Efficiency Evaluation on the Influence of Washing Methods for Biodiesel Produced from High Free Fatty Acid Waste Vegetable Oils through Selected Quality Parameters

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Abstract: The increasing use of alternative energy sources has brought benefits like the recycling of waste vegetable oils (WVO) for biodiesel production; however, this practice presents challenges derived from a highly variable raw matter. Our research focused on the washing techniques usually employed by small to medium scale producers which may lack the infrastructure to perform high-grade purification and often relay on the employment of "artisanal" technology. We report biodiesel production from pure canola oil (C1) and a mix of soybean/canola oil (C2) as well as WVO from sample C2 with high free fatty acid content (2.7 FFA %) oil. We selected seven quality tests: pH determination, cloud, pour and flash point, acid number (AN), water/sediments and soap content considering the most commonly failed checkpoints in biodiesel production. From the use of four washing techniques, we determined that the sawdust filtration had the highest recovering yield with 89%. The majority of the washed biodiesel met the America Society of Testing Material (ASTM) standard limits for the tests selected. Biodiesel stability was tested from week 0 to week 6, determining a shelf life of three weeks in optimal conditions without the addition of antioxidants or specific storage conditions.

Keywords: biodiesel; quality; oil; biofuel; washing; purification; misting

1. Introduction

The accelerated decline in the availability of fossil fuels and their direct impact on the fluctuating market of bioenergetics has driven the need to develop and implement new and sustainable strategies [1]. Among these new trends, alternative sources of energy have been presented through the use of solar panels, wind turbines and modified combustion engines, which has allowed the use of biofuels, among others. The concept of biofuels by itself is not new, with reports as early as the 1900s, when Rudolf Diesel presented his engine design fashioned to run on peanut oil. Biofuels, such as biodiesel, are based on biomass use, and are traditionally produced chemically through transesterification of a vegetable oil or animal fat with a short chain alcohol and a catalyst.

Biodiesel holds an energetic content of 120,000 British Thermal Units (BTU) per gallon [2], is miscible in diesel fuel and it can be used on diesel engines with minimal modifications and is expected to represent 20% of the total fuel consumption by 2020 in the European Union [3].

However, the main barrier for biodiesel is cost, since the use of raw matter options like vegetable oils, of first and second generation, animal fats and algae, can represent a feedstock value that can count up to 75% when establishing the manufacturing processes [4].

In this regard, a raw matter considered as a feasible substitution for food crops virgin oils has been waste cooking oils. This source presents a viable option given their inherent uniformity aligned to maintain food flavor and quality control in order to convene the standards required for its consumption [5].

As for the availability of waste cooking oil, there are reports of 1,149,000,000 L/yr. (L/years) in Europe [6], 378 541 178.4 liters produced per day in the United States of America [7] and 100,000 L per month (L/month) in Mexico City with an estimation of a mean of 11.3 L per capita by the year 2019 [8], giving a potential for use of this valuable resource. Given that the characteristics of the fuel obtained are correlated with the type and quality of the raw matter used, when using waste cooking oil it is critical to evaluate its quality to avoid damaging engines. Most biodiesel production methods base their quality performance on two main standards, the European Committee for Standardization (EN)14214 and the ASTM D675, both of which share international acceptance. The standards include physical and chemical characteristics, including contaminants, such as free fatty acids, water, glycerol, and methanol contents.

To achieve this degree of refinement on biodiesel and to eliminate the residues inherent to biofuel production, two strategies are used to refine cooking oils for the production of biodiesel. The most common uses water as a dissolvent [9] and extraction with either mixing, misting or bubble washing operations [10]. Otherwise, dry extraction methods are used including magnesium silicate, ion exchange resins or cellulose packed columns [11].

The objective of this work was to compare different washing methods based on the procedures usually applied by biodiesel producers that employ waste cooking oil in a semi-industrial manner and correlate quality parameters in order to determine the effectiveness of the refining method on the biofuel produced.

2. Materials and Methods

2.1. Collection of Oil Samples

Waste vegetable oil (WVO) samples were collected from a local restaurant, filtered through a sieve to remove food residues, dried in a heating plate at 100 °C for 15 min and placed in storage in glass containers at environmental temperature. The samples were characterized through gas chromatography to determine their fatty acid profile using the Association of Official Analytical Chemists (AOAC) standards AOAC 969.33 [12] and the FFA by the AOAC 940.28 method [13].

The oil used as control 1 (C1) corresponded to a commercial pure canola oil sample and for control 2 (C2) the same brand of vegetable oil used by the company that provided the WVO was analyzed in order to have a base point of comparison. The negative control (NC) consisted of a WVO biodiesel sample that remained unwashed.

