

# Extraction of caustic potash from spent tea for biodiesel Production

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**Abstract.** Biodiesel is an alternative to non-renewable fossil fuels due to its low gas emission and economical value. This study aims to extract caustic potash (KOH) from spent tea and to optimize the transesterification process based on parameters such as amount of catalyst, reaction temperature and methanol to oil ratio. The spent tea was first dried at 60°C prior to calcination at 600°C for two hours. Caustic Potash were extracted from the calcined spent tea. The transesterification process was done based on Design of Experiments (DOE) to study the effects of amount of catalyst ranging from 0.5 wt % to 2.5 wt %, reaction temperature from 55°C to 65°C and methanol to oil ratio from 6:1 to 12:1 at a constant agitation rate of 300 rpm for three hours. The calcined spent tea produced was recorded the highest at 54.3 wt % and the extracted catalyst was 2.4 wt %. The optimized biodiesel yield recorded was 56.95% at the optimal conditions of 2.5 wt % amount of catalyst, 65°C reaction temperature and 9:1 methanol to oil ratio.

## 1. Introduction

The utilization of diesel fuel in the world is tremendous, ranging from agriculture and transport towards commercial and domestic industries in generating power and mechanical energy. However, diesel fuel is a non-renewable source of energy that is running low on natural sources. This could be a major issue if no new alternatives can replace diesel fuel. In this research, biodiesel fuel made from vegetable oils or animal fats are studied for its promising features that attract huge attention all over the world for its availability, eco-friendly, non-toxicity and biodegradability [1].

The process of converting waste cooking oil (WCO) to biodiesel is called transesterification, a process that requires catalyst in order to react. By using WCO for biodiesel production, it can reduce the cost of raw materials for biodiesel as well as the problem of environmental pollution. Phan & Phan, 2008 study shows that the amount of usage of the waste cooking oil affects the amount of catalyst needed in the production process [2]. In their study, the WCO acid values ranges from 0.67 to 3.64 mg KOH/g when the sample WCOs are used once or a few times. Low free fatty acid content results in lower concentration of potassium hydroxide (KOH) needed to be added. On the contrary, the higher free fatty acid requires an addition of KOH to compensate for this acidity. Current biodiesel production uses conventional catalysts such as strong acid-based or basic catalysts. These catalysts are highly corrosive to the equipment, relatively expensive due to the production and downstream separation required, and has serious environmental effect.



Another occurring problem is the usage of homogenous catalyst in the process of transesterification. Homogenous catalysts cannot be regenerated or reused, as it is consumed during the reaction. The separation of catalyst will also be much harder due to the homogeneity, which uses more equipment thus increasing the production cost.

Spent tea was chosen as the restaurant waste for low cost catalyst in transesterification because tea is one of the most abundant wastes in Malaysia. Annually, Malaysia produces less than five million kg of tea while the per capita consumption of tea in Malaysia is about half kg and has the annual demand of nine million kg. Such biowaste can be reused as precursor in catalyst production can bring significant change to the environment. Kumar [3] found that materials containing alkali metals such as potassium, calcium and sodium may produce potassium carbonate upon heating [3]. When burnt and oxidized in the presence of air, metal oxides are formed. Carbon dioxide produced through burning will react with potassium oxide to produce potassium carbonate while in the presence of water vapour will produce potassium bicarbonate and potassium hydroxide. Thus, by calcination of tea spent, KOH can be produced to be later used in the transesterification process.

In this study, spent tea, which is a waste from restaurant, was chosen as catalyst. Caustic potash was extracted from spent tea in order to render the transesterification process achievable and able to attain high yield of biodiesel. This paper studies the extraction of caustic potash from spent tea as low cost catalyst and the optimization of methanol to oil ratio, amount of catalyst and temperature in biodiesel production from WCO.

## 2. Experimental procedures

For extraction of catalyst, used teabags were collected from cafeterias around International Islamic University Malaysia (IIUM), the spent tea was first washed with tap water and then distilled water a few times. Then, it was dried in an oven at 60°C for five hours. After drying, calcination of the spent tea was conducted as the final step of extracting caustic potash from the waste. The dried tea was put in an aluminum foil and into the furnace at 600°C for two hours. After that, it was left to cool inside the furnace for five hours.

