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Thermal Analysis and Characteristics of Refine/Waste Canola Biodiesel under Long-Term storage in Ambient Condition

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Abstract

Canola is an oilseed crop that is adapted to soil type and climatic conditions in Cyprus and is considered a viable rotational biofuel crop. Increasing demand for canola as biodiesel feedstock has encouraged us to investigate its main fuel properties. In this paper, the effects of long-term storage on kinematic viscosity, density, and cold flow properties for waste canola oil (WCOME) and refined canola oil methyl ester (RCOME) were investigated experimentally. Biodiesel samples were kept in ordinary ambient storage conditions for 30 months. The samples were taken from the biodiesel feedstock in every 2/4 months and the properties of the samples were tested periodically. It was found that during 30-months storage, no significant deterioration was observed in cold flow properties. Moreover, Computer Aided Cooling Curve Analysis (CA-CCA) technique for characterizing thermal energy phase change was proposed. Newtonian technique was used to predict the phase transition temperatures, the latent heat of fusion and characterize biodiesel's freezing over a range of temperature. Solidification of biodiesel samples was studied using CA-CCA method. The results showed that CA-CCA method can generate all necessary information to cold weather characterization and study phase changing of biodiesel samples.

Keywords: Cold flow properties; Cooling curve; Density; Kinematic viscosity; Refined Canola biodiesel; Waste canola biodiesel

1. INTRODUCTION

Canola, an oil-seed crop is used as raw material for production of numerous food and industrial products such as cooking oil, animal feed, biodiesel, and lubricants [1]. Globally, canola ranks second in oil-seed production and vegetable oil production [2]. Global canola production is concentrated away from the equator in areas with dry weather and shorter growing seasons [3]. Canola cultivars may be winter or spring types [4]. Canola is the most dominant crops grown in Turkey with annual plantings of more than 8 million hectares of land [5]. Oil seed crops considered as biodiesel alternatives include canola [6]. Canola biodiesel is a good alternative fuel, and has great advantages compared with other biodiesel fuels [7]. Canola has experienced a resurgence of interest as a biodiesel Turkey and Cyprus, an alternate to petro-diesel using renewable saddle crops [8][9].

Generally, biodiesel, as an alternative diesel fuel consisting of alkyl monoesters of fatty acids prepared from vegetable oils or animal fats, has attracted more and more focuses because it is renewable and environmental-friendly [10]. Biodiesel is completely miscible with diesel and thereby can also improve certain fuel qualities. However, commercial use of biodiesel has been limited because of some drawbacks including corrosivity, instability of fuel properties, higher viscosity, higher density[11-14]. Stability of fuel properties is especially important to ensure expected engine performance as well as engine life [13, 14].

Furthermore, low temperatures cause clogging in fuel pipes and the fuel system filters diesel engines [15], the cold flow properties of biodiesel are significant parameters, especially in cold climates. Cold flow properties of diesel fuels are usually determined by the properties of cloud point (CP), pour point (PP), and cold filter plugging point (CFPP). The cooling curve recorded in a thermal analysis is a temperature versus time graph of a melt during freezing; hence it keeps the whole solidification history. Each phase change causes a thermal event which is displayed as a plateau on the cooling curve [11].

The storage stability of biodiesel is one of the most important concerns for improving the sustainability of biodiesel fuel. Since unsaturated ester components of biodiesel fuel are oxidized by atmospheric conditions, effects of long-term storage of biodiesel on the fuel properties need to be clarified in terms of different quality criteria of the fuel. Kinematics viscosity is an important parameter to determine biodiesel stability [16]. There are several studies conducted on the stability of biodiesel in long term storage under different external conditions [17][18].

In the present study, two pure biodiesel (RCOME and WCOME) were produced from refined and waste canola oil collected from domestic sources through the transesterification reaction**.** Therefore, the main aim of this study was to investigate the effects of long-term storage on RCOME and WCOME under an ambient temperature condition. In this work, kinematic viscosity and density of biodiesel over the temperature range from -10℃ to 300℃

were examined during the long-term (30 months) storage. Additionally, this work aims to investigate the effects of long-term storage on the cold flow properties of biodiesel samples. Furthermore, the suitability of a simple Computer Aided Cooling Curve Analysis (CA-CCA) technique for characterizing thermal energy phase change biodiesel was proposed in the present work. A Newtonian technique was used to predict the phase transition temperatures and the latent heat of solidification. The method records the temperature changes in the sample as it cools through various phase transformations. Information on the latent heat of solidification, phase transition temperatures, solid fraction amounts and types of phases due to under-cooling were obtained**.** The results showed that paper is shown that the effect of aging on RCOME and WCOME under ambient temperature during 30 months is not significant because the changes of the properties are still within acceptable ranges of biodiesel standard.

