Biodiesel Production from Waste Cooking Oil Using Sequential Process Intensification Technique (Ultrasound and Microwave)

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Abstract. The use of biodiesel as a renewable fuel has increasingly become more attractive due to its environmental advantages such as non-toxicity and biodegradability. While biodiesel has the capability to mitigate the energy crisis, its production is hindered by the availability of feedstock. The general drawbacks of the transesterification-based synthesis include high energy consumption, high operating cost, high separation times, and low production efficiency. The present study demonstrates an innovative and industrially viable biodiesel production process from waste cooking oil (WCO) as feedstock and KOH as a catalyst using sequential ultrasound (22kHz + 35kHz) and microwave (0 – 1000W) reactor. The sequential approach of ultrasound and microwave removes the barrier of mass transfer (high-velocity liquid jets and turbulence induced by ultrasound) and enhances the heat transfer (rapid dipolar moment induced by microwave) by their synergism while significantly improving the reaction efficiency and overall economics of the process. The reaction conditions that can attain a maximum yield of 96.9% were methanol/oil molar ratio, 7:1, catalyst concentration, 0.9 wt%, and reaction temperature, 62°C in 5 minutes of reaction time for a sample size of 4500 mL.

1. Introduction
Currently, global heating and environmental deterioration are threatening the world. The gases emitted by burning fossil fuels are the major source of pollution. Therefore, alternative energy sources need to be identified that are feasible, ecologically friendly, and commercially sustainable. In addition, the world-wide exhaustion of petroleum has also encouraged the search for alternative sources.

Biodiesel as an alternative fuel holds great potential. It is a biodegradable, renewable, non-toxic fuel, and a reducer of carbon monoxide emissions that can be recycled by photosynthesis. Besides, biodiesel has the benefit of good fuel properties such as good lubricity, reduced exhaust gas emissions, low sulfur, and aromatic contents, carbon-neutral and lower carbon dioxide emissions in the atmosphere, a cetane number and cloud point that is largely dependent on the feedstock and a high flash point that tends to make it volatile and easy to handle [1].

For biodiesel productivity, edible oils are categorized as first-generation feedstock. Nonetheless, due to the clash with the accessibility of food and waste disposal in the environment, there is currently a switch towards the use of low-grade feedstock (second-generation) such as inedible oils and waste cooking oil (WCO) to produce biodiesel [2].

Various characteristics of edible oil are modified by frequent frying. Therefore, controlling the quality of the oil is necessary to avoid using deteriorated oil for cooking purposes. The Food Safety
and Standards Authority of India (FSSAI) stipulates a threshold of 25\% for Total Polar Compounds (TPC), above which vegetable oil is not appropriate for use [3]. Vegetable oil with a TPC of more than 25\% is referred to as waste cooking oil.

The properties of WCO vary globally. WCO is collected from large-scale food processing industries, households, and business establishments. The usage of WCO as a substitute feedstock has the potential to mitigate the environmental concern of waste disposal [4]. The other benefits of using waste cooking oil over edible vegetable oil include availability, better oxidation stability, and a greater percentage of saturated fatty acids [5]. Currently, 850 crore litres of diesel is used in India every month. The government intends to blend 5\% of biodiesel into diesel by 2030 in line with the National Policy on Biofuels -2018 [3]. There is therefore a requirement for 500 crore litres of biodiesel per annum. The current consumption of vegetable oil in India is 2700 crore litres, of which 140 crore litres of WCO may be obtained from the majority of consumers like restaurants, canteens, and hotels, which could provide approximately 110 crore litres of biodiesel annually [3].

Approximately 75\% of the biodiesel’s production price corresponds to the price of virgin vegetable oil [6]. On an average, WCO is considered to be 2 to 3 times cheaper than that of virgin cooking oil [7]. The higher concentration of free fatty acid (FFA) makes WCO less expensive.

Biodiesel fuels are generally produced through a transesterification process, a method in which triglyceride (oil/fat) reacts with an alcohol involving a catalyst to produce esters (biodiesel) and glycerol (waste by-product). Transesterification comprises three successive reversible reaction steps, with each step resulting in one ester molecule [8] as shown in equations (1), (2), and (3).

\[
\text{Triglyceride} + \text{R'}\text{OH} \leftrightarrow \text{Diglyceride} + \text{R'COOR} \quad (1)
\]

\[
\text{Diglyceride} + \text{R'}\text{OH} \leftrightarrow \text{Monoglyceride} + \text{R'COOR} \quad (2)
\]

\[
\text{Monoglyceride} + \text{R'}\text{OH} \leftrightarrow \text{Glycerol} + \text{R'COOR} \quad (3)
\]

The traditional approach of mechanical stirring for biodiesel production from WCO as a feasible feedstock has been reported by several researchers in their studies [9][10]. This method has many drawbacks like low product conversion, an excess concentration of reactants, slow and inefficient heating, and higher by-product yield [11]. Thus, resulting in excess power consumption for the production of biodiesel.

