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Biodiesel Production through Transesterification of Poultry Fat at 30 °C

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Biodiesel is an alternative fuel to fossil diesel whose major disadvantage is the dubious economical viability of the production process, due to the high raw material costs. In the work here reported, biodiesel was produced from poultry fat, a potentially good alternative raw material due to its low price and availability. The specific objectives of the work were (i) to study biodiesel production from poultry fat by homogeneous alkaline transesterification; (ii) to evaluate and improve the parameters affecting the reaction, namely, temperature, time, methanol to fat molar ratio, and concentration of the catalyst; and (iii) to evaluate the quality of the obtained biodiesel, according to the European biodiesel quality standard EN 14214. The product yield varied from 73 to 86 wt %. Regarding the product quality, independently of the reaction conditions used, density, acid value, flash point, copper corrosion, linolenic methyl ester content, and iodine number fulfilled EN 14214; however, viscosity and purity were not fulfilled at all conditions. Considering the parameters studied, the results obtained showed that the biodiesel produced at 30 °C during 90 min with a methanol to fat molar ratio of 6:1 and 1.0 wt % of NaOH as the catalyst obeyed all the quality specifications evaluated.

1. Introduction

More than 80% of the world energy consumption depends on traditional energy sources such as coal, petroleum, and natural gas. Petroleum alone represents about 39%, being a nonrenewable source responsible for much environmental damage, including greenhouse gases emissions.[1] For this reason, it is essential to find alternative energy sources.

Biodiesel is an alternative fuel to fossil diesel and its use contributes to attenuate current energetic problems. It is renewable, being produced from triglycerides sources such as vegetable oils and animal fats. Commonly, a transesterification reaction with methanol is performed to produce biodiesel (a mixture of fatty acid methyl esters, FAME) and glycerol as byproduct. Because of the fact that biodiesel presents similar properties as fossil diesel, it can be mixed to be used in compression-ignition engines; the biodiesel percentage in the mixtures generally varies from 5% (B5) to 30% (B30), being also possible to be used in the pure form (B100). This biofuel is biodegradable, nontoxic and has a more favorable combustion emission profile,[2] leading to a reduction of carbon dioxide, carbon monoxide, particulate matter, and unburned hydrocarbon emissions as compared with fossil diesel. Only NO_x emissions have been reported to increase, which is due to the high oxygen content of biodiesel that provides additional oxygen for NO_x formation.[3]

One of the major disadvantages of biodiesel production is its dubious economical viability, essentially due to the high costs of the currently used raw materials,[4,5] usually high quality food-grade vegetable oils like soybean and rapeseed oils.[6] The use of low cost feedstock, such as animal fats, will reduce biodiesel production costs and additionally might be a friendly environmental alternative to recycle wastes that have a very limited commercial value.

The production of poultry meat has increased significantly in recent years, and the recycling of fatty wastes resulting from its slaughter has been the subject of study since their incorporation in animal diets was restricted;[7] consequently, poultry fat is being considered currently a potentially good low cost raw material for biodiesel production.

Up until now, there are few publications regarding the production of biodiesel from poultry fat; the most relevant are presented in Table 1, showing the reaction conditions including temperature, time, molar ratio of methanol to fat, and concentration of catalyst used. Wyatt et al. produced biodiesel from poultry fat through alkaline transesterification using the most common temperature (65 °C) and a reaction time of only 0.5 h; the molar ratio of methanol to fat was 6:1 and 0.4 wt % of sodium hydroxide was used as the catalyst.[8]

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Table 1. Studies Regarding the Production of Biodiesel from Poultry Fat

T (°C)	t (h)	molar ratio of methanol to fat	catalyst type	catalyst amount	ref
65	0.5	6:1	NaOH	0.4%	Wyatt et al.8
25	6−7	72:1	NCO	1 mmol/15 mL	Reddy et al.9
120	8	30:1	MgAlH	10%	Liu et al.7
50	24	30:1	H_2SO_4	25%	Bhatti et al.10

