

Article

# Biodiesel Production from Waste Cooking Oil Using Different Types of Catalysts

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**Abstract:** The global energy demand is increasing day by day. Fossil fuels such as crude oil, coal and gas are the main source of energy worldwide. However, fossil fuels, which cause acid rain, the greenhouse effect and other such environmental problems, will eventually be depleted, and renewable energy seems to be the most reasonable solution in this regard. Renewable biofuels have significant potential and can meet the world's current energy demand. One of the important biofuels is biodiesel, and in the future it can replace petroleum. Waste cooking oil was used as a raw material in biodiesel production in order to reduce the production cost of the offered additive. In this study, the aim was to optimize the process parameters for biodiesel production within the acceptable limit values in the literature. Therefore, the molar ratio of methanol to waste cooking oil (9:1–15:1), catalyst concentration (1–5% by weight) and reaction time (60–120 min) were studied for two catalyst types, potassium hydroxide and ion exchange resin Amberlyst 15. The biodiesel obtained with maximum efficiency for each catalyst was also compared with the international biodiesel standards.

**Keywords:** biodiesel; waste cooking oil; renewable energy; catalysts; transesterification

## 1. Introduction

Due to the increase in world population and rapid technological developments, the energy need of humanity is increasing. The world's main energy source is fossil fuels such as oil, natural gas and coal, and energy production is generally provided by burning these fuels. It is a well-known fact that fossil energy reserves are gradually decreasing and will be depleted in the near future. In addition, fossil fuels are regional energy sources, they lead to increased environmental pollution and create greenhouse gas emissions as a result of the combustion reaction; thus, energy research is focused on renewable energy sources [1–3].

Biomass, one of the renewable energy sources, constitutes about 13% of the world's energy consumption and is the most diverse and easily utilized renewable energy source. Biomass resources obtained from easy-to-grow plants and wastes are an excellent option for meeting modern energy needs such as electricity and fuel production. In this context, biodiesel, which is produced from oils, is one of the biomass fuels that has been reported as a potential alternative to petroleum-derived diesel for diesel engines. Biodiesel is a renewable, non-toxic and biodegradable liquid fuel, and it has recently exhibited promise for use separately or in combination with conventional diesel fuel in existing diesel engines without the need for major modification. It also reduces exhaust emissions such as carbon monoxide, unburned hydrocarbons, particulate matter and sulfur dioxide. Thus, different mixing ratios of biodiesel and diesel fuels are increasingly important due to environmental reasons on the one hand and compliance with fuel standards and agricultural needs on the other [4–6].

Biodiesel is a transesterification product that is formed as a result of the reaction of oil (vegetable or animal oils) with a short-chain alcohol (methanol, ethanol or other suitable alcohols) in the presence of a catalyst. Figure 1 shows the transesterification reaction; the free fatty acids in the oil react with the alcohol and fatty acid ester mixture which is biodiesel, and biodiesel and glycerine are produced [7,8].



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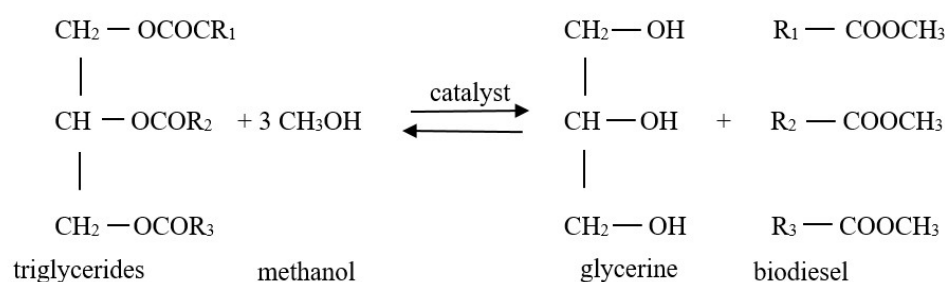
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**Figure 1.** Transesterification reaction.

The catalyst plays an important role in the transesterification reaction. It increases the speed of the slow transesterification reaction as well as the biodiesel product yield. Catalysts used in biodiesel production are divided into three main basic types: homogeneous, heterogeneous and biocatalysts. Homogeneous catalysts are substances that can exist in the same phase as the reaction medium. For example, sulfuric acid and sodium hydroxide belong to the group of homogeneous catalysts. Homogeneous basic catalysts are preferred in industrial application because the reaction conditions are preferable and the reaction times are shorter. In the homogeneous basic catalyst processes, oil and alcohol should not contain water, as this causes soap formation. Heterogeneous catalysts are catalysts that are not in phase with the reaction medium. When heterogeneous catalysts are used to produce biodiesel, high temperature, high pressure, a large amount of catalyst and high mole ratio of alcohol to oil are needed. Biological substances such as enzymes, algae, fungi and microbial bacteria are included in the class of biocatalysts, and they can be homogeneous or heterogeneous. There are some advantages and disadvantages of all these catalyst types. After biodiesel production, the catalyst must be removed from the reaction mixture in order for the biodiesel to be ready for use. Homogeneous catalysts remain in the reaction medium and require extra processes, such as washing and drying, in order to be removed, whereas heterogeneous catalysts can be removed from the reaction medium by simple physical methods such as filtration. The separated catalyst can be reused. On the other hand, acidic heterogeneous catalysts are preferred over basic heterogeneous catalysts because unwanted soap formation is avoided during transesterification [9–17].