The concept of process intensification refers to the implementation of innovative techniques and/or equipment that can achieve substantially higher benefits or yields compared to the existing techniques and/or equipment in operation [12]. In the perspective of the production of biodiesel, process intensification refers to the rise in heat and mass transfer rates between reaction products at various stages of biodiesel production such as purification and/or separation and/or transesterification and/or extraction. Various techniques have been proposed to improve biodiesel productivity like reactive distillation [13], membrane reactor [14], microwave, and ultrasonic irradiation (independently or in a combination) [15][7]. Among them, the most noticeable results were observed when microwave or ultrasonic-assisted methods were employed for process intensification.

Several kinds of research involving microwave and ultrasound as techniques for intensifying the processes have demonstrated increased biodiesel yields, reduced energy consumption, better biodiesel quality, and reduced reaction times owing to the thermal and non-thermal effects of these radiations. In particular, microwaves produce fast and appropriate heating but have a mass transfer limit [16] while ultrasound induces intense mechanical mixing through cavitation but has heat transfer limitations [17]. These restrictions of heat and mass transfer can be resolved simultaneously by integrating the two separate methods into a single apparatus leading to a balanced operation that could lead to energy-efficient biodiesel productivity. Ultrasound will produce the mechanical mixing that microwave needs to boost mass transfer and minimize localized overheating; and microwaves will produce thermal energy that is lacking in ultrasound, hence enhancing the process and increasing the benefits [18].

The studies conducted by Yan et al. [19], Thakkar et al. [20], Yu et al. [21], Safieddin Ardebili et al. [22], Gole et al. [23], and Hsiao et al. [18], [24] showed use combined microwave-ultrasound and achieved the yield up to 98\%. Studies on waste cooking oil using combined microwave-ultrasound
were reported by Martinez-Guerra et al. [12], [22] reported the yield around 95%. The production of biodiesel by this method meets the standards for biodiesel. While the impacts of ultrasound and microwave irradiations on the process of transesterification have been studied separately in several studies but their combined effect on biodiesel productivity has been stated in relatively few studies. The purpose of this research was to investigate the sequential impact of ultrasound followed by microwave on the biodiesel productivity from waste WCO and potassium hydroxide (KOH) as feedstock and catalyst respectively. The optimum reaction conditions of various process parameters such as methanol: oil molar ratio, catalyst content, reaction temperature, and time were also examined.

2. Materials and Methods

2.1. Overview of Experimental Configuration

Figure 1 demonstrates the system developed for the batch synthesis of biodiesel employing sequential ultrasound and microwave. The developed sequential reactor set-up comprises an oil tank, water tank, heater, stirrer, rotameter, flow control valves, pump, ultrasound generator, ultrasonic reactor, microwave, by-pass valves, and control panel. The system is composed of ultrasound and microwave connected in series. It is possible to use the ultrasound and the microwave separately, or both in series. The output of the ultrasound is supplied to the microwave in the series connection.

At first, a known quantity of oil is fed in the tank. It is heated to a required temperature utilizing a hot water jacket. A mixture of catalyst and solvent is supplied into the tank after the oil attains the required temperature, and then this mixture is stirred by means of a stirrer. This mixture then passes to the ultrasound through a pump. It passes to the microwave from ultrasound and then back to the tank. The mixture also keeps on rotating inside the reactor for a stipulated amount of time after which it is collected employing a drain valve at the bottom of the tank.

2.2. Reagents and Materials Used

WCO was collected from the canteen of PDPU, methanol (Emplura Grade, 99% purity) was used as a solvent and potassium hydroxide (KOH) flakes (Emplura Grade, 85% purity) were utilized as a homogeneous base catalyst.