2.2. Biodiesel Production

Alkaline transesterification was carried out for biodiesel production under optimized conditions established by previous work (data not shown) with 100 g of vegetable oil, using 1.5% potassium hydroxide (KOH) as the catalyst, at a 10:1 molar ratio (alcohol:oil) and was maintained at 60 °C. The samples were reacted for 60 min with stirring before being left at an ambient temperature for 24 h before decanting. The biodiesel recovered was saved in sealed glass containers, exposed to light and was stationary at 23 ± 2 °C for six weeks.

2.3. Washing Methods

All washing methods were performed on nonblended biodiesel samples in three independent rounds of experimentation with 9 replicates using regular tap water at room temperature (23 ± 2 °C).

2.3.1. Mechanical Agitation

The samples were subjected to vertical agitation for 5 continuous minutes in a closed container with a 3:1 water/biodiesel ratio followed by a 60 min pause. Once the established time had elapsed, the bottom phase (water with impurities) was removed with a 10 mL (milliliters) glass pipette, and a new cycle was initiated for a total of 3 washing cycles per sample.

2.3.2. Bubble Washing

The washing system used consisted of an air pump with a 2 L/min capacity connected to an air diffuser. Water was added in a 1:1 ratio to the biodiesel samples obtained and air was passed through the diffuser for 60 min until a clear phase separation was achieved.

2.3.3. Misting

A mist washing system was adapted using a water pump with a capacity of 1500 L/h (liters per hour) and a nozzle with a flow diameter opening of 1.5 mm. For this washing technique, the water/biodiesel ratio was not considered, instead a total volume of 2300 mL was sprayed from above the biodiesel sample at a distance of 10 cm for a 5 min period.

2.3.4. Sawdust Filtration

For the dry washing, a mixture of pine wood shavings and kiln-dried sawdust (<2 mm particle size) was packed inside a 30 × 115 mm plastic column with a plastic mesh of 3 cm (centimeter) in diameter and a pore opening of 1 mm (millimeter). Each sample was filtered through and left to stand for at least 60 min (minutes).

2.4. Sample Drying

All samples (with the exception of the negative control) were dried using a heating plate with gradual temperature increments until 120 °C was reached and maintained for 5 min.

2.5. Quality Parameters

To determine the quality of the biodiesel produced, the selected tests were performed according to ASTM international standards. The cloud point and pour point were determined by the ASTM D2500 and ASTM D97-17b respectively, pH was measured by the ASTM E70 method, Acid Number was determined using the ASTM D974, Water and Sediments using the ASTM D2709 protocol, Soap content through the ASTM D1962 method and flash point with a modified technique for the International Organization for Standardization (ISO) 3679 standard [14–20].

2.6. Statistical Analysis

The data obtained from each quality tests (with the exception water and sediments test), were analyzed and all assays results were collected and subjected to a multiple mean comparison using Tukey and Scheffé tests with IBM SPSS STATISTICS® for Macintosh, version 23. Armonk, NY, USA.

3. Results and Discussion

3.1. Biodiesel Production

The waste vegetable oil (WVO) sample used to obtain biodiesel (WVOB) was collected from a restaurant that serves fried food. The sample was composed of soybean and canola oil with three predominant fatty acids present; oleic acid (31%), linoleic acid (45%) and palmitic acid (13%) (data not shown). The biodiesel obtained had a yield of 84.95% ± 1.73% without previous esterification.

The fatty acid profile and amount of free fatty acids of the beginning oil are particularly important in determining the final biodiesel quality; both considered parameters of ample significance when correlating the origin of the oil with the quality of the biofuel produced [21–24].

In this regard, the FFA (free fatty acids) percentage was 2.7%, higher than the recommended value (<1%) for use with direct transesterification, however with the procedure referred by Ortiz et al. [5] and with the use of a direct neutralization of the sample, the esterification step was omitted.

The transesterification reaction itself does not alter the fatty acid composition of the feedstocks [24] but characteristics like chain length and double bond presence have been seen to affect yield and quality of the biodiesel produced; with an average value of 17 atoms in length and a double bond value of 1.16, Pinzi et al. [23] obtained favorable results. Whereas esters of saturated medium chain acids were reported as suitable [25] given that the absence of double bonds gives excellent oxidative stability and an adequate low temperature flow property by the lack of the high melting points related to the presence of long-chain saturated fatty acid esters.

Furthermore, Ganduglia et al. [26] and Ramos et al. [24] reported that the amount of each fatty acid and their pattern of saturation and distribution could have a possible impact on chemical and physical properties of the biofuel produced, directly correlating with properties such as ignition, stability, flow at low temperature and iodine number.

3.2. Biodiesel Washing Methods and Quality Determination

When producing biodiesel by chemical transesterification, a washing step is used to remove residual contaminants that could reduce the quality of the final biodiesel. Low quality biodiesel can lead to excess engine corrosion from excess free fatty acids and/or water, or blockages when there are soaps and/or glycerides, as well as other problems [9,27].

Given this fairly common scenario, a diversity of washing/purification techniques have been developed, including natural sedimentation [28] or water washing techniques such as mechanical shake-up, misting or bubble washing [2,29]. However, the use of water entails a number of disadvantages, such as possible development of emulsions, fuel degradation, the generation of large amounts of water residues that require treatment and the addition of a necessary drying step in order to remove residual amounts of water [30].