In biodiesel production, vegetable oil seeds, animal oils, waste, used or non-edible oils and all types of biological originated oils can be used as feedstock. According to the World Health Organization, waste cooking oil constitutes 25% of wastewater pollution, and one liter of waste oil can contaminate one million liters of water. When waste cooking oil mixes with water, it covers the water surface, prevents oxygen transfer from air to the water, accelerates the depletion of oxygen in water and damages the entire aquatic ecosystem such as birds and fish. This causes long-term water and soil pollution, deterioration of the marine ecosystem and sewage blockage [18].

The use of waste cooking oil as a raw material for biodiesel production has the potential to solve the problem of waste oil disposal. In order to reduce biodiesel production costs, cheap raw materials such as waste cooking oil can be used. If the total free fatty acid (FFA) amount of waste edible oils is more than 2% by weight when homogeneous alkaline catalysts are applied, soap formation occurs. A two-stage esterification process has been developed for the conversion of waste oils with high FFA content. Firstly, FFA is converted into fatty acid methyl esters and water using an acid catalyst such as sulfuric acid. Then, the transesterification reaction is carried out using alkali-based catalysts. Acid catalysts can catalyze both esterification and transesterification reactions [3,16,19,20].

The reactions with homogeneous acid catalysts are much slower than the reactions with homogeneous alkaline catalysts. Therefore, acid-catalyzed transesterification is not preferred. The corrosiveness of the acidic homogeneous catalysts, the saponification problem of basic homogeneous catalysts, separation after biodiesel production in the presence of both types of homogeneous catalysts and the difficulties and cost increasing factors in purification processes are important factors and the main disadvantages of these catalysts.

In order to eliminate all these disadvantages of homogeneous catalysts, ion-exchange resins, heteropoly acids, carbon-based catalysts, zeolites or catalyst supports are suggested in the literature. Among them, ion-exchange resins are frequently preferred because they can catalyze the reaction in milder conditions due to their high acid concentrations, are highly selective, do not harm the environment and are easily available commercially. In addition, they are heterogeneous catalysts that can be easily separated from the product as well as regenerated [21–26].

There are many studies about biodiesel production from waste cooking oil by using homogeneous- and heterogeneous-type catalysts. Suzihaque et al. [20] and Thirumarimurugan et al. [27] studied waste cooking oil by using NaOH catalyst by applying molar 6:1 methanol/oil. Banerjee et al. [17] used the same catalyst by applying 15:1 methanol/oil molar ratio. They achieved a biodiesel yield between 80 and 94%. Komintarachat and Chuepeng [28] and Mohadesi et al. [29] obtained nearly 98% biodiesel yield by using KOH catalyst with a methanol/oil 6.18 and 12. Boz et al. [22] used waste cooking oil as a feedstock and A15 and modified A15 as a catalyst for biodiesel production. They achieved a biodiesel yield of 78%. Another study by Pradhan et al. [30] showed that 97% biodiesel yield was achieved by using mustard oil and A15 catalyst.

There are also many articles on biodiesel production in the literature. Since the production conditions for biodiesel can vary greatly, it is anticipated that these studies will take longer. The variety range in the applied raw material for the biodiesel production and the different types of catalysts in the transesterification reaction can be shown as the reason for this. Conditions such as methanol oil ratio, temperature, amount of catalyst and reaction time determine the differences between studies. Moreover, it has to be ensured that produced biodiesel complies with the determined international biodiesel standards. Accordingly, in this study, two different types of catalysts were used for the same oil type by applying the same operating conditions. This means a new point of view for providing a comparison of the two different types of catalysts.

In this study, the aim was to optimize the process parameters for production of biodiesel within acceptable limit values in the literature, which is given in Section 3.2. For this reason, molar ratio of methanol to waste cooking oil (9:1, 12:1, 15:1), catalyst concentration (1 wt.%, 2.5 wt.%, 5 wt.%), reaction time (60 and 120 min) and catalyst types potassium hydroxide and ion exchange resin Amberlyst 15 were studied. The biodiesel obtained with the maximum yield for each catalyst was further analyzed and compared with international biodiesel standards.