2. MATERIALS AND METHODS

2.1. Materials

In available literature there are numerous feedstocks reported which can be used to produce biodiesel. Among them, soybean, palm, sunflower, rapeseed oils have been considered in earlier times but their negative impact on food crops have hindered their usage. The selection of raw material mainly depends upon the availability and cost. Cyprus and Turkey for example are self-dependent in production of edible oils. As a result edible oil such as canola is the mostly used oils. In this study, transesterification method is preferred for biodiesel production. Two different types of vegetable oils (refined canola and waste canola) were used as raw feedstock. The refined canola oil was purchased from a local market, while waste canola oil was collected from different restaurants. Methyl alcohol (CH_3OH) as alcohol and potassium hydroxide (KOH) as a catalyst were used in the production of biodiesel samples. The reaction temperature was kept at 56 ℃ and the reaction duration was 60 min. A mixture consists of biodiesel, glycerol, and other minor products (tri-di-mono glyceride, catalyst, excess methyl alcohol) was obtained after the reaction. Afterwards, three stage water washing process filtered out the contaminations from the biodiesel. First, glycerol that has a higher specific gravity than the biodiesel is separated. In the second stage, 10% pure water by volume was added to the mixture in order to wash out the polar impurities from the mixture. Water and methyl alcohol in the mixture is removed by evaporation in the last stage. The final product was pure biodiesel and was ready to use as fuel. The fatty acid composition of RCOME and WCOME were obtained by gas chromatography (GS) and given in Table 1. Furthermore, the physical and chemical properties of the obtained biodiesel samples are shown in Table 2.

Table 1. Fatty acid methyl ester composition (wt%)

Fatty acid	Structure	RCOME	WCOME
Caprylic acid	C8:0	0	0
Capric acid	C10:0	0	0
Lauric acid	C12:0	0	0.08
Myrictic acid	C14:0	0.05	0
Palmitic acid	C16:0	4.14	5.63
Stearic acid	C18:0	1.91	1.57
Oleic acid	C18:1	58.9	62.97
Linoleic acid	C18:2	20.57	21.34
Linolenic acid	C18:3	9.34	6.99
Arachidic acid	C20:0	0.63	0.46
Eicosenoic acid	C20:1	1.35	1.04

Table 2. Properties of biodiesel samples

Property	Unit	Test method	Limits	RCOME	WCOME
Kinematic viscosity at 40℃	mm^2/s	EN 14214	3.5–5.0	4.48	4.545
		ASTM D445	1.9-6.0		
Density at 15 ℃	kg/m^3	EN 14214	860–900	883.98	895.44
		ASTM D854	867-.....		
Cloud Point	℃	EN 14214	Location & season dependant	-4	-2
		ASTM D2500			
Cold filter plugging point	℃	EN 116	Location & season dependant	-8	-5
		ASTM D6371			
Pour Point	℃	EN 14214	Location & season dependant	-12	-10
		ASTM D97			

2.2 Storage Conditions

Biodiesel samples were stored in closed glass bottles for 30 months and were kept indoors, at ambient temperature under light in the laboratory. Samples were taken out periodically every 2/4 months to study the properties of biodiesel.

3. EXPERIMENTAL SETUP

3.1 Kinematic Viscosity and Density

Kinematic viscosity (mm^2/s) and density (kg/m^3) were determined with Ubbelohde viscometer following the ASTM D-445 and Pycnometer with a bulb capacity of 25ml following the ASTM D854. Kinematic viscosity and density of eleven biodiesel fuel samples were measured at high temperature (from 20℃ to 300℃ in steps of 10℃) and low temperature (from -10℃ to 20℃)[1]. The measurements for viscosity and densities were done three times for each sample and the results were averaged.

3.2 Cold Flow Properties (CFP)

The biodiesel under cold climatic conditions may lead to its condensation and gel formation resulting in the crystallization of fuel particles in liquid fuel due to strong intermolecular interaction below their melting point. Inferior cold flow properties may cause clogging of fuel pipes and pump leading to improper engine operation. As the temperature drops down, more solid is formed and biodiesel achieves the pour point which is the minimum temperature at which it will not be able to flow. The cold flow properties are explained in terms of cloud point (CP), pour point (PP), and cold filter plugging point (CFPP). Cloud Point (CP) was measured according to automated method ASTM D2500. In addition, CFPP and PP were estimated according to the ASTM D97 For a greater degree of accuracy, PP measurements were done with a resolution of 1℃ instead of the specified 3℃ increment.

