# Analysis of the biodiesel production yield from waste frying oil

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# Analysis of the biodiesel production yield from waste frying oil

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**Abstract**. This research sought to produce biodiesel from waste frying oil (WFO) from chicken grills by using chemical transesterification to evaluate quality conditions and the yield of the biodiesel obtained. For this, acid esterification and basic transesterification were applied under the following conditions: reaction temperature $60°C$, catalyst concentration of 1% (m/m) KOH, oil:alcohol 1: 6 $molar\ ratio$, and two reaction times (55 $and$ $70min$) for the transesterification. The physicochemical properties of the raw material were analyzed (*i.e.*, density, humidity, kinematic viscosity, fatty acid profile, acidity index, peroxides, and saponification) where the WFO showed high contents of oleic acid (42.45%) and palmitic acid (33.52%), which are fundamental for biodiesel production. Chemical transesterification under the conditions of $60°C$, 1% KOH, and $70min$ obtained the best yield by presenting a high conversion percentage (96.15%) and an acid number of $1.33mmKOH/g$, according to ASTM D6751 and EN 14214 international standards.

## 1. Introduction

Currently, it has become necessary to implement biofuels as renewable energy sources to reduce emissions and concentrations of greenhouse gases, which increased in 2012 to $258.8Mton$ of $CO_2$ eq. at the national level, with the transportation sector being responsible for the emission of $28.2Mton$ of $CO_2$ eq. and carbon dioxide as the main pollutant gas in the country, representing 74% of the total emissions [1]. Additionally, reports of the International Energy Agency (IEA) expose that $32 - billion\ tons$ of $CO_2$ per year are released into the atmosphere throughout the world [2]. Hence, with the development of biofuels, new alternatives of energy supply may be provided, offering solutions to the global energy crisis caused by the reduction of fossil fuel reserves. Data provided by diverse organizations, such as IEA, estimate that when oil reserves in the United States decays in 2020 and the production from the countries in the Organization of Petroleum Exporting Countries (OPEC) falls back, large-scale investment will be needed to exploit $670 - billion$ of barrels of new resources until 2040 to compensate the decline in existing deposits [3]. After the oil reserves peak was reached in the 1960s and 1970s, the amount of reserves discovered has decreased [4], where 95% of the reserves discovered in the last three decades produce less than $200 - thousand$ barrels per day [5].

In 2013, the Colombian association of oils and edible fats (Asograsas) denounced the existence of an informal sector that purchases the used oil to be bottled and labeled as new oil, supplying around 30% of the oil sold in stores and supermarkets [6]. According to the data from Asograsas, in 2016

consumption of edible vegetable oil in the country was $621 - thousand\ tons$; after its use, the oil becomes a residue discarded into bodies of water or drains [7]. This action causes a negative environmental impact on the water sources, generating cost overruns in water treatment plants or oil films on surface of water bodies, which affect its oxygen exchange capacity, affecting ecosystems [7]. In addition, oil reused three times, or more can affect human health, due to its carcinogenic potential associated to the production of acrylamides [8].

Due of the aforementioned aspects, the Resolution 316 of 2018 [7] promulgated by the Ministry of the Environment and Sustainable Development, established dispositions on the management of used kitchen oils, introducing the obligations and responsibilities of producers, distributors, and marketers of edible vegetable oils, and generators and managers of used cooking oil. Keeping in mind these dispositions, several investigations have been conducted in Colombia in the field of biodiesel production by using waste frying oil as raw material. Some results of these investigations show conversions of $99.53\%$ and $97.69\%$ of WFO into biodiesel with basic catalysis [9]. Hence, this research studied the chemical transesterification process for biodiesel production using WFO from a grilled chicken restaurant to determine the operational variables that can provide better yield and quality results in the biodiesel obtained; thus, contributing to the diversification of the energy basket and venture into the production of biofuels to avail of this agro-industrial residue.

## 2. Materials and methods

### *2.1. Physicochemical characterization of the waste frying oil*

Initially, the WFO sample was collected from a grilled chicken restaurant, taking directly *in situ* $40 liters$ of oil generated under normal operating conditions. In the laboratory, the WFO was filtered to remove particles and food residues. To evaluate the type and amount of fatty acids present in the WFO sample and to establish its quality, some physicochemical characteristics were analyzed by applying different methods detailed in Table 1. These analyses were done in triplicate.

