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ORIGINAL PAPER

Biodiesel production from three mixes of oils with high free fatty content: quality evaluation and variable analysis

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Abstract Due to the excessive use of fossil fuels around the world, more environmentally friendly alternatives have been studied. Technologies for the production of ethanol, biogas and biodiesel are focusing on the importance of improving costs and efficiency. Biodiesel can be used in automotive internal combustion, is biodegradable and has no presence of metals, however, it lacks competitiveness versus petrodiesel mainly by the high cost of the pure oils used for its production. The aim of this study was to obtain biodiesel from oil samples with high content of free fatty acids (>1 %) obtained from three fast food restaurants using their molecular weight and acidity index values in order to neutralize the free fatty acids in a one-step reaction and perform a screening for optimal conditions for transesterification. The experimental design consisted of two reaction times (60 and 90 min); four methanol–oil molar ratios—6:1, 10:1, 15:1 and 20:1; and two catalysts (NaOH and KOH) at three concentrations 0.5, 1 and 1.5 % with a constant temperature of 60 °C and 500 rpm. The optimum conditions for the different waste cooking oil feedstocks were established reaching a final yield up to 85.53 % of biodiesel, concluding that there is viability of production through the use of this raw material and free fatty acids neutralization technique, obtaining a biofuel that meets international quality standards.

Keywords Alkaline · Recycling · Transesterification · Waste

Introduction

The energy crisis that has reigned the world in recent years has driven the efforts to find new and more environmentally friendly energy sources such as biofuels. By definition, biofuels are alcohols, ethers, esters and another organic compounds (Stratta 2000) produced from crops, herbaceous or woody plants, agricultural residues, forestry activity and in minor degree industrial waste. Out of the three major biofuels, biodiesel consists in monoalquilic esters from fatty acids derived from renewable sources like vegetable oils or animal fats (Ramadhas et al. 2004), and is biodegradable, renewable, with nontoxic emissions and superior lubrication properties (Lotero et al. 2005), it presents a higher cetane number than diesel, it does not present aromatic compounds or sulfur, contains 10–11 % of oxygen per weight (Canacki 2007), and it is synthesized through transesterification among a vegetable oil or animal fat in combination with a short chain alcohol combined with a catalyzer (Dana 2009). Considering that any fatty acid source may be used to obtain biodiesel, the substitution of raw matter for others that are less expensive like nonedible or waste cooking oils (WCO) (Behzadi and Farid 2007) has been taken into consideration; here, the food industry presents an interesting option given that in order to maintain the same flavor and quality of the food prepared, the oil used has a determined number of cycles to be used, and once this are surpassed, it becomes a waste. The Energy Information Administration in the United States estimates that approximately 100 million gallons of waste cooking oil is produced per day in the USA (Radich 2006). Until recent years, waste cooking oil was usually discarded through sewerage which can lead to corrosion of metal and concrete elements of wastewater treatment plants increasing the cost of treating effluents (Szmigielski et al. 2008),

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and now it can be used as a high potential source for recycling processes. Considering that the use of these residues reduces the cost of biodiesel production up to 70–95 % (Connemann and Fischer 1998; Zhang et al. 2003), the aim of this study was to investigate the optimal parameters for biodiesel production with WCO from the fast food industry with various content of free fatty acids (FFA) using chemical transesterification with alkaline one-step neutralization and two-step catalysis, along with the analysis of different washing methods and assessment of quality of the obtained biodiesel. All the samples were taken and the procedures were carried out within a period of 1 year from August 2012 to August 2013 in the Biotechnology Institute of the Biological Sciences Faculty in the Autonomous University of Nuevo León, México.

Materials and methods

Obtainment of waste cooking oil and preparation

The WCO was collected from three fast food restaurants that use continuous frying processes, the samples (3–5 L) of oil were obtained monthly during 6 months. The WCO was filtered through mesh to separate food residues. After this process, 1 L of samples was taken in a beaker and placed on a heating plate to eliminate water. Finally, the WCO samples were placed on closed plastic containers for storage at environmental temperature (25 °C). The samples were donated for the realization of the work.