## 2. Materials and Methods

### 2.1. Materials and Chemicals

Waste cooking oil was taken from local restaurants. The main chemicals methanol ( $\text{CH}_3\text{OH}$ ) and potassium hydroxide (KOH) were acquired from Merck Co., Ltd. (Darmstadt, Germany). The cation exchange resin in the  $\text{H}^+$  form, Amberlyst 15 (A15), was purchased from Rohm and Haas Co., Ltd. (Philadelphia, PA, USA).

### 2.2. Analyses

The waste cooking oil sample was analyzed using the standard titration method to determine the acid value and the percentage of FFA in the oil. The standard titration method was applied by using KOH. If the percentage of FFA is lower than 2%, the transesterification reaction may occur directly without the esterification reaction. The acid value is defined as the amount of KOH required for neutralizing the free fatty acids in 1 g of fat. It is determined by titrating the oil with a pinch of phenolphthalein against the KOH. The acid value of samples was determined in accordance with the formula given in [31]. The obtained acid value and the free fatty acid percentage values of the waste cooking oil were 1.98 and 0.99 mg KOH/g, respectively. The chemical composition of the feedstock was determined by using gas chromatography (HP 5860 series 2 type) and is shown in Table 1.

**Table 1.** Chemical composition of waste cooking oil.

| Fatty Acid Composition |  |        |
|------------------------|--|--------|
| Name                   | Formula  | (wt.%) |
| Oleic acid             | C <sub>18</sub> H <sub>34</sub> O <sub>2</sub> | 40.78  |
| Linoleic acid          | C <sub>18</sub> H <sub>32</sub> O <sub>2</sub> | 34.63  |
| Myristic acid          | C <sub>14</sub> H <sub>28</sub> O <sub>2</sub> | 12.5   |
| Palmitic acid          | C <sub>16</sub> H <sub>32</sub> O <sub>2</sub> | 3.98   |
| Stearic acid           | C <sub>18</sub> H <sub>38</sub> O <sub>2</sub> | 3.33   |
| Eicosenoic acid        | C <sub>24</sub> H <sub>48</sub> O <sub>2</sub> | 1.98   |
| 11-Eicosenoic acid     | C <sub>20</sub> H <sub>38</sub> O <sub>2</sub> | 1.32   |
| Behenic acid           | C <sub>22</sub> H <sub>44</sub> O <sub>2</sub> | 0.73   |
| Tetracosanoic acid     | C <sub>24</sub> H <sub>48</sub> O <sub>2</sub> | 0.45   |
| Others                 |  | 0.3    |

### 2.3. Transesterification Experiment Runs

In order to remove residues, the oil was filtered using coffee filter paper. The water content of the oil may negatively affect the quality of the biodiesel. Thus, the oil sample was heated to 110 °C for 1 h to eliminate the water content. The transesterification reaction of oil with methanol was carried out at 65 °C for KOH catalyst and 70 °C for A15 in a three-necked, refluxed 250 mL batch reactor. Magnetic stirring was applied, and 600 rpm was used as the stirring speed. Experiments were carried out with 1%, 2.5% and 5% catalyst amounts by weight of the oil; reaction times of 60 min and 120 min and methanol/oil molar ratios of 9:1, 12:1 and 15:1. The experiments' parameters are shown in Table 2. The liquid mixture obtained after the reaction was taken into a separatory funnel for phase separation. After the glycerine was separated, firstly, the remaining methanol was removed at 65–70 °C, and then produced ester was washed with distilled water. After washing, drying was carried out at 110 °C for 1 h to remove excess alcohol and excess water in the ester.

**Table 2.** Operating conditions for the experiments.

| Catalyst Type | Methanol/Oil Ratio (Molar) | Catalyst Amount (wt.%) | Time (min) |
|---------------|----------------------------|------------------------|------------|
| KOH and A15   | 9                          | 1                      | 60         |
|               |                            |                        | 120        |
|               |                            | 2.5                    | 60         |
|               |                            |                        | 120        |
|               |                            | 5                      | 60         |
|               |                            |                        | 120        |
|               | 12                         | 1                      | 60         |
|               |                            |                        | 120        |
|               |                            | 2.5                    | 60         |
|               |                            |                        | 120        |
|               |                            | 5                      | 60         |
|               |                            |                        | 120        |
|               | 15                         | 1                      | 60         |
|               |                            |                        | 120        |
|               |                            | 2.5                    | 60         |
|               |                            |                        | 120        |
|               |                            | 5                      | 60         |
|               |                            |                        | 120        |

### 3. Results and Discussion

It has been previously stated that there are many studies on biodiesel in the literature. Since biodiesel can be obtained in many different ways, the intensity of studies on this subject proves this. Especially in the production of biodiesel by transesterification, studies can vary, as the working conditions can differ greatly. Catalyst type, catalyst amount, methanol to oil ratio, temperature and reaction time are among these working conditions. In biodiesel studies, the yield of biodiesel can be examined through operating conditions. However, whether the obtained biodiesel meets the international standards criteria or not is also important. In some studies, the biodiesel yield and optimum working conditions were examined, and the analysis of the physical properties of the obtained biodiesel was not included. Recently, besides the efficiency of biodiesel, providing and developing the necessary conditions has been taken into account, especially for its use in the engines. In this study, this situation has been into taken account.

