

Low temperature behavior of poultry fat biodiesel:diesel blends

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ABSTRACT

As the worldwide consumption of poultry meat rises the use of poultry fat as a feedstock for biodiesel production becomes attractive considering economical and environmental reasons. However, poultry fat biodiesel still faces some restrictions due to its poor flow properties at low temperatures. In this study ethylic and methylic poultry fat biodiesels and their blends with diesel were evaluated in terms of flow properties. Modulated Temperature Differential Scanning Calorimetry (MT-DSC) was used to understand the physical meaning of properties as Cold Filter Plugging Point (CFPP), Pour Point (PP) and Cloud Point (CP), widely used in biodiesel characterization. Based on the MT-DSC studies, it was observed that the first crystallization peak temperature had values similar to CFPP and CP. This way CP was found to be associated with the first solidified material and not with the early formation of the first nuclei, as normally reported. On the other hand, these crystals already lead to the flow decrease, as indicated by the CFPP results. PP values were close to the second crystallization peak temperature, not being related to the complete solidification of the fuel.

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1. Introduction

Brazil occupies the third world position in the poultry meat production, after China and United States, with an average production estimated at 10.9 million tons per year. Occupying a leader position in terms of overseas exportation, Brazil is responsible for supplying 40% of the poultry meat world market.

Such position favors the availability of a high amount of inedible residual fats. This by-product is usually not reused by industries and traders enhancing solid waste problems and environmental pollution. The use of poultry residues as a fatty acid-rich feedstock of biodiesel production is promising since this sort of raw material is of relatively low cost, readily available, very rich in lipids and exhibits very attractive physico-chemical properties, such as high calorific power and cetane number [1–7].

The use of poultry fat biodiesel is however of limited application, mainly due to its solidification tendency, particularly in cold weathers. The presence of a high amount of saturated fatty acids in its composition is responsible for such low temperature performance as it tends to solidify even at room temperatures in cold regions. Thus, it becomes essential to evaluate the low temperature properties of biodiesel in order to gain a further insight into this

problem and develop new ways to overcome these difficulties which lead to engine damage due to the presence of micro-crystals in the fuel.

Geller et al. [1] studied the viscosity variation of poultry fat biodiesel and their biodiesel/diesel blends (B20, B40, B60 and B80) after a 12-month storage period under varied temperature ranges. Viscosity alterations have been noticed particularly for B80 blends while no meaningful changes in viscosity for blends up to B60 were observed. Sediment formation was found in all blends whose amount increased with biodiesel concentration.

Rheological properties of biodiesels from poultry fat, beef tallow, lard and yellow grease were already evaluated [2,3]. It was observed that all fats showed liquid–solid transitions between 40 and 48 °C being completely liquid at 50 °C. These studies also indicated that this sort of fats exhibited pseudo-plastic behavior.

Cold flow properties, such as Cold Filter Plugging Point (CFPP), Pour Point (PP) and Cloud Point (CP) were determined in order to investigate the formation of precipitates in biodiesel/diesel blends produced from poultry fat (PFB), soybean oil and cottonseed oil. Cold flow performance is related to the saturation and unsaturation degree of fatty acids. Saturated acids were found in higher proportion in PFB comparing with soybean and cotton biodiesels leading to lower flow at higher temperatures [7].

The crystallization temperature is a very important factor to be considered for applications in low temperature environments. Differential

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Scanning Calorimetry (DSC) has been shown to be an efficient method to investigate liquid–solid transitions, crystallization and melting characteristics including the crystallization temperature of biodiesel [8]. This technique has been used to characterize biodiesels from babassu, soybean, tallow, corn and linseed [8–10] but no works were found related to poultry fat.

In the present work, poultry fat ethylic and methylic biodiesel's (PFEB and PFMB, respectively) and their corresponding binary biodiesel–diesel blends (B5, B10, B15, B20 and B50) were studied. An attempt was made to gain a better understanding of their rheological and cold flow properties by associating these results with informations obtained from Modulated Temperature Differential Scanning Calorimetry (MT-DSC) and from Gas Chromatography/Mass Spectrometry (GCMS).

2. Experimental

2.1. Sample preparation

The pieces of poultry waste were locally supplied. Heating was done in an oven at 70–80 °C for fat extraction followed by occasional stirring for 2 h to complete liquefaction. Subsequently, the fatty blend was subjected to vacuum filtration to remove impurities.

