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OPTIMIZATION OF BIODIESEL PRODUCTION USING A STIRRED PACKED-BED REACTOR

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ABSTRACT

The use of waste cooking oil (WCO) as feedstock and in microwave heating technology are favored to reduce the cost of biodiesel. In this study, a continuous flow transesterification of waste cooking oil (WCO) by microwave irradiation for biodiesel production using calcium oxide (CaO) as a heterogeneous catalyst, calcined from cockle shells, has been used. The catalyst was packed inside a plastic perforated container that mounted on a stirrer shaft and inserted inside the reactor. The thermocouple inside the reactor was connected to a temperature controller and microwave power input to maintain the temperature. Response surface methodology (RSM) was employed to study the relationships between power input, stirrer speed and liquid hourly space velocity (LHSV) on the WCO methyl ester (WCOME) conversion at a fixed molar ratio of methanol to oil of 9 and a reaction temperature was set at 65°C. The experiments were designed using the Box-Behnken design (BBD) for optimum conditions. The transesterification of the WCO was produced at 72.5% maximum WCOME conversion at an optimum power input of 445 W, stirrer speed of 380 rpm and LHSV of 71.5 h⁻¹. The energy consumption at a steady state condition was 0.594 kWh for the production of 1 litre WCOME, for this heterogeneous catalyst is much faster compared with conventional heating.

Keywords: Box-Behnken design; Calcium oxide; Microwave; Optimization; Waste cooking oil

1. INTRODUCTION

Biodiesel belongs to the category of alternative diesel fuels and can be produced from animal fats and vegetable oils, which are generally referred to as renewable biological sources. Research activities on the production of biodiesel have been on the increase in recent times due largely to the surge in the price of petroleum products and the environmental benignity of the biodiesel. The use of biodiesel blends has led to a reduction in the levels of greenhouse gases, such as CO₂ (Balat & Balat, 2010). A major impediment to the full commercialization of biodiesel is the high cost of production. Thus, one way of reducing the cost of production is through the use of inexpensive raw materials such as WCO (Hamze et al., 2015). From the environmental point of view, the use of WCO reduces the disposal problem.

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The environmental problem caused by ~~the~~ disposal of WCO through direct discharge into ~~the~~ water drainage systems ~~has~~ led ~~the~~ policy-makers in many developed countries ~~set to develop~~ policies that stipulate stiffer penalties ~~for the~~ violators (Kathirvel et al., 2016). ~~So that~~ ~~With this~~ ~~in mind~~, the use of WCO as feedstock can create a safe and clean environment (Kusrini et al., 2015).

Hence, ~~the~~ researchers ~~have been~~ focusing on using WCO as ~~a~~ feed-stock for diesel fuel (Chen et al., 2012; Kannan et al., 2011; Liu et al., 2010; Muralidharan & Vasudevan, 2011; Patil et al., 2010). The conversion of fats and oils into biodiesel ~~has~~ been carried out by researchers ~~who~~ employ~~ing~~ various methods, such as pyrolysis, catalytic cracking and transesterification (Rathore et al., 2016). ~~However,~~ ~~Among~~ these methods, transesterification is noted for the production ~~of~~ high quality biodiesel (Talebian-Kiakalaieh et al., 2013). Transesterification, also known as alcoholysis, involves the formation of glycerol and esters through the reaction of alcohol with either oil or fat. The reaction type and temperature, type of alcohol, agitation speed, concentration of catalyst, water content, alcohol to oil molar ratio, and FFA of the feedstock are the core process factors that usually influence the transesterification process (Hamze et al., 2015). Compared ~~d~~ with conventional heating, the applications of microwave irradiation in biodiesel production have led to ~~a~~ considerable reduction in the reaction time and ~~an~~ increase in the biodiesel yield (Motasemi & Ani, 2012). Besides that, enhancement in the rate of reaction and the yield can also be facilitated through the use of a catalyst. Unlike conventional heating, this method can operate at over 90% efficiency and ~~is~~ eco-friendly in a variety of chemical reactions (Lam et al., 2016).