#### 3.1. Biodiesel Yield

The experiments were duplicated for the calculation of biodiesel yield. Biodiesel yield is percent mass ratio of the biodiesel produced to the total mass of the waste cooking oil consumed [32,33]. It was calculated using formula as shown below:

$$\text{Biodiesel yield}(\%) = \frac{\text{weight of biodiesel}}{\text{weight of oil}} * 100$$

##### 3.1.1. Effect of Methanol/Oil Ratio

During the experimental study, to investigate the effect of each parameter, the specified parameter was changed as the others were kept constant.

The molar ratio plays an important role in achieving the maximum conversion of oil to biodiesel via the transesterification reaction. Stoichiometrically, the molar ratio of methanol to oil is 3:1, but to drive the forward reaction and achieve maximum yield, high volumes of methanol are required [34]. In this study, it was determined that the biodiesel yield increased from 70% to 95% when the mole ratio of methanol to waste cooking oil was increased from 9 to 15 for KOH catalyst. This value was a little lower for A15 catalyst. In some studies, it was determined that when the methanol/waste cooking oil molar ratio was increased above 25, a decrease in biodiesel yield may have also occurred. This may be due to the reconversion of biodiesel to triglyceride [33]. In addition, when increasing the total volume of methanol, the decrease in the catalyst concentration in the reaction medium could be effective in decreasing the biodiesel yield. In this study, no such situation was encountered (Figures 2–5).

##### 3.1.2. Effect of Catalyst Amount

It is known that the effect of the catalyst amount is important in biodiesel production reactions. It has been observed that in the absence of a catalyst, the reaction almost never takes place. However, if the amount of catalyst is excessive, the reactants cannot interact sufficiently with the catalyst because the mixing in the reaction mixture is inadequate and the reaction efficiency decreases. When a small amount of catalyst is used, the reaction efficiency cannot reach the desired values. Therefore, it is necessary to investigate the appropriate amount of catalyst. Özbay et al. [35] studied the ion exchange resins in the esterification of waste cooking oil to decrease free fatty acid level. They used A15, A16 and A35 catalysts with 1–2 wt.% amounts and observed that as catalyst amount was increased, FFA conversion increased. In addition, Gan et al. [36] investigated the catalyst concentration in heterogeneous esterification of waste cooking oil by using A15. They found that the optimum catalyst concentration was 4 wt.%, and the maximum FFA conversion was 60.2%.