Oil analysis

The fatty acid profile of the samples was determined through gas chromatography (AOAC 969.33, 2000) from which their molecular weight was calculated.

$$\text{Fatty acids molecular weight(FAMW)} = \sum f_i \Big/ \sum \left(\frac{f_i}{\mathrm{MW}_i} \right)$$

where f_i equals the fraction of the fatty acid weight (%), $\sum fi$ equals the fatty acid total in a sample and MW_i equals the molecular weight of the fatty acid.

$$\text{Oil molecular weight} = 3 \times \text{FAMW} + 38.049$$

where 38.049 equals the weight of the glycerol molecule in the triglyceride.

Additionally, the acidity index was determined using percentage of oleic acid through titration method (AOAC 940.28, 1990).

Experimental design

To determine the optimal conditions for biodiesel production (Table 1), type and catalyst concentration were considered, selecting NaOH and KOH at 0.5, 1 and 1.5 %; alcohol-to-oil molar ratio, testing 6:1, 10:1, 15:1 and 20:1; and two reaction times, 60 and 90 min, with an established constant temperature of 60 °C and 500 rpm. As a primary screening, 48 experiments consisting of the total of the combinations presented in the experimental design were conducted (data not shown) selecting KOH as the best catalyst. All the positive results were performed in triplicate.

Acid esterification and titration

For the samples provided by company No. 3 (C3), the alkaline neutralization was not achieved. An additional step of esterification pre-treatment was added, where the oil was heated to 60 °C adding 60 % of the amount of methanol previously established in the experimental design, adding slowly to the mixture the specific amount of sulfuric acid according to the acidity of the oil, mixed for 30 to 40 min and proceed to leave it overnight (McFarling, 2012). To finalize the pre-treatment, the acidity index was re-determined. In order to re-test the oil subjected to acid esterification, a titration was necessary. For this technique, 1 g of the previously esterified oil was measured in a

Table 1 Experimental design considering catalyst concentration (%CC), molar relation (MR), reaction time (Rx. Time), agitation velocity (RPM) and temperature (Temp.)

Test number	%CC	MR	Rx. time (min)	Test number	%CC	MR	Rx. time (min)	Test number	%CC	MR	Rx. time (min)	RPM	Temp. (°C)
1	0.5	6:1	60	9	1.0	6:1	60	17	1.5	6:1	60	500	60 °C
2		6:1	90	10		6:1	90	18		6:1	90		
3		10:1	60	11		10:1	60	19		10:1	60		
4		10:1	90	12		10:1	90	20		10:1	90		
5		15:1	60	13		15:1	60	21		15:1	60		
6		15:1	90	14		15:1	90	22		15:1	90		
7		20:1	60	15		20:1	60	23		20:1	60		
8		20:1	90	16		20:1	90	24		20:1	90		

beaker, adding 2–3 drops of phenolphthalein as indicator and 10 ml of isopropyl alcohol. The titration was made with a 0.1 N solution of KOH marking the end point when the solution turned pink for ten seconds (Sudhir et al. 2007).

Transesterification

Samples of 100 g of WCO were preheated in a beaker to add the methoxide and covered with aluminum foil to prevent hydration. The temperature was measured, and once 60 °C was reached, the reaction time was started with constant agitation (500 rpm) for final yield evaluation at 60 or 90 min, respectively, according to the reaction conditions for each experiment (Table 1). Once two phases were clear and defined (upper phase: biodiesel; lower phase: glycerol), the separation was carried out by decantation funnel followed by measure of the product of the upper phase and storage in plastic containers. Glycerol was stored separately.

The final conversion yield was determined using the theoretical value of the biodiesel and the biodiesel obtained after the washing process with its molar weight and density, using the following formula:

$$\%\text{yield} = \frac{\text{Real yield}}{\text{Theoretical yield}} \times 100$$

where the real yield equals the volume of biodiesel obtained in each sample and the theoretical yield equals the theoretical volume of produced biodiesel, which is calculated from the molar weight (X g/mol) and density (X g/ml) of the samples (Sales, 2011).