In addition, the catalyst plays ~~a very~~ decisive role in the process of transesterification. Catalysts are classified as either homogeneous or heterogeneous, based on the type of chemicals that are present in the transesterification reaction. Catalysts are said to be homogeneous when they ~~catalysts~~ act in ~~the a~~ liquid phase ~~which is the~~ same as the reaction mixture. On the other hand, heterogeneous catalysts are ~~catalysts those~~ that act in a phase that is different from the reaction mixture, commonly as a solid (Borges & Díaz, 2012). Heterogeneous catalysts ~~are~~ a green process, ~~are~~ eco-friendly and non-corrosive. ~~Heterogeneous catalysts~~ ~~They~~ have good recyclability, ~~and~~ as such can be used ~~as many times as~~ possible, thus offering a cost-effective way for the production of biodiesel (Said et al., 2015). ~~The a~~ Application of calcium oxide (CaO) derived from waste carbonaceous materials is a potential solid catalyst in ~~the~~ biodiesel production process (Yusuff et al., 2018).

The use of conventional transesterification in batch operations ~~was~~ ~~has been~~ considered rigorous, tedious, slow, and unsuitable for automation adaptation. Besides that, it has ~~a~~ ~~low~~ cost of production, ~~the~~ ability to ~~only~~ produce ~~trifling very small~~ quantities of biodiesel, ~~the~~ capacity to make improvements to the design of equipment for the optimization of the quality of biodiesel, and the ability to produce greater quantities of biodiesel per unit of labour are some of the advantages of the continuous flow ~~transesterification~~ (Chen et al., 2010).

During ~~the~~ transesterification process, the reactants, ~~triglycerides?~~ (oil) such as WCO and methanol, are considered immiscible due to their inability ~~to~~ properly mix together and form a mixture with a single layer. The liquid-solid mass transfer resistance between ~~the~~ solid catalyst and reactants can be reduced by increasing the mass transfer and micromixing efficiencies using ~~a~~ ~~stirring~~ packed-bed reactor (Chen et al., 2010; Li et al., 2013).

~~Several~~ continuous flow reactors using heterogeneous catalysts to produce biodiesel from WCO (Borges & Díaz, 2013; Buasri et al., 2012). Application of RPB in ~~the~~ conventional heating has been widely used, particularly the use of the WCO as a raw material. It is clear that residence time is relatively ~~larger long~~ compared ~~d~~ to microwave heating. Although using microwave

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irradiation can produce biodiesel with a low residence time, but conversion could be obtained from this system is still low.

The response surface modelling (RSM) has been applied widely for in the development and optimization of various synthesis processes and for the optimization of analytical chemistry processes. RSM is a powerful statistical and mathematical tool with several advantages (Pinzi et al., 2010). Dwivedi and Sharma (2015) in their study carried out optimization of the four process factors through the implementation of Box-Behnken RSM in order to obtain maximum biodiesel yield from Pongamia oil.

The objective of this paper is to report on a continuous flow transesterification of WCO using microwave irradiation for biodiesel production, where in which the solid catalyst was placed in a catalyst container stirrer. It is interesting to optimize conversion of three process variables, namely power input, stirrer speed and LHSV, for the transesterification process of WCO using RSM-based BBD in 15 experimental runs, with the help of Design Expert version 7.0 software.

2. MATERIALS AND METHODS

2.1. Materials

Methanol analytical reagent (AR) Grade was purchased from QR&C, Republic of New Zealand. The WCO obtained from the restaurants containing FFA (0.41-0.48%) usually contains food waste in the form of solids. Therefore, filtering is necessary for filtering. Besides, water content contained in the WCO needs to be removed by evaporation. Evaporation of any water can be done by heating the oil in an electric oven at 110°C for 4 h. After that, the WCO is ready for the transesterification process.

The catalyst was CaO, it was made from the cockle shells. They were crushed using a hammer and sieved with to a size of 2 mm to 4 mm. Furthermore Subsequently, cockle the shells were decomposed in the a furnace at a temperature of 900°C for 3.5 h by the method performed by Nair et al. (2012). After the cold solids were obtained, they were stored in a desiccator before use in the transesterification process. Characterization was performed using X-ray diffraction (XRD) to see observe the structure of the metal oxide formed. The characterization results obtained were compared with data-JCPDS data, which are the standards for XRD diffraction pattern data and decomposition of cockle shells.

2.2. Transesterification process of waste cooking oil

2.2.1. Experimental set-up

The experiment was carried out using a 1 kW domestic modified microwave system having with a frequency of 2450 MHz frequency. The microwave power can be selected at the different levels from 100 to 1000 W and for various exposure times (1–90 min). A reactor made of borosilicate with a diameter of 50 mm and a length of 210 mm was placed in the microwave.