**Table 1.** Physicochemical characterization of the WFO.

| Parameter | Method/Technique |
|---|---|
| Density ($g/cm^3$) | Pycnometer method |
| Refraction index | Refractometric |
| Humidity content (%) | NTC 287 |
| Kinematic viscosity ($mm^2/s$) | Hoopler Method |
| Acidity index (mgKOH/g) | NTC 218 |
| Peroxide index ($meqO_2/g$) | NTC 236 |
| Saponification index (mgKOH/g) | NTC 335 |
| Fatty acid profile | NTC 4967 |

### *2.2. Biodiesel production process*

To convert fatty acids into biodiesel, $12\ liters$ of WFO were used. The process was carried out in two steps; the first one was an acid esterification of free fatty acids with methanol at $60°C$ and agitation at $200 rpm$ during $60 min$, using an oil:alcohol $1:6\ molar\ ratio$, in the presence of $1\%$ (w/w) sulfuric acid ($H_2SO_4$) as acid catalyst. In this step the triglyceride chains are broken to facilitate the conversion into biodiesel in the second step. The second step is the transesterification phase, which consisted in the reaction of the esterified mixture with methanol in oil:alcohol $1:6\ molar\ ratio$, using potassium hydroxide (KOH) as catalyst with $1\%$ (w/w) concentration at two reaction times of $55\ (Q_1)\ and\ 70\ (Q_2)\ minutes$. After completing the reaction time, the phases generated were separated (*i.e.*, biodiesel, glycerol) by static decantation for $12h$, where the upper phase was biodiesel and the second, at the bottom of the funnel, was glycerol. The biodiesel separated and obtained from the chemical transesterification process was neutralized by adding phosphoric acid ($H_3PO_4$). This neutralization was carried out at $60°C$ during $15 min$, through constant agitation of the sample. As

consequence of the neutralization, potassium phosphate salts are produced and are separated from the biodiesel.

The mixture generated in the transesterification reaction has a high amount of impurities, hence, the purification phase is essential to eliminate unwanted material and achieve quality standards (*e.g.*, ASTM D6751 [10] and EN 14214 [11]). The washing was done by spraying distilled water at room temperature, eliminating remains of impurities and catalysts in the biodiesel. The amount of distilled water used for each washing was a fifth part of the volume of biodiesel produced. Five washings were carried out, with the first four accomplished with decantation times of $15min$, and the last with a $12h$ decantation time. In the final step, to reduce water content and traces of methanol in the biodiesel, the sample was taken to evaporation, using a Rotavapor R-210/215 at $60°C$ and $180rpm$ rotation of the sample.

*2.3. Evaluation of the quality of the biodiesel obtained*

The physicochemical characteristics of the biodiesel were measured by applying different methods described in Table 1 for analyses of density, refraction index, and kinematic viscosity. Besides, flash point, moisture content, copper strip corrosion, acid number, and ester content, were determined according to the international standards ASTM D92 [12], ASTM E1064 [13], ASTM D130 [14], ASTM D664 [15] and EN 14103 [16], respectively.

## 3. Results and discussion

*3.1. Physicochemical characterization of the WFO*

The waste frying oil used as raw material had high viscosity, dark color and strong odor. According to the physicochemical characterization shown in Table 2, the WFO has a density slightly higher than that reported by [17] with a value of $0.901g/cm3$. The refraction index is similar to that reported by [18] with a value of $1.4670$; moisture content is lower compared to that calculated by [9] with a value of $0.1406\%$ for residual oil. Regarding kinematic viscosity, WFO reports a very high value according to that exposed by [19] who determined viscosities between $30.05mm2/s$ and $33.47mm2/s$ for different samples of used cooking oil. This difference in the kinematic viscosity may be due to the WFO used in this research could be utilized several times for cooking. When oil is used for cooking, oil has several chemical reactions. One of these are polymerization reactions forming compounds with high molecular weight and increasing the viscosity. Also, the excessive use of the oil for cooking can explain the low moisture content of the WFO used in this study. The acidity index was quite similar to that reported by [20] with a value of $5.61mgKOH/g$; lastly, the saponification index was quite similar to that reported by [21] with a value of $170.94mgKOH/g$ for chicken fat residues.

**Table 2.** Physicochemical characterization of the WFO.

| Parameter | Result |
|---|---|
| Density (g/cm$^3$) | 0.9623±0.000035 |
| Refraction index | 1.4628±0.000289 |
| Moisture content (%) | 0.0549±0.011316 |
| Kinematic viscosity (mm$^2$/s) | 52.8921±0.305588 |
| Acidity index (mgKOH/g) | 5.1158±0.031228 |
| Peroxide index (meq$O_2$/g) | 12.5055±3.022429 |
| Saponification index (mgKOH/g) | 174.6862±3.025660 |

A high value of the oil's moisture content can affect biodiesel production yield, given that the presence of water leads to the formation of soaps during the triglyceride reaction with the basic catalyst [18]. It is also stated that the ideal acidity index for the raw material (WFO) must be $< 5mgKOH/g$ [22]. A high acidity value affects directly the transesterification reaction, diminishing the conversion of triglycerides into biodiesel. The saponification index measures the amount of total fatty acids (fatty acids

and free fatty acids), indicating that a higher saponification index means a lower yield, given that it favors the organic salt formation reaction [23].