Biodiesel washing

Two washing methods were selected for this work. The shaking technique consists in preparing a 1:3 dilution with distilled water:biodiesel to be maintained at constant agitation for 5 min followed by rest periods of 60 min. The second technique was bubble washing, in which air bubbles were passed through the sample via aeration stone and air pump for 60 min followed by 60-min rests, both techniques were repeated in triplicate or until the washing water was clear.

Quality determination

To determine the quality properties of the biodiesel produced (B100 and B20 mix with petrodiesel), a series of test reported by several authors were selected as some of the most important parameters in the finished biodiesel (Table 2), such as: physical description (color and appearance), conversion test 27/3 (used as a complementary test to the FTIR analysis), pH (ASTM D6423), density (ASTM D 1298-99), total acid number (ASTM D 6751), cloud point (ASTM D 2500), pour point (ASTMD 97), viscosity (ASTM D445), flash point (ISO 3679:2015), water and sediment (ASTM D 1796), all with standard values given in Table 2 (Canacki and Sanli 2008; Chhetri et al. 2008; Hossain and Boyce 2009).

Fourier transform infrared spectroscopy (FTIR)

In order to confirm the reliability of the biodiesel samples obtained, they were analyzed through a Nicolet$^{\text{TM}}$ iS 10 FTIR spectrometer in a range of 400–4000 cm^{-1}. Each sample had two replicates and was loaded placing one drop of the sample on the ATR and cleaned with solvent using a tissue. All the spectra were normalized to eliminate the differences in intensity from concentration variations.

Results and discussion

Free fatty acids and neutralization

The three mixes of oils used in this work were exposed to high temperatures, different heating patterns and turnover

Table 2 B100 standard limits

Test	Method	Standard limit
Color and appearance	Small-scale production	Yellow to brown
Conversion	27/3	1 clear phase
pH	ASTM D 64 23	6–8
Density (at 15 °C g/cm^3)	ASTM D 1298	0.875–0.900
Viscosity	ASTM D445	1.9–6.0 mm^2/s
TAN (mg KOH/g)	ASTM D 664	Max. 0.8
Cloud point (°C)	ASTM D 2500	NA
Pour point (°C)	ASTM 97	NA
Flash point (°C)	ISO CD 3679	Min. 130
Water and sediment (vol%)	ASTM D 1796	Max. 0.05

rates, all, affecting directly in the degradation of the oil composition, formation of new compounds and elevation of the value of the FFA (Rodrigues Machado et al. 2007), a key parameter when producing biodiesel.

Variation in the acid number was found among the samples ranging from 8.79 for sample 1 (C1), 2.74 for sample 2 (C2) and 7.22 for sample 3 (C3) all expressed in % of oleic acid. These values can be explained from the differences in cooking processes and the quality of the original feedstock. According to Kulkarni and Dalai (2006) if the FFA content exceeds 1–3 %, the use of a pre-treatment is highly recommended in order to succeed in the transesterification process; among the pre-treatment of choice, acid esterification is considered the best route to convert the FFA into esters; however, it can lead to the formation of water and to sample loss of up to 4 % (Ahmad et al. 2010; Van Gerpen 2005; Kumar et al. 2007; Kombe et al. 2012).

Although alkaline neutralization can lead to values of sample loss of up to 20 % of volume, when done adding the exact amount of catalyst, it can lead to no sample loss. This technique requires precision and an adequate fatty acid profile of the oil sample (Table 3), for both, an excess as an insufficiency in the amount of catalyst added results in the formation of soaps (Refaat 2010). Considering that there is a direct relation between the FFA percentage and the amount of catalyst that has to be added to the reaction to compensate the acidity and its deactivation (Freedman et al. 1986), alkaline neutralization was considered for the three samples as pre-treatment method for its effectiveness in lowering the FFA content, along with substantial quantities of mucilaginous substances, phospholipids and color pigments (Bhosle and Subramanian 2005; Sudhir et al. 2007).