WCO was also collected from the cafeterias and methanol was bought from Sigma Aldrich Sdn Bhd. Transesterification was done by mixing WCO with the calcined spent tea and methanol with the amount based on the DOE. The catalyst caustic potash KOH is stirred in methanol and dissolved in a 250 ml beaker. 20-30g of WCO is then transferred into the biodiesel incubator shaker along with the KOH/methanol mixture. The incubator was set to be running for 3 hours at 300 rpm with varying parameters set based on the DOE designed by the Design Expert 10 software as shown below.

**Table 1.** Reaction parameters as per generated by DOE

Run	Factor 1: Catalyst amount, A (wt %)	Factor 2: Temperature, B (°C)	Factor 3: Methanol:oil	Response: Yield (%)
1	1.5	60	6	
2	2.5	55	12	
3	1.5	60	9	
4	0.5	60	9	
5	2.5	55	6	
6	2.5	65	12	
7	0.5	55	6	
8	1.5	55	9	
9	1.5	60	9	
10	1.5	60	9	
11	1.5	60	9	
12	2.5	60	9	
13	0.5	55	12	

14	2.5	65	6
15	1.5	60	12
16	1.5	60	9
17	1.5	60	9
18	0.5	65	12
19	1.5	65	9
20	0.5	65	6

Next, the samples were transferred into a 50ml disposal tube for centrifugation and then heated in the oven overnight at the temperature of 68°C. It is then left overnight to cool down and separated by gravity in a separating funnel. The layer formed on the top is the methyl ester while the bottom layer is the by-product glycerol. The bottom layer is removed and put in a separate container while the upper layer was heated to remove excess methanol.

Based on Hosseini et al. [4], to analyze the biodiesel produced gas chromatography (GC) was conducted to find the value of biodiesel produced yield content and the value of conversion from triglycerides to methyl esters. According to Cheng et al. [5], using hexane as the solvent, the chromatography was done under the following conditions: Helium as carrier gas, oven temperature of 150°C initially for 1 minute and then increased to 250°C at the rate of 15 °C /min. The FAME content percentage is calculated using the formula below:

$$FAME\ Content\ \% = \frac{\text{weight of FAME contained in crude biodiesel}}{\text{weight of biodiesel}} \times 100\% \quad (1.0)$$

### 3. Results and Discussion

#### 3.1. Extraction of Caustic Potash from Spent Tea

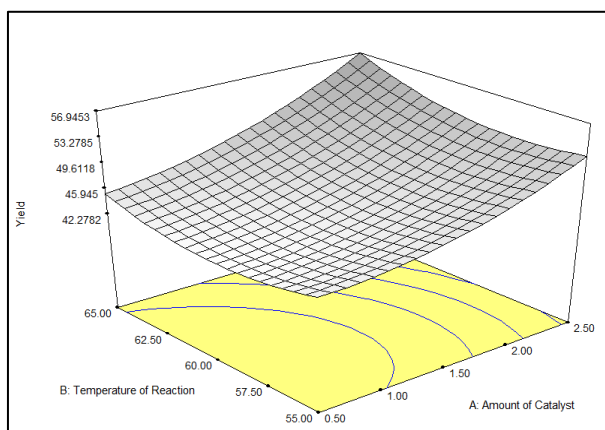
Based on the titration done, the calcination of catalyst from spent tea produced low amount of KOH, only 3.43 wt %. This lead to low conversion of biodiesel during the transesterification process as the amount of catalyst used in the reaction was weighted based on the weight of calcined spent tea, not the weight of KOH.

