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Comparison of Physicochemical Properties and Energy Consumption Between Microwave and Conventional Heating For Fatty Acids Production

[1]Nuraini Raman Yusuf, [1]Ruzaimah Nik Mohammad Kamil, [2]Suzana Yusup

[1]*Department of Fundamental & Applied Sciences, Universiti Teknologi PETRONAS, 32610 Bandar Seri Iskandar, Perak, Malaysia.*
[2]*Department of Chemical Engineering, Universiti Teknologi PETRONAS, 32610 Bandar Seri Iskandar, Perak, Malaysia.*

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ABSTRACT

Fatty acids were prepared by alkaline hydrolysis from *Jatropha Curcas sp.* oil conducted under two different methods; microwave assisted irradation system and conventional heating. It was found relative to conventional heating; microwave irradiation has been proved to be a faster method as increased the reaction rate and yields of fatty acids. Under optimum conditions, a 98 % yield of fatty acids could be achieved in 15 min under microwave irradiation compared to 2 h under conventional heating. The physicochemical properties such as density and viscosity of Jatropha Fatty acids shows decreased as both heating applied.

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INTRODUCTION

Conventional heating is a popular way to produce and prepare any fats and oil product such as biodiesel and fatty acids. However, this method consume large of energy therefore will lead to slow reaction (Roberts and Strauss, 2005). This because the method transferring energy into a reaction system as it depends on the thermal conductivity of the media and the temperature of the reaction vessel is higher than that of the reaction mixture. Latterly microwave irradiation (MW) is becoming worldwide popular method of heating which replace the conventional method (CV) because it proved to be clean, convenient and most important it affords higher yields and used in shorter time (Thostenson and Chou, 1999); (Chiavaro *et al*., 2010). Microwave radiation causes molecular friction of electric dipole, which results in heating. Lipids have low specific heat, which makes them susceptible to this radiation. This also generates permanent pores in the oil, makes higher yield. This method of heating has been extended to almost all area of chemistry with the exception of the carbohydrate ie fatty acids but it testified by the limited number of applications.

In vegetable oils exposed to microwave energy, the higher the amount of polyunsaturated fatty acids in the oils, the greater was the rate of quality deterioration of the oils (Yoshida *et al*., 1992). Jatropha oil (JO) is one of important oil seed crops in the world. Research indicates that the fatty acids composition of JO is discovered to have great potential in biodiesel, fatty acids production also in bio lubricant production. The quest for renewable energy sources has since dominated most manufacturing industries with much emphasis on bio products. Several researchers have agreed on the possibility of obtaining more efficient lubricants from such (bio products) sources. Hence, there is need to investigate the possibility of obtaining an environmentally friendly and economically viable lubricant from one of such sources (Bilal *et al*., 2013).

The earliest known study involving microwave pre-treatment of C18 fatty acids esterification. The results showed conversion up to 60% in 60 min of reaction under relatively mild conditions and noncatalylitic system (Melo-Junior *et al*., 2009). It has been proposed that in case of low power of high-frequency electromagnetic field the nonthermal activation of enzyme may be observed. However, little research has been done so far on application of microwave irradiation in alkaline hydrolysis for fatty acids production. In this study we propose the comparison of efficiency of pre-treatment alkaline hydrolysis between microwave irradiation and conventional heating on fatty acids production also on their physicochemical characterization and energy consumption.

Corresponding Author: Nuraini Raman Yusuf, Department of Fundamental & Applied Sciences, Universiti Teknologi PETRONAS, 32610 Bandar Seri Iskandar, Perak, Malaysia.

MATERIALS AND METHODS

Materials:

Jatropha oil selected for the experiment was purchased from Bionas Sdn Bhd, Malaysia. All the chemicals and solvents used were analytical grade and were used directly without any purification. The experiments using conventional method were performed in a 250 mL round-bottom flask equipped with a water-cooled reflux condenser. A hot plate with magnetic stirrer arrangement was used for heating the mixture in the flask. As comparison, microwave treatments were carried out using a domestic microwave synthesis reactor (Fabricated by Dixson company) with power levels of 3000 W.