The biodiesel samples were obtained by alkali-catalyzed (KOH) transesterification of the poultry fat, using methanol and ethanol routes [10,11]. A molar ratio of poultry fat:alcohol of 1:6 and 1.0% of catalyst were employed. The blends were prepared by addition of 5, 10, 15, 20, and 50% (v/v) of pure PFMB and PFEB into diesel (0.02% sulfur).

2.2. Chemical characterization

The poultry fat (PF) and the biodiesel samples (PFMB and PFEB, for the methylic and ethylic poultry fat biodiesels', respectively) were analyzed by Nuclear Magnetic Resonance Spectroscopy (¹H NMR) and Gas Chromatography/Mass Spectrometry (GCMS).

The results of ¹H NMR (200 MHz) were obtained in a VARIAN MERCURY spectrometer. Tetramethylsilane (TMS) was used as the internal standard and deuterated chloroform (CDCl₃) as the solvent.

GCMS analysis were done in a Shimadzu, model GCMS-QP2010, spectrometer, equipped with split injector and with auto-sampler. The capillary column used was Durabond – DB-SHT (Agilent Technologies). The carrier gas used was helium, using a flow rate of 3 mL min⁻¹ and an injection volume of 1 mL. The MS detector temperature of 250 °C was used. The characterization of the fatty acid occurred by comparing the mass spectra with existing patterns in the software library of the equipment (Mass Spectral Database NIST/EPA/NIH).

2.3. Physicochemical characterization

The cold flow properties and MT-DSC analyses were determined for biodiesel samples, diesel and their blends. Dynamic and kinematic viscosities were determined for all samples.

Regarding the cold flow properties, Cold Filter Plugging Point (CFPP), Pour Point (PP) and Cloud Point (CP) were determined. For CFPP analyses a TANAKA Model AFP-102 equipment was employed according to ASTM Test Method D6371-05 [12]. For CP and PP a TANAKA Model MPC – 102 L equipment was used. CP measurements were done according to ASTM D2500-09 [13] while PP measurements were done according to ASTM D 97-09 [14].

The dynamic viscosity measurements were conducted in a Brookfield LV-DVII viscometer, with a small sample adapter, using

an isothermal bath at 25 °C. The kinematic viscosity determinations were performed in accordance to the ASTM D445-97 [15] Test Method, using a Cannon Fenske calibrated viscometer.

The MT-DSC curves were obtained in non-isothermal conditions in a TA Instruments DSC 2920 using 10 mg of sample in nitrogen atmosphere heated from 40 to –60 °C and from –60 to 100 °C with a temperature modulation of ±1 °C min⁻¹.

3. Results and discussion

Fig. 1 shows ¹H NMR spectra. The triplet at $\delta = 2.32$ – 2.24 appeared in all fatty acid esters with small shifts to higher or lower values according to the chain attached to the ester oxygen. The signal at $\delta = 2.74$ was assigned to the bis-allylic hydrogens being specific of linoleic and linolenic fatty chains. The double doublet at $\delta = 4.31$ – 4.23 and from 4.15 to 4.06 was related to the H of the glyceride portion of the triglycerides. The signals at $\delta = 5.35$ – 5.24 were associated to the olefin protons confirming the presence of unsaturated C=C in all fatty chains.

As expected, the ¹H NMR spectrum of methyl and ethyl esters presented a profile similar to PF in the region of the hydrogens of the hydrocarbon chains ($\delta \sim 0.83$ to 3.00) considering the structural permanence of the chains after the transesterification reactions. The most characteristic sign of the methyl ester was the methoxyl singlet located at $\delta = 3.64$ distinguishing it from the PF and the ethyl ester.

The ¹H NMR spectrum of ethanol biodiesel confirmed the formation of ethyl ester by the presence of a quartet at 4.15 ppm that is assigned to the ethylene group protons of the alcohol ester. Almost no glycerides were found.

The fatty acid compositions of PF, PFEB and PFMB are shown in Table 1. Comparing with other animal fats such as lard and beef tallow, the fatty acid composition of poultry fat showed a higher proportion of unsaturated and polyunsaturated fatty acids in agreement with the literature [16]. The saturation degree of fatty acids may vary especially when the poultry feed diet composition contains a high percentage of saturated or unsaturated fats [17].