The catalyst was packed inside a perforated plastic container mounted on a stirrer shaft and inserted inside the reactor. Three holes were drilled in the top reactor cover of the domestic microwave oven in order to place the reactor inside it. Two K-type metallic thermocouples were used to measure the temperature of the methanol mixture and the WCO; one was inserted from the bottom of the reactor (T1) and one at the top of the reactor (T2). The thermocouples were connected to a Pico data acquisition system, which in turn was connected to a personal computer for continuous recording of the data using Picolog software. The thermocouple (T1) was also connected to a temperature controller and microwave power input to maintain the temperature.

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The microwave and thermocouple ~~should-were-be~~ grounded to avoid any discrepancy in temperatures readings or sparks.

The rotation of the packed bed was ~~done-made~~ using an overhead high-speed stirrer (WiseStir model HS-30D) equipped with a digital speed regulator. Stirrer speeds of 200, 300 and 400 rpm ~~were-as~~ used in this research. The rotation of the packed bed helped to increase the contact between the catalyst and the reactants. The experimental set-up for single step transesterification ~~continue-ds~~ the process with ~~the~~ catalyst container stirrer, as shown in Fig 1. The flowrate of WCO ~~was~~ regulated using a peristaltic pump, ~~whose~~ speed can be varied ~~which-ins~~ directly proportional to the flow rate. The molar ratio of alcohol to oil and reaction temperature ~~in this study~~ were fixed at 9:1 (Aworanti et al., 2013; Buasri et al., 2013) and 65-°C (Buasri et al., 2013), respectively. The weight of ~~the~~ catalyst ~~was~~ varied ~~between~~ 10.00, 12.73 and 20.00 g. ~~The f~~low rate of WCO ~~was~~ regulated by setting the speed of ~~the~~ peristaltic pump ~~from-at~~ 24, 28 and 32 rpm, ~~w~~hile the alcohol ~~was~~ put into the reactor using a syringe.

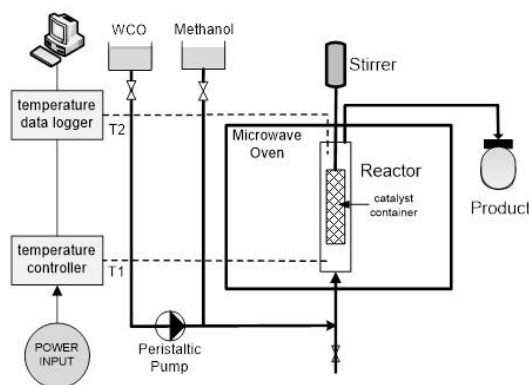


Figure 1 ~~E~~The experimental set-up for ~~continuous?~~ transesterification ~~continue-s~~ process with ~~stirring~~ packed-bed

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2.2.2 Experimental procedure

~~Weigh~~The catalyst ~~was weighed~~ and inserted ~~catalyst~~ into the container. ~~Fix a~~ container ~~was fixed onto~~ the stirrer shaft and inserted ~~it~~ into the glass reactor. ~~Pour~~ Methanol ~~was then poured~~ into the glass reactor ~~and~~ ~~Attach the~~ moveable cover ~~attached for-to~~ the top of a reactor. ~~Set~~The stirrer speed ~~was set~~ and ~~the~~ temperature controlled. ~~Make~~The process of dispersing ~~of-the~~ methanol and catalyst ~~was made~~ at 65-°C for 20 min. ~~Then stream~~The the WCO ~~was then streamed~~ through the bypass valve until the reactor ~~was~~ full. ~~Close~~The bypass valve ~~was closed~~ and ~~turn on~~ the peristaltic pump ~~turned on~~. Sampling was ~~done-made~~ after ~~the~~ process time ~~reache-ds~~ the space-time. The sample was collected in 10 mL glass vials and centrifuged at 4000 rpm for 10 min. The product mixture was ~~then~~ exposed to open air for 30 min. to evaporate ~~the~~ excess methanol (Aworanti et al., 2013). ~~Then~~Subsequently, ~~they were analyzed~~the chemical and physical properties ~~were analyzed~~.

