



## RESEARCH PAPER

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## The changes of physical properties in transesterification of three different vegetable oils to biodiesel; comparison and evaluation to determine reaction conversion

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Article published on July 12, 2014

**Key words:** *Biodiesel, Refractive Index, Viscosity, Specific Gravity, Pour Point*

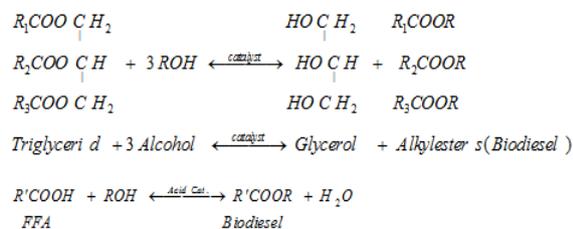
### Abstract

In recent decade, since the fossil fuel sources are diminished, many researchers have proposed to use a pure, non-toxic and biodegradable fuel which is called biodiesel, instead of traditional diesel fuel. The progress of transesterification of oil to biodiesel could be determined by monitoring the changes of physical properties during reaction. This approach could be used as an alternative to expensive and time-consuming methods. In this study transesterification of sunflower, canola and corn oils was carried out at 65°C, MeOH to oil molar ratio of 6:1, 1 wt% of KOH as catalyst under vigorous mixing for 60 min. Yield of the reaction was 95.2, 94.7, and 95.6, respectively. Six blends composed of produced biodiesel and fresh oil were prepared in different wt%, for each oil, as incomplete reaction mixture. Specific gravity, viscosity, refractive index, cloud point, pour point and flash point were measured and the appropriate functions were fitted on the extracted data. Results have shown that the physical properties of fresh oil change during the reaction with a constant rate and the slope of the changes are independent of oil type. Depending on results, refractive index, specific gravity and viscosity are highly recommended to predict the reaction progress.

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**Introduction**

In recent years, because of the world energy crisis, reduction of the world’s petroleum reserves and on the other hand increasing energy requirement and also environmental concerns, there is a great demand for alternative sources of energy. Therefore, many countries have started to perform a series of investigation on new fuel resources (Samart *et al.*, 2009; Leung *et al.* 2010; Huang *et al.* 2010). Among many possible sources, non-toxic, biodegradable and renewable biofuel which can be obtained from bio-resources such as vegetable oils or animal fats which is called “biodiesel”, has attracted much attention as a promising alternative for fossil diesel fuels. Biodiesel is very attractive due to its environmental benefits and especially the renewable resources. Significantly, it has lower emissions in compare with fossil diesel. In addition, biodiesel is better than diesel fuel because of no sulfur content and aromatic content, high flash point, and biodegradability (Huang *et al.*, 2010; Park *et al.*, 2008; Bozbas, 2008; Patil and Deng, 2009). It does not rise the level of carbon dioxide in the atmosphere and leads to minimize the intensity of greenhouse effect (Patil and Deng, 2009; Dias *et al.*, 2008; Guan *et al.*, 2009). Biodiesel consists of mono alkyl esters derived from either the transesterification of materials usually composed of C<sub>12</sub>–C<sub>22</sub> fatty acid triglycerides (TGs) or the esterification of free fatty acids (FFAs) with short-chained alcohols such as methanol. Glycerol is a valuable byproduct of this reaction. Fig. 1 shows schematic of these reactions (Guan *et al.*, 2009; Helwani *et al.*, 2009; Fernando *et al.*, 2007; Hameed *et al.*, 2009).



**Fig. 1.** Schematic of transesterification of triglycerides (TGs) and esterification of free fatty acids(FFAs).

Transesterification of triglycerides can be catalyzed using both homogeneous (acid or base) and heterogeneous (acid, base and enzyme) catalysts. Homogeneous base catalysts (such as sodium and potassium hydroxides or methoxides) are the most common used catalysts due to their high activity and the other advantages that make them economically superior to liquid acids and heterogeneous catalysts. Although, there are some problems when waste oil or fresh oil with high acidity is used like soap formation. Also, during biodiesel purification lots of hazardous and corrosive waste water is produced (Hameed *et al.*, 2009; Sun *et al.*, 2010; Arzamendi *et al.*, 2006; Murugesan *et al.*, 2009; Jacobson *et al.*, 2008). Thus, in order to eliminate these difficulties it is essential to do a lot of experiments that obviously some of them are incomplete and analysis is necessary (Arzamendi *et al.*, 2006; Ellis *et al.*, 2008). For this purpose, several methods have been developed, such as chromatography and spectroscopy methods. Among these approaches Gas Chromatography (GC) and High Performance Liquid Chromatography (HPLC) are the most common techniques (Arzamendi *et al.*, 2006; Murugesan *et al.*, 2009; Jacobson *et al.*, 2008; Ellis *et al.*, 2008). Each approach has abilities and disabilities, so that they are used according to user’s object, but all of them need especial equipments that often they are expensive. On the other hand there are a lot of transesterification product samples that should be qualified and quantified, in each research. Moreover, to select the most appropriate approach in order to analyze the samples, analysis quality, accessibility, costs, and required time are very important. Consequently, introduction of simple, rapid, inexpensive and accessible methods is very useful.