Reddy et al. studied the conversion of poultry fat to biodiesel using a significantly lower temperature (25 °C), but the reaction time was much longer (6−7 h); the molar ratio of methanol to fat (72:1) was very high and nanocrystalline calcium oxide (NCO) was used as heterogeneous catalyst (1 mmol dissolved in 15 mL of methanol).[9] Liu et al. also studied the transesterification of poultry fat with methanol; higher reaction temperature and time were used (120 °C, 8 h), as well as high molar ratio of methanol to fat, 30:1; Mg−Al hydrotalcite (MgAlH) derived heterogeneous catalysts were used.[7] Bhatti et al. studied the production of biodiesel from chicken tallow at 50 °C, during 24 h, using a high molar ratio of methanol to fat (30:1) and 25 wt % of sulfuric acid as the catalyst.[10] Regarding the production of biodiesel from poultry fat, the analysis of these publications allowed concluding that the current knowledge concerning the better conditions for transesterification, aiming to produce a biodiesel according to the quality standards, is yet very scarce. Aiming to improve that knowledge, namely, concerning the reduction of energetic and material costs to produce biodiesel, the objective of the work here reported was (i) to study biodiesel production from poultry fat by homogeneous alkaline transesterification; (ii) to evaluate and optimize the parameters affecting the reaction, namely, temperature, time, methanol to fat molar ratio, and concentration of the catalyst; and (iii) to evaluate the quality of the obtained biodiesel, according to the European standard EN 14214.

2. Experimental Section

2.1. Materials. Waste poultry fats were provided by a local butchery "Talho Carlota", located in Santa Maria da Feira, Portugal. Fats were melted by heating at a temperature higher than 100 °C and then filtered under vacuum to remove suspended matter. The reagents used in the synthesis and purification of biodiesel were sodium hydroxide powder 97% (reagent grade, Aldrich), methanol 99.5% (analytical grade, Fisher Scientific), and anhydrous sodium sulfate 99% (analytical grade, Panreac).

2.2. Methods. *2.2.1. Raw Material Characterization.* The following properties of the raw material were determined: (i) acid value, by volumetric titration according to the standard NP EN ISO 660 (2002) and (ii) iodine number, by volumetric titration with Wijs reagent according to the standard ISO 3961 (1996).

2.2.2. Biodiesel Production. Biodiesel production was performed in two steps: synthesis and purification. Alkali transesterification of poultry fat was carried out using different experimental conditions by varying the catalyst concentration, methanol to fat molar ratio, reaction time, and temperature.

Table 2. Reaction Conditions Used in the Experiments

catalyst concentration (wt %)	methanol to fat molar ratio	time (min)	temperature (°C)
0.4	6:1	60	60
0.6	6:1	60	60
0.8	6:1	60	60
1.0	6:1	60	60
0.8	3:1	60	60
0.8	9:1	60	60
0.8	12:1	60	60
0.8	6:1	30	60
0.8	6:1	90	60
0.8	6:1	120	60
0.8	6:1	60	30
0.8	6:1	60	40
0.8	6:1	60	50

Reference conditions were chosen: 60 °C, 60 min, 6:1 methanol to fat molar ratio, and 0.8 wt % of NaOH.[11,12] When one parameter varied, the other three were maintained in the reference conditions according to what is presented in Table 2.

2.2.2.1. Synthesis. All reactions were performed in one reactor consisting of 1 L flat-bottom flask immersed in a temperature controlled bath equipped with a water-cooled condenser and a magnetic stirrer.[11] The fat was melted at 110 °C to remove the residual water, cooled to the reaction temperature, weighed, and then added to the reactor. The reaction started when a defined amount of methanol premixed with the catalyst was added to the reactor containing 150 g of fat at the reaction temperature. The reactor was always kept under vigorous mixing. At the end of the reaction, products were left to settle for 1 h for separation between biodiesel and glycerol.