The final yield results from the four methods tested were obtained as follows: the sawdust technique presented a final yield of 89%, followed by the bubble washing and misting methods with a final yield of 86% and mechanical agitation with 74%. This is in conjunction with the results obtained from specific quality tests selected for their importance as key indicators that biodiesel producers seek in their final product.

Considering that this work is mainly focused on small- to medium-scale producers, the quality test criteria selection was directed to the tests that allowed an evaluation of a complete transesterification reaction, the efficiency of the washing techniques, as well as the stability of the biodiesel obtained.

3.3. Pour Point and Cloud Point

Cloud point and pour point are parameters that are related to the fatty acid composition and the degree of double bonds directly correlating to the crystallization temperature because of their disruptive effect on packing capacity [31]. The importance of monitoring these parameters lies in the fact that both conventional diesel and biodiesel, unlike gasoline, freeze at low temperatures [26]. These phenomena cause fuel filtering systems to obstruct fuel flow and consequently, cause severe damage to the injection system [32].

The pouring or fluid point is defined as the lowest temperature at which the paraffin conglomerates show resistance and the gravitational force does not have an effect on the flow, while the cloud point is the temperature at which the first small paraffin crystals are observed as they precipitate into the fuel [33].

The negative control presented a cloud point of 16 °C and a pour point of 14 °C, with no significant difference between the method or its effect on the control samples (data not shown). For C1 and C2 the best result came from the use of bubble washing with a cloud point of −2 °C and −1 °C, respectively (Figure 1).
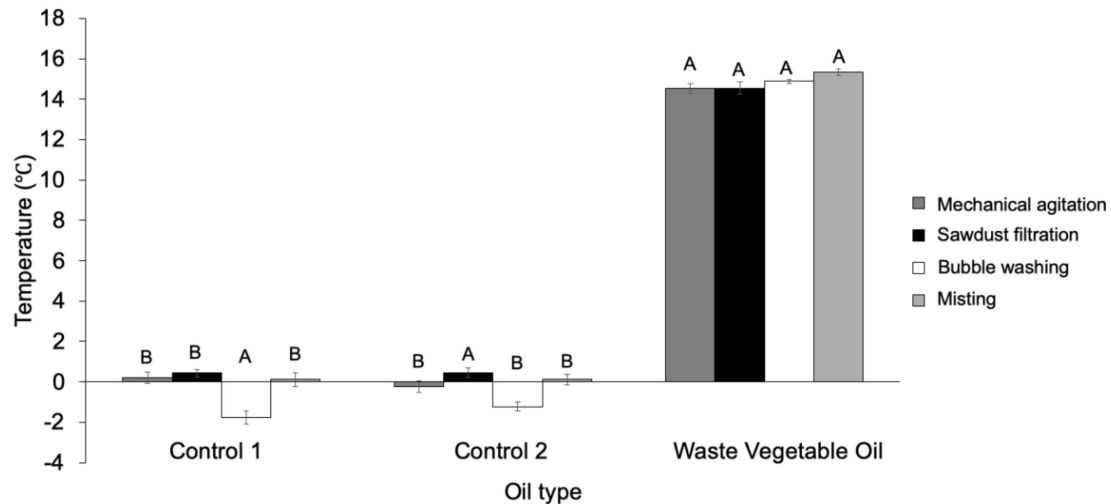

Figure 1. Cloud point.

The results obtained for both the cloud and pour points of the washed samples by the four different methods are presented in Figures 1 and 2, with the significant difference established by grouping the results obtained from the statistical analysis with letters A–C. Focusing only on the product of the oil transesterification, the fatty acid methyl esters (FAME), these results show us that the amount of saturated or unsaturated fatty acids on the original samples had no direct impact on these parameters even when unsaturated fatty acids represented more than 60% in the WCO sample, a factor that was in agreement with Reaume and Ellis [34] and Canoir et al. [35] and could have an effect on lowering the temperatures for both cloud and pour points and thus remarks the importance of the raw matter quality for this specific test.
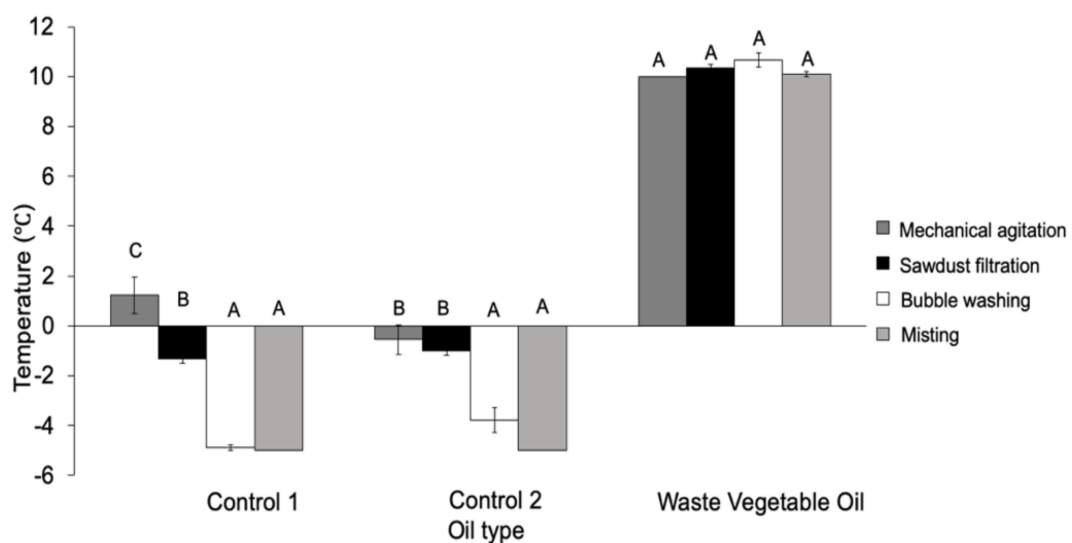