3.3.3 Computer-Aided Cooling Curve Thermal Analysis (CA-CCA)

Computer aided cooling curve thermal analysis (CA-CCA) presents useful information about the solidification latent heat, fraction of solid during solidification, and the amount of different phases [19]. CA-CCA can be classified into Newtonian and Fourier analysis.

In this study, Newtonian analysis was performed with a sample size of 45ml biodiesel in glass test jar. Figure 1 schematically presents the experimental setup for CA-CCA. An insulated cooling bath was filled with ethanol as the coolant. Ethanol was cooled down while its temperature was controlled by an automated refrigeration unit. A stirrer was

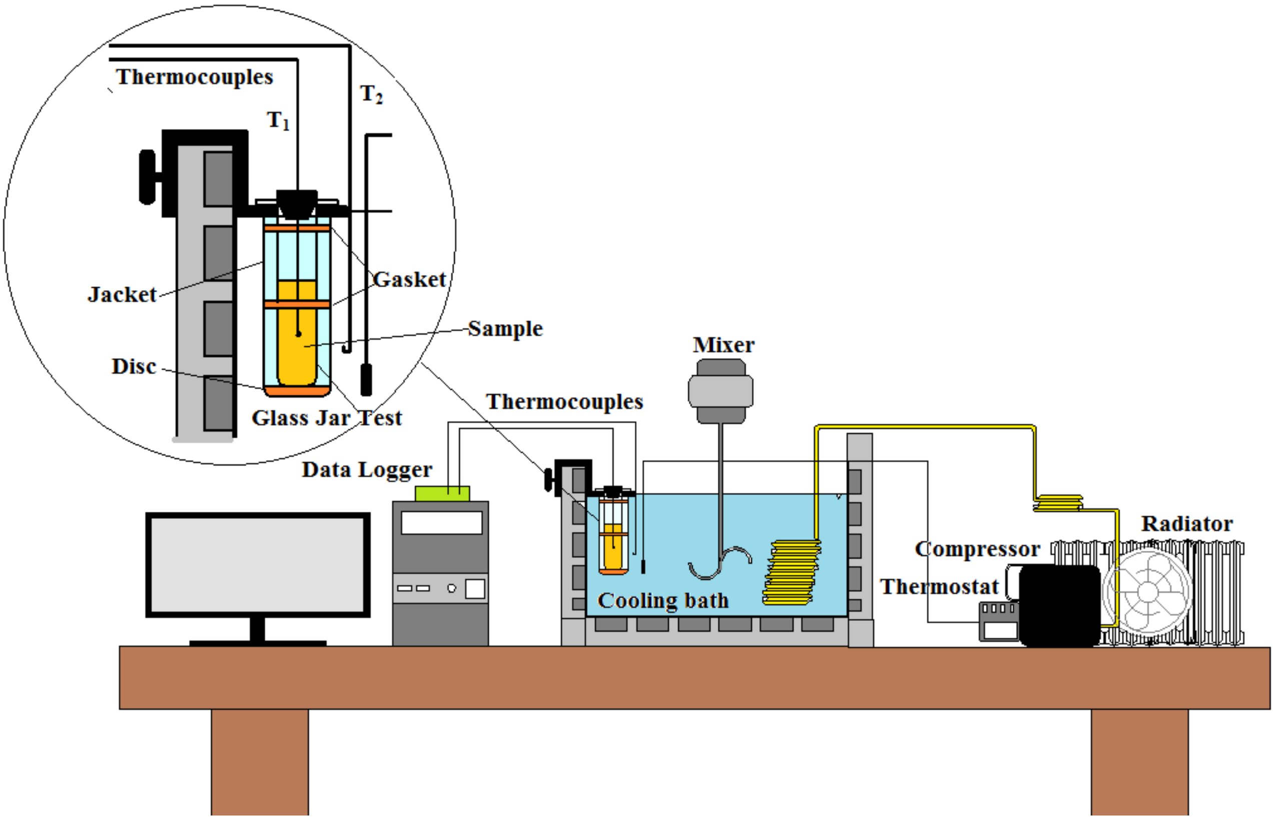

Figure 1. Experimental setup for CA-CCA

[1] The experimental setup of measuring the viscosity and density from -10 to 300℃ is described according to Kassem & Çamur (2016).

used for thermal homogeneity of the ethanol in the cooling bath. An aluminum cylinder jacket was placed in the middle of the cooling bath. A 6 mm thick cork disk was placed at the bottom of the jacket as a thermal insulator. The glass test jar was filled with biodiesel sample to a level of 54 mm corresponding to a sample volume of about 45 ml. The test jar was then fitted into the jacket and a uniform air gap of 5 mm in the radial direction between the test jar and the jacket was ensured by two gaskets. Moreover, two T-type thermocouples position for measuring the temperature of biodiesel sample (T_1) and cooling bath temperature (T_2). It should be noted that the thermocouples were calibrated before the tests. Prior to data collection for CA-CCA, the cooling bath was cooled down to -20 °C and the biodiesel sample was heated up to 65℃, which was about 40 °C above the expected CP value.