Vegetable oils have some physical properties that make them different from biodiesel. During transesterification reaction these properties change between oil and biodiesel properties, progressively. The properties variation in this process can be used as an index to determine oil conversion. In the present work, the change of physical properties for sunflower,

canola and corn oil in transesterification to biodiesel have been investigated. Then, some relations between oil conversion (methyl ester wt%) and physical properties have been established. In order to generalize these relations, we have compared the existing trends among three different oils. Ultimately, the appropriate functions were fitted and evaluated.

## Materials and methods

### Materials

Refined sunflower, canola and corn oil prepared by brand "Famila" was purchased from local store. These oils were in agreement with the specifications for food oil according to ISO 5508. The reagents used during synthesis and purification procedures were as follow: methanol 99.9% (chromatography grade, Merck), potassium hydroxide 85% pellets (GR for analysis, Merck), Hydrochloric Acid 37%(GR for analysis, Merck), n-Hexane 95% (extra pure, Merck) and some other chemicals were purchased from national companies. Methyl Laurate (Methyl Dodecanoate)  $\geq 99.7\%$  were supplied from Sigma as standard for GC analysis.

### Methods

#### Biodiesel synthesis

Transesterification of refined sunflower, canola and corn oil were carried out in a 1000 ml three necks flat bottom flask equipped with a condensation system and thermometer. The reactor was placed on a hot plate magnetic stirrer. Operational conditions were as follows: atmospheric pressure; catalyst weight: 1% based on initial oil weight; methanol/oil molar ratio: 6/1; reaction temperature, 65°C; under vigorous mixing made by magnet stirrer and reaction time was 60 min. The reactor was initially charged by 500 g of oil and heated up to the reaction temperature. Afterwards, the catalyst was dissolved in methanol that was weighed before. Finally, the methanol solution was added to the reactor and reaction was started. After the mentioned reaction time, the flask was cooled immediately and reaction mixture was transferred into separator funnel.

#### Separation and purification of biodiesel

Following enough time for settling, the residual methanol and glycerol, as heavier phase, was separated from produced biodiesel and unreacted oil. After draining of bottom layer, oil layer was washed out with hot distilled water for at least three times to remove remained catalyst and glycerol. Washing was continued until drainage pH became as same as distilled water. Then, the product was heated up to 110°C for about 15 minute to eliminate residual water.

#### Preparation of biodiesel and vegetable oil blends

Synthesized biodiesel, at the above mentioned conditions was used to make the blends with fresh vegetable oil, after purification. Six different blends were prepared by mixing of 0, 20, 40, 60, 80, and 100 weight percent of produced biodiesel with fresh oil, for each oil. These samples were assigned as B/Sun, B/Can, and B/Cor, respectively for Sunflower, Canola and Corn oil. All samples have a number that indicate weight percent of biodiesel in mixture.

#### Biodiesel characterization

The composition of fatty acid methyl ester (FAME) in biodiesel was determined utilizing a HP 6890 Gas Chromatograph with a flame ionization detector (FID). Capillary column was a BPX-70 high polar column with a length of 120 m, a film thickness of 0.25  $\mu\text{m}$  and an internal diameter of 0.25 mm. Nitrogen was used as carrier gas and also as an auxiliary gas for the FID. One micro-liter of sample was injected by a 6890 Agilent Series Injector. Lauric acid methyl ester (methyl laurat) was added as a reference into the crude biodiesel and the samples were analyzed according to Wang *et al.* (2006).

$$FAME \text{ wt}\% = \left( \frac{\text{area of all FAME}}{\text{area of reference}} \times \frac{\text{weight of reference}}{\text{weight of Biodiesel sample}} \times \frac{f_{FAME}}{f_r} \right) \times 100 \quad 1$$

Where,  $f$  is correction factor for transforming area percent to weight percent and it can be obtained from calibration.

