Investigation on continuous FAME production using rotor reactor with bumpy number variation under theoretical methanol to oil molar ratio condition

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Abstract
Continuous methyl ester production process from waste cooking oil (WCO) was studied by using a transesterification process at room temperature. The aim of this research was to investigate the optimized production conditions and specific energy consumption (SEW*h/kg of neat biodiesel) of the bumpy surface rotor reactor. There are 6.9%, 13.8%, and 27.6% of area fractions (AF) respectively. The reaction was under the condition being theoretical methanol to oil molar ratio and the reactor volume was 0.5L. At 2,000-4,000rpm of rotor speed, 1.0-1.5%w/w of KOH and 1.132-5.110L/min of precursor flow rate are used. It was found that the highest FAME content (fatty acid methyl ester in neat biodiesel, %) being 98.6%. It was achieved by using AF 27.6% rotor reactor under experimental conditions including 3,000rpm, 1.50%w/w of KOH and 2.027L/min of precursor flow rate. In addition, this condition gave SEC of 12.5W*h/kg. It was lower than that of orifice hydrodynamic cavitation reactor, power ultrasonic reactor, and mechanical stirring reactor around 93.2%, 95.0%, and 97.5% respectively. The properties of produced FAME met both EN 14214 and ASTM D 6751 standards.

Keywords: Continuous biodiesel production, Bumpy surface rotor reactor, Specific energy consumption, Retention time

1. Introduction
Information Methyl ester or Biodiesel is an alternative fuel to compensate diesel. Biodiesel can be produced from edible oil such as palm, coconut, soybean, sunflower oil or animal oil. In addition, it can be produced from non-edible oil such as used vegetable oil, Jatropha oil, and it also contains oil from rubber granules [1, 2]. About 95% of biodiesel production in the world is derived from edible oil. The price of oils for edible was the main cost of biodiesel production and made the cost increase by 1.5-2.0 times the diesel fuel price [3, 4].

Currently, the raw material cost is a major part of the total biodiesel production cost which is about 75-78% [5]. Biodiesel production on a commercial scale was necessary to reduce raw material cost. For this reason, used oils such as waste cooking oils have become more attractive to the alternative raw
material. Therefore, the energy efficiency and raw material cost were major contributors to decrease the total cost of biodiesel production [6, 7].

It was estimated that the WCO amount in Thailand had 74 million L/year approximately [8]. After the suspension was filtered out of WCO and passed oils to the dehumidification process that the treated oils had properties nearly the pure refined vegetable oils since it was low in percentage of free fatty acid (%FFA). It could be used to produce biodiesel by transesterification process [9, 10].

The optimized production conditions for mechanical stirring (MS) including 6:1 of MeOH to oil molar ratio, 1.0 %wt. of KOH, 60˚C of reaction temperature, and 60 min of reaction time [11, 12]. The research by Ji, J. [1] showed the comparison to the biodiesel productions among MS, hydrodynamic cavitation (HC), and power ultrasonic cavitation (UC) which the optimized condition including 45˚C of reaction temperature, using 6:1 of MeOH to oil and 1% w/w of KOH as a catalyst. MS operated at 900rpm, PU used the power150W at 19.7MHz, and the single-edged plate HC worked at 0.7MPa. They compared the SEC that sorted by MS, PU, and HC at 500 W*h/kg, 250 W*h/kg, and 183W*h/kg respectively. HC had a slightly lower reaction rate than PU while MS had the lowest reaction rate. The production of biodiesel by the MS via transesterification process was interrupted by the reaction rate limited by mass transfer of the oil and alcohol [13]. They could not be mixed homogeneously and the process was a reverse reaction as Equation 1 which had a limit for reversing by the discontinuity in the product circulation. It didn’t benefit from continuous production.

\[
TGR + 3MeOH \rightarrow GL + 3FAME
\]

Where a mole of triglyceride (TGR) reacted with 3 moles of the methanol (MeOH) by using KOH as a catalyst that changed to a mole of glycerol (GL) and 3 moles of the fatty acid methyl ester (FAME).