4. RESULTS AND DISCUSSIONS

4.1 The Effects of Testing Temperature and Ageing on Kinematic Viscosity of RCOME and WCOME

The kinematic viscosities of biodiesel fuels measured from 20℃ to 300℃ are shown for selected storage periods in Figure 2. It can be seen that viscosity decreases as the temperature increases and this behavior can be explained by the kinetic molecular theory for liquid state [20]. The kinematic viscosity is increased when the storage period increased.

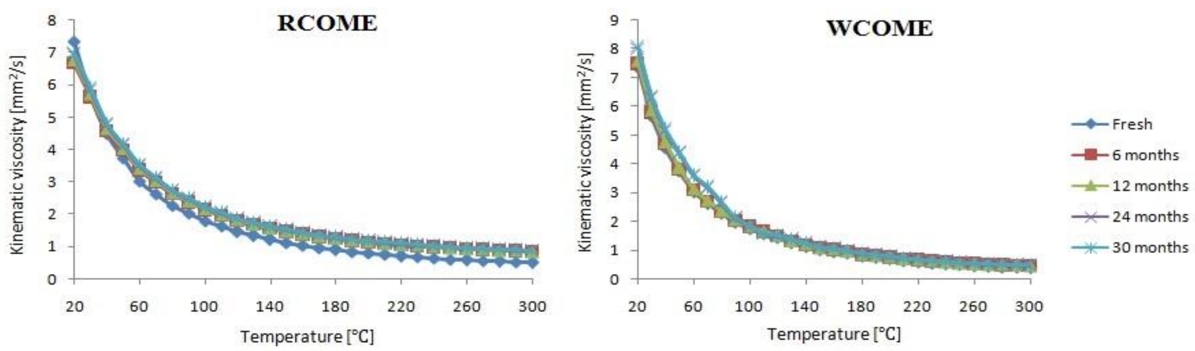

Figure 2. Measured kinematic viscosities of biodiesel samples from 20℃ to 300℃

Figure 3 shows the kinematic viscosity measured from -10℃ to 20℃. The sharp increase in viscosity at temperatures might be an attribute to poor cold flow properties of biodiesel blends, although the viscosity increase identified below cloud point temperature somewhat differed among blends with different contents of biodiesel [13].

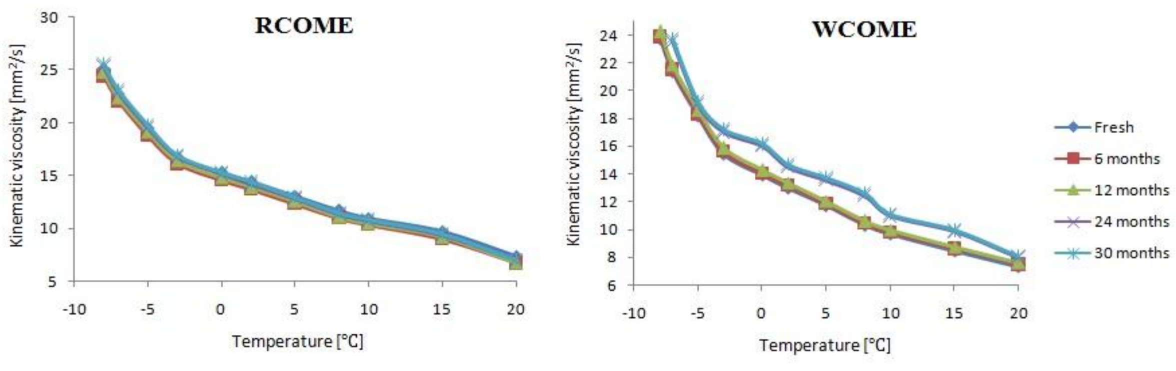

Figure 3. Measured kinematic viscosities of biodiesel samples from -10℃ to 20℃

Table 4 shows the variations of kinematic viscosity at 40℃ as a function of storage period of 30 months at ambient temperature. As Table 4 shows there is an increase in kinematic viscosity of biodiesel samples with the increase of storage period. Moreover, all methyl esters remained within the ranges listed in ASTM D6751 for the duration of the study at ambient temperature. According to EN 14214, RCOME remained below the maximum limit specified in the biodiesel standards with the exception of WCOME after 16 months. In general, viscosity may be considered as the integral of the interaction forces between molecules. When energy or heat is applied up to a certain level, molecules can then slide over each other or become melted. Initially, they slide over each other very slowly. If the amount of heat or temperature greatly exceeds the melting point, they move pass each other very rapidly and the liquid becomes less viscous [21]. In addition, the properties of waste oils can be changed depending on the fatty acid composition methyl ester and on using or frying conditions, such as temperature, cooking time and cooking process [22].