Table 3 Fatty acid profile, oil molecular weight and %FFA

Fatty acid	Sample C1	Sample C2	Sample C3
Lauric acid	0.11		
Meristic acid	0.71		0.82
Palmitic acid	11.61	13.16	34.76
Palmitoleic acid	0.75	1.27	2.08
Margaric acid	0.28		
Margaroleic acid	0.25		
Stearic acid	5.47	4.73	5.16
Oleic acid	52.65	30.92	44.52
Linoleic acid	23.52	45.14	12.67
Linolenic acid	2.24	4.78	
Araquidic acid	0.36		
Gadoleic acid	0.71		
Molecular weight	872.16	868.84	846.24
%FFA	8.79	2.74	7.22

This neutralization method was only successful for samples C1 and C2 allowing successful transesterification in 15 and 11 of the 24 experiments designed for each, respectively. However, for sample C3 the high amount of saturated fatty acids in the sample prevented the use of this technique and an acid esterification was needed as pre-treatment.

Acid esterification

For sample C3, no biodiesel was obtained from the oils neutralized with an alkaline catalyst. The samples were subjected to an acid esterification as used by several authors (Canacki and Van Gerpen 2001; Talebian-Kiakalaieh et al. 2013) in order to obtain esterificable molecules and in turn reduce the FFA to continue with alkaline transesterification. From this process, a reduction in the FFA content from 7.22 to 2.73 % and obtainment of biodiesel for 4 of the 24 experiments for C3 through alkaline transesterification was achieved.

Biodiesel yield

Biodiesel was obtained from the samples provided by the three companies. From a primary screening (data not shown), KOH was selected as the best catalyst (Refaat et al. 2008) and proceed to continue with it for the optimization process. Out of the 72 reactions, the highest initial yield results were of 76.99 % for C1, 95.58 % for C2 and 89.95 % for C3, and selected this biodiesel samples to test for quality parameters. All the optimum parameters established are presented in Table 4.

Optimization parameters

Temperature and stirring time

While the temperature has no detectable effect on the production of esters, high temperatures reduce the reaction time to reach maximum conversion (Pinto et al. 2005), and since the transesterification can be carried out both at room temperature and up to the boiling point of methanol (64.7 °C) (Van Gerpen 2005), the range commonly used for transesterification is 60–65 °C which presents results in a time range between 30 and 90 min (Agbajelola et al. 2015). These results were consistent with the observations of maximum conversion of methyl esters establishing 60 °C and 90 min as optimal obtainment parameters for companies C1 and C2 and 60 °C and 60 min for company C3.

Table 4 Optimal parameters determined for the highest biodiesel yield

Sample number	Catalyst	%CC	MR	Time (min)	RPM	Temp. (°C)	Initial yield (%)	Final yield[a] (%)
C1	KOH	0.5	10:1	90	500	60	76.99	66.37
C2		1	10:1	90			95.58	85.53
C3		0.5	15:1	60			89.95	81.12

[a] The final yield specified refers to the biodiesel obtained after the washing process

Molar ratio

Alcohols are the most frequently used acyl-acceptors in transesterification reactions and include methanol, ethanol, propanol, butanol, amyl alcohol and octanol (Fukuda et al. 2001). According to the stoichiometry of the transesterification reaction, 3 mol of alcohol is needed for each mole of triglyceride to obtain 3 mol of methyl esters and 1 mol of glycerol (Highina et al. 2011), and since the reaction is reversible, it is recommended to use an excess of alcohol, increasing the molar ratio to 6:1 ranging up to 35:1 or greater depending on the vegetable oil used (Freedman et al. 1986; Meka et al. 2007; Gupta et al. 2007; Kang and Wang 2013).

The alcohol selected for its short chain, cost and performance in the transesterification reaction was methanol, which was handled in four molar ratios: 6:1, 10:1, 15:1 and 20:1. The analysis began with 6:1 for which no production of biodiesel in any of the samples analyzed was attained; due to the nature of the raw material (WCO with high value of FFA), the molar ratios were increased testing 10:1, 15:1 and 20:1. Such scaling was based on the relation between the increasing molar ratios and the increase in biodiesel yield observed by Singh et al. (2006).

The highest production yields (Fig. 1) for sample C1 and sample C2 had a molar ratio of 10:1, whereas for sample C3, the optimum molar ratio was 15:1 from which a trend was observed that evinces a decrease when the highest molar ratio (20:1) was tested, likely due to an excess of alcohol in the reaction that makes the separation of glycerol and the recovery of methyl esters more complicated (Schuchardt et al. 1998) and in time decreasing the methyl ester yield.