Preparation of Fatty Acids:

The process was carried out as described by (Salimon *et al*., 2011). Briefly, the batch mixture containing Jatropha curcas seed oil and ethanolic KOH was mixed in a 500 ml round bottom flask reactor. The reactor was placed in a microwave synthesis reactor. In addition, due to high temperature, a reflux system was established for condensing sample into reactor. After the reaction was completed, the sample was cooled and 200 ml water was added to the mixture to enhance separation of fatty acids. A 100 ml of hexane was added to extract the unsaponifiables. The aqueous alcohol phase, containing the soaps, was acidified to pH 1 with HCl 6N, and the FFA was recovered by extraction with hexane. The extract was washed with distilled water until neutral. The FFA-containing upper layer was dried with anhydrous magnesium sulfate, and solvent was evaporated in a vacuum rotary evaporator at 50 °C.

Characterization of fatty Acids:

The quality of fatty acids is expressed in terms of the physicochemical properties such as free fatty acids percentage (% FFA), density and viscosity. The % FFA determined according to acid value test as (AOCS, 1999) reference method. The density was measured by using density meter DMA 4500 Anton Paar and viscosity test were performed using Anton Paar DMA 5000 viscometer. The Fourier Transform Infra-Red (FTIR) were determined using spectrophotometer (Perkin Elmer Model Spectrum One/BX) in the range of 4000–400 cm^{-1} using a KBr disc containing 1% of finely ground samples.

Energy Consumption:

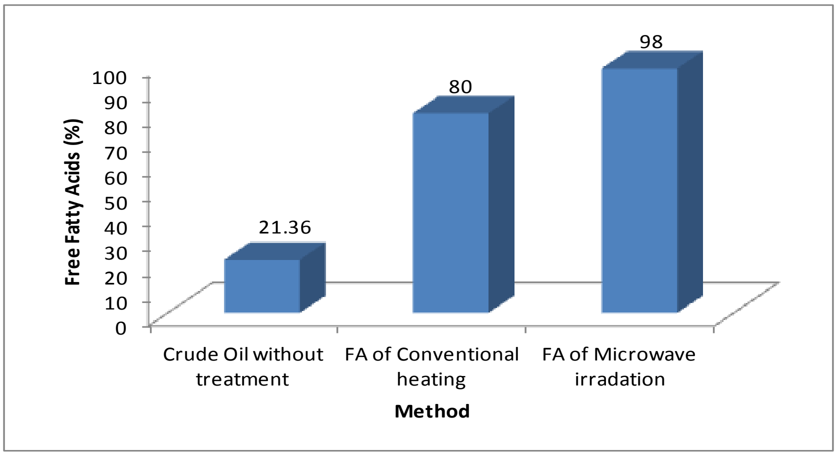
Comparison between two method were calculate based on the following equation. The energy E in kilowatt-hours (kWh) per day is equal to the power P in watts (W) times number of usage hours per day t divided by 1000 watts per kilowatt;

$$E_{(kWh/day)} = P_{(W)} \times t_{(h/day)} / 1000_{(W/kW)} \qquad (1)$$

RESULT AND DISCUSSION

Free Fatty Acids %:

The FFA% derived from two difference method was compare as shown in **Figure 1**. The increase in the free fatty acids from crude oil is attributed to hydrolysis of triglyceride by both heating method to produce fatty acids. The effect of MW irradiation on the FFA% is slightly increase compared to conventional method. Nevertheless, as optimum condition were different, MW irradiation have shown using shorter time (15 minutes) compare to CH, 2 hours heating with low oil/solvent ratio; 1:68 compare to 1:78 and with different reaction temperature, MW; 90 °C compare CH ; 65°C.



Physical and Chemical Properties:

Table 1 shows the comparison physiochemical characteristic of Jatropha curcas oil, Jatropha curcas fatty acids using CH and Jatropha curcas fatty acids using MW method on the effect of specific gravity, viscosity and free fatty acids value. The specific gravity (SG) is an important characterization and corresponds to the ratio of the weight of the substance's given volume to the weight of an equal volume of water. All the SG value is near to SG of water and within the range reported in literature (Uquiche *et al*., 2008). The viscosity of fatty acids

MW was significant less viscous than sample prepare by conventional heated that clearly observed. The low viscosity may attribute by fatty acids and solvent are materials with high dielectric constant. The ability of this compound absorbed energy under MW irradiation and converts the absorbed energy into heat results in a short dissolution time and produce low viscosity of sample (Idris and Iqbal, 2008). Despite that, both methods will reduce the viscosity from the crude oil.