4.2 Effects of Testing Temperature and Ageing on Density of WCOME and RCOME

The density of RCOME and WCOME was measured at a range of temperature from 20℃ to 300℃, and the results of selected storage periods are shown in Figure 4. It was observed that the density of the biodiesel decreased with increase in temperature for all the tested fuels.

Table 4. Influence of storage time (months) and temperature on kinematic viscosities (at 40℃) in mm^2/s

Months	0	2	4	6	8	10	12	16	20	24	28	30
RCOME	4.500	4.505	4.511	4.566	4.593	4.618	4.640	4.667	4.695	4.742	4.789	4.794
WCOME	4.545	4.550	4.556	4.678	4.706	4.732	4.753	4.782	5.105	5.145	5.185	5.202

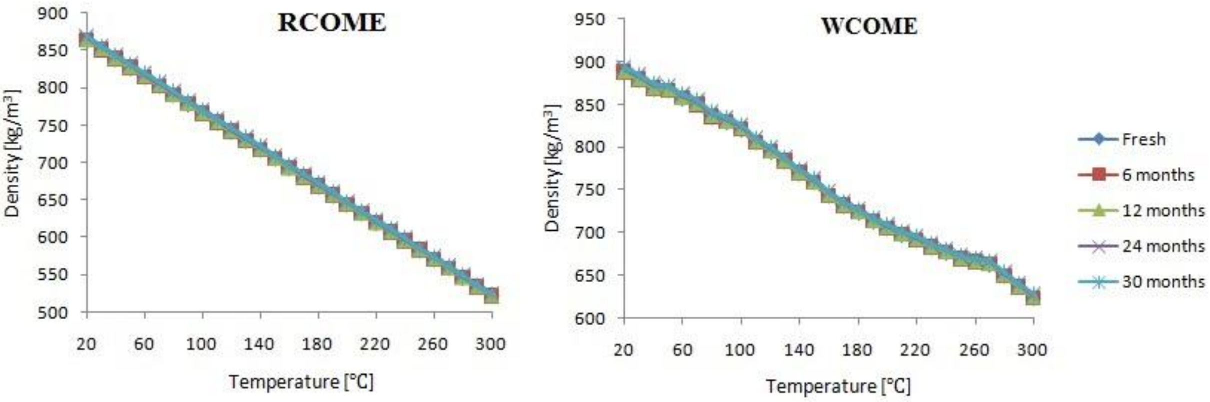

Figure 4. Measured Density of biodiesel sample from 20℃ to 300℃

Moreover, the experimental results of density at ranging temperature of -10℃ to 20℃ are presented in Figure 5 for some selected storage periods of all biodiesel samples. The results showed that for each of the two biodiesel the densities were high at low temperature and decreases as the temperature increased. That is, density decreases non-linearly with temperature. Comparing RCOME and WCOME, it was observed that RCOME crystallized at higher temperatures compared to WCOME. As a result, the formation of crystals of the blends showed at a different temperature. The standard for biodiesel states that the fuel should have a density between 860 kg/m^3 and 900 kg/m^3. Hence, the results obtained showed that for fresh biodiesel samples had a density in the range of 860-900 kg/m^3.