Figure 2. Pour point.

As for the WVO sample, by analyzing the results obtained we could assume that the modifications made by the heating processes on the oil, as well as the contact with food and water were determinant

factors for which the cloud and pour temperatures were as elevated as shown within a range of 14–16 °C. Whereas the fatty acids undergo a number of chemical reactions when exposed to high temperatures often ranging from 170 to 200 °C for extraneous periods of time, we can correlate that the modifications could come from a conformational change in the structure of the fatty acids. As Knothe and Dunn [36] remark a cis/trans isomerization can modify the biodiesel molecule into a straight configuration and thus affect negatively on parameters such as cloud and pour point, given that trans double bonds are more stable and at higher temperatures can shift to adjacent positions [37]. However, a more in-depth analysis is required in order to confirm these results.

3.4. Acid Number

Acid number in biodiesel is almost entirely determined by fatty acid content (FAC), which can either be present in the oil or formed by hydrolysis of the esters throughout the transesterification process.

The accepted value for biodiesel is set at 0.5 mg KOH/g corresponding to approximately 0.25 wt% (weight percentage) and determined by titration, a reliable technique [38].

The negative control had an acid number of 0.56 mg and the washed samples needed a mean of 0.61 ± 0.005 milligrams (mg) of KOH (potassium hydroxide) to neutralize 1 g of FAME with no significance based upon the type of oil or washing step. In accordance with international standards such as ASTM D6751 and EN 14214, none of the washing techniques met the quality criteria established; however, the aforementioned standards have been considered as overly cautious; therefore, some countries have higher permissible limits (e.g., Austria accepts a maximum of 0.8 mg KOH/g) [39]. High levels of acidity in fuels are associated with corrosion and formation of deposits in the engine, particularly in fuel injectors [40].

3.5. pH

The pH of biodiesel samples should be neutral; if lower, the FAME product could affect the integrity of plastic materials where the fuel is used, such as in hoses and injectors. The negative control had a pH of 11.6 and the results obtained for C1 ranged from 6 to 8, these being the best methods; bubble washing and mechanical agitation technique. C2 ranged from 5–8 only, obtaining a significant difference from the sawdust filtration method and WVOB from 6–10 with a more heterogeneous behavior, forming 3 differenced groups (Figure 3), with the significant difference established by grouping the results obtained from the statistical analysis with letters A–C.
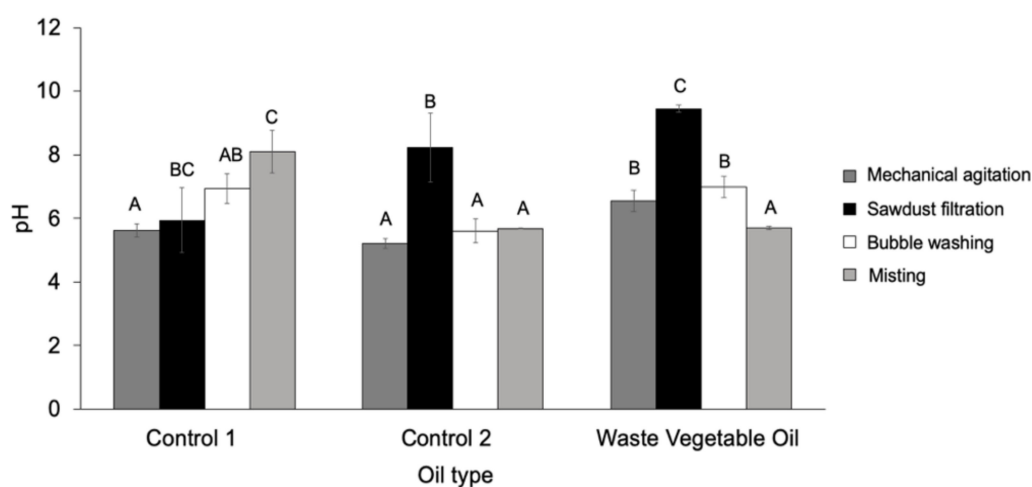

Figure 3. pH from biodiesel samples.