HC was started with the cavitation phenomenon caused by the transition of status from liquid to vapor. Because the cavitation beginning point pressure was less than the vapor pressure over the surface of that substance. In order to the cavitation could be achieved by using the reactor at an approximately constant temperature within the precursor flow. Cavitation was similar to boiling which it was caused by a change in pressure that was controlled by the fluid dynamics, but it was not due to the change in temperature [14, 15]. The intensification technology, such as a bumpy surface rotor reactor (BSRR) could be increased the mass transfer rate between oil and alcohol at room temperature. It has some challenges for this research that still could be reduced the MeOH quantity, the SEC, and retention time respectively.

In this study, the WCO methyl ester was produced by using the BSRR via transesterification process and using theoretical MeOH to oil molar ratio. The optimized production conditions were investigated by studying the variables including rotor speed, [KOH] %w/w, precursor flow rate, and the investigated parameter was the SEC. The FAME content was compared when using a reactor with rotors AF 6.9%, 13.8%, and 27.6% that the properties of biodiesel produced met both of EN 14214 and ASTM D 6751 standards.

2. Materials and methods

2.1 Material
The waste cooking oil was bought from a local fried chicken shop nearby Ubon Ratchathani University, Thailand. The suspensions particles in WCO were removed by filtration through a filter 30 sieve and the moisture was removed by boiling at 110˚C which treated WCO has 1.89% FFA. It was packed in a closed container when cooled down at 30-40˚C. The WCO properties were summarized in Table 3. Methanol used anhydrous grade with 99.9% purity. Potassium hydroxide was used with industrial grade 90% purity.

2.2 Methods
The experimental equipment and setup show in Figure 1 that consisted of a precursor storage tank with
a 40L capacity (1). The storage tank inlet was connected to the WCO feeder (2) and feeder of MeOH mixed with KOH (3). The precursor flowed pass to inlet valve (4) through pump model PMD-331B (5). The precursor flow rate into BSRR could be adjusted by opened ball valve (6) 16.5-25.5° to vary the flow rate from 1.132-5.110L/min. Within BSRR (7), it consisted of three type rotor AF 6.9%, 13.8%, and 27.6% of area fractions that relationship was shown in Equation 2, and equipment detail was shown in Figure 2. Inside the reactor had a gap between the rotor surface and the contact surface jacket within 4.5mm. At the bottom of the reactor was connected with a ball valve (8) for storing product samples. The inner temperature was measured by thermocouple Type K 0-399°C (9). The reactor was driven by Induction Motor 5HP model SF-JR (10). The speed control device was the inverter model VB5N 43P7G-S / 45P5P (11). The energy consumption was measured with LCD Watt-Hour 3 phase Energy Meter model DTM 024 (12).

![Diagram of the Bumpy Surface Rotor Reactor (BSRR)](image)

**Figure 1.** The Bumpy Surface Rotor Reactor (BSRR) with 0.5L capacity

**Figure 2.** The bumpy surface rotors with area fraction 6.9%, 13.8%, and 27.6% respectively

\[
\text{Area Fraction} = \frac{A_{\text{Holes}}}{A_{\text{Rotor Surface}}} \times 100
\]

Where Area fraction (%) is the percentage of the total area of holes \( A_{\text{Holes}} \) per the surface area of the rotor \( A_{\text{Rotor Surface}} \)
The properties of WCO and biodiesel were checked under standards that shows in Table 3. The percentage of FAME was evaluated according to EN14214 under EN14103. Regarding with gas chromatography (GC) analysis by CLARUS 680 GC equipped with CLARUS SQ 8 CMS which was shown in Figure 3.

Figure 3. A CLARUS 680 GC equipped with a CLARUS SQ 8 C MS

The gas chromatography graph (GC graph) of neat biodiesel was analyzed by using the area under curve (AUC) method as show in Figure 4 which %FAME was investigated by using the gas chromatography technique. The FAME content in each experiment was calculated by the following expression as shown in Equation 3.

\[
FAME\ content = \frac{A_{FAME}}{\sum A} \times 100
\]

(3)

Where \( FAME\ content \) (%) was the percentage of FAME in neat biodiesel which \( A_{FAME} \) (%) was the area under the methyl ester curve, \( \sum A \) (%) was the total area under the curve acquired by GC.