2.3. Procedure for Experimental Run

The process of transesterification was performed in an oil tank (capacity: 20 liters) equipped with a condenser and thermocouple using a batch size of 4500 mL. A precise quantity of pre-mixed catalyst and methanol mixture was introduced into the tank. US power, MW power, and stirrer speed are 600W, 1000W, and 500 rpm respectively were fixed during all the reaction runs while varying the catalyst content from 0.9 to 1.2 wt%, methanol: oil molar ratio from 4.5:1 to 12:1, temperature from 52 to 62 °C, and reaction time varying from 5 to 15 minutes. Two phases of reaction mass, i.e., the top phase of biodiesel and the bottom phase of glycerol involving a small amount of unreacted methanol and oil, were found upon completion of the reaction. The extra methanol in the liquid product was recovered. After segregation of glycerol layer and recovery of methanol, the synthesized biodiesel was washed four times with distilled water to eliminate traces of the catalyst. The phase rich in biodiesel was washed again with distilled water to eliminate any remaining oil traces and then heated to 82 °C to extract any moisture.

2.4. Analysis and Characterization of Biodiesel

The samples of biodiesel were analyzed on GC (Agilent Technologies, 5975C) with HP-5MS capillary column possessing a flame ionization detector (FID) by employing the GC method defined for in-house laboratory use. The FAME identification [25] in the samples was done by comparison with the standard retention times. Biodiesel yield was determined using Equation (4):

\[
Yield (\%) = \frac{\text{Biodiesel (grams)} \times \text{FAME (\%)}}{\text{Raw Oil (grams)}} \times 100
\]

(4)
Figure 1. Schematic illustration of biodiesel production employing sequential ultrasound and microwave. 1: Oil tank, 2: Heater, 3: Water tank, 4: Stirrer, 5: By-pass valve, 6: Pump, 7: Ultrasound, 8: Microwave

Figure 2. General view of the pilot-scale unit.

2.5. Energy Analysis

Energy consumed during biodiesel production is analyzed during the following steps: heating of waste cooking oil to extract moisture, conversion of raw oil to biodiesel in the reactor, extraction of extra methanol utilizing rotary evaporator, and heating of washed biodiesel to eliminate any trapped moisture.

Specific energy consumption (SEC), efficiency factor ($\eta$), and energy use index (EUI) [26]–[28] are the various performance parameters used in the energy analysis of the experiments. Energy can be categorized into supplied energy ($Q_{MW+US}$) and dissipated energy ($Q_{th}$).

$$Q_{MW+US} = P_{MW+US}t \quad (5)$$
$$Q_{th} = mC_p\Delta T \quad (6)$$

where $Q_{MW+US}$ is the energy input to the reactor, $P_{MW+US}$ is available reactor power (W), $t$ is the time duration for which power is supplied (sec), $Q_{th}$ is the energy needed to attain a particular temperature,
\( m \) is the reactant mass (kg), \( C_p \) is the heat capacity of the reactants (J/kg°C) and \( \Delta T \) is the difference between mixture’s initial and final temperature (°C).

Efficiency factor \( \eta \) as shown in Equation (7) can be determined as the proportion of dissipated energy to the supplied energy:

\[
\eta = \frac{Q_{\text{dissipated}}}{Q_{\text{supplied}}} \tag{7}
\]

Specific Energy Consumption (SEC) for the synthesis of biodiesel is determined by Equation (8):

\[
SEC = \frac{\text{Total Energy Consumption (Wh)}}{\text{Biodiesel produced (kg)}} \tag{8}
\]

The energy consumption of laboratory equipment is the focus of energy analysis in this study. However, a comprehensive study of energy flow needs to be conducted in order to optimize and control the energy employed in the process to improve the efficiency of biodiesel production.

3. Results and Discussions

3.1. Waste Cooking Oil Characterization

The fatty acid structure of waste cooking oil (WCO) was examined utilizing gas chromatography (Table 1). Palmitic acid, oleic acid, and linoleic acid are the main components of WCO according to this analysis. The acid value of WCO has been identified to be 2.25 mg KOH/g that corresponds to a free fatty acid concentration of 1.13%, considered suitable for single-step conversion (direct transesterification without esterification) of WCO to biodiesel. The measured moisture content was less than 0.05%.

| Fatty Acids            | Composition (%) |
|------------------------|-----------------|
| Palmitoleic acid (C16:1)| 0.96            |
| Oleic acid (C18:1)     | 24.68           |
| Linoleic acid (C18:2)  | 40.89           |
| Palmitic acid (C16:0)  | 28.66           |
| Stearic acid (C18:0)   | 2.56            |
| Linolenic acid (C18:3) | 1.27            |
| Myristic acid (C14:0)  | 0.98            |