2.3. Conversion of FFA

The effect of different parameters on ~~the~~ FFA conversion of WCO methyl ester (WCOME) that ~~are~~ investigated are catalyst weight, flowrate, stirrer speed and power input, ~~with the~~ molar ratio and reaction temperature kept constant. The reaction products were analyzed by gas chromatography (GC) ~~using the?~~ Perkin Elmer Auto system GS FID equipped with a flame-

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ionization detector (FID) through the capillary GC column BP5, 30 m in length, 0.25 mm ID and 0.25 micrometers. Comparison with authentic samples was carried out in order to identify the reactants and the products. The palmitic acid (hexadecanoic acid methyl ester) conversion is given by the following equation (Han et al., 2016):

$$\text{Conversion (\%)} = 100\% \times (1 - (a_1 / a_2)) \quad (1)$$

where a_1 and a_2 represent the initial and final stages of the reaction mixture, respectively.

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2.4. Space-time and space-velocity

According to Levenspiel (1999), for batch reactors the natural performance measure is the reaction time, while for flow reactors the proper performance measures are the space-velocity and the space-time. For a negligible change in volume, the residence time and the space-time (τ) in the batch reactor and ideal tubular reactor, respectively, are considered the same and is given by:

$$\tau = \text{reactor volume/volumetric feedrate} \quad (2)$$

The space velocity, which is the inverse of the space time, is given by:

$$\text{Space velocity} = 1 / \tau \quad (3)$$

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The liquid hourly space velocity (LHSV) in h^{-1} is a measure of the feed rate and is calculated using Eq. (4) (Ancheyta, 2011).

$$\text{LHSV} = \text{total volumetric flowrate/total catalyst volume} \quad (4)$$

$$\text{LHSV} = 60F / V_c \quad (5)$$

where F is flowrate in ml min^{-1} and V_c is the catalyst volume (ml), so that Eq. (5) can be expressed in the following equation:

$$\text{LHSV} = 60F \times \rho_c / W_c \quad (6)$$

where W_c is the catalyst weight (g) and ρ_c is the catalyst density (g ml^{-1}).

3. RESULTS AND DISCUSSION

3.1. Biodiesel production

The results of the biodiesel transesterification process of WCO using the packed bed stirrer are shown in Table 1. The kinematic viscosity of WCOME is lower than that of WCO. One major specification that a manufacturer of biodiesel needs to comply with is the kinematic viscosity. This indicates the amount of fatty acid methyl esters (FAME) contained in the biodiesel, thus signifying the degree of the reaction.

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Table 1 Selected properties of WCO and WCOME

| Properties | Units | WCO | WCOME |
|------------------|---------------------|--------|--------|
| Calorific value | kJ kg^{-1} | 43,522 | 38,049 |
| Density at 15 °C | kg l^{-1} | 0.917 | 0.870 |

| | | | |
|------------------------------|-----|-------|-------|
| Flash Point | °C | 318.0 | 158.5 |
| Kinematic Viscosity at 40 °C | cSt | 47.51 | 5.48 |

The results of the test using GCMS showed that one sample contains 97.91% methyl ester. Using a molar ratio of methanol to oil of 9:1, a temperature of 65°C, power input of 450 W, a stirrer speed of 400 rpm and LHSV of 57.31 h⁻¹. One of the biggest components is hexadecanoic acid methyl ester, which is then to be used in the calibration calculation of calibration.

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3.2. Experimental design results

The experimental response and predicted response for the transesterification of WCOME at a time collected to space time ratio of =1 are shown in Table 2. The predicted values of the output parameter are calculated using the following regression equation given as follows:

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$$C = 69.8236637 + 0.00341229 A + 0.0155426 B - 0.95253203 C - 1.644414 \times 10^{-5} AB + 0.000126601 \times AC + 9.4621 \times 10^{-5} BC - 9.4299 \times 10^{-7} (A^2) - 2.08229 \times 10^{-5} (B^2) - 0.000307651 \times (C^2) \quad (8)$$

Table 2 Responses for transesterification of WCOME

| Run | A:Power input (watts) | B:Stirrer speed (rpm) | C:LHSV (h ⁻¹) | Conversion (%) | |
|-----|-----------------------|-----------------------|---------------------------|-----------------------|--------------------|
| | | | | Experimental Response | Predicted Response |
| 1 | 450 | 400 | 57.17 | 72.48 | 72.51 |
| 2 | 315 | 300 | 57.17 | 72.09 | 71.93 |
| 3 | 450 | 300 | 83.18 | 72.18 | 72.34 |
| 4 | 315 | 400 | 31.19 | 72.11 | 72.11 |
| 5 | 315 | 400 | 83.18 | 71.77 | 71.59 |
| 6 | 315 | 200 | 31.19 | 71.75 | 71.94 |
| 7 | 450 | 200 | 57.17 | 72.46 | 72.29 |
| 8 | 180 | 300 | 83.18 | 70.05 | 70.07 |
| 9 | 315 | 300 | 57.17 | 71.96 | 71.93 |
| 10 | 180 | 200 | 57.17 | 70.49 | 70.47 |
| 11 | 450 | 300 | 31.19 | 72.48 | 72.46 |
| 12 | 315 | 300 | 57.17 | 71.75 | 71.93 |
| 13 | 180 | 300 | 31.19 | 72.13 | 71.97 |
| 14 | 315 | 200 | 83.18 | 70.43 | 70.44 |
| 15 | 180 | 400 | 57.17 | 71.40 | 71.57 |