The pH analysis of the biodiesel samples assists in the determination of the corrosive potential that the biofuel can present, and in turn it functions in correlation with the AN.

Nevertheless, both parameters are directed to measure different aspects of the biodiesel sample; the AN test does not embody a complete representation of the acidic components of the sample (organic or inorganic) but it is of ample use in detecting indirectly the products of a degradation process, such as oxidation. The pH determination detects how the effects of the said reaction affects the characteristics of the product of interest (biodiesel). As the biodiesel is oxidized, it derives a series of modifications in properties such as increasing the acidity index and the peroxide value, among others [21], and in turn lowers the pH.

Another major factor effecting the composition of the oil sample for WVO is that the acidity levels in biodiesel are likely to drop over time. For this, it is of high importance to consider the water content, as it promotes the hydrolysis of esters and the production of acids when exposed to air and high temperatures [41].

3.6. Soap Content

Generally, samples will present higher levels of soap when waste oils and animal fats are used instead of refined vegetable oils for biofuel production. These soaps are the result of the process of saponification of free fatty acids present in the feedstock, and are mainly attributed to an incomplete transesterification reaction [42].

The results obtained in our work indicate that there was a significant difference ($p < 0.05$) between the soap content presented among the biodiesel samples. For C1, the two main groups were formed: group A, made up by mechanical agitation and sawdust; and group B, constituted by bubble washing and misting. For C2, three groups were formed: group C, constituted by bubble washing and misting; group A, correspondent to mechanical agitation; and group B, composed of the sawdust washing technique (Figure 4).
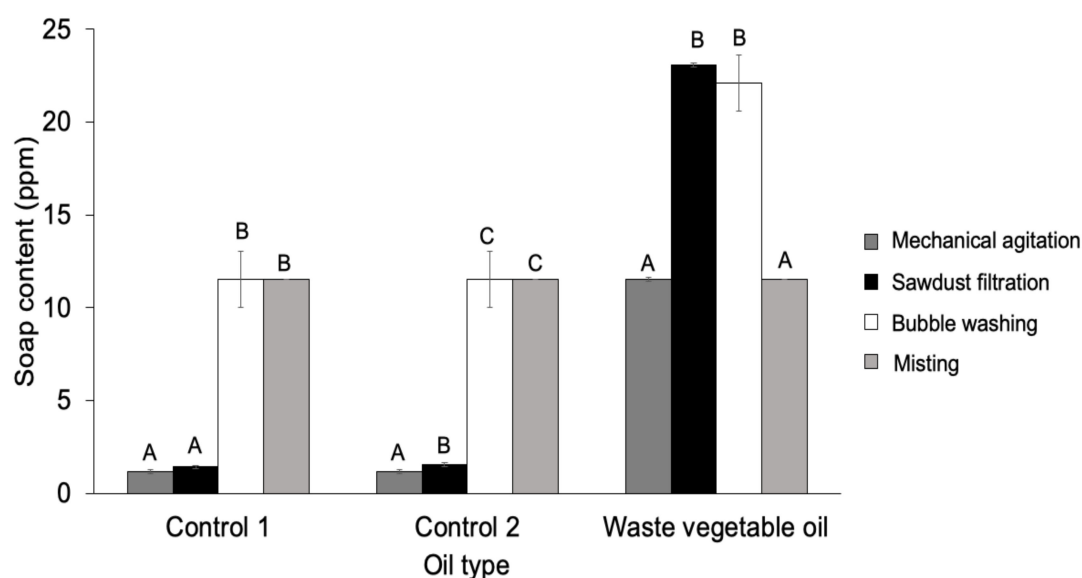

Figure 4. Soap content from biodiesel samples.

Similarly, for WVOB, two groups were formed: group A, composed of mechanical agitation and misting; and group B, made up of sawdust and bubble washing. For the negative control the results were reported as < 500 ppm.

When comparing the washing techniques, washing by misting and bubbling is reported to be more effective than agitation or mixing to eliminate soap residues from biodiesel. These two techniques mobilize water in drops, which have a larger contact area within the biodiesel, thus helping the soap to dissolve more easily; unlike the mechanical agitation in which the presentation of the water reduces the contact area and therefore its efficiency to eliminate soaps [2]. On the other hand, it has been reported

that by using a dry wash technique the soap content in biodiesel was reduced from 2000 ppm to a level below the detection limit close to 10 ppm using only 1 g of sawdust for every 45 g of biodiesel [30].