3.2. Assessment of Process Parameters

3.2.1 Impact of Methanol: WCO Molar Ratio

The method of transesterification involves reacting the triglycerides (fat/oil) with alcohol utilizing a catalyst for the formation of alkyl esters and crude glycerol. Stoichiometrically, 1 mole of triglyceride and 3 moles of alcohol are required for a complete transesterification reaction. In practice, this ratio must be greater than the stoichiometric ratio to attain a maximum ester yield. As this process is reversible, surplus alcohol is employed to transfer the balance to the synthesis of esters (product) [8]. The impact of various M:O molar ratios i.e., 5:1, 6:1, and 7:1, on the transesterification reaction at a pre-set temperature of 62 °C and catalyst content of 0.9 wt% was evaluated. As shown in Figure 3(a), the yield of biodiesel increases with increasing M:O molar ratio and time. A molar ratio of 7:1 was determined to achieve a maximum yield of 96.9% in 5 minutes. The biodiesel yield improved marginally as the reaction time increased further.

3.2.2 Impact of Catalyst

The catalysts utilized in the production of biodiesel are usually acids or bases (homogeneous or heterogeneous) due to which their properties are associated with different advantages and
disadvantages [29], [30]. The reaction rates induced by base catalysts are higher than those by acid-catalyzed reactions which makes them the most preferred catalyst in biodiesel production. Sodium hydroxide (NaOH) and potassium hydroxide (KOH) flakes are among the extensively utilized base catalysts in the biodiesel industry. Here, we have selected KOH which is the most frequently used homogeneous base catalyst. The impact of the catalyst content (wt%) on the yield of biodiesel can be seen in Figure 3(b). For these experiments, the temperature was set at 62 °C and the methanol: oil molar ratio at 7:1. A catalyst content of 0.9 wt% was determined to achieve a biodiesel yield of 97.9% in 10 minutes. A lower amount of catalyst at less reaction time was not sufficient to accelerate the reaction forward, possibly

![Figure 3](image-url)

**Figure 3.** Impact of process parameters on yield (%): (a) M:O molar ratio at 0.9 wt% of KOH catalyst and 62 °C temperature; (b) catalyst concentration (wt%) at 7:1 M:O molar ratio and 62 °C temperature; and (c) reaction temperature at 7:1 M:O molar ratio and 0.9 wt% of KOH catalyst, on ultrasound and microwave irradiated sequential transesterification.
due to a reduction in the mass transfer induced by methanol resulting in lower yields. Increasing the catalyst content from 0.7 to 0.9 wt%, increased the biodiesel yield, while increasing catalyst concentration to 1.1 wt% reduced the biodiesel yield. Two explanations can be given to explain why a lower conversion rate was reached with the catalyst amount at 0.7 and 1.1 wt%. Some amount of the catalyst was neutralized with traces of FFA present in WCO during the process of transesterification. Thus, increasing the catalyst content will increase the biodiesel yield. This may justify why the biodiesel yield was only 82.3% in 5 minutes of reaction time when 0.7 wt% of the catalyst was used. Increases in the catalyst amount from 0.7 to 0.9 wt% improved the biodiesel yield. Second, a saponification reaction could be triggered by too much of the catalyst concentration. This could explain why 0.9 wt% of the catalyst was more effective than 1.1 wt%. In short, the catalyst must be used in an optimal amount.

3.2.3 Impact of Reaction Temperature
Temperature is a vital factor in biodiesel production since it affects the rate and yield of transesterification. [31]. At higher reaction temperature, a reduction in reaction time and an upsurge in reaction rate are observed due to a decrease in oil viscosity. It also minimizes the limitation of mass transfer leading to increased yield of methyl ester and FFA conversion. However, higher reaction temperatures (above the optimum level) promote triglycerides saponification and methanol vaporization which results in decreased biodiesel yield. To inhibit the vaporization of alcohol, transesterification must occur at a temperature lower than the boiling point of alcohol. The optimum temperature limit for the reaction can fluctuate between 50°C and 60°C, depending on the type of oil used. Figure 3(c) shows the relation between reaction temperature and biodiesel yield. Constant process parameters were methanol: oil molar ratio of 7:1 and catalyst content of 0.9 wt%. The reaction temperatures used were: 52, 57, and 62 °C. It was observed that the biodiesel yield increased with the temperature rise. An optimal temperature of 62°C was determined to achieve a biodiesel yield of 96.9% in 5 minutes. The biodiesel yield improved marginally as the reaction time increased further.