The significance of each coefficient in Eq. (8) was evaluated by the P-value, as shown in Table 3. The smaller the magnitude of the P-value, the more significant is the corresponding coefficient. Considering the linear effect, only two operating parameters were found to be significant terms in increasing the conversion in the final product. The power input term had the most significant linear effect, followed by LHSV and stirrer speed, respectively. In terms of the quadratic effect, no parameter was significant at the 5% level. However, for the interaction effect, it was found that only power input-LHSV interaction was significant at 5% level. The other interaction terms had no significant effect on the WCOME conversion.

Table 2 shows that the value of the Model F-value of 19.23 means that the model is significant. Under this condition the "Prob > F" value of less than 0.0500 indicates a significant model term, three of which are the main parameters of power input, stirrer speed and LHSV. The interaction effect of power input-LHSV is also significant. If there are many insignificant model terms (not including those needed to support the hierarchy), model reductions can improve your model. The "Lack-of-Fit F-value" of 2.11 implies that Lack of Fit is insignificant compared to pure error. There is a possibility of 33.70% that this "Lack-of-Fit F-value" could be due to noise. The absence of an insignificant fit is good enough. The results in Table 3 also show that this model is acceptable, because the model is significant and lack of fit are not significant. In addition, the power input parameters, stirrer speed, LHSV, and (power input & LHSV) are also significant.

Table 3 ANOVA for response surface quadratic model analysis of variance table

| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F | |
|-----------------|----------------|----|-------------|-------------|------------------|-----------------|
| Model | 8.262845 | 9 | 0.918094 | 19.22902983 | 0.0023 | significant |
| A-Power Input | 3.830211 | 1 | 3.830211 | 80.22190583 | 0.0003 | |
| B-Stirrer Speed | 0.861057 | 1 | 0.861057 | 18.03442567 | 0.0081 | |
| C-LHSV | 2.045512 | 1 | 2.045512 | 42.84225667 | 0.0012 | |
| AB | 0.197064 | 1 | 0.197064 | 4.127410066 | 0.0979 | |
| AC | 0.789524 | 1 | 0.789524 | 16.53619519 | 0.0097 | |
| BC | 0.242002 | 1 | 0.242002 | 5.068608073 | 0.0742 | |
| A ² | 0.001091 | 1 | 0.001091 | 0.022841231 | 0.8858 | |
| B ² | 0.160096 | 1 | 0.160096 | 3.353141191 | 0.1266 | |
| C ² | 0.159572 | 1 | 0.159572 | 3.342154705 | 0.1271 | |
| Residual | 0.238726 | 5 | 0.047745 | 2.114964134 | 0.3370 | |
| Lack of Fit | 0.181511 | 3 | 0.060504 | | | not significant |
| Pure Error | 0.057215 | 2 | 0.028607 | | | |
| Cor Total | 8.501571 | 14 | | | | |

The R² value for WCOME conversion is 0.9719. This means that 97.19% of the variability in the data accounted to model, thereby validating the reliability of the model developed for establishing a correlation between the process variables and the WCOME conversion.

3.2.1. Interactive effects of the variables on WCOME conversion

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The interaction of power input and stirrer speed at an LHSV of 71.5 h^{-1} on WCOME conversion is exhibited in Figure 2. It shows that for low power input, WCOME conversion increases with increasing stirrer speed. At the high power input, WCOME conversion increases with stirrer speed until a certain point, and then decreases. The minimum WCOME occurs at minimum power input and minimum stirrer speed. This is due to the fact that lower agitation and power transmission from microwave to mixture decrease the conversion. The yield value clearly increased when the speed was increased, because of the increase in the micro-mixing intensity between the oil and methanol phases (Chen et al., 2010).