By the ASTM standards for soap content (<66 ppm) all the washing techniques were effective, especially mechanical agitation which reduced soap content from <500 ppm to less than 10 ppm. For the WVOB samples we obtained what might be considered as contradictory results. We separated the washing methods into two groups. Group A, consisting of mechanical agitation and misting, with its main characteristic being that these methods use water as a contaminant extraction adjuvant pushed through the sample, this could be considered disadvantageous given that this approach is prone to form emulsions; and group B, corresponding to the washing methods that are labeled as "gentle", the bubble washing and sawdust filtration. We observed that by comparing and contrasting the results we had a system saturation phenomenon [43], this meaning that in both the bubble washing and sawdust filtration, the amount of water or filter material was kept as a constant and was not renewed as in the case of mechanical agitation or misting techniques. Therefore, we can infer that the washing systems came to a saturation point from which no more soap was able to be retained or linked and was therefore removed, contrary to what was observed in the systems that had a flow of water available by means of repetitions.

3.7. Flash and Ignition Point

The flash point is defined as the lowest temperature at which the vapors of a material will ignite when an ignition source is applied to it [44]. The importance of this test for biodiesel lies in the safe handling, transport and storage of the biofuel.

The results of the statistical analysis indicated that there was no significant difference on the temperatures recorded on neither the samples nor the washing methodologies employed.

The flash point is related to the concentration of residual methanol in biodiesel decreasing it to a point where purification processes are necessary in order to remove it [9,45]; the negative control presented a flash point of 150 °C, which indicates a higher methanol content than the samples following the washing process. The percentage of methanol must be below 0.2% (wt%) to meet ASTM standards of 130 °C as the minimum flash point temperature [46]. Based on this, we can establish that all biodiesel washing methods used allowed us to obtain a fuel within the acceptable flash point criteria (Figure 5) showing no significant difference between groups (A).
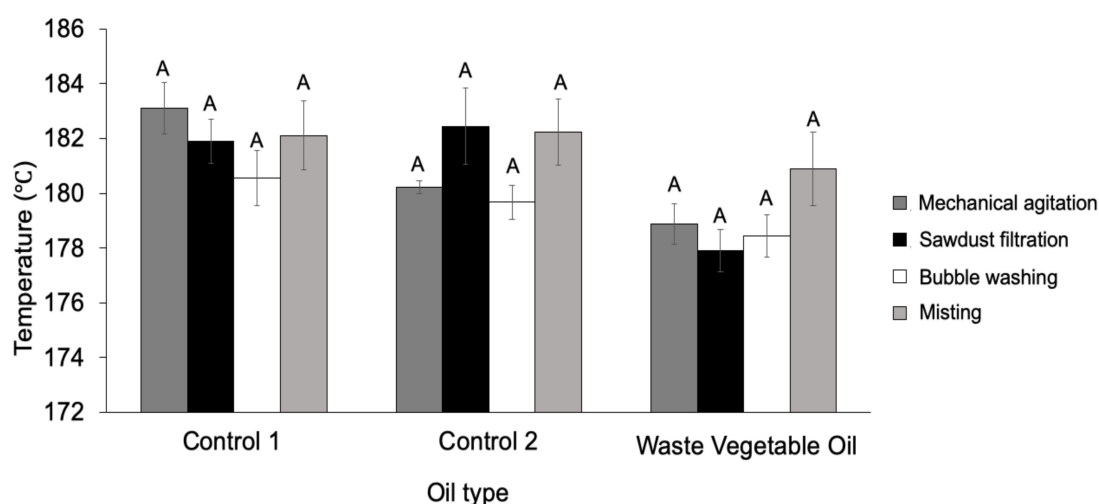

Figure 5. Flash point from biodiesel samples.

3.8. Water and Sediments

Water content is a major aspect that should be determinant of fuel quality. Biodiesel is composed of hygroscopic molecules (fatty acid methyl esters) that can augment the soluble water content of the sample by absorbing moisture [47,48].

The test to determine water and sediments is carried out to find out the percentage of water present in the fuel as well as contaminants like soap and glycerol residues. The limit established by the ASTM D2709 is 0.05% vol., meaning that excesses in the sample can lead to deterioration of the fuel and possible bacterial spoilage [30].