3.3. Biodiesel Fuel Properties
Biodiesel fuel properties like calorific value, density, flash point, oxidation stability, and kinematic viscosity are calculated in compliance with ASTM standards. Table 2 shows the evaluated biodiesel properties synthesized using waste cooking oil, compared to ASTM standards.

| Properties                  | ASTM Standard Values | ASTM Standard | This Study |
|-----------------------------|----------------------|---------------|------------|
| Density (kg/m³)             | 860 - 900            | ASTM D4052    | 876        |
| Flash point (°C)            | 130 (min)            | ASTM D93      | 179        |
| Kinematic viscosity (mm²/sec)| 1.9 - 6.0            | ASTM D445     | 3.9        |
| Oxidation stability (h)     | 3 hrs (min)          | EN 14112      | 7.6        |
| Calorific value (MJ/kg)     | 35 - 45              | ASTM D240     | 37.6       |

3.4. Energy Analysis
The energy consumption during the biodiesel production process is shown in Table 3. Equations (6) and (7) were used to calculate energy dissipation in liquid mixtures and efficiency factor respectively. A system energy meter installed in the control panel of the reactor was used to measure the energy delivered to the set-up.

The energy requirement is driven by variables such as the sample volume, ultrasound/ microwave radiation intensity, nature of the solvent, and the reaction volume depth. The energy requirements for the various heating and mixing methods are compared in Table 4. The observations demonstrate that the energy required for processing and separation by the sequential approach has been reduced multi-
fold compared to individual and simultaneous operation. The outcome of the sequential ultrasound and microwave approach has a substantial impact on the overall energy savings and production of biodiesel with greater economic prospects.

### Table 3. Energy analysis of sequential reactor for 4500 mL batch size.

| Run Order | M:O, Catalyst, Temp, Time | Measured Parameters | Performance Parameters |
|-----------|----------------------------|---------------------|------------------------|
|           |                            | Heating Oil (Wh)    | Sequential Reactor (Wh)| Rotary Evaporator (Wh) | Q_{MW+US} (Wh) | Q_{th} (Wh) | η (%) | SEC (Wh/kg) |
| 1         | 4.5:1, 0.9, 57, 5          | 625                 | 218                    | 2000                   | 843           | 204        | 24.2  | 876         |
| 2         | 4.5:1, 0.9, 57, 15         | 625                 | 538                    | 2000                   | 1163          | 204        | 17.5  | 954         |
| 3         | 7:1, 0.9, 52, 15           | 521                 | 527                    | 2000                   | 1048          | 199        | 19    | 799         |
| 4         | 7:1, 0.9, 62, 5            | 799                 | 263                    | 2000                   | 1062          | 249        | 23.4  | 766         |
| 5         | 7:1, 1.2, 57, 5            | 619                 | 257                    | 2000                   | 876           | 199        | 22.7  | 733         |
| 6         | 7:1, 1.2, 57, 15           | 619                 | 554                    | 2000                   | 1173          | 199        | 17    | 801         |

### Table 4. Comparison of energy consumption by different methods of biodiesel production.

| Technique                          | Experimental settings                                                                 | Energy consumed (kJ/g) | Reference |
|------------------------------------|----------------------------------------------------------------------------------------|------------------------|-----------|
| Heating                            | Camelina sativa oil (9 g of oil, 15 min, 500 W)                                         | 50                     | [32]      |
| Ultrasonic-assisted reactor        | Vegetable oil (52 g of oil, 30.7 min, 130 W)                                           | 4.6                    | [33]      |
| Microwave-assisted reactor         | Nagchampa oil (160 W, 30 min, 20 g oil)                                                | 6.7                    | [23]      |
| Simultaneous microwave/ultrasound reactor | Waste vegetable oil (18 g of oil, 2 min, 100 MW/100 US) | 1.3                  | [34]      |
| Sequential ultrasound and microwave| Waste cooking oil (4122 g of oil, 15 min, 300 US/1000 MW)                             | 2.66                   | This study |

### 4. Conclusion
The present study analyzed the transesterification of waste cooking oil (WCO) using KOH as a catalyst employing sequential ultrasound and microwave approach for biodiesel production. A substantial degree of process intensification is provided by the synergistic relationship between the mechanical effects of micro-emulsification and the acoustic propagation of ultrasound combined with the quick heating and dipolar rotation of molecules caused by microwaves. Results show that under the optimum process parameters of 7:1 methanol: oil molar ratio, 0.9 wt% catalyst content, and 62°C reaction temperature, the biodiesel yield achieved was 96.9% in 5 minutes of reaction time for a sample size of 4500 mL with energy consumption of 2.66 kJ/g. This study offers an insight into the progress of biodiesel production technologies to not only take the advantage of synergistic effect and potential energy and cost savings, but also in terms of time, robustness, safety, and environmental effects.
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