*3.2. Fatty acid profile of the WFO*

The determination of the fatty acid profile of the waste frying oil indicated that the predominant fatty acids in the oil are oleic acid with 42.45%, palmitic acid with 33.52%, and stearic acid with 7.44%, as evidenced in Table 3. The chromatography profile confirms the strong presence of monounsaturated fatty acid (oleic acid) characteristic of vegetable oils, which along with palmitic acid are the principal components of palm olein [24]; unlike soybean oil that has a higher proportion of linoleic acid (56.6%), oleic acid (21.5%), and palmitic acid (12.2%) in its composition [22]. Results of fatty acid profile indicates that the oil sample analyzed comes from palm olein. Likewise, this oil is similar to the chromatography profile reported for raw palm oil, which has 42.4% oleic acid, 37.1% palmitic acid and 5.4% stearic acid [25]. According to the fatty acid profile, it is ratified that WFO becomes a potential raw material for biodiesel production, considering that palm oil is one of the most prevalent raw materials for biodiesel production.

**Table 3.** Fatty acids predominant in WFO.

| Fatty acids | Amount (%Area) |
|---|---|
| Oleic acid, Methyl ester (C18:1) | 42.70 |
| Palmitic acid, Methyl ester (C16:0) | 33.52 |
| Stearic acid, Methyl ester (C18:0) | 7.44 |
| Myristic acid, Methyl ester (C14:0) | 3.96 |
| Palmitoleic acid, Methyl ester (C16:1) | 2.41 |

The molecular weight of the WFO was estimated using the stoichiometric calculations. An equation taken from [19] was used to determine the molecular weight, obtaining that the WFO sample has a molecular weight of $753.25 g/mol$.

*3.3. Physicochemical characterization of the biodiesel obtained*

The acid number in samples $Q_1$ and $Q_2$ evidences an important decrease compared to the value presented by the raw material according to Table 4. However, the acid number is higher than the value required by the quality standards for biodiesel. A high acid number in biodiesel can lead to engine corrosion and in which the rate of degradation [26]. The unsatisfactory results obtained for this parameter in this study denotes flaws in the biodiesel neutralization stage.

**Table 4.** Physicochemical characterization of the biodiesel obtained from WFO.

| Parameter | Treatment | | ASTM | EN |
|---|---|---|---|---|
| | $Q_1$ | $Q_2$ | D6751[11] | 14214[12] |
| Density at 25°C (g/cm$^3$) | 0.918 | 0.920 | - | 0.86-0.9 |
| Moisture content (%) | 0.449 | 0.387 | 0.05 Max. | 500mg/kg |
| Refraction index | 1.446 | 1.447 | - | - |
| Kinematic viscosity (mm$^2$/s) | 9.303 | 9.483 | 1.9-6 | 3.5-5 |
| Flashpoint (°C) | 178.400 | 174.100 | 130 Min. | 101 Min. |
| Copper strip corrosion | 1a | 1a | N°3 Max. | N°1 Min. |
| Acid number (mgKOH/g) | 1.582 | 1.3270 | 0.5 Max. | 0.5 Max. |
| Ester content (%) | 94.210 | 96.150 | - | 96.5 Min. |

With respect to the other parameters evaluated in the biodiesel, treatments $Q_1$ and $Q_2$ had a considerable decrease of density ($0.92 g/cm3$) and kinematic viscosity ($9.39 mm2/s$), compared with the conditions presented by the raw material, like that reported in the study by [22] where the kinematic viscosity diminished after carrying out the transesterification reaction. Regarding the moisture content, the samples had a significant increase in water percentage, unlike that reported by [27] where the water

content diminished considerably to $0.02\%$ because they developed the evaporation phase in a discontinuous column under vacuum ($0.05bar$), which is more efficient in the biodiesel dehydration. On average, the flashpoint for biodiesel is found at $176.25°C$, similar to that reported by [18] with a flashpoint of $170.67°C$. Copper strip corrosion showed satisfactory results by being in level (1a) according to the ASTM D130 [14] pattern strips. The study of the prior physicochemical characteristics permitted identifying that the biodiesel obtained only complies with two of the specifications required by ASTM D6751 [10] and EN 14214 [11], such as flashpoint and corrosion on copper sheet, as shown in Table 4.