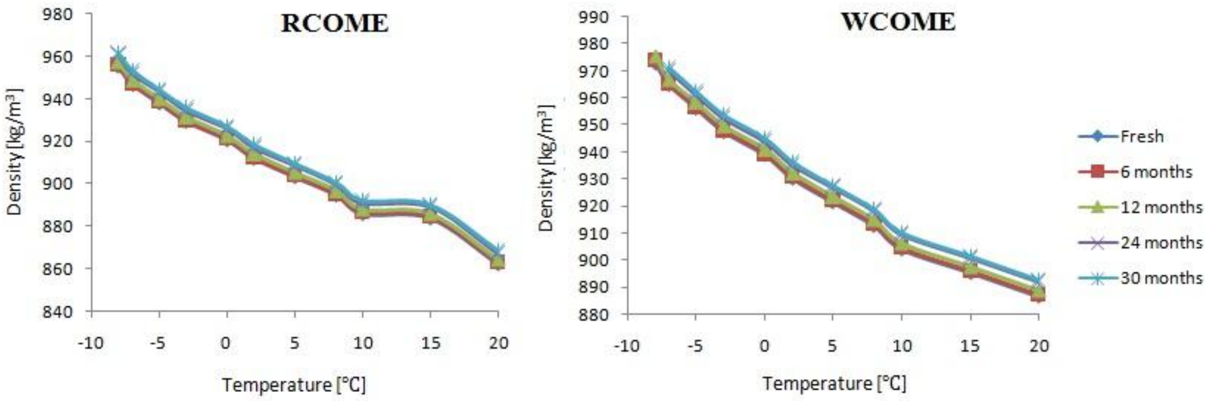

Figure 5. Measured Density of biodiesel sample from -10℃ to 20℃

The initial densities of biodiesel samples (Table 5) were in the range of standard limit of 860–900 specified in ASTM and EN 14214 (Table 2). Storage over an extended period (30 months) resulted in higher density for all methyl esters.

Table 5. Influence of storage time (months) and temperature on densities (at 15°C) in kg/m^3

Months	0	2	4	6	8	10	12	16	20	24	28	30
RCOME	883.98	884.38	884.29	884.42	884.92	885.42	885.91	887.01	888.20	888.91	889.62	889.89
WCOME	895.44	895.85	895.76	895.89	896.39	896.89	897.40	898.51	899.71	900.43	901.15	901.42

4.3 Cold Flow Properties of Biodiesel Samples

The cold flow properties of fresh biodiesel samples (0 month) are shown in Table 6. It can be seen from that RCOME has higher cold flow properties comparing to WCOME. This difference may be due to origin of oil and quality also. It is here to be noted that both these properties are dependent on fatty acid composition of oil that is saturated and unsaturated fatty acids present in oil [23]. Therefore, the length of the fatty acid chain as well as its composition plays a substantial role in the cold flow properties of biodiesel. Also, Storage for 30 months did not significantly impact the CP, PP or CFPP values of the methyl esters. It was speculated that extended storage would result in an accumulation of fatty acids and other degradation products that would increase CP, PP and CFPP.

Table 6. Cold flow properties in °C of biodiesel samples

Months		0	2	4	6	8	10	12	16	20	24	28	30
RCOME	**CP**	-4	-4	-4	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5
	CFPP	-8	-8	-8	-7.5	-7	-7	-7	-6.5	-6	-6	--5.5	-5.5
	PP	-12	-11	-11	-10	-9.5	-9	-8.5	-8	-7.5	-7.5	-7.5	-7.5
WCOME	**CP**	-2	-2	-2	-2	-2	-1.5	-1.5	-1.5	-1.5	-1.5	-1.5	-1.5
	CFPP	-5	-5	-5	-4	-4	-4	-4	-3.5	-3	-3	-2.5	-2.5
	PP	-10	-9.5	-9.5	-9	-8.5	-8	-7.5	-7.5	-7	-6.5	-6.5	-6.5

4.4 Computer-Aided Cooling Curve Analysis of Biodiesel Samples[2] at Storage Period of 30 Months

As shown in section 4.3, storage for 30 months did not significantly impact the cold flow properties values of the methyl esters. Consequently, CA-CCA analysis method to study phase change biodiesel for thermal energy storage (30 months) applications is discussed in this section.

The *dT/dt* vs *(T−T$_0$)* curve plotted in Figures 6 and 7 exhibited linearity both in liquid and solid phases during freezing of RCOME and WCOME, respectively. The curve deviated from linearity in the two-phase region in which the liquid and solid biodiesel coexist together. Excluding the two-phase region, a straight line was fitted through the data points of fully liquid and fully solid regions.

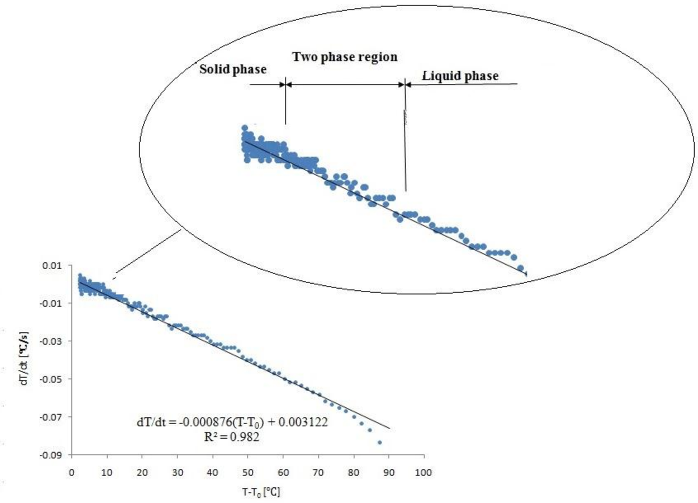

Figure 7. *dT/dt* versus (*T*−*T$_0$*) curve of WCOME during cooling

Figures 8 and 9 show the cooling curve thermal analysis results of RCOME and WCOME, respectively, including cooling curve, first derivative curve (dT/dt), and zero curve. The average cooling bath temperature was fixed at -19.55°C with a minimum of -21°C and a maximum of -18.1 °C. A plateau is observed on the cooling curve (T vs t) at about -5