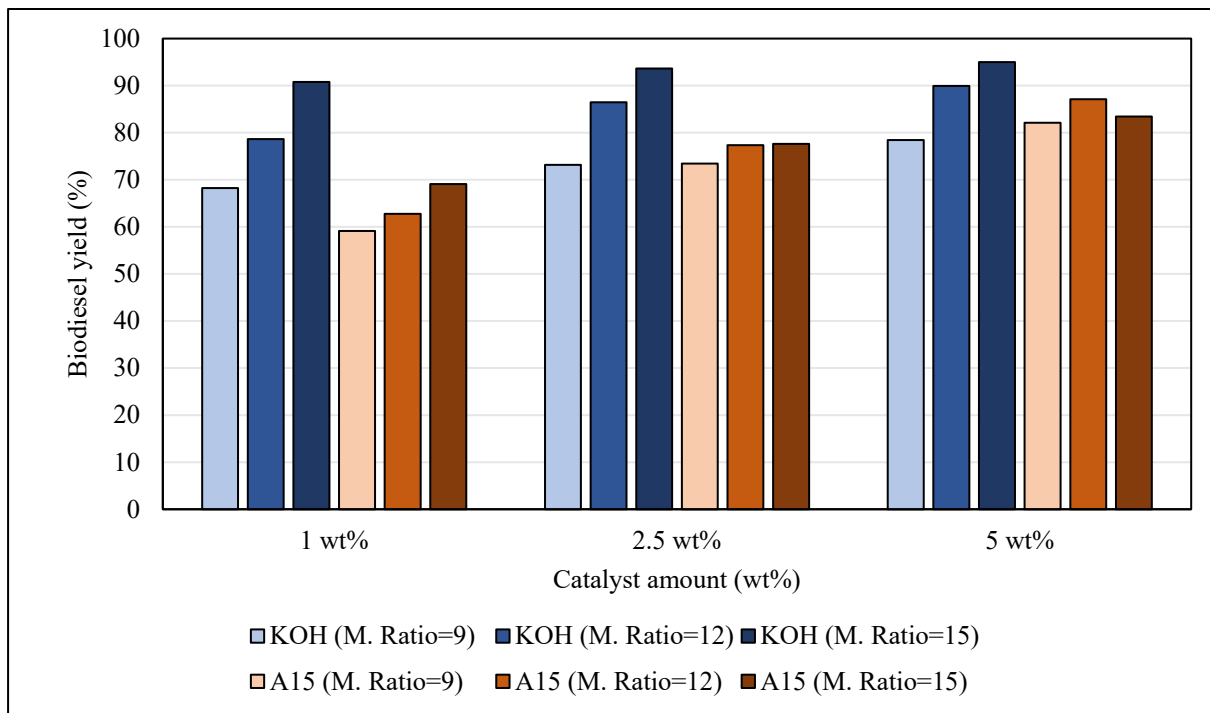


Figure 2. Biodiesel yield with respect to KOH and A15 catalysts for reaction time of 60 min.

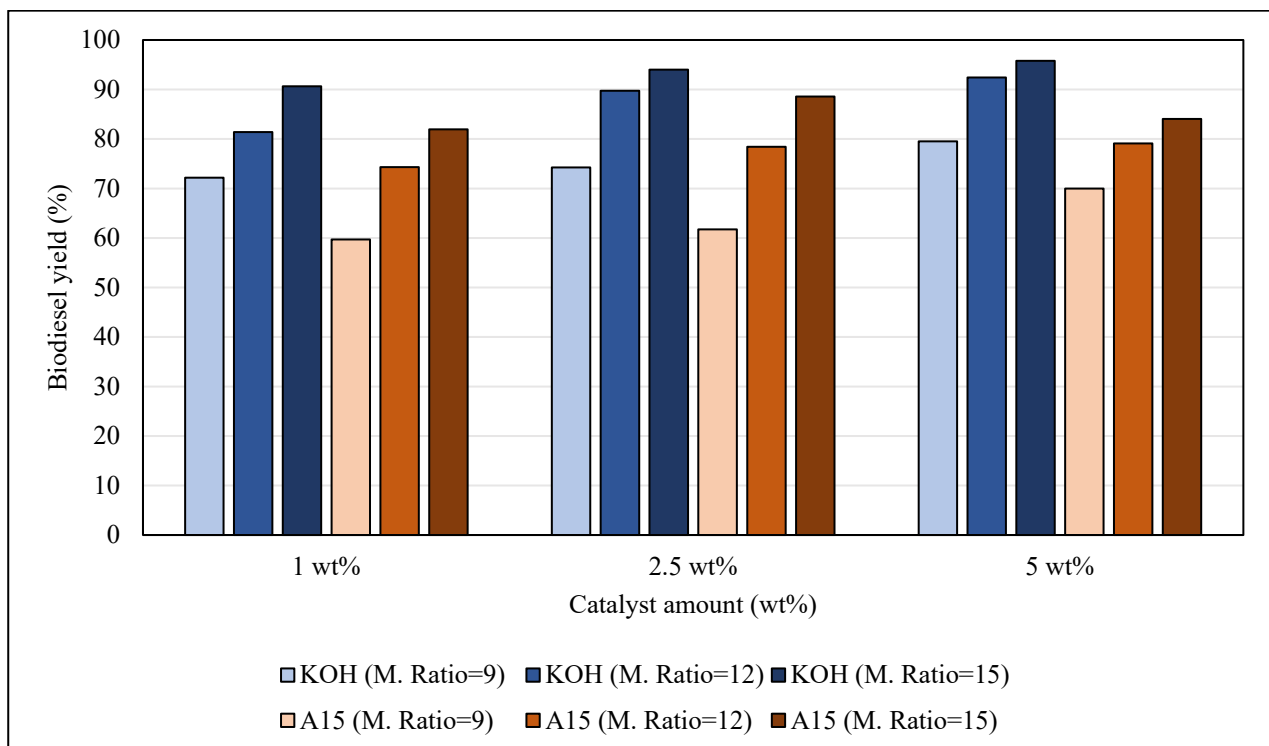


Figure 3. Biodiesel yield with respect to KOH and A15 catalysts for reaction time of 120 min.

In this study, the catalyst was studied in the range of 1–5% by weight, keeping the methanol/oil molar ratio and time constant. The biodiesel yield increased when the catalyst weight was increased from 1% to 5% for both catalyst types. Biodiesel yield value was nearly 95% for KOH catalyst and 87% for A15 catalyst. This can be attributed to the fact that

the 5 wt.% catalyst offers the highest number of active sites for reactant interaction, which means increasing the mass transfer between the components and catalyst (Figures 2–5).