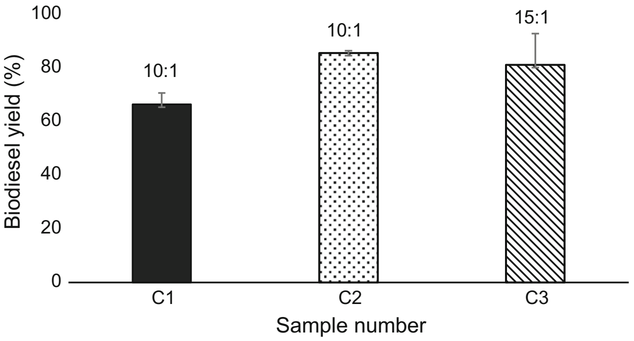

Fig. 1 Molar relation from the samples with the highest final biodiesel yield

Type and catalyst concentration

Two homogeneous alkaline catalysts, sodium hydroxide (NaOH) and potassium hydroxide (KOH), were tested considering that are the most widely used in transesterification for its low cost, high catalytic activity and ease in their operating and storage conditions (Refaat 2010). As stated earlier, a primary screening with both catalysts was performed, obtaining the best results with KOH (Tomasevic and Siler-Marinkovic 2003, Encinar et al. 2007; Demirbas 2009).

The catalyst (KOH) was tested at three concentrations, 0.5, 1 and 1.5 %, observing a trend when increasing the concentration at 1.5 % (Fig. 2), in which the methoxide and the mixture of oil acquired a viscous concentration that presented difficulty in agitation. This indicated that the reaction had reached the saturation point and tended to favor the formation of gels that hinder the recovery of glycerol and decrease biodiesel yield (Encinar et al. 2005). These results corresponded to those reported by Refaat, (2010) from which the best performance is obtained within a range of 0.5–1 %.

Biodiesel washing

Once the transesterification is complete and the two phases are separated, it is highly recommended to wash the biodiesel samples to eliminate any residual inorganic salts.

Two washing methods were used in this study: bubbling and shaking (Fig. 3). Both methods were selected for their low cost and accessibility and used with distilled water at environmental temperature (25 °C).

From the comparison, the first method presented a higher volume loss equivalent up to 15 % of biodiesel volume after the third wash (Fig. 4). As reported, in this method, the bubbles created do not necessarily generate an aqueous interface between the air and the biodiesel and can hide an incomplete reaction (Homer and Hunter 2014); however, due to its gentle approach, this technique does not create emulsions. With the second method, minor