2.2.2.2. Purification. After biodiesel and glycerol separation, excess methanol was recovered from each phase separately, by rotary evaporation under reduced pressure. Then, biodiesel was purified to remove the dissolved NaOH, first washing with 50 vol % of an acid solution (0.2 wt % HCl) and then successively washing with 50 vol % of distilled water until the pH of the washing water was the same as the distilled water. Afterwards, biodiesel was dehydrated with 25 wt % of anhydrous sodium sulfate; the biodiesel and the salt were stirred for 30 min and then the solution was filtered under vacuum to obtain the purified product. After purification, biodiesel production yield (weight of product/weight of fat × 100) was determined.

2.2.3. Biodiesel Quality Evaluation. The biodiesel quality was evaluated according to the European standard EN 14214 (2003). The following properties were determined: (i) density at 15 °C, using a hydrometer method according to the standard EN ISO 3675 (1998); (ii) flash point, using a rapid equilibrium closed cup method, according to the standard ISO 3679 (2004); (iii) copper corrosion, using a copper strip test according to the standard ISO 2160 (1998); (iv) acid value, by volumetric titration according to the standard EN 14104 (2003); (v) water content, by Karl Fischer coulometric titration according to the standard NP EN ISO 12937 (2003); (vi) kinematic viscosity at 40 °C using glass capillary viscometers according to the standard ISO 3104 (1994); (vii) ester and linolenic acid methyl ester contents, by GC according to the standard EN 14103 (2003); and (viii) iodine value, determined from ester content according to annex B of EN14214 (2003). Regarding chromatographic analysis, a Dani GC 1000 DPC gas chromatograph (DANI Instruments S.p.A.) with an AT-WAX (Heliflex capillary, Alltech) column was used.

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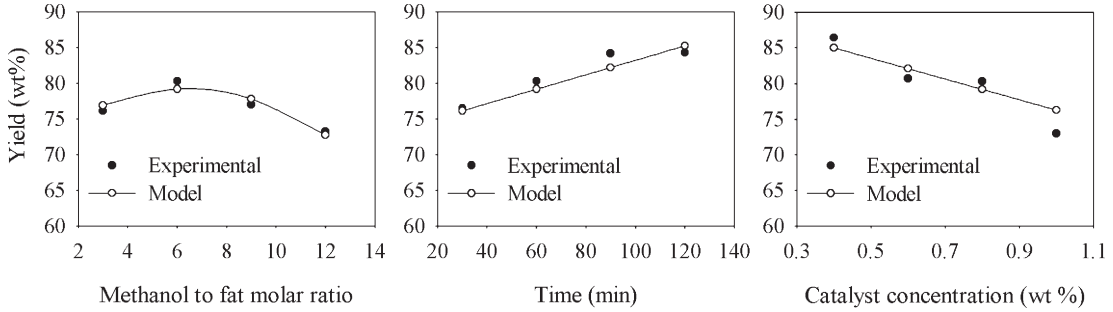

Figure 1. Experimental and model results concerning the influence on biodiesel yield of the methanol to fat molar ratio (0.8 wt % catalyst, 60 °C, 60 min), time (0.8 wt % catalyst, 60 °C, methanol to fat molar ratio of 6:1), and catalyst concentration (60 °C, 60 min, methanol to fat molar ratio of 6:1).

Table 3. Yield of Biodiesel Produced under Different Reaction Conditions

catalyst concentration (wt %)	T (°C)	t (min)	methanol to fat molar ratio	yield (wt %)
0.4	60	60	6:1	86
0.6	60	60	6:1	81
0.8	60	60	6:1	80
1.0	60	60	6:1	73
0.8	60	60	3:1	76
0.8	60	60	9:1	77
0.8	60	60	12:1	73
0.8	60	30	6:1	77
0.8	60	90	6:1	84
0.8	60	120	6:1	84
0.8	30	60	6:1	77
0.8	40	60	6:1	83
0.8	50	60	6:1	79

The injector temperature was set at 250 °C, while the detector (FID) temperature was set at 255 °C. The following temperature program was used: 120 °C, heating at a rate of 4 °C min^{-1} until 220 °C and holding at that temperature for 10 min.[11]

2.2.4. Statistical Analysis. The experimental results were considered to be statistically valid at $p < 0.05$ (using the Student's t- test). In order to understand the influence of the studied variables on biodiesel yield and purity, a statistical analysis using JMP5 software was performed; the developed models were considered to be statistically valid when the obtained parameters showed a p value less than 0.05 using an F-test.