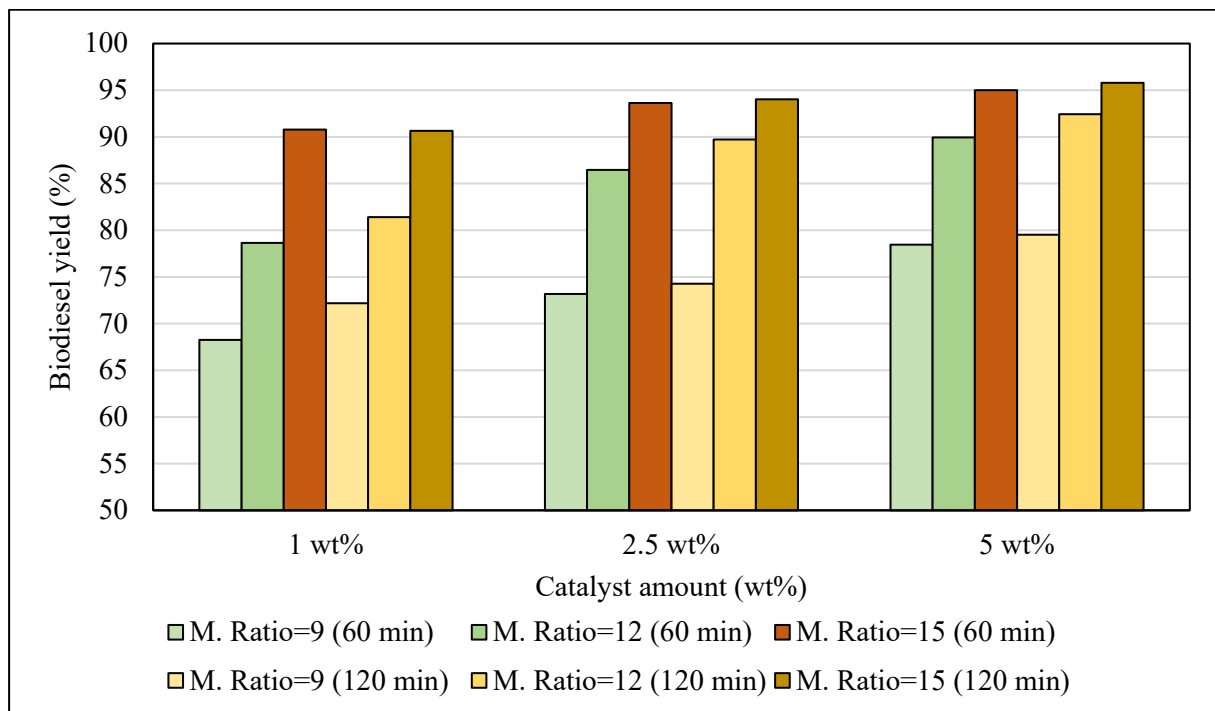


Figure 4. Biodiesel yield for KOH catalyst amount and time.