The chromatogram profiles of methanol and ethanol biodiesels displayed some quantitative differences. For instance, the linoleic acid content (C18:2) present in PFMB and especially in PFEB was significantly smaller than in PF while a higher amount of stearic acid (C18:0) was obtained. This indicates that the proportion of unsaturated chains reduced significantly after the transesterification reaction, especially in ethanol route.

Comparing the chromatograms with ¹H NMR spectra, similar results were obtained suggesting that the reduction in the chain size and in the unsaturation degree occurred making PF biodiesels more prone to crystallization.

Previous studies [7] demonstrated that several factors may affect the precipitated formation in biodiesel–diesel blends, such as storage temperature, storage time, biodiesel blend level, and

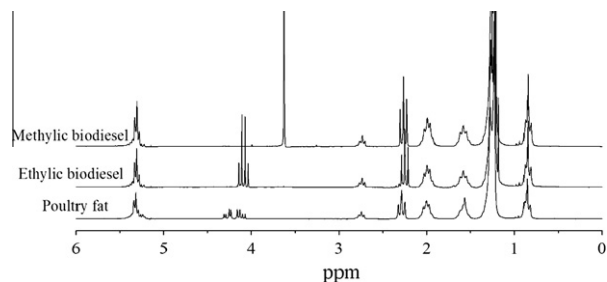


Fig. 1. ¹H NMR spectra of poultry fat and ethanol and methanol poultry fat biodiesels.

Table 1
Fatty acid composition of PF, PFEB and PFMB.

Fatty acid	Composition (% m/m)		
	PF	PFEB	PFMB
Myristic (C14:0)	0.70	0.94	1.07
Palmitic (C16:0)	25.57	29.74	29.05
Palmitoleic (C16:1)	3.62	4.16	4.23
Stearic (C18:0)	7.75	11.21	10.49
Oleic (C18:1)	36.64	40.53	39.56
Linoleic (C18:2)	24.69	7.40	14.13
Linolenic (C18:3)	1.02	–	–
Others	–	6.02	1.47
Saturated	34.03	41.89	40.61
Unsaturated	65.98	52.09	57.92

feedstock. This authors [7] suggested different precipitation mechanism from B20 and B100, and for the methylic poultry fat biodiesel the B50 blend showed the high amount of precipitated mainly at low temperature, attributed to monoglycerides. Although our results of ^1H NMR did not show these components on biodiesel, the CFPP value of B50 (PFMB) was higher than B100 (PFMB), which may be related to the easier formation of precipitates in this blend. According to the CFPP values obtained in this work, these biodiesels could not be used in cities where low temperatures are observed.

The rheological results of ethanol and methanol biodiesels, their blends and unblended diesel are presented in Table 2. Viscosity values of biodiesel were in agreement with the requirements: $1.9\text{--}6.0\text{ mm}^2\text{ s}^{-1}$ [18]. They also indicated that the transesterification of the poultry fat was satisfactory, since the values of the biodiesel viscosities were 10–15% of the viscosity of the poultry fat. These results are in agreement with the literature data [2,3].

Comparing the biodiesel and diesel samples, it should be observed that the presence of the ester functional groups in biodiesel tends to increase the dipole–dipole intermolecular forces and consequently the flow resistance. In diesel, the hydrocarbon chains display weaker London-type intermolecular forces. The esters also present stronger London forces due to their bigger chains, as compared to diesel. As a consequence the biodiesel viscosity was about 30% higher than diesel one.

It should be noted that the higher the viscosity the lower the efficiency of fuel injection, since the spray process is poorer for bigger droplets. This is followed by a decrease of the contact area of pressurized gases within the combustion chambers inhibiting fuel evaporation and favoring an incomplete combustion [19].

It is noteworthy that the blends containing from 5% to 50% of PFEB displayed lower dynamic and kinematic viscosities than the corresponding PFMB blends in spite of the higher PFEB viscosity. One possibility that might deserve further studies is that the longer ethylic biodiesel chains might increase the spacing among diesel chains decreasing London forces among them.

According to some authors, the tests that best describe the fuel behavior at low temperatures are the cold flow parameters, CP, CFPP and PP [8,20,21], presented in Table 3. In this work, CFPP and CP values were very close for both biodiesels. CP values were consistent with a previous work carried out with poultry fat biodiesel [7]. PP values had a meaningful difference between the methanol and the ethanol biodiesels. CFPP values were lower than beef tallow ones obtained by Cunha et al. [22], due to its higher percentage of saturated fatty acids when compared to poultry fat biodiesel. Wyatt et al. [23] observed the same behavior and found CFPP values around $8\text{ }^\circ\text{C}$ and $1.3\text{ }^\circ\text{C}$ for biodiesel from beef tallow and poultry fat, respectively.