*3.4. Analysis of biodiesel yield*

A way of determining the efficiency of the biodiesel production process consists in analyzing the ester content in the final product. According to Table 4, it was found that experiment $Q_2$ reached a value of $96.15\%$, evidencing that the chemical transesterification process with $70min$ reaction time obtains better yield in biodiesel production from WFO, although the biodiesel obtained did not reach the minimum value stipulated by the quality standards.

As illustrated in Figure 1, two samples of biodiesel produced from similar raw materials did not fulfill with the minimum percentage required; thus, the study using alkaline catalysis with ethanol had the lowest ester content, $88.9\%$ [28], compared with the study using methanol, with esters at $92\%$ [19]. This indicates that the biodiesel produced in both processes contains fatty acids that did not react, that is, the transesterification reaction was incomplete. The biodiesel produced from palm oil [25], through chemical transesterification, complies with ester content required by EN 14214 [11], reporting $99.4\%$ ester content, revealing a high yield in the process. However, in studies using WFO as raw material, only one accomplishes the minimum ester content, reporting $98.45\%$ ester content [29].

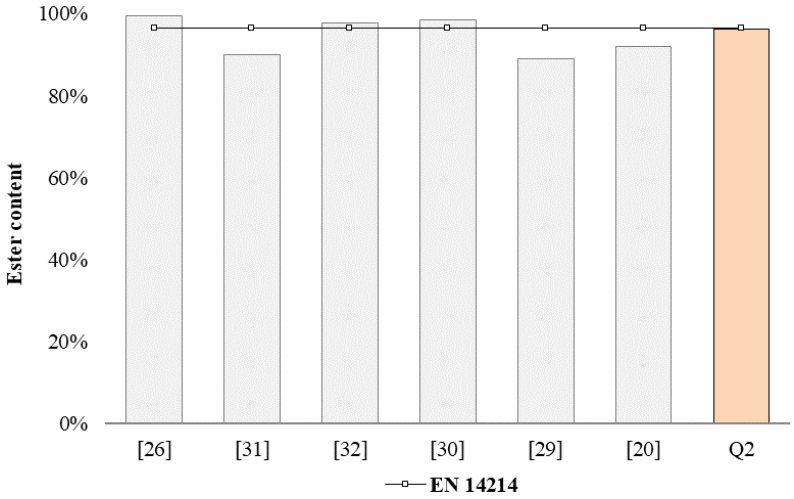


**Figure 1.** Analysis of ester content.

In studies of biodiesel production through enzymatic catalysis from WFO, ester conversion percentage is found around $90\%$ [30]; however, only one achieves the minimum value required and fulfills with the norm, reporting $97.8\%$ ester content by using the *Novozym 435* immobilized enzyme in an anhydrous medium together with long reaction times, highlighting that the raw material used was a mixture of used oil with canola oil [31].

## 4. Conclusions

The acid number presented by the biodiesel obtained through chemical transesterification ($1.327mgKOH/g$), exceeds the maximum amount permitted by the quality standards for biodiesel ($0.50mg/KOH/g$). This demonstrates that the product still has residues of free fatty acids, thereby, it

is possible that the washing or neutralization stage was not performed satisfactorily. On the contrary, the ester content for experiment $Q_2$ has a high conversion percentage of fatty acid methyl esters (96.15%), which indicates a good conversion in the reaction, however, it does not meet the minimum value required by the EN 14214 (96.5%), probably due to the raw material's quality conditions.

The high viscosity, dark color, strong odor, and remains of accumulated fats of the WFO characterized in this study evidenced that frying oil in some chicken grills is excessively reused. This is ratified by the low moisture content of the sample, compared with frying oils from other studies. However, this result is favorable for biodiesel production, given that low moisture content avoids the presence of soap in the transesterification process. The frying conditions (*e.g.*, high temperatures, prolonged times, excessive reuse) affecting considerably the WFO quality, increasing density ($0.962g/cm3$), kinematic viscosity ($52.89mm2/s$), and acidity index ($5.116mgKOH/g$), compared to raw vegetable oil. Finally, the analysis of the fatty acid profile of the WFO showed a composition of 42.7% oleic acid, 33.52% palmitic acid and 7.44% stearic acid, corroborating that this material can be used for biodiesel production, having a profile similar to the composition of palm oil, which is in Colombia the major source to produce this biofuel.

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