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## Statistical Optimization for Esterification of Waste Coffee Grounds Oil using Response Surface Methodology

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#### Abstract

Biodiesel is alternative bio-fuel to substitute for diesel fuel which produces harmful emission causing environmental problems. The common feedstock for biodiesel production was edible oil which effected to high production cost. In order to conquer this problem, waste material was appeared to be an attractive choice. The oil from waste coffee grounds (WCGs) has the potential to use as raw material. However, high free fatty acid (FFA) concentration (>1%) in extracted oil from WCGs requires pretreatment prior to transesterification. Therefore, FFA was treated by using sulfuric as acid catalytic. In this study, the optimization of esterification of extracted oil from WCGs was investigated by using response surface methodology (RSM). The effect of operation parameters to pre-treatment of FFA was conducted using laboratory scale. The predicted optimum condition results indicated that %FFA of the waste coffee grounds oil (WCGO) was decreased 94.97% when the condition was carry out with methanol 10 mole per mole of FFA in the presence of 15% w/w of %FFA-to-catalyst ratios for 115 min reaction time at 65°C.

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Keywords: Waste Coffee Grounds Oil; Esterification; Response Surface Methodology

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#### 1. Introduction

Biodiesel is an alternative, renewable and biodegradable energy produced from biological resources using transesterification or esterification reactions. Its properties are close to that of petroleum which can be used alone or blended with diesel in unmodified diesel-engine vehicles. Moreover, it also reduces both the amount of fossil fuels burned and the emission of greenhouse gases such as  $CO_2$  [1]. Currently, the main feedstock for biodiesel production is edible vegetable oils because they get high biodiesel yield due to low FFA content. However, the using of edible oils as the feedstock is a major hurdle to commercialization because it is high cost and 75% of biodiesel production cost is raw material [2]. In addition, the biodiesel production from edible oils can lead to food oil crisis [3]. Thus, these factors have the negatively effects on its production from edible oils. Therefore, many non-edible oils, e.g. rubber seed oil, Jatropha oil, sheabutter, chicken fat and WCGO have become more attractive as an alternative feedstock because of priceless and easy processing [4].

Coffee has a great commercial in Thailand because 75,000 tons of coffee bean were produced in 2014 [5]. Considering this huge amount of WCGs, some attempts for reutilization should be made. The WCGs contain 11 to 20 wt% of oil depending on its type [1]. Therefore, WCGO is expected to be a new resource for the biodiesel production process. However, non-edible oils usually contain high FFA (>1%) and are too far beyond the level that could be conducted into one step transesterification. Because FFA could react with the base catalyst which leading to the soap formation [12] through the undesirable saponification reaction. Pretreatment by esterification reaction was mentioned in order to remove the FFA content before biodiesel production.

In the present study, the esterification of WCGO in the presence of acid catalyst was conducted to reduce the %FFA. The aim of this investigation was to study the effect of methanol to FFA mole ratio, amount of catalyst, reaction temperature and reaction time on the esterification reaction. The central composite design (CCD) of RSM was used to design of experiments for generating the model and optimizing the operational parameters.

#### 2. Material and methodology

#### 2.1 Materials

This study, WCGs were collected from Southern coffee shop (Tesco Lotus, Nakhon Si Thammarat branch) which were a mixture of Arabica and Robusta. AR grade methanol was purchased from Fisher Scientific. Concentric sulfuric acid was from Loba chemie. Hexane was purchased from J.T. Baker.

#### 2.2 Extraction of WCGO

The WCGs were dried in an electrical oven at 105°C for 24 hrs. [6] to get rid of its moisture content. Dried WCGs were extracted using hexane as a solvent. The solid-liquid mixture was stirred for 5 mins. The extracted solvent was separated from the WCGs by filtration. The WCGO was separated from the solvents by using a vacuum rotary evaporation. The WCGO was left in the oven for 6 hrs. at 105°C to remove any hexane that remains in the extracted oil. The fatty acid composition was analyzed by Gas Chromatography-Flame Ionization Detector (GC-FID) according to In-house method of Scientific Equipment Center, Prince of Songkla University.

#### 2.3 Esterification reaction

The esterification of WCGO was carried out in 250 mL round-bottomed flask connected to reflux condenser, thermometer and hot plate with magnetic stirrer. The WCGO was reacted with methanol in the presence of sulfuric acid. The variables for optimization were selected and given in Table 1. The reaction product mixture was poured into a separating funnel in order to separate the layer of oil and water. The upper layer was washed with hot water to remove acid catalyst and heated to remove water content.

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