2.3 Experiment

The experiment included two parts. In the first part, the methanol to oil molar ratio was set at 3:1 with a constant speed of 3000rpm. The BSRR with rotor AF 6.9%, 13.8%, and 27.6% were investigated by comparison the effect to FAME content. In addition, the variables were checked by chose the optimal variables that were flow rate (1.132, 2.027, 3.000, 4.128, and 5.110L/min) and [KOH] (1.00, 1.25, and 1.50%w/w).

The second part was an experiment to find a suitable speed by using the [KOH] %w/w and the flow rate from the first part with theoretical molar ratio. The sampling collection time was 1min for all two
parts of the experiment. The optimal variable of the best result was set up to repeat three times. The electrical energy consumption and mass of neat biodiesel were recorded and used to calculate parameter SEC as shows in Equation 4.

The specific energy consumption, \( SEC \) (W*h/kg) was calculated by the following expression.

\[
SEC = \frac{EC}{m_{NB}}
\]  

(4)

Where energy consumption, \( EC \) (W*h) was the total energy used to produce a net mass of neat biodiesel, \( m_{NB} \) (kg).

3. Results and discussion

3.1 Effect of KOH concentration

![Figure 5. Effect of flow rate on FAME content with different area fraction: [KOH] 1.00%w/w](image1)

![Figure 6. Effect of flow rate on FAME content with different area fraction: [KOH] 1.25%w/w](image2)

Figures 5 to 7 show the [KOH] %w/w affected the conversion of triglycerides to methyl esters with various flow rates at different area fraction. They show the influence of [KOH] %w/w on the evolution of ester content with varied flow rate as shown in Figure 5, 6, and 7 by increasing of FAME content.
varied on the increasing of AF. The best results were reached with further increased in [KOH] %w/w and the optimal was [KOH] 1.50%w/w. All of the experiment results FAME content were greater than 96.5% which the best FAME content of rotor AF 6.9%, 13.8%, and 27.6% were 97.8%, 98.2%, and 98.6% respectively with a flow rate 2.027L/min as shown in Figure 7.

![Figure 7. Effect of flow rate on FAME content with different area fraction: [KOH] 1.50%w/w](image)

3.2 Effect of rotor speed
Figure 8 shows the rotor speed affected the FAME content to vary on the increasing of AF. It shows the influence of speed on the FAME content that it increased when speed was increased. The experiment gave the best results at 3000rpm that FAME content was 98.6% by rotor AF 27.6% and [KOH] 1.50%w/w.

![Figure 8. Effect of speed on FAME content with different area fraction](image)

3.3 Effect of rotor speed on temperature
Figure 9 shows the increased speed affected the increasing of temperature which varied on each AF. When AF was constant, the influence of speed increased in the range of 2000-4000rpm and affected the temperature to increase 2°C. All temperatures were recorded after operated for 1 min at initial temperature of 30°C without an external heat source. When the rotor speed was constant and AF was increased while temperature was increasing in range 1-4°C.
3.4 Specific energy consumption
Table 1 shows the increasing of SEC when speed was increased and AF was constant. At the same speed shows the decreasing of SEC since $m_{NB}$ increased when AF was increased. The best results at the optimal conditions showed that SEC with AF 27.6% was 12.50 W*h/kg.

Table 1. Effect of speed on SEC with MeOH to oil ratio 3:1 and [KOH] 1.50%w/w by different AF

| Speed (rpm) | AF 6.9% | AF 13.8% | AF 27.6% |
|-------------|---------|---------|---------|
| | EC W*h/kg | $m_{NB}$ kg | SEC W*h/kg | EC W*h/kg | $m_{NB}$ kg | SEC W*h/kg | EC W*h/kg | $m_{NB}$ kg | SEC W*h/kg |
| 2000 | 10 | 1.35 | 7.41 | 10 | 1.50 | 6.67 | 10 | 1.55 | 6.45 |
| 2500 | 15 | 1.50 | 10.00 | 15 | 1.55 | 9.68 | 15 | 1.55 | 9.68 |
| 3000 | 20 | 1.50 | 13.33 | 20 | 1.55 | 12.90 | 20 | 1.60 | 12.50 |
| 3500 | 25 | 1.50 | 16.66 | 25 | 1.55 | 16.13 | 25 | 1.60 | 15.63 |
| 4000 | 30 | 1.50 | 20.00 | 30 | 1.55 | 19.35 | 30 | 1.60 | 18.75 |