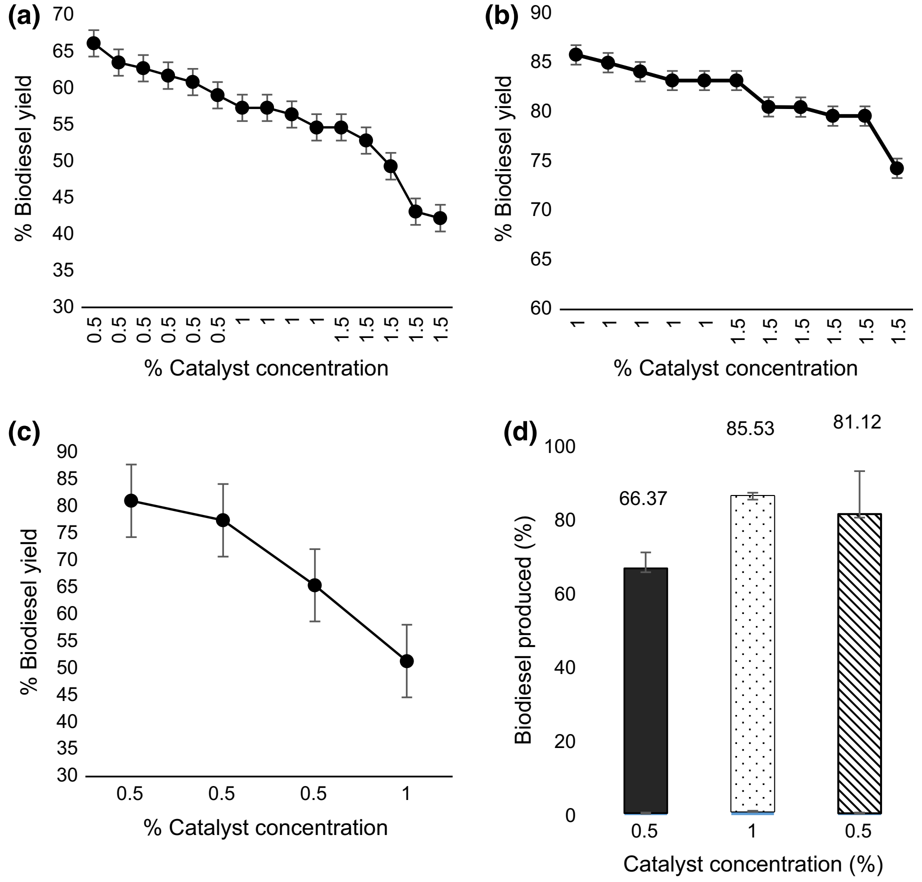

Fig. 2 Catalyst concentration trend: **a** sample C1, **b** sample C2, **c** sample C3 and **d** highest biodiesel yields obtained (%)

percentage losses were obtained, even when the contact area of the biodiesel and water was reduced. The results had a range from 6.5 to 10 % of the total volume sample after the third wash, and considering that this method requires less time and is less expensive, it was chosen for further experiments.

Quality tests

The evaluation of quality parameters was done in the samples with the highest final production yield (66.37 % for C1, 85.53 % for C2 and 81.12 % for C3), right after the samples were washed. Quality for B100 and B20 mixes for the three samples was evaluated. The results obtained are given in Table 5 for B100 and B20.

Regarding color and appearance, all samples were clear ranging from yellow to brown.

For the conversion test also known as the 27/3, all samples were positive, making use of this test as a complement to the analysis made by FTIR. This test is used often by small-scale biodiesel [Parker (no year)] producers and is based on the principle of miscibility of biodiesel in

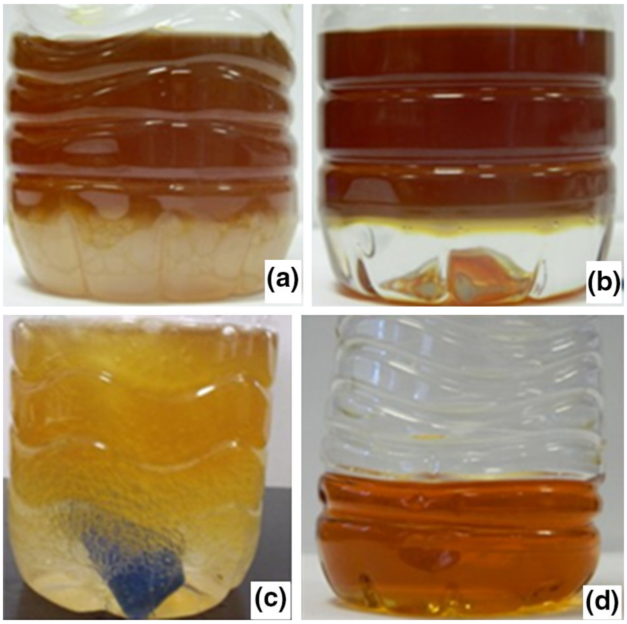

Fig. 3 Washing methods: **a** agitation method (first wash), **b** agitation method (third wash), **c** bubbles passing through sample and **d** biodiesel after final bubble wash