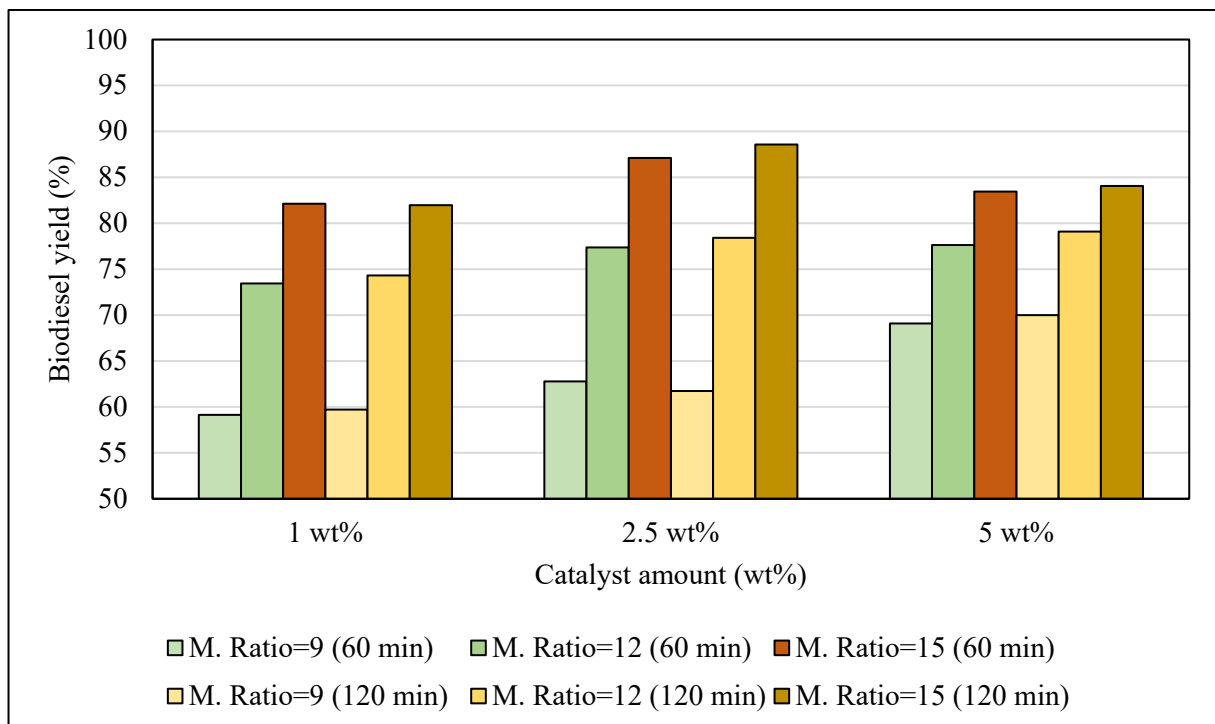


Figure 5. Biodiesel yield for A15 catalyst amount and time.

### 3.1.3. Reaction Time

Since energy consumption in the biodiesel production process is directly related to time, it is important to determine an adequate reaction time. In the literature, biodiesel

reaction times between 30 min to 9 h have been studied. Thirumarimurugan et al. [27] studied the transesterification reaction of waste sunflower oil with NaOH catalyst at 60 °C for 1–3 h. They found 80% biodiesel yield. Furthermore, Farooq et al. [37] found 89% biodiesel yield for the same reaction by using heterogeneous catalysts derived from waste chicken bones by taking the reaction time as 4 h. In this study, the 60 min and 120 min reaction times were evaluated. It was determined that there was no significant effect of contact time on biodiesel yield with respect to other parameters (Figures 4 and 5).

### 3.2. Biodiesel Characteristics

For the biodiesel characterization experiments, the experiments which gave the maximum yields for both catalyst types were repeated. The experimental conditions were as follows: for KOH catalyst, molar ratio was 15, 5 wt.% catalyst and 120 min reaction time; for A15 catalyst, the molar ratio was 15, 2 wt.% catalyst and 120 min reaction time. Afterwards, the obtained biodiesels were subjected to analysis. Chemical composition of the waste cooking oil biodiesel obtained by using both catalysts was analyzed at Bursa Tubitak BUTAL and at Ege University and is given in Table 3. Measurements on the properties were performed in accordance with TS EN ISO12966 standards.

**Table 3.** Chemical composition of waste cooking oil biodiesel.

| Fatty Acid Composition |  | (wt.%) |       |
|------------------------|--|--------|-------|
| Name                   | Formula  | KOH    | A15   |
| Oleic acid             | C <sub>18</sub> H <sub>34</sub> O <sub>2</sub> | 40.78  | 39.72 |
| Linoleic acid          | C <sub>18</sub> H <sub>32</sub> O <sub>2</sub> | 34.63  | 36.69 |
| Myristic acid          | C <sub>14</sub> H <sub>28</sub> O <sub>2</sub> | 12.5   | 10.45 |
| Palmitic acid          | C <sub>16</sub> H <sub>32</sub> O <sub>2</sub> | 3.98   | 4.02  |
| Stearic acid           | C <sub>18</sub> H <sub>38</sub> O <sub>2</sub> | 3.33   | 4.25  |
| Eicosenoic acid        | C <sub>24</sub> H <sub>48</sub> O <sub>2</sub> | 1.98   | 1.97  |
| 11- Eicosenoic acid    | C <sub>20</sub> H <sub>38</sub> O <sub>2</sub> | 1.32   | 1.45  |
| Behenic acid           | C <sub>22</sub> H <sub>44</sub> O <sub>2</sub> | 0.73   | 0.61  |
| Tetracosanoic acid     | C <sub>24</sub> H <sub>48</sub> O <sub>2</sub> | 0.45   | 0.65  |
| Others                 |  | 0.3    | 0.19  |

In order to increase its commercial value, there exist some standards for the physical properties of the synthesized biodiesel [38–42]. These physical properties affect performance in engines. The importance of these properties is explained below in detail. The produced biodiesel was analyzed to investigate these properties. The analyses were carried out at Uludag University and Bursa Tubitak BUTAL in accordance with ASTM D 6751 and EU methods.