3. Results and Discussion

3.1. Raw Material Characterization. The yield of fat extraction from poultry wastes was 40 wt %. The characterization of the fat showed an acid value of 0.92 mg KOH/g and an iodine number of 80 g of I_2/100 g. The acid value of the raw material suggested a good alkaline transesterification due to reduced risk of catalyst consumption by free fatty acids; the iodine number was in agreement with other published results for poultry fat.[13]

3.2. Biodiesel Production Yield. Biodiesel production yields varied between 73 and 86 wt %. The results for all experiments are presented in Table 3.

The results showed that an increase of the catalyst concentration led to lower yields which might be associated with higher soap formation through reaction between sodium hydroxide and free fatty acids of the raw material. Higher soap formation will make the washing step difficult, leading to product losses and therefore lower yields.[6] The influence of reaction temperature on the yield did not show a trend, but reaction time significantly increased the yield. The highest methanol to fat molar ratio used was associated with the lowest yield; the increase of methanol content increases the glycerol solubility in the ester phase, making the separation more difficult and consequently reducing the yield.[14]

The obtained results confirmed that different variables influenced biodiesel production yield; a statistical analysis using JMP5 software was further performed aiming to find the best model describing the influence of the studied variables, individually or combined. A predictive model was obtained and the corresponding coefficients were calculated from the experimental responses through least-squares regression using standardized data. The model which better described the data was a second-degree polynomial with $r^2 = 0.85$. The statistically significant ($p < 0.01$) coefficients obtained were −0.704, 2.14, and −2.49, respectively, for the quadratic effect of methanol to fat molar ratio and for the linear effect of time and catalyst concentration; the intercept was 80.9. The second degree term relative to the methanol to fat molar ratio showed that a maximum yield could be achieved, after which an increase in the methanol to fat molar ratio caused a yield decrease. The predictive model showed that, within the experimental range, the yield varied linearly with both time and catalyst concentration, increasing with time and decreasing with catalyst concentration. Figure 1 shows the experimental results for each variable as well as the results predicted by the model.

The models which accounted for the temperature effect and also the ones which considered the combined effects of the studied variables led to coefficients that were not statistically significant ($p > 0.05$). Therefore, considering the use of the obtained predictive model, no conclusions could be taken regarding the influence on the biodiesel yield either of the temperature or of the combined effects of the studied variables; accordingly, the results seemed to indicate that the individual effects had the greatest impact on the biodiesel yield.

3.3. Biodiesel Composition. Biodiesel composition (Table 4), which relates with the composition of the waste poultry fat used, was determined by gas chromatography. The methyl esters contained in the biodiesel were identified by comparing retention times with those of chromatographic standards, the results being according to previously published results.[13,15]

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(14) Sulisto, H.; Rahayu, S. S.; Winoto, G.; Suardjaja, I. M. Biodiesel Production from High Iodine Number Candlenut Oil. *World Acad. Sci., Eng. Technol.* **2008**, *48*, 485–488.

(15) Tang, H.; Salley, S.; Simon, K. Y. Fuel properties and precipitate formation at low temperature in soy, cottonseed, and poultry fat-based biodiesel blends. *Fuel* **2008**, *87*, 3006–3017.