Table 1: Comparison of physicochemical characteristics of Fatty acids Jatropha Oil.

Property	Crude Oil	Conventional heating	Microwave irradation
Specific gravity (g/cm^{-3}) at 40°C , 60°C	0.9085,0.903	0.8820, 0.8680	0.8816, 0.8678
Viscosity (mPa) at 40°C , 60°C	35.229,18.718	21.974, 11.853	16.794, 9.638

Thermal Analysis:

Thermogravimetric analysis (TGA) is a technique for characterizing thermal stability of a material (compound or mixture) by measuring changes in its physicochemical properties expressed as weight change as a function of increasing temperature (Santander *et al*., 2012). **Figure 2** shows TGA plot for fatty acids from microwave irradiation, fatty acids conventional and crude oil. The mass of the fatty acids for both heating starts to decrease at approximately after 200- 230°C, and this step was attributed to vaporization of carboxylic acid group (Prafulla *et al*., 2012). TGA curve of evaporation crude oil starts at approximately 300° C shows decomposition residue becomes greater when alcohol with longer chain is used (triglyceride) (Todaka *et al*., 2013).

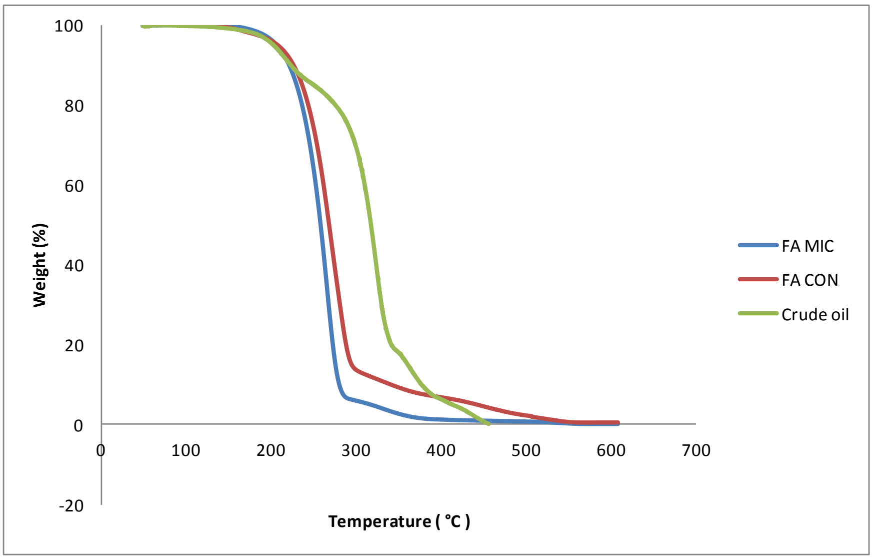


Fig. 2: Thermogravimetric curves for Fatty acids microwave, Fatty acids conventional and crude oil.

FTIR Analysis:

The comparison of FT-IR spectra between jatropha fatty acids of MW, jatropha fatty acids of CH and jatropha curcas oil are shown in **Figure 3**. A chemical shift at a wavelength of 1746 cm^{-1} (oil) to wavelength of 1713 cm^{-1} and 1710 cm^{-1} (FA MW and FA CON) were observed. The shift indicates esters functional group from triglyceride has been converted to fatty acid for both methods (Lambert *et al*., 1987). This conversion was amplified by broad O-H stretching of the carboxylic acid functional group at 3200 cm^{-1} – 2500 cm^{-1} and also O-H bend in the region 950-910 cm^{-1}. Peaks at 3009, 3008 cm^{-1} and 2923 – 2854 cm^{-1} indicated that stretching of C=C, CH_2 and CH_3 scissoring of both oil and Fatty acids. The C-O bending at 1298 -1240 cm^{-1} for crude oil and fatty acids (Salimon and Abdullah, 2009). Compared for both methods, the spectra is not alteration despite of one stretching at 1831 cm^{-1} indicates group anhydride C=O.