#### Physical properties measurement

Specific gravity is defined as ratio of sample density (unit volume weight) of sample to density of pure

water in 60°F. This property was measured by standard E100 hydrometer in 60°F bath according to ASTM 1298. The hydrometer was immersed in enough amount of sample placed in the bath. The number showed by hydrometer at the liquid surface determines specific gravity. Viscosity is the ability of a material to flow. ASTM D 445 provides a method for obtaining kinematic viscosity and a calculation method to determine dynamic viscosity (Fernando *et al.*, 2007). Cannon glass capillary kinematic viscometer in 40°C water bath was used for determining viscosity of all biodiesel samples. Flash point is the temperature at which the fuel becomes a mixture that will ignite when exposed to a spark or flame. This property was determined according to ASTM D 93. A Pensky-Martens closed-cup tester of fuel oils (ASTM, 1994a) was used (Fernando *et al.*, 2007). Cloud point is the temperature that initial

crystalline particles are formed. It is the starting point to freeze. Cloud point of the samples was measured according to ASTM 2500. Pour point is defined as temperature that first liquefied droplet of solid sample can flow. It is the starting point to melt. This property was measured for the samples according to ASTM D97. For cloud point and pour point, E1 thermometer and cold bath made by ethanol and solid carbon dioxide, were used. Refractive index is defined as ratio of light speed in sample to vacuum (or air) for an especial wavelength. The data was obtained by ABBE Refractometer in 40°C, according to ISO 6320.

**Results and discussion**

The composition of sunflower, canola and corn oils, according to ISO 5508, and some characterization data have been summarized in Table 1.

**Table 1.** Composition and some characterization data of Famila® sunflower, canola and corn oil.

<i>Characterization</i>	<i>Sunflower oil</i>	<i>Canola oil</i>	<i>Corn oil</i>
Free Fatty Acid (wt%)	0.1	0.196	0.324
Water & Volatile content (wt%)	0.07	0.07	0.08
Refractive Index (at 40 °C )	1.4669	1.4665	1.4666
Soap content (ppm)	66	67	71
Saponification index (mg KOH/g oil)	195.3	189.5	195.72
Mean Molecular Weight (g/mol)	861.7	887.9	859.9
<i>Fatty Acid Composition</i>	<i>(wt%)</i>	<i>(wt%)</i>	<i>(wt%)</i>
Lauric (C12:0)	0.53	---	---
Myristic (C14:0)	---	0.14	0.05
Palmitic (C16:0)	6.14	4.86	11.79
Palmitoleic (C16:1)	0.09	0.19	0.04
Margaric (C17:0)	0.09	0.11	0.10
Margaroleic (C17:1)	0.06	0.05	0.07
Stearic (C18:0)	4.11	2.39	2.44
Oleic (C18:1)	34.30	60.75	31.06
Linoleic (C18:2)	51.17	21.13	52.92
Linolenic (C18:3)	2.23	7.27	1.35
Arachidic (C20:0)	0.17	0.51	0.08
Eicosenoic (C20:1)	0.17	0.44	---
Behenic (C22:0)	0.41	0.71	---
Erucic (C22:1)	0.53	1.41	---

Transesterification of the above mentioned oils was carried out in given operating conditions. Conversion of triglyceride to methyl ester was calculated for each reaction according to equation 1 and the results have been reported in Table 2. From the reported results, it is obvious that reaction rate is very fast and nearly 90% of reaction has occurred in the first five minutes. First, methyl ester concentration is changed intensively during a short time and then concentration is almost constant. In fact, after these points reactions continue very slowly. Therefore, most

of the variations of each physical property should happen in the first step of reaction. In order to monitor the physical properties changes during the reaction it is needed to take samples before 5 minutes. But it is too much difficult to take a sample in this step, due to the fast reaction rate. Therefore, to solve this problem, the blends consist of unconverted oil and produced biodiesel in different levels, mentioned in “Methods” section, were used as an incomplete reaction product.

**Table 2.** Weight percent of methyl ester in transesterification products in different reaction time.

Reaction time (min.)	wt% methyl ester in produced biodiesel		
	Sunflower	Canola	Corn
0	0.0	0.0	0.0
5	89.8	90.1	92.3
10	93.9	94.9	94.1
20	94.3	94.5	94.3
30	94.5	94.6	94.7
45	94.9	94.6	95.1
60	95.2	94.7	95.6

All physical properties measurements have been done on these samples as described above. Measured data for sunflower, canola and corn biodiesel blends have been summarized in Table 3. Variations of physical properties have been shown in Figs. 2-7 as a function of weight percent of total methyl ester in blends. Depending on the presented data except cloud point and pour point all of the other reported properties decrease during transesterification, by increasing in methyl ester content in products. In all cases, linear regression was used to correlate data, because of its simplicity and linear behavior of data points. All fitted linear equations and their parameters have been reported in Table 4. In Fig. 2 specific gravity, at 60°F, has been drawn as a function of mass percent of methyl ester. According to the results for all oil types, slope of equations are equal to -0.0004 and the intercept of each equation is very close to oil specific gravity, and, consistency criteria (R-squared value) are more than 0.99. Since, variation of viscosity was