Table 2. Comparison of BSRR performance with different reactors [1]

| Description | Reactor Type | BSRR | HC | PU | MS |
|-------------|--------------|------|----|----|----|
| SEC (W*h/kg) | 12.5 | 183 | 250 | 500 |    |
| Methanol to Oil by mol | 3:1 | 6:1 | 6:1 | 6:1 |    |
| Temp. °C | Room Temp. | 45 | 45 | 60 |    |
| KOH wt.% | 1.50 | 1.00 | 1.00 | 1.00 |    |
| Time (min.) | Retention time | Reaction Time | 0.25 | 30.0 | 10.0 | 60.0 |    |

3.5 Performance
The optimal conditions gave SEC of 12.5W*h/kg which less than HC reactor, PU reactor, and MS reactor to 93.2%, 95.0%, and 97.5%. The retention time was 0.25min which less than HC, PU, and MS reactor to 29.8 min, 9.8 min, and 59.8 min respectively as shown in Table 2. The produced FAME physicochemical properties are met both ASTM D6751 and EN 14214 standards as shown in Table 3.
Table 3. Physicochemical properties of WCO and biodiesel in comparison to biodiesel standard

| Properties                        | WCO | WCO biodiesel | ASTM D6751 | EN 14214 | Standards /Method |
|-----------------------------------|-----|---------------|------------|----------|------------------|
| Density (kg/m³, 15°C)             | 915.0 | 880.0        | -          | 860-900  | ASTM D1298       |
| Kinematic viscosity (cSt,40°C)    | 40.50 | 3.52         | 1.9-6.0    | 3.50-5.00 | ASTM D445       |
| Sulfur content (mg/kg)            | 10.50 | 10.00        | 15 max     | 10 max   | ASTM D2622       |
| High heating value (MJ/kg)        | 38.65 | 39.74        | -          | -        | ASTM D240        |
| FAME content                      | -    | 98.6         | -          | 96.5 min  | EN 14103         |

3.6 The comparison of production conditions

In early studies of the FAME production compared to the production conditions studied as shown in Table 4 found that BSRR can be operated at room temperature. This reactor didn’t want to use a reactant preheating as the conventional method, it used the lowest methanol to oil molar ratio only 3:1, using 1.50%w/w of KOH, 3000rpm of rotor speed, and 2.027L/min of flow rate. This continuous production used the SEC only 12.5W*h/kg of neat biodiesel and conversed high FAME content 98.6%.

Table 4. The FAME production conditions summary by using WCO with methanol as reactant