3.2.2 Optimization of response parameters

The objective of optimization is to find the best setting value conditions based on the power input, stirrer speed and LHSV that produce the highest conversion value. The optimum conversion is obtained by setting the lower and upper limits of the variable process, namely power

Design-Expert® Software
 % CONVERSION
 X1 = A: Power Input
 X2 = B: Stirrer Speed
 Actual Factor
 C: LHSV = 71.500

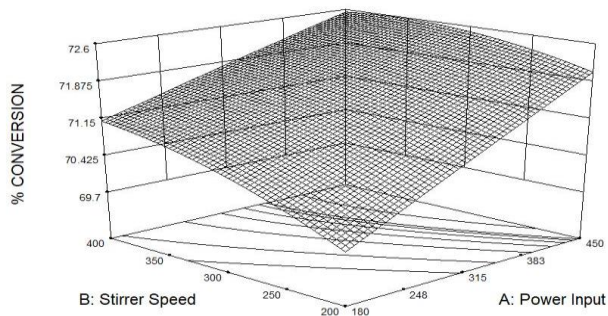


Figure 2 WCOME conversion versus power input and stirrer speed at LHSV= 71.5 h^{-1}

input, stirrer speed and LHSV. The percentage of conversion was set to maximum. Based on the model it is implied that the model is significant and the lack-of-fit F-value which is not significant, hence indicating that the model can be used to predict the conversion.

The response was optimized to maximize the WCOME conversion conditions for collected time same, as space time was based on the mathematical equation that was developed. The optimum value of the WCOME conversion of 72.52% was achieved with a power input of 445 W, stirrer speed of 380 rpm and LHSV of 71.5 h^{-1} . The experiment was then validated at in optimization conditions of a power input of 450 W, stirrer speed of 380 rpm and LHSV of 71.5 h^{-1} . The result of WCOME conversion at this condition at these levels was 72.5%.

Some continuous flow biodiesel production research using heterogeneous catalysts are shown in Table 4. It shows that in which the residence time of MW heating is relatively shorter as compared to when using conventional heating. Microwave irradiation can produce biodiesel with a shorter residence time, depending on the design requirements for fuel consumption in diesel engine power generation.

Table 4. Continuous flow biodiesel production of WCO using heterogeneous catalyst and its condition and performance.

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| Catalyst used | Technologies—H-heating method technology | Reaction conditions | | | Performance | Ref. |
|---|--|---------------------|--|-----------------------------------|---------------------------------|---------------------|
| | | Temperature (°C) | Type of alcohol (alcohol to oil molar ratio) | Residence time- (LHSV) | | |
| Calcium oxide (CaO) from cockle shells (lump size 2-4 mm) | Stirring packed-bed reactor Microwave heating | 65 | Methanol (9) | 50.35 min (71.5 h ⁻¹) | Conversion of FFA= 72.57% (max) | Present work |
| Potassium-loaded pumice material (K-Pumice) | Packed-bed 8.2 cm packed-bed length conventional heating | 55 | Methanol (28) | 2 h | Yield=95.9% | Borges et al (2013) |
| Calcium oxide supported on activated carbon (CaO/AC) | Fixed bed reactor 295mm packed bed height conventional heating | 60 | Methanol (25) | 8 h | Conversion =94% | Buari et al (2012) |

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In an earlier study, for the a microwave biodiesel production system using a homogeneous catalyst which is equipped with a mechanical stirrer, the energy for electrical consumption was 0.468 kWh kg⁻¹ of biodiesel (Motasemi & Ani, 2012). Later, a similar study, was done conducted using a microwave reactor for the continuous production of palm oil biodiesel by Choedkiatsakul et al. (2015). The energy consumption for this process include the premixing tank, peristaltic pump, microwave system and cooling system were measured by a plug-in power meter which required 0.1167 kWh l⁻¹ of biodiesel. In this study, the energy consumption for the optimal steady state condition is 0.594 kWh for the production of 1 litre of biodiesel using a heterogeneous catalyst.

4. CONCLUSION

The application of the continuous flow transesterification of WCO using microwave technology with a perforated plastic container for the solid catalyst on the stirrer could be used to convert WCO to biodiesel. The effects of variables such as power input, stirrer speed and LHSV on the heterogeneous transesterification reaction are very significant. The results obtained clearly showed that the RSM-based BBD is a valuable tool that can be used to establish the relationships among-between the process factors and the responses, with a minimum number of experiments, in a very efficient way. Furthermore, the RSM-based BBD can be used to determine the recommended optimum conditions for the production of the-biodiesel.

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