Table 4. FAME Composition of Biodiesel and Mean Molecular Weight

FAME	composition (wt %)
myristate (C14:0)	0.60
palmitate (C16:0)	2.4
palmitoleate (C16:1)	5.8
stearate (C18:0)	5.5
oleate (C18:1)	41
linoleate (C18:2)	21
linolenate (C18:3)	1.4
arachidate (C20:1)	0.40
$M_{\text{Biodiesel}}$ (g/mol)	290

Table 5. Biodiesel Quality Properties

properties	range of results	EN 14214
density at 15 °C (kg/m^3)	878−885	860−900
acid value (mg KOH/g)	0.091−0.24	< 0.50
flash point (°C)	170−178	> 120
copper corrosion	class 1a	class 1
linolenic acid methyl ester (wt %)	1.36−1.50	< 12.0
iodine number (g of I_2/100 g)	80.1−81.6	< 120
water content (mg/kg)	727−1390	< 500
kinematic viscosity at 40 °C (mm^2 s^{-1})	4.52−6.13	3.50−5.00
methyl ester content (wt %)	78.9−96.3	> 96.5

The mean molecular weight (290 g/mol), also shown in Table 4, corresponds to the weighted average (through the molecular fraction) of the molecular weight of the fatty acid methyl ester that constitute biodiesel.

3.4. Biodiesel Characterization. Table 5 presents the range of values obtained for the determined biodiesel properties. Density, acid value, flash point, copper corrosion, linolenic methyl ester content, and iodine number fulfilled the European biodiesel standard EN 14214, meaning that the selected process, independently of the variation of the studied parameters, was effective in obtaining biodiesel with acceptable quality regarding those properties. Water content was always higher than the maximum set by EN 14214 independent of the transesterification conditions used; this fact showed that the drying method selected should be improved or replaced by others showing greater effectiveness, such as evaporation under reduced pressure. Kinematic viscosity and methyl ester content varied among experiments and the reason why will be discussed in more detail this point forward.

3.4.1. Kinematic Viscosity at 40 °C. Kinematic viscosity of biodiesel is a measure of the internal resistance of the fuel to flow; therefore, it controls engine performance related with injection and combustion characteristics.[13] Higher viscosity leads to higher drag in the injection pump, causing higher pressures and injection volumes, especially at low engine operating temperatures. Consequently, the timing for fuel injection and ignition tends to be slightly advanced for biodiesel, compared with mineral diesel, which might in turn lead to increased emissions of nitrogen oxides.[13] Viscosity has been correlated with methyl ester content, being possible to use it as a biodiesel purity indicator.[16] It is closely related with the biodiesel composition, increasing with increasing length of both the fatty acid chain and the alcohol group. On the other hand, viscosity is inversely related to the number of double bonds of biodiesel constituents.[13]

(16) Allen, C. A. W.; Watts, K. C.; Ackman, R. G.; Pegg, M. J. Predicting the viscosity of biodiesel fuels from their fatty acid ester composition. *Fuel* **1999**, *78*, 1319–1326.

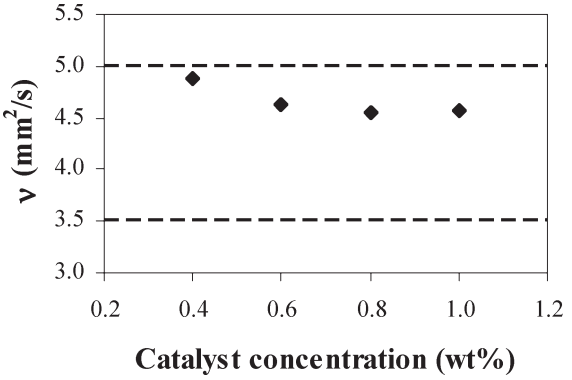

Figure 2. Influence of the catalyst concentration on biodiesel viscosity (60 °C, 60 min, methanol to fat molar ratio of 6:1); dashed lines indicate maximum and minimum limits according to European standard EN 14214.

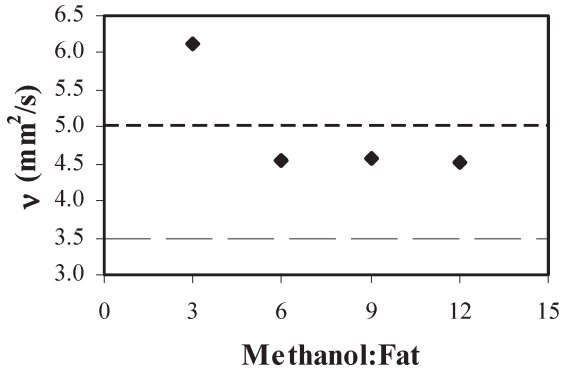

Figure 3. Influence of methanol to fat molar ratio on viscosity (60 °C, 60 min, 0.8 wt % NaOH); dashed lines indicate maximum and minimum limits according to European standard EN 14214.