[2] Newtonian technique based on CA-CCA to characterize biodiesel properties is also discussed in details in Ref. (Evcil et al., 2018).

and -2°C which correspond to the experimentally determined CFPP value for RCOME and WCOME, respectively, given in Table 6. The CP (-3.5°C for RCOME and -1.5°C for WCOME) is located before the plateau that corresponds to a sharp change in the slope of *dT/dt* vs t curve (indicated by the letter "A") and is related with the nucleation of solid crystals in the freezing RCOME as shown in Figures 8 and 9, respectively. At point "B" close to the data point for PP another remarkable change in the slope of *dT/dt* curve can be noticed. The change in the slope occurs at -7 and -6°C which is about -7.5 and -6.5°C below the experimentally determined PP value of RCOME and WCOME, respectively.

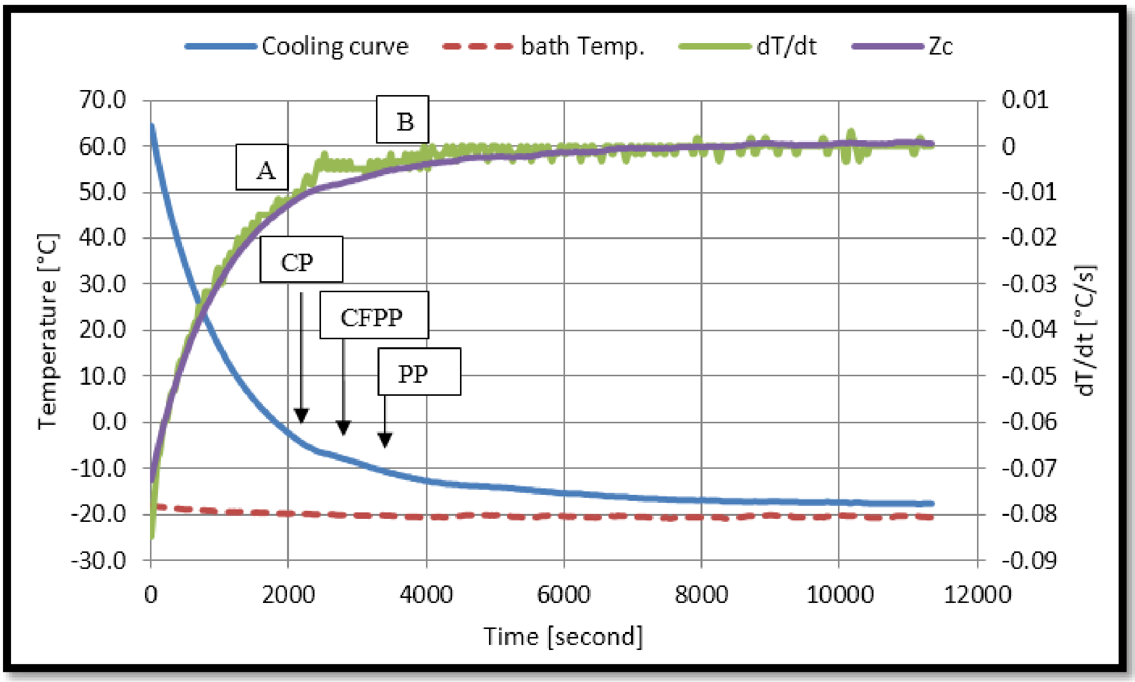

Figure 8. Cooling curve analysis and Newtonian zero curve of RCOME

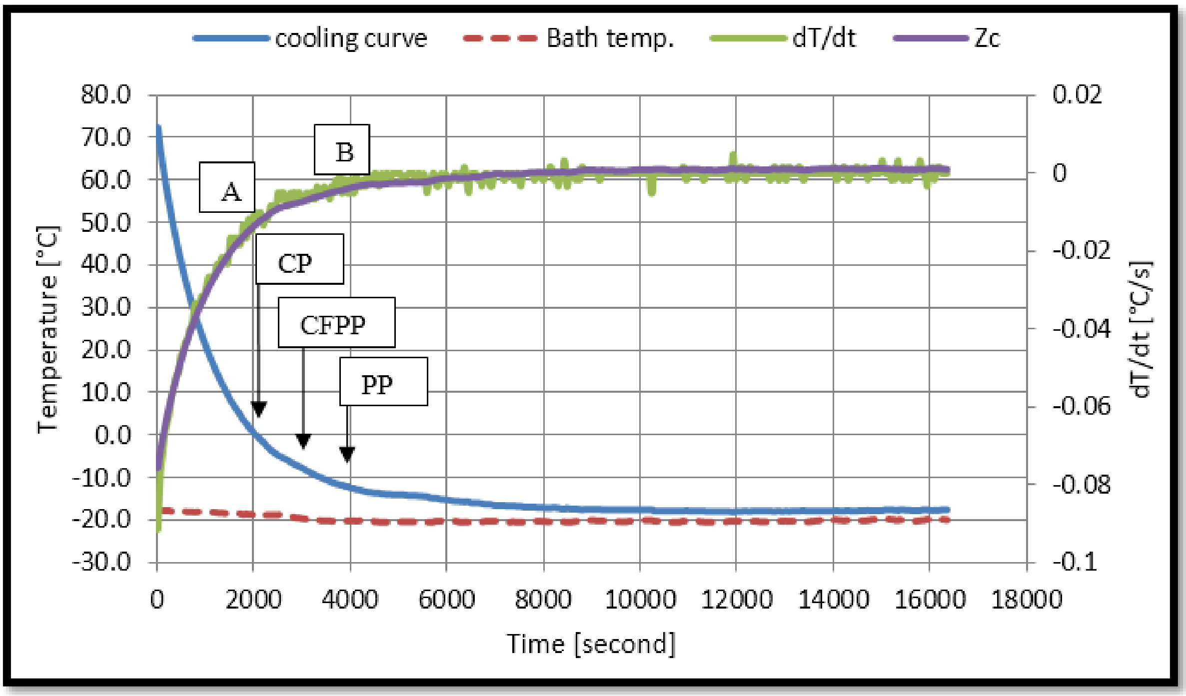

Figure 9. Cooling curve analysis and Newtonian zero curve of WCOME

Generally, the physical properties of the biodiesel are dependent on the fatty acid composition of biodiesel that is saturated and unsaturated fatty acids present in the fuel. Therefore, the lengths of the fatty acid chain, as well as its composition, play a substantial role in the cold flow properties of fuel [23]. The solid phase can be classified into two phases: crystalline and amorphous phases (Speight, 2010). Based on the experimental results, the final solid-liquid transition is about -3 and-1 ℃ for RCOME and WCOME, respectively, for 9.5℃ for biodiesel obtained from waste frying oil [11]. This is in agreement with the amount of saturated fatty acids in each sample. The differences in crystallization behavior between waste frying oil-based biodiesel [11] and canola biodiesel of current study suggested that canola biodiesel is composed of a amorphous phase while waste frying oil-based biodiesel is composed crystalline phase only.

6. CONCLUSIONS