| Researchers                  | Catalyst (%w/w) | alc./Oil Molar Ratio | Reaction Condition | FAME Content (%) | Other details |
|------------------------------|-----------------|----------------------|--------------------|------------------|---------------|
| Chen G [16]                  | CH₃ONa, 0.75    | 6:1                  | 65°C 3min          | 97.9             | MC 1200W FFA<1% |
| Azcan N [17]                 | NAOH, 0.75      | 6:1                  | 60°C 5min          | 98.8             | MC Power not available |
| Barnard T M [18]             | KOH, 1.00       | 6:1                  | 50°C 0.5min        | 97.9             | MC 1.6kW 0.11% |
|                             |                 |                      |                    | 2.0 L/min        | FFA Con. Pro. with Teflon tubing coil 8cm ID |
| Maghami M [19]               | KOH, 1.00       | 6:1                  | 55°C 30min         | 79.9             | 100% UC 400W 20kHz |
| Pukale D D [20]              | K₃PO₄, 3.00     | 6:1                  | 50°C 90min         | 92.0             | 50% UC 375W 22 kHz |
| Anuar M R [21]               | Hydrotalcite, 0.08 | 15:1              | 57°C 1min          | 76.5             | 55% UC 400W 20kHz |
| Maddikeri G L [22]           | CH₃OK, 1.00     | 12:1                 | 40°C 30min         | 90.0             | 60% UC 450W 22 kHz |
| Ghayal D [6]                 | KOH, 1.00       | 6:1                  | 60°C 10 min        | 95.0             | HC 7.5kW Cap. 10L |
| Maddikeri G L [23]           | CH₃OK, 3.00     | 12:1                 | 40°C 30min         | 90.0             | HC 3 bar Cap.15L |
|                             |                 |                      |                    | 90.0             | UC 60%-450W 22kHz |
|                             |                 |                      |                    | 70.0             | MS 1000rpm |
|                             |                 |                      |                    | 98.0             | HC4kW 2bar50L |
| Chuah L F [24]              | KOH,1.00        | 6:1                  | 60°C 15 min        | 97.9             | MW 1.2kW FFA<1% |
|                             |                 |                      | 60°C 90 min        | 97.0             | BSR Reactor Cap0.5L |
|                             |                 |                      |                    |                  | speed 300rpm, SEC12.5W*h/kg |
| Chen K S [25]                | CH₃ONa, 0.75    | 15:1                 | Room temp.         | 98.6             |               |
| Thaiyasuit P                 | KOH,1.50        | 3:1                  | Continuous Production | 2.027 L/min |               |
4. Conclusion
In summary, a transesterification process with BSRR was applied to carry out the experiment to produce high purity biodiesel with WCO which having a low %FFA. The continuous production condition is an efficient method that saved a lot of energy, time, and cost.

The optimum conditions for continuous production of WCO methyl ester under BSRR conditions and theoretical molar ratio of 3:1 were as follows; the first independent variable was the alkaline catalysed concentration of KOH %w/w which affected the conversion of TGR to FAME when [KOH] %w/w was constant, increasing of FAME content varied on the increasing of AF. When AF was constant, the best results were reached with a further increased in [KOH] %w/w and the optimum was [KOH] of 1.50%w/w. The second variable was the rotor speed that affected the FAME content when the speed was constant and AF was increased and affected the FAME content to be increased. The best result was a rotor speed of 3000 rpm at the precursor flow rate of 2.027L/min with 0.25 min of retention time. The highest FAME content of rotor AF 6.9 %, AF 13.8 %, and AF 27.6% under the achieved optimum condition were 97.8%, 98.2%, and 98.6% respectively.

When BSRR was operated at room temperature that HC was started with the cavitation caused by the transition of precursor flowed. The status of methanol in the mixture of KOH and WCO was changed from liquid to vapour. At the beginning point of cavitation, the pressure in the reactor was less than the vapour pressure over the surface of that substance. The cavitation could be achieved by using the BSRR which was caused by a change in pressure at an approximately constant temperature with the changing in the narrow range. After operating the reactor for 1 min at an initial temperature of 30°C when speed was constant and the AF increased, the result was showed that the temperature increased in the narrow range of 1-4°C. For all of the AF when the speed was changed to increase in the range of 2000rpm to 4000rpm which affected the temperature to be increased in the range of 2°C. Therefore, this reactor was not required to use reactant preheating as the conventional method.

The SEC was a parameter that increased when AF was constant and rotor speed was increased. EC was dependent on the rotor speed when the rotor speed was constant and AF was increased in which affected the neat biodiesel mass to increase, but EC was constant with the rotor speed, so SEC was decreased. The optimum conditions showed that SEC with AF 27.6% was 12.50 W*h/kg with EC of 20 W*h but could be produced neat biodiesel 1.6kg in 1 min. Finally, the performance comparison of SEC with the other reactor found that BSRR was less than the HC reactor, PU reactor, and MS reactor to 93.2%, 95.0%, and 97.5%. The retention time was 0.25min which less than HC, PU, and MS reactor to 29.8 min, 9.8 min, and 59.8 min, respectively. The results were showed that the FAME content increased due to an increase in the AF that the most optimized BSRR was achieved by using AF 27.6% (120 holes) rotor reactor. It was found that the highest FAME content at 98.6% was met both ASTM D6751 and EN 14214 standards.

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