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Methanolysis of Castor Oil and Parametric Optimization

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Abstract

The rate of transesterification reaction is very slow as oil is sparingly soluble in methanol. In the present work, castor oil as non-edible oil source was investigated for the synthesis of fatty acid methyl ester (FAME). To enhance transesterification reaction, purified castor oil was treated with microwave energy prior to alkaline transesterification reaction. The experimentally observed FAME yield was compared with FAME yield achieved with microwave energy untreated castor oil at the same reaction condition. 95.6 wt. % FAME yield was exhibited with oils treated with microwave energy as compared to 84.034 wt. % FAME yield untreated with microwave energy while the reaction time was reduced from 60 min to 30 min. Central composite Design (CCD) technique of response surface methodology (RSM) was used to investigate the interaction effect of reaction variables and optimize these variables for maximum FAME yield. At optimal condition 96.12 wt. % FAME yield was predicted from model equation while 95.4± 0.3 wt. % FAME yield was demonstrated by the experiments conducted twice at optimal condition.

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*Keywords:*Castor oil; Transesterification; Microwave energy; Fatty acid methyl ester

1. Introduction

The rising energy demand from fossil fuel sources associated with its environmental impact triggers the search for alternative renewable fuels. Biodiesel is one of the promising alternative renewable energy sources to complement petroleum based diesel fuel. It was reported that mixing of small amount of biodiesel with petro-diesel drastically reduces the emission of pollutants caused by the use of diesel fuel alone. Biodiesel is produced by the transesterification reaction of vegetable oil or animal fats with low molecular alcohol (methanol and ethanol) in the presence of catalyst or at supercritical condition [1]. Investigation conducted by different researchers demonstrated at present more than 95 percent of biodiesel is produced from edible oil sources. On the other hand, the global demand of edible oil sources is increasing every year. Diversion of edible vegetable oil for biodiesel production is difficult to justify as it affects the cost of biodiesel as well as food industry. Use of alternative non-edible oil sources such as castor oil, jatropha oil, algae oil, karanja (*Pongamia pinnata*), tobacco (*Nicotiana tabacum*), rubber plant (*Hevea brasiliensis*), waste cooking oil etc. are gaining increasing attention and are broadly under investigation [2 - 4].

The limited solubility of oil in methanol affects the rate of transesterification reaction even though catalysts are used to enhance the reaction. Application of different techniques such as ultra-sonication, use of co-solvent, phase transfer agents and microwave

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irradiation can increase the mass transfer between the reacting components ultimately increasing the reaction rate of transesterification [5-7].

Castor oil is non-edible oil sources produced from *ricinus communis* or castor tree. It grows all over the world. India is the leading producer and exporter of castor oil while China and Brazil comes second and third, respectively. Of non-edible oil, castor oil is the second high oil yield plant which is about 1.1 tons per hectare while jatropha gives the highest which is about 1.9 tons per hectare [8]. On average castor seed contains 40 to 55 % oil [9]. Castor oil appears as pale yellow in colour, and clear liquid at room temperature. Castor oil mostly composed of Ricinoleic acid (85 to 90 %) while linoleic acid, oleic acid, palmitic acid, stearic and linolenic acid makes the remaining composition. Therefore, castor oil can be used to produce biodiesel without any side effects [10, 11].

Use of microwave energy as energy source for organic reactions have been an attractive method in the middle of 1980s [12]. Different studies on microwave heating system indicated that it is an efficient method of heat supply in which the reaction occurs rapidly, safely and with higher product yields. Microwave energy accelerates chemical reaction because of selective absorption of microwave energy by polar molecules. Fast and evenly distributed internal heating through all samples as compared with the conventional was observed in microwave irradiation heating [13].The basic principles of microwave energy heating involve mixing of polar molecules or its ions that oscillate under the influence of an oscillating electric or magnetic field. In the presence of oscillating field, particles try to re-align themselves or be in phase with the field. The motion of oscillating particles is hampered by resistance force that is inter-particle interaction and electric resistance forces, and it restrict the movement of particle while generating random motion that produces heat [14].

In this study, the effect of microwave irradiation heat treatment of castor oil prior to alkaline transesterification reaction was investigated. The reaction variables interaction effect and optimal condition were determined using central composite design of response surface methodology.

2. Materials and Methods

2.1 Materials

Castor oil was procured from local supplier (Best Formula Industries, Kuala Lumpur, Malaysia). Chemicals used were methanol with $\geq$ 99.7% purity, n-hexane with a purity of purity $\geq$ 99%), NaOH (purity $\geq$ 99%), KOH with a purity of $\geq$ 85%, iso-propanol ($CH_3CHOHCH_3$, purity 99.8%), reagent grade acetic acid, diethyl ether ($C_4H_{18}O$) and other pro-analysis chemicals supplied by Avantis Laboratory Supply, Malaysia. Standard chemical kits for analysis of biodiesel in gas chromatographic were procured from Sigma Aldrich, Malaysia.

2.2 Oil characterization and purification

AOCS official method cd 3d-63(2003) titration method was employed to investigate the acid value. A calibrated pycnometer (Jayteck, UK) was used for measuring the density of oil and fatty acid methyl ester (FAME). Brookfield with model cap 2000+ viscometer (USA) was utilized to determine the viscosity of oil and FAME. Pensk-Martens automatic flash point analyser was used to determine the flash point, the heat content (calorific value) of castor oil was measured using bomb calorimeter**.**

2.2.1 Oil Purification

Since castor oil free fatty acid (FFA) content is greater than 2%, the oil need to be purified by esterification reaction in order to reduce the FFA content to about 2% or less. In the oil purification process, 100 gm of oil, methanol (12:1 molar ratio of methanol to oil), and 2 wt % sulfuric acid concentration as acid catalyst were added into a two neck round bottom reactor with a reflex condenser. The reaction was conducted at 80^{o}C, 400 rpm for one hour. After one hour, the mixtures in the reactor were separated and purified oil was recovered for further transesterification reaction.

2.3 Design of experiment

The reaction variables individual and interaction effects during transesterification reaction on the yield of FAME and the optimum reaction condition for maximum FAME yield were investigated using design of experiment (DOE). Central composite design (CCD) technique of response surface methodology (RSM) was used to design the experiments at different variable combination. The experimental results were fitted using a polynomial quadratic equation (1) for detail analysis of reaction variables on the response variables (FAME yield). MINITAB 14 software packages were used for DOE. The statistical significance of the mathematical model equation was tested using analysis of variance (ANOVA) with 95% confidence intervals.