The results found in our work (Figure 6) show that free water and glycerol were the two residues presented in the washed samples. For the WVOB two methods had residual contaminants; for the bubble washing method, a 29.63% of the total samples had free water and 25.93% of samples presented free glycerol as a sediment when washed by the sawdust method, however none of the samples exceeded the 0.05% standard limit.
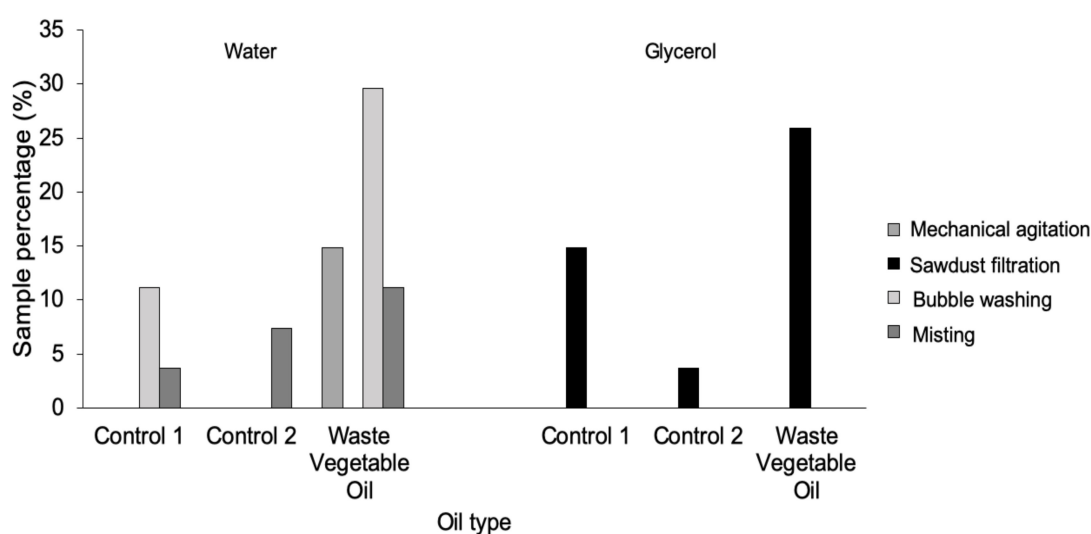

Figure 6. Residual contaminant presence in biodiesel samples.

3.9. Biodiesel Shelf Life

The average shelf life of a biodiesel sample will depend on the storage conditions. Biodiesel, like petroleum diesel, degrades over time, it has stability monitoring specifications for a 6-month time frame, however its maximum functional time will depend entirely on its correct handling and storage, avoiding factors such as exposure to light, chemical contamination and exposure to air, among others [49].

Regarding the composition of the samples, the larger the presence of unsaturation, the more the biodiesel will have a major susceptibility to a faster oxidation process, on occasion this aspect being a more impactful factor than the environmental conditions to which the sample is exposed to [41].

The oxidation of biodiesel occurs in three stages: initiation, propagation and termination. During these three phases, hydroperoxides are formed through a chain mechanism of free radicals. The radicals produce short-chain aldehydes, ketones and carboxylic acids, thus increasing the acidity as well as the viscosity. Under certain conditions, radical-induced polymerization can form insoluble polymers which can plug and clog fuel lines, filters and pumps [25,40].

While storage conditions and production can have an effect on the stability of biodiesel; sample purification can be a major aspect that acts directly on the characteristic and quality parameters in the long-term scenario [50].

Table 1 displays the quality parameter measurements taken after the third and sixth week of storage.

Table 1. Quality determinations for biodiesel samples during storage.

	Week 0	Week 3	Week 6
pH			
WCO			
Mechanical agitation	6.6	7.1	4.1
Sawdust filtration	9.4	8.3	4.4
Bubble washing	7.0	7.2	3.6
Misting	5.7	6.1	3.9
Cloud point			
Mechanical agitation	14.6	14.1	15.4
Sawdust filtration	14.6	15.3	15.0
Bubble washing	14.9	15.4	15.0
Misting	15.3	14.7	14.8
Pour point			
Mechanical agitation	10.0	9.6	9.9
Sawdust filtration	10.3	10.1	10.0
Bubble washing	10.7	9.7	10.1
Misting	10.1	10.1	10.3
Flash point			
Mechanical agitation	178.9	179.9	177.0
Sawdust filtration	177.9	178.1	180.0
Bubble washing	178.4	178.9	180.0
Misting	180.9	180.6	180.6
Soap content			
Mechanical agitation	11.5	11.5	11.5
Sawdust filtration	23.1	11.5	11.5
Bubble washing	22.1	11.5	12.8
Misting	11.5	11.5	11.5

For pH measurements, the results showed a significant difference between the first and third sampling, obtaining results lower than 5 by the sixth week regardless of the raw matter or the washing method employed. The pH reduction of the samples is a sign of the oxidation process on the fuel. Regarding the acid number, the samples presented no significant difference in either the second or third sampling points. However, these results do not ensure that the acid number will not be affected over time, with increasing acid number reports made on biodiesel samples stored for 52 weeks at 20 °C (<1.00 mg KOH/g) and highly elevated results when exposed to air [41].

The cloud and pour point of the samples had the same behavior which was maintained throughout the storage stage, displaying minimal variations among the sampling points, all within a behavior range showed by samples of similar origin [49–52].

The flash point as already stated complied with the minimum temperature requirements (>130 °C) and all the samples were able to be maintained during the storage stage of the experimental work [53,54].