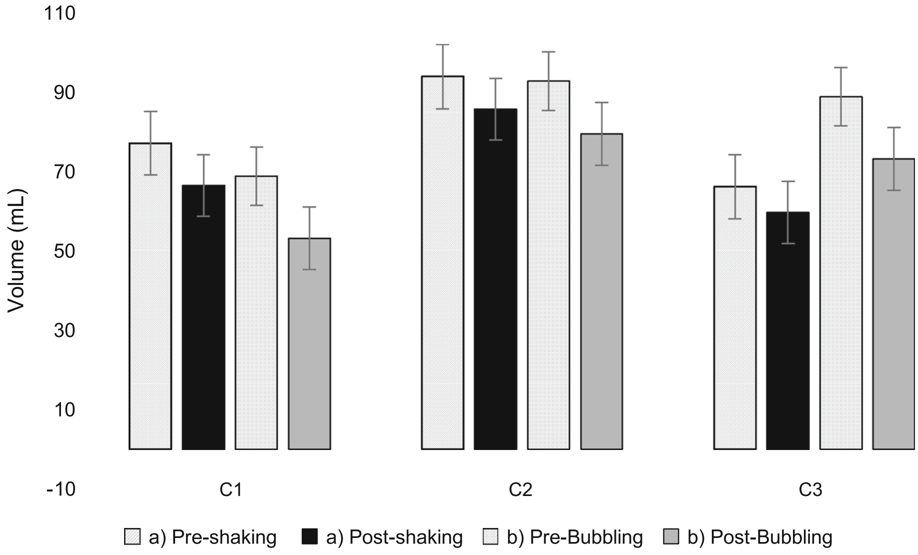

Fig. 4 Washing methods yield: **a** shaking method and **b** bubbling Method

Table 5 Quality parameters for B100 and B20 samples

Tests	B100[a]			B20[b]		
	Samples					
	AC1	AC2	AC3	BC1	BC2	BC3
Color and appearance	Yellow	Brown	Yellow	Yellow	Brown	Yellow
Conversion (27/3)	√	√	√	√	√	√
pH	6.5	6	6.5	6.5	6	6.5
Density (at 15 °C g/cm^3)	0.873	0.867	0.867	0.843	0.843	0.845
Viscosity (mm^2/s)	5.8	5.7	5.8	4.0	4.1	4.0
TAN (mg KOH/g)	0.3	0.4	0.3	0.4	0.4	0.4
Cloud point (°C)	2	13	12	1	3	1
Pour point (°C)	−2	5.5	6	−7	−7	−7
Flash point (°C)	163	177	168	80	78	83
Water and sediment (vol%)	0.01	0.02	0.02	0.01	0.02	0.02

[a] B100: pure biodiesel samples (AC

[b] B20: biodiesel mix (BC) (80 % petrodiesel, 20 % biodiesel)

√, positive conversion

methanol, when positive, 1 clear phase is observed and indicates the absence of unreacted oil. All samples had pH and density within the normal limits.

Viscosity

The high viscosity of oils make them not suited for direct use in diesel engines (Chhetri et al. 2008); however, the conversion process (transesterification) allows the fuel viscosity and quality properties to shift and be comparable to those of petrodiesel. The results obtained from the samples with the highest biodiesel yield, all fell within the established limits of the ASTM (1.9–6–0 mm^2/s), with a range between 5.7 and 5.8 mm^2/s for B100 and 4.0–4.1 mm^2/s for B20 samples.

TAN

All the results attained for this parameter were found below the maximum level permitted in the ASTM D 664 norm. Measuring total acid number (TAN) is one of the most important parameters measured given that a high acidity can affect the injection system specifically rubber hoses (Hossain and Mazen 2010).

Cloud and pour point

In this work, cloud point was evaluated as the temperature at which wax crystals are formed (Ali et al. 1995) and pour point as the lowest temperature at which a fuel can flow (Lee et al. 1995).

This parameter is directly related to the fatty acid composition of the sample, the higher the unsaturation rate, the lower the cloud point. Considering that a high degree of unsaturation influences the crystallization temperature by having double bonds in their structure, the spatial arrangement is disruptive and alters their packing ability and in consequence affects the crystallization capacity (Nascimento et al. 2005).

For the samples C1 and C3, the predominant fatty acid was oleic acid, while for sample C2, the predominant fatty acid was linoleic acid. Both fatty acids present instaurations, and as predominant compounds, it is expected a direct improvement in this particular parameter (Canacki and Sanli 2008). The sample from C1 had the lowest cloud and pour point out of the three which corresponded with the highest degree of instaurations in the fatty acid.