The difference between the results of the present work and literature may be assigned to the methodology used in the poultry fat extraction. Moreover, variations in fatty acid composition may

Table 2
Absolute values of dynamic and kinematic viscosity of all samples.

Sample/biodiesel amount (%)	Dynamic viscosity (MPa s)		Kinematic viscosity ($\text{mm}^2\text{ s}^{-1}$)	
	PFEB	PFMB	PFEB	PFMB
PF	64.98 ± 0.4		35.97	
Diesel	4.28 ± 0.02		3.46	
5	4.12 ± 0.04	4.10 ± 0.02	3.50	3.62
10	4.15 ± 0.03	4.35 ± 0.04	3.52	3.62
15	4.21 ± 0.03	4.32 ± 0.03	3.54	3.66
20	4.25 ± 0.02	4.33 ± 0.03	3.58	3.69
50	4.68 ± 0.03	5.03 ± 0.04	4.16	4.05
100	5.65 ± 0.03	5.41 ± 0.02	4.75	4.71

Table 3
Values of CP, CFPP and PP of all samples.

Sample/biodiesel amount (%)	CP ($^\circ\text{C}$)		CFPP ($^\circ\text{C}$)		PP ($^\circ\text{C}$)	
	PFEB	PFMB	PFEB	PFMB	PFEB	PFMB
Diesel	5	–	4	–	–5	–
5	7	5	8	4	1	–2
10	7	5	8	4	2	–2
15	7	6	8	5	2	–2
20	7	6	8	5	2	–1
50	6	6	7	7	–4	–2
100	6	7	7	5	–4	1

occur as a result of different factors as genetic origin, age and sex of the poultries. The vitamin used in the poultry nutrition is another factor for the variation in fatty acid composition and in lipid deposit, especially in monogastric animals [24]. As a consequence of the variation in composition, different behaviors may be observed in the cold flow properties.

In relation to blends, the CFPP values were also in agreement with the literature. In assessing the CFPP of biodiesel from beef tallow and its binary blends, the values found for B2 to B30 blends were similar to the values for diesel while the B40, B50 and B100 samples displayed values varying from $2\text{ to }14.3\text{ }^\circ\text{C}$ [25]. In the present work, compositions from B50 to B100 were not studied because there is a low probability of use of these blends as biofuels.

In spite of being widely used, physical properties of the cold flow parameters are not well known. Aiming at a better understanding of these low temperatures properties, the MT-DSC curves were obtained (Figs. 2 and 3).

Fig. 2 illustrates the reversible and irreversible MT-DSC curves for diesel and PF biodiesels. It is noteworthy that for all samples, the non-reversible curves were used to evaluate the fuel behavior, as they showed a better peak definition. In these curves, baseline transition was associated to a liquid–liquid transition (T_{l-l}) and exothermic events on cooling were assigned to peak crystallization temperatures (T_c) [8]. It was also possible to measure the temperature at which crystallization began (onset temperature – T_o) that corresponded to the point in which the exothermic peak started to deviate from baseline [26]. Crystallization of a liquid occurs when some molecules approach each other, forming a crystalline core, which serves as a nucleus for crystal growth.

The biodiesel curves shown in Fig. 2b and c demonstrated liquid–liquid transitions around $26\text{ }^\circ\text{C}$. The first crystallization peaks were observed at $9.0\text{ }^\circ\text{C}$ and $8.0\text{ }^\circ\text{C}$ for PFEB and PFMB, respectively. Diesel MT-DSC curves (Fig. 2a) presented the T_{l-l} at $12.0\text{ }^\circ\text{C}$ and four exothermic transitions below $3.2\text{ }^\circ\text{C}$, associated with crystallization. It is noteworthy that saturated molecules tend to crystallize more readily than unsaturated molecules. As the biodiesel from the poultry fat was composed by similar amounts of saturated

