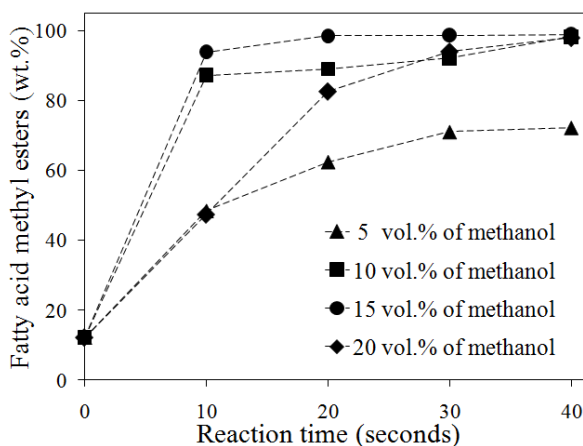


Fig. 4 Effect of methanol concentration on FAME conversion with ultrasonic irradiation in the presence of 5 g KOH/liter of oil and temperature of 30 °C

Conclusions

The fatty acid methyl ester (FAME) conversion from the esterified oil can be produced quite efficiently by the 1,000 W ultrasonic homogenizer at a low frequency of 18 kHz. The FAME of more than 98 wt.% was achieved when using the catalyst of 5 g KOH/liter of oil, 15 vol.% of MeOH, the reaction time of 20 seconds at temperature of 30 °C. This condition is the cost-savings condition to reduce the chemical cost (the amount of MeOH and KOH), electricity cost, and reaction time.

Acknowledgements

The authors would like to thank the National Research University Project of Thailand's Office of the Higher Education Commission for financial support, the Energy Policy and Planning Office of Thailand (EPPO) and the Faculty of Engineering, Prince of Songkla University for providing research funds for this study.

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**2012 International Conference on Frontiers of Mechanical Engineering,
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