The determination of this parameters is of importance even when a limit is not established in the international standard, especially when the region where the fuel is produced can periodically have low temperature that affects flowing capacity.

Flash point

The results for all the samples analyzed fell within the normal limits of flash point. This parameter is of significance when it comes to safe management of biodiesel; having a higher flash point than petrodiesel gives it an advantage when it comes to storage and use.

Water content

By using WCO as feedstock, the presence of water in the samples was expected due to handling processes, and knowing that the water in the WCO accelerates the hydrolysis and simultaneously reduces the formation of esters (Arun et al. 2011), it is vital a filtering and heating process in order to ensure an anhydrous medium which allows us to avoid the negative effects of the compounds formed during frying (Refaat 2010). The water content was determined for all the biodiesel samples within the normal limits.

FTIR analysis

Analysis through infrared spectroscopy is useful as a detector of conversion of oil to biodiesel given that the characteristic groups that integrate this biofuel have strong absorbance at distinctive regions. As reported, the detection of this peaks (1744, 1436 cm^{-1}) don't give a direct measure from which the transesterification can be monitored; however, it gives a clear indication of the union of the alcohol with the fatty acids forming the desired methyl esters (Lin-vien et al. 1991; O'Donnell et al. 2013).

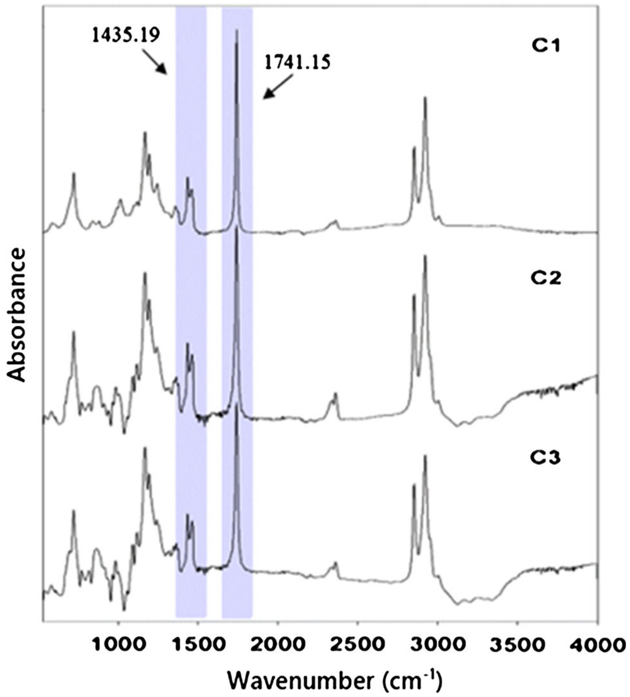

Fig. 5 Infrared spectra for all three samples of biodiesel B100: *C1* sample 1, *C2* sample 2 and *C3* sample 3

The B100 samples that presented the highest yields were analyzed by FTIR at final yield time (Fig. 5). Every sample had two replicates that were normalized. Characteristic groups that present in biodiesel, such as the carbonyl ester group with an infrared absorption of 1745 cm^{-1} and a methyl ester group at 1435 cm^{-1} were observed for all three samples of pure biodiesel (Bradley Appl. Note).

Conclusion

Biodiesel production from waste cooking oils with high FFA% derived from fast food companies was successful using both alkaline neutralization and two-step catalysis. Variables like type and concentration of catalyst, molar ratio and temperature played a key role on the yield of the transesterification reaction. The optimum amount of catalyst was within the 0.5–1 % range. The molar ratios for highest production were 10:1 and 15:1. Initial biodiesel yields were 76.99, 95.58 and 89.95 % for samples C1, C2 and C3, respectively, all with quality parameters within the standard limits. The agitation method for washing the samples was selected having a loss range between 6.5 and 10 % in volume. The use of a low-cost oil feedstock conversion to biodiesel was confirmed by FTIR where the characteristic ester and carbonyl groups were detected, concluding that the one-step alkaline neutralization method for waste cooking oil with high FFA% content can be a viable option for biodiesel production obtaining a production price of $2.65 dollars per gallon of biodiesel produced.

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Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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