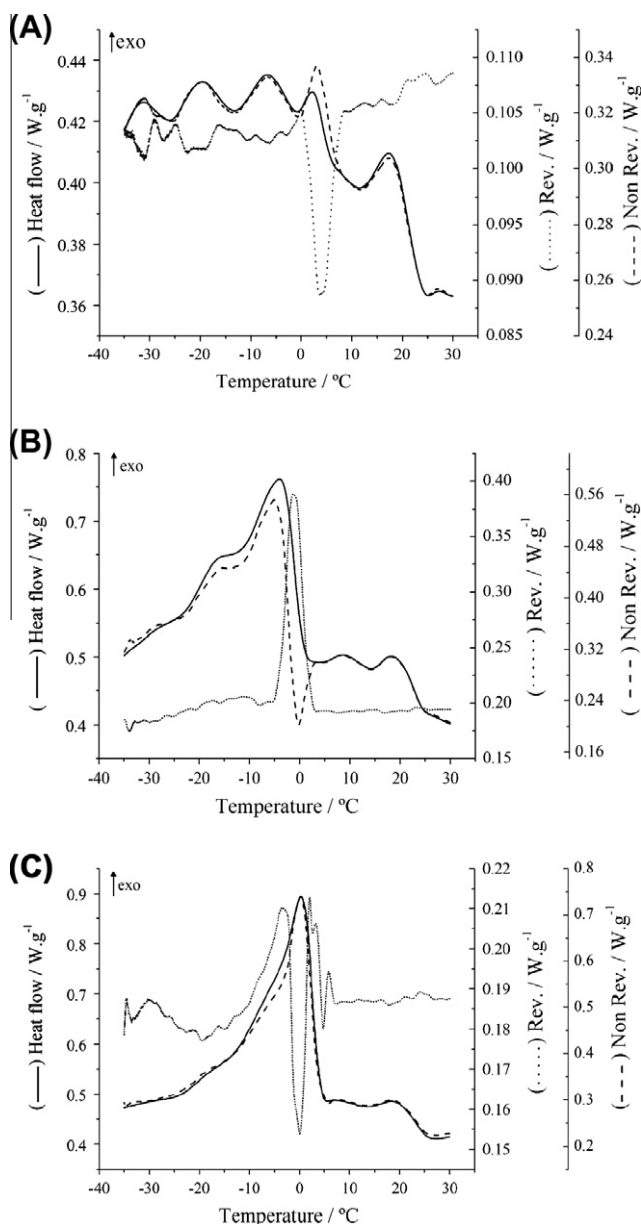


Fig. 2. MT-DSC curves of (A) diesel, (B) PFEB and (C) PFMB.

and unsaturated molecules, as presented in Table 1, the biodiesel had a high tendency to crystallization.

Comparing the methanol and ethanol biodiesels it was observed that PFMB crystallized at higher temperatures. As the methanol biodiesel displayed a smaller chain than ethanol biodiesel, the molecules interacted more easily. The temperatures of the PFEB second and third crystallization peaks were below 0 °C, while for the PFMB, the temperatures were below or close to 0 °C. These values were consistent with the fact that these esters were also formed by unsaturated fatty acids, linoleic, linolenic and palmitic acids, that displayed low melting points, of -5, -17 and -0.5 °C, respectively, as shown in Fig. 4a and b.

For blends (Fig. 3), T_{i-1} was observed at about 12.0 °C, besides three to four crystallization peaks. Each of these peaks corresponded to the crystallization of different fractions of biodiesel/diesel blends. The blends showed different thermal behaviors, according to the concentration of biodiesel in the blend. According to Baldotto [27], upon cooling, during the formation of the first crystal from a blend, the heavier components, which present the

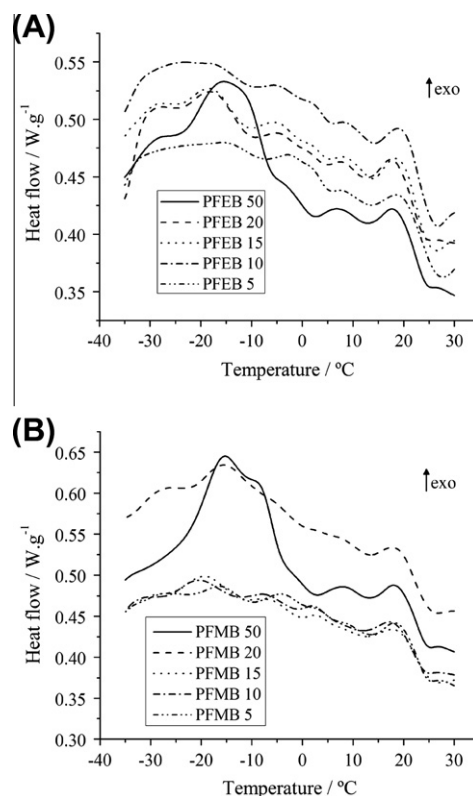


Fig. 3. MT-DSC curves of diesel blends with (A) PFEB and (B) PFMB.

highest melting point, precipitate first and are present in a higher proportion.