The kinematic viscosity was always in agreement with the EN 14214 (3.5−5.0 mm^2 s^{-1}), ranging from 4.56 and 4.88 mm^2 s^{-1}. Figure 2 shows the influence of the catalyst concentration on the kinematic viscosity (ν) at 40 °C. As expected, it decreased with increasing catalyst concentration, more evident when the concentration increased from 0.4 to 0.6 wt %.

The influence of the methanol to fat molar ratio on viscosity is presented in Figure 3. The highest value was observed for a ratio of 3:1, and this fact was attributed to the incompleteness of the reaction when the stoichiometric ratio was used (it should be referred that in the end of this reaction over biodiesel some foam was observed, presenting a color and aspect (doughy) different from the usual); for the other molar ratios, viscosities did not vary considerably, showing that considering the biodiesel viscosity, 6:1 is an advantageous ratio.

Regarding reaction time, the increase from 30 to 120 min did not significantly influence viscosity that varied from 4.56 to 4.69 mm^2/s, always being in agreement with EN 14214.

As for reaction time, reaction temperature did not significantly influence viscosity; when temperature decreased from 60 to 30 °C, viscosity varied from 4.56 to 4.81 to mm^2/s, always in agreement with EN 14214. This was an important observation because most publications refer that the transesterification reaction should be processed between 60 and 70 °C; the results obtained showed that the transesterification reaction can be processed at 30 °C (allowing significant energetic savings) producing a biodiesel obeying the quality standard for viscosity.

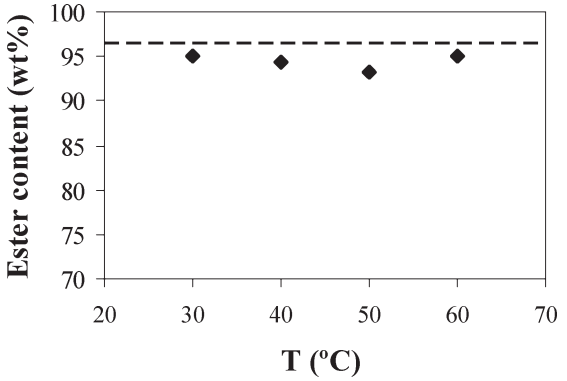

Figure 4. Influence of reactions temperature on ester content (methanol to fat molar ratio of 6:1, 60 min, 0.8% NaOH); dashed line indicates minimum limit according to European standard EN 14214.

3.4.2. Ester Content. The fatty acid methyl ester content, or ester content, is an indicator of the purity of biodiesel, and EN 14214 indicates a minimum of 96.5 wt % to ensure quality. This parameter is very important to prevent illegal mixture of other substances.[13]

The increase of catalyst concentration from 0.4 to 1.0 wt % did not significantly influence the ester content of biodiesel (94.4−95.3 wt %), which was very close to the minimum required but never reached it. With respect to the methanol to fat molar ratio, in agreement with what occurred for the kinematic viscosity, it was found that when the stoichiometric ratio (3:1) was used, the conversion was low and therefore the ester content was also very low (78.9 wt %). For all the other studied ratios, the ester contents were similar (95.1−95.7 wt %), showing more effective conversion. As for the viscosity, these results showed that molar ratios higher than 6:1 did not bring significant advantages concerning conversion.

The influence of reaction temperature on ester content (Figure 4) was not significant for the range of temperatures considered. When the reaction was performed at 30 °C, the ester content was 95 wt %, very close to the minimum required (96.5 wt %) but not reaching it.