In this study, the biodiesel was produced from refined and waste canola oil and the fuel properties were investigated for a long-term storage period of 30 months under the climate conditions in Cyprus. In addition, the results of their analysis were compared to the ASTM standard specifications and EN14214. The kinematic viscosity and density of biodiesel are measured at wide range temperatures from-10℃ to 300℃. A Computer-Aided Analysis of the cooling curve in conjunction with the Newtonian thermal analysis was carried out for cold weather characterization and also to examine the variation of solid fraction in the biodiesel while it was freezing. The following conclusions can be drawn from the results of the present study.

- In all cases, the viscosity and density of biodiesel samples decrease as temperature increases; and both of them increase because of the increase in storage periods.
- The fuel properties of the produced biodiesel were compared with international standards and the results confirmed a high quality of the produced biodiesel.
- The kinematic viscosity of RCOME and WCOME is stable under ordinary storage conditions for 30-month storage with the exception of WCOME which was out of the range of EN 14214 after 16 months.
- The cold flow properties were essentially unaffected by extended storage. It concluded that the refined/waste canola oil-based biodiesel can be stored in an ordinary storage environment without a significant deterioration in cold flow properties.
- The solid fractions at CP, CFPP and PP could be estimated as a result of the Newtonian thermal analysis following the cooling curve analysis. Also, the estimations value of cold flow properties were justified with the visual observations experimentally.
- The results suggested that the current approach may be considered as a potential tool to estimate rapidly the cold flow properties and also solid fractions in biodiesel fuels of crystalline solidification nature particularly, when they are prone to freeze in mild cold weather.
- Canola biodiesel exhibited superior fuel properties and CFP's over a long-term storage period under the climate conditions in all seasons on the island.

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