The cold flow properties were compared with the thermal analysis results (Fig. 4). Some authors correlated CP values with the onset temperature of the peak related to the biodiesel crystallization, obtained from DSC cooling curve [8,21]. In the present case, the results indicated that the Cloud Point was related to the first crystallization peak, and not with the onset temperature related to the first crystal formation. This different behavior may be due to the size of crystals formed in each case. We believe that in the present work, crystals must grow further in order to be detected in the Cloud Point analysis. As a consequence, CP values correspond to the crystallization peak and not to its onset. CFPP values were close to the first crystallization peak just as CP ones, indicating that crystallization made flowing more difficult.

The PP values were located between the first and the second crystallization peaks being closer to the second one, pointing out that a significant portion of the fuel solidified before it stopped flowing.

Pérez et al. [28] studied the cold flow properties (CP, CFPP and PP) and DSC at low temperatures of five biodiesels obtained from several vegetable oils – namely soybean, rapeseed, sunflower, palm and peanut oil, with and without winterization – and verified a linear correlation between CP and T_{onset} ; and between CFPP and T_{max} , where T_{max} is the point of maximum heat release during the higher temperature exothermic peak.

Teixeira et al. [29] also studied biodiesel samples obtained from blends containing different amounts of beef tallow, babassu oil, and soybean oil by the corresponding conventional techniques and by Temperature Modulated Differential Scanning Calorimetry (TMDSC), and observed that CP and CFPP values correlate well with the crystallization temperature T_{onset} , while PP correlates with the peak temperature, in agreement with the present study for poultry fat biodiesel.

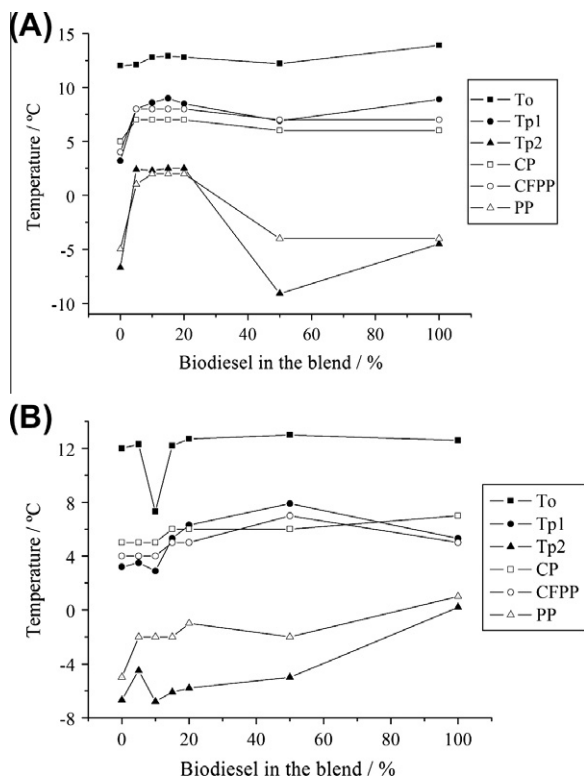


Fig. 4. Comparison between cold flow parameters and MTDSC curves data for diesel, biodiesel and their blends (A) PFEB and (B) PFMB.

4. Conclusions

Biodiesel was obtained from transesterification reaction of poultry fat, as confirmed by ^1H NMR, gas chromatography and by the viscosity reduction. Viscosity values were in agreement with the requirements of the ASTM D445 standard. The presence of small saturated chains increased the crystallization tendency of biodiesel.

MTDSC curves showed that the CP and the CFPP were related to the first crystallization peak, with temperature values varying from 6.9 to 8.9 °C and from 2.9 to 9.5 °C for PFEB and PFMB, respectively. The PP values corresponded to a temperature between the first and second crystallization peaks (4–8 °C; –5 to 1 °C) indicating that a significant portion of the fuel solidified before it stopped flowing.

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