Regarding reaction time, it was concluded that it also did not significantly influence the ester content; when reaction time increased from 30 to 90 min, the ester contents varied from 94.4 to 96.3 wt %, not reaching the minimum quality specification. In conclusion, further analysis should be conducted to ensure fulfilment of the quality standard regarding ester content. A statistical analysis using the JMP5 software was performed for the ester content, similar to what was done for the yield; however, because of the small variations found for most properties, none of the experimental obtained models were statistically valid.

3.5. Selection of Reaction Conditions. The results presented in the previous sections allowed concluding that using 0.8 wt % of catalyst, a molar ratio of methanol to fat of 6:1 and conducting the transesterification reaction at 30 °C during 90 min, the biodiesel obtained had an ester content very close to minimum required by EN 14214. With an attempt to improve the ester content, the catalyst concentration was increased conducting the reaction at 30 °C, during 90 min, and using a methanol to fat molar ratio of 6:1. The results obtained for catalyst concentration of 0.4, 0.6, and 1 wt % presented in Table 6 showed that at lower temperatures the influence of catalyst concentration is much more significant, as expected; increasing the catalyst concentration increased the ester content and decreased the viscosity. The biodiesel produced, conducting the transesterification reaction at 30 °C during 90 min with a methanol to fat molar ratio of 6:1 and 1.0 wt % of NaOH as the catalyst, obeyed the European quality standards, namely, in terms of viscosity and ester content.

Table 6. Influence of Catalyst Concentration on Kinematic Viscosity, Ester Content, and Yield (30 °C, 90 min, Methanol to Fat Molar Ratio of 6:1)

catalyst concentration (wt %)	kinematic viscosity at 40 °C (mm^2/s)	ester content (wt %)	yield (wt %)
0.4	5.54	85.2	87
0.6	4.72	92.5	84
1.0	4.53	96.5	81

It is known that biodiesel production from recycled wastes presents lower yields than when, for instances, virgin oils are used. Accordingly, when using poultry fat as raw material, the yields were not very high, being from 73 to 86 wt %, but such disadvantages can be compensated by the value added to the wastes through their recycling, the cost reduction on waste treatment, and the reduction of greenhouse gases emissions.

Compared with the revised works (Table 1), Liu et al. and Bhatti et al. used much more extreme reaction conditions (namely, concerning reaction temperature, time, methanol to fat molar ratio, and catalyst concentration) to produce biodiesel from poultry fat. Most publications state that the transesterification reaction should be processed between 60 and 70 °C. Only the work performed by Reddy et al. used a slightly lower temperature (25 °C) than the one used in the present work (30 °C) to produce biodiesel from poultry fat; however, the reaction time was much longer (6−7 h) and the amount of methanol used was also much higher (72:1), which will increase the production costs; the advantage of the heterogeneous catalysis related with catalyst reusing is also limited because after three cycles it was deactivated. Regarding the reaction time, Wyatt et al used only 0.5 h but at much higher temperature (65 °C); the energetic costs of heating tend to be determinant for the selection of reaction conditions, namely, when wastes are being recycled as raw material, the reason why the results obtained from this study are innovative and promising; in fact, significant reductions of energetic and material costs can be possible using poultry fat for biodiesel production under the selected reaction conditions.

This low temperature process might be extremely appealing for meat processing industries that produce this type of wastes and usually need to pay for its management. Nevertheless, the authors consider that the transesterification of poultry fat at low temperature for biodiesel production can and should be improved, aiming at a yield increase and reaction time reduction, the reason why further analysis will proceed, namely, through a more detailed kinetic study and by considering other type of catalysts.

4. Conclusions

The results obtained showed that the biodiesel produced, conducting the transesterification reaction at 30 °C during 90 min with a methanol to fat molar ratio of 6:1 and 1.0 wt % of NaOH as catalyst, obeyed the European quality standards, namely, in terms of viscosity and ester content. As most publications refer that the transesterification reaction should be processed between 60 and 70 °C, this conclusion showed that waste poultry fat might be extremely appealing as a raw material for biodiesel production because significant reductions of energetic and material costs can be possible.