Energy Consumption:

Table 2 represent comparison of energy consumption between MW irradiation and CH on production of fatty acids. It can be observed that energy consumption of MW irradiation more efficient compare to CH, as low consumption (0.74 kWh/day) compare to 2.04 kWh/day. The energy efficiency of the MW assisted reactions depends on sample volume, nature of the medium (solvents) and others. Poor efficiencies can be observed when a high power microwave device is used for a very small sample volume. This observation suggests that effective utilization of microwave power can lead to process energy savings (Gude *et al*., 2013).

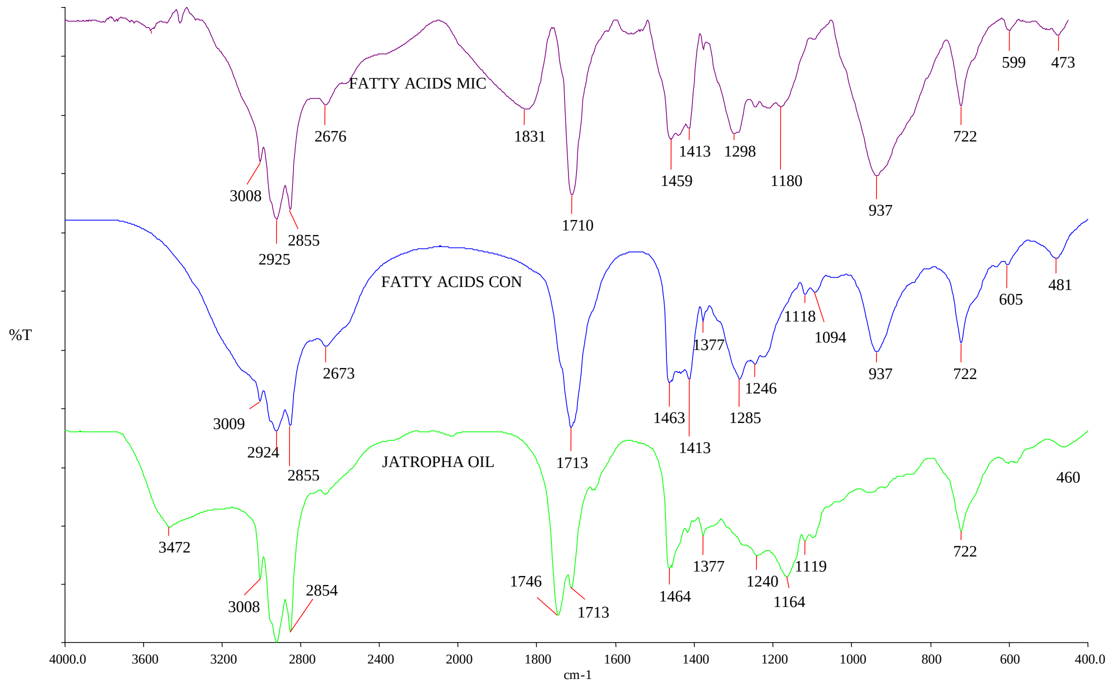


Fig. 3: Comparison of FTIR spectra for fatty acids of microwave, fatty acids conventional and jatropha oil.

Table 2: Comparison of energy consumption between microwave irradiation and conventional heating.

Property	Power (Watt)	Time (h)	Energy Consumption (kWh/day)
Microwave Irradiation	3000	0.25	0.75
Conventional Heating	1020	2	2.04

Conclusion:

As a conclusion, the productions of fatty acids were successfully derived by alkaline hydrolysis of jatropa curcas oil using MW irradiation and CH heating. The CH and MW heating were differ in their effects on the amount of free fatty acids percentage as MW produce slightly higher (98 %) compared to conventional method (80 %). Furthermore, the parameters involve were differ; MW irradiation have shown using shorter time (15 minutes) compare to CH, 2 hours heating with low oil/solvent ratio; 1:68 compare to 1:78 and with different reaction temperature, MW; 90 °C compare CH ; 65°C. The effect on physicochemical between MW and CH shows no significant differences in the levels but some slightly contrast such as fatty acids of MW heated was significant less viscous than viscosity of CH. Others is energy consumption; MW is more efficient compare to CH. The thermal degradation shows for both heating starts to decrease at approximately after 200- 230°C and the FTIR spectra shows no significant differences except for one functional group at 1831 cm^{-1} indicates group anhydride C=O for MW heating.

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