Viscosity can be defined as the resistance of fluids to flow against gravity. For diesel fuels, viscosity has an important role in lubricating and injecting the fuel into the combustion chamber. The viscosity increases as the chain length of hydrocarbons increases and decreases as the number of double bonds increases. The viscosity value of the obtained biodiesel varies between 3.5 and 5 mm<sup>2</sup>/s at 40 °C for both catalyst types. Density is another important parameter in biodiesel fuels. In the study, the densities of the biodiesel samples comply with the fuel standard. The density value should be between 0.86 g/cm<sup>3</sup> and 0.90 g/cm<sup>3</sup>. The saponification value is the amount in milligrams of KOH required to saponify 1 g of oil. A higher saponification value indicates the presence of a greater proportion of low molecular weight fatty acid chains. The saponification value was reported as 203 mg KOH/g for KOH catalyst and 190 mg KOH/g for A15 catalyst in this study. This value varies between 175 and 234 mg KOH/g in [20]. The importance of acid value is explained above, and this value was measured before and after the experiments. It is expressed in mg KOH/g. For diesel fuels, high acid values cause sedimentation and a corrosive effect, so these values should be within the specified standard limits. It can be clearly seen that WCO biodiesel has an acid value in the order of 0.2–0.23 mg KOH/g



for both catalysts, which is very low and acceptable. The iodine number is a measure of the total unsaturation of the oil and is based on the oil's properties and depends on the number of double bonds. Fuels with a high iodine number block the injector holes or cause damage to the combustion chamber. For KOH catalyst, the iodine number value is within the limits, whereas for the biodiesel produced by using A15 catalyst this value is higher than the standard value. The peroxide number is a measure of the amount of active oxygen found in oils. The presence of peroxide is based on the principle of proof and determination of iodine separated from potassium iodide in an acidic solution.

It was observed that most of the main physical properties of the obtained biodiesel were determined within set limits. The biodiesel synthesized by A15 catalyst gave some results, which are out of limits. These values are density, which exhibited a very small deviation, and iodine value. However, the results with KOH catalyst show that the biodiesel is of high quality and has the potential for use as a fuel. The standard limit values and the derived biodiesel values are given in Table 4.

**Table 4.** Physicochemical properties of standard limits and WCO biodiesel produced by using KOH and A15 catalysts.

| Property   | Limits [4,12,14,29,43,44] | WCO Biodiesel (KOH) | WCO Biodiesel (A15) |
|--|---------------------------|---------------------|---------------------|
| Density (kg/m <sup>3</sup> )                     | 860–900                   | 879                 | 901                 |
| Kinematic viscosity (40 °C) (mm <sup>2</sup> /s) | 3.5–5                     | 4.79                | 4.98                |
| Saponification value (mg KOH/g oil)              | ---                       | 203                 | 190                 |
| Acid value (mg KOH/g oil)                        | <0.5                      | 0.2                 | 0.23                |
| Iodine value (Wijs g/100 g oil)                  | <120                      | 91.73               | 132                 |
| Peroxide value (meq g/kg)                        | 2.1–4.1                   | 2.8                 | 2.9                 |

#### 4. Conclusions

Today, although the fuel properties of biodiesel render it unsuitable for use purely in new generation diesel engines, especially agricultural vehicles, heavy work machines and electricity generating generators have diesel engines that allow the use of biodiesel as a blended or pure fuel at a high rate. However, in order for the use of biodiesel to become attractive, it is very important to minimize the raw material cost, which is very high. The high production cost of biodiesel is seen as the largest obstacle to its commercialization. In particular, 60–80% of the total production cost is the raw material cost. For this reason, frying oils, waste cooking oils, waste animal oils, soap residues and yellow and brown greases are increasingly preferred as raw materials for biodiesel production. The main important aspects of produced biodiesel are its fuel properties, such as density and kinematic viscosity, which provide an indication of its usability as a diesel engine fuel.

The aim of this study was to find the optimum conditions in the production of biodiesel, which is in the class of alternative energy sources, and to determine the compliance of fuel properties with standards. Although there are several biodiesel production methods, the catalytic transesterification reaction is mostly used due to the ease of the reaction and the high yield of biodiesel. This work provides a point of the operating conditions used for biodiesel production, including homogeneous and heterogeneous catalysts. In this study, positive results were obtained with both types of catalysts.

In previous studies, biodiesel production was studied with the same catalysts. Although there are few studies in which ion exchange resins are used as catalysts in the production of waste cooking oil and biodiesel, the obtained biodiesel yield values for A15 catalyst are higher than the literature values. Regarding the detailed literature search, while approximately the same biodiesel yield results were obtained with the KOH catalyst, there exist no results obtained with the A15 catalyst similar to those of this study. As a result of this study, it was concluded that the efficiency of the work with A15 is slightly lower than that of the basic catalyst. However, heterogeneous catalysts are preferred to

homogeneous catalysts due to their ease of recovery and reuse. In addition, they are more environmentally friendly and can be operated in continuous mode. The properties of the derived biodiesel were found between the standard values. Considering the obtained suitable physical properties of the derived biodiesel, it is clear that this study will contribute to the literature.

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