As for the soap content, the control samples presented a significant difference between the washing methods. For C1, mechanical agitation and sawdust were the most effective during week 0; by the second sampling point (three weeks), the sawdust method had a significant increment and meanwhile the mechanical agitation technique maintained the lowest soap content levels; however, by the sixth

week, the soap content in all treatments and samples was recorded between the range of 11.5–19.2 ppm, probably due to the deposition of polar compounds that agglomerated over time [55], regardless, all the samples presented acceptable results in accordance to international quality standards.

3.10. Biodiesel Analysis through Fourier Transform Infrared Spectroscopy (FTIR).

We performed FTIR testing for all the WVOB samples resultant from the four washing methods in order to corroborate the transesterification process stability (Figure 7). The differences between the methyl ester and refined oil samples regarding FTIR are considered small and subtle since the chemical structure of both products is very similar. Nevertheless, there is a region that allows chemical discrimination between oil and methyl esters known as the "fingerprint" region and it covers a range of 1500–900 cm^{-1}. The peak at 1446 cm^{-1} corresponds to the asymmetric stretch of $-CH_3$ found in biodiesel spectra and absent in refined oils; similarly, the present peak, the stretching of O−CH at 1196 cm^{-1}, is also considered typical of biodiesel [56] both were observed in all the samples processed. The FTIR analysis confirmed the identity of the obtained samples as biodiesel through the detection of characteristic signals.
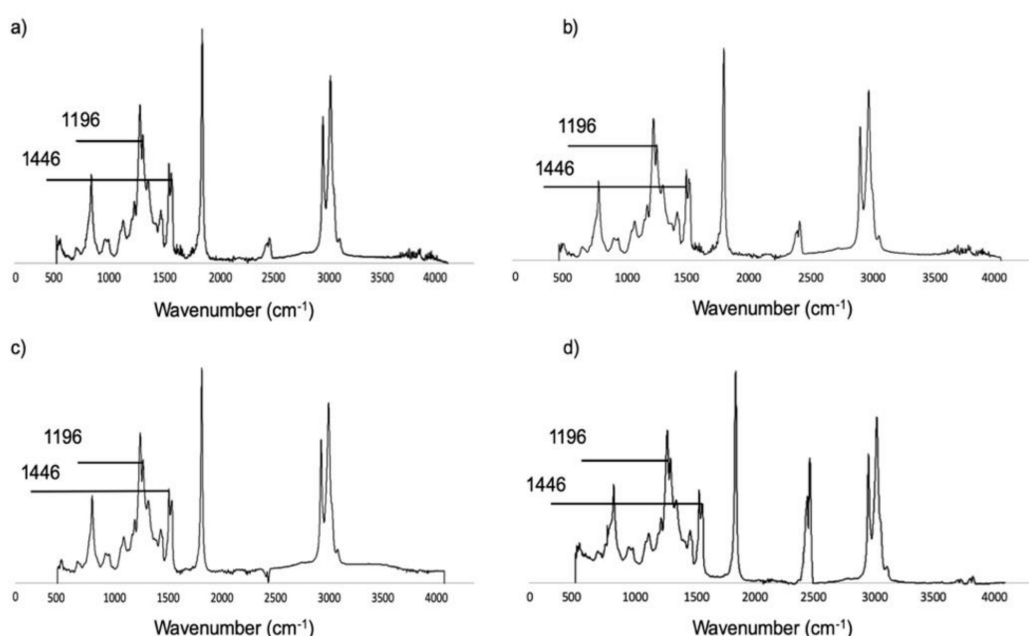

Figure 7. FTIR from washed WVOB. (**a**) Mechanical agitation, (**b**) sawdust filtration, (**c**) bubble washing, (**d**) misting.

4. Conclusions

Biodiesel production using waste vegetable oil was effective, obtaining a final yield of 89% post-washing through the sawdust method. We were able to compare the effectiveness of the washing techniques during a storage time of six weeks, which gave us a general understanding of the transesterification reaction and the biodiesel stability.

By using only a selection of tests, we obtained results that highlighted the importance of biodiesel washing, showing the clear difference an additional purification step could represent on the quality and stability of the biodiesel produced.

Most of the biodiesel samples, except for the negative control, met the quality guidelines established on international standards (ASTM) at week 0 and at week 3; however, by the sixth week, we observed the first phase of fuel oxidation with a decrease in pH and a rise in the soap content, showing that without further storage adaptation or addition of antioxidants, the biodiesel maintained its quality with a shelf life of at least 3 weeks.

Regarding the quality determinations, cloud and pour point temperatures, although there are no specific values concerning these tests for biodiesel, the results obtained could potentially present difficulties for its direct use; however, to avoid any use difficulty, we recommend the use of blended biodiesel.

The acid number test was complied with a less rigorous criterion and is related to the pH determination, and with both tests we could establish a more robust picture of how the biodiesel could increase its acidity index and peroxide value over time. For soap content, we obtained one of the most interesting results, and from the sawdust filtration and bubble washing, we reduced the soap content to <10 ppm. Both flash and ignition points were not affected by any washing technique and finally, for the water and sediment tests, we found that no washing method exceeded the maximum limits.

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