#### 3.2. Optimization of the Transesterification Process

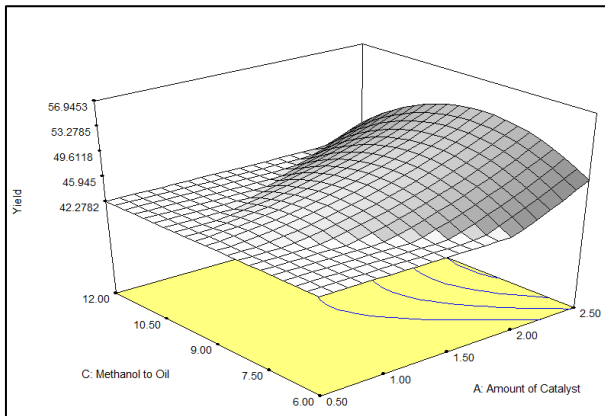
Based on the ANOVA study, the relationship of the three parameters was observed. In figure 1, while keeping the methanol to oil ratio at 9:1, the three dimensional response surface showed that the biodiesel yield increases with the amount of catalyst and temperature of reaction. From 55°C to 65°C, the yield of biodiesel showed a significant increase to 56.945% when the amount of catalyst is at 2.5 wt %. According to Vyas et al. [6], high amount of KOH leads to saponification and decreases the conversion yield [6]. However, in this case the amount of KOH in each gram of catalyst is only 0.034g thus the biodiesel yield is the highest when 2.5wt % of catalyst was used.

Figure 2 shows that at 0.5 wt % to 1.0 wt % catalyst amount the biodiesel yield did not deviate much from the value of 42.3% when the reaction temperature was kept at 60°C. However, when the amount of catalyst was at 2.5 wt %, the highest biodiesel yield was shown to be at 49.61% with methanol to oil ratio at 9:1. The yield value dropped when the methanol to oil ratio deviates further from the 9:1 ratio despite the amount of catalyst is at 2.5 wt %. Excess methanol is required to shift the reaction equilibrium forward. However when high amount of methanol was mixed the basic catalyst, it produces water that will cause saponification to occur [7]. Another reason to the decrement of biodiesel yield is the excess methanol interfering with the methyl ester and glycerol separation by increasing glycerol solubility [8].

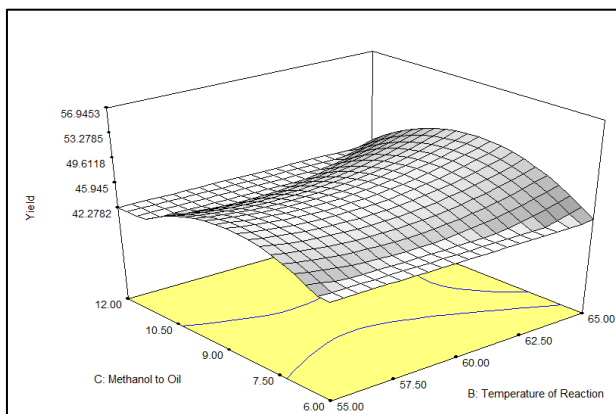
In figure 3, when the amount of catalyst was kept constant at 1.5 wt %, the increment in conversion yield occurred when the methanol to oil ratio increased from 6:1 to 9:1. The yield reached its peak at 9:1 but decreased when the ratio was further increased to 12:1. Relative to the reaction temperature, the biodiesel conversion yield increased as the temperature was increased to 65°C. Generally, at high temperature speeds up the transesterification reaction and increases the yield [2]. Low yield at lower temperatures may due to incomplete reaction. Low yield at high temperature was probably caused by acceleration of the saponification reaction [9].



**Figure 1.** Effect of Amount of Catalyst and Temperature of Reaction



**Figure 2.** Effect of Amount of Catalyst and Methanol to Oil Ratio



**Figure 3.** Effect of methanol to oil ratio and temperature of reaction

#### 4. Conclusion

This research was conducted to extract caustic potash from spent tea as a low cost catalyst and to optimize the process of transesterification for biodiesel production by varying the methanol to oil ratio, amount of catalyst and temperature of the reaction based on the DOE. The amount of catalyst extracted from the spent tea averaged about 53.32% in weight. However, titration was done on the basic catalyst and it was found only 0.34 KOH was found in 10g of extracted catalyst. This low value of KOH resulted in low conversion of WCO to biodiesel. For the transesterification process, the optimum condition for the reaction was proposed by ANOVA to be 2.31 wt % of catalyst, 10.32 methanol to oil ratio at 53°C for three hours reaction time.

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