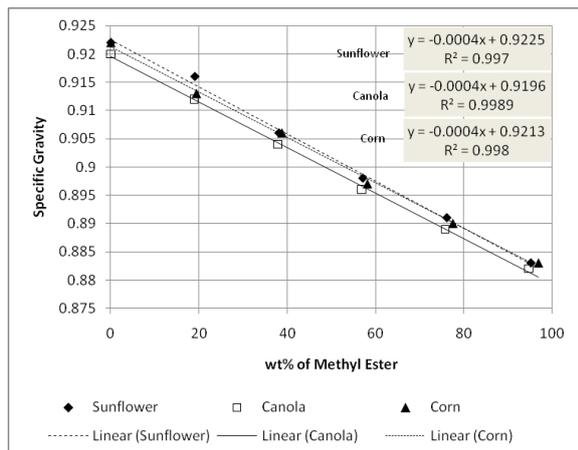
nonlinear and it was very similar to exponential function, natural logarithm of viscosity, at 40°C, was fitted as linear function of methyl ester content (Fig.3). According to the results for all oil types, slope of equations are close to -0.019 and the intercept of each equation is very close to natural logarithm of oil viscosity. R-squared value is around 0.99. Fig. 4 shows refractive index of mentioned samples, at 40°C, as function of mass percent of methyl ester. According to the results for all oil types, slope of equation is equal to -0.0002 and the intercept of each equation is very close to oil refractive index. R-squared values are close to 0.999. Variations of cloud point versus methyl ester content have been presented in Fig. 5. Slope of all equations are around 0.1 and there are few differences among them. R-squared values are more than 0.9. The intercepts of these equations are around one degree less than cloud point of pure oil.

**Table 3.** Physical properties of sunflower, canola and corn biodiesel blends in different percentages.

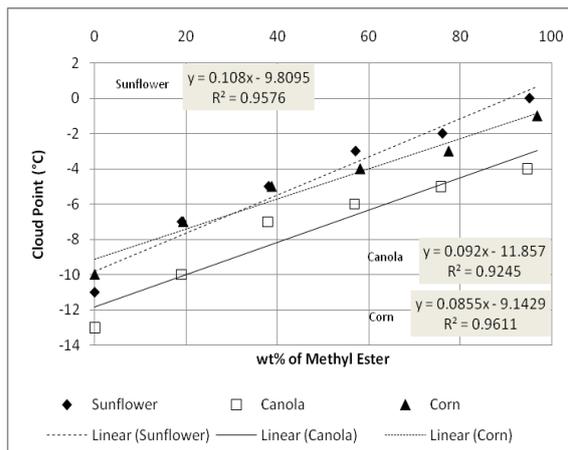
Sample name	wt% of total Methyl Ester	sp.gr. at 60°F	Viscosity at 40°C (cP)	Refr. Index at 40°C	Cloud Point (°C)	Pour Point (°C)	Flash Point (°C)
Sunflower biodiesel blends							
B/Sun 0	0	0.922	25.62	1.4669	-11	-12	195
B/Sun 20	19.04	0.916	17.46	1.4640	-7	-9	191
B/Sun 40	38.08	0.906	10.53	1.4598	-5	-6	185
B/Sun 60	57.12	0.898	7.44	1.4563	-3	-6	182
B/Sun 80	76.16	0.891	5.33	1.4528	-2	-3	176
B/Sun 100	95.2	0.883	3.98	1.4494	0	-3	170
Canola biodiesel blends							
B/Can 0	0.0	0.920	26.59	1.4655	-13	-18	210
B/Can 20	18.94	0.912	15.91	1.4620	-10	-15	196
B/Can 40	37.88	0.904	10.72	1.4589	-7	-12	190
B/Can 60	56.82	0.896	7.53	1.4550	-6	-12	184
B/Can 80	75.76	0.889	5.25	1.4516	-5	-9	177
B/Can 100	94.7	0.882	4.01	1.4481	-4	-6	172
Corn biodiesel blends							
B/Cor 0	0.0	0.922	26.08	1.4666	-10	-12	197
B/Cor 20	19.38	0.913	14.57	1.4628	-7	-9	186
B/Cor 40	38.76	0.906	10.25	1.4594	-5	-9	182
B/Cor 60	58.14	0.897	7.36	1.4559	-4	-6	176
B/Cor 80	77.52	0.890	5.21	1.4524	-3	-6	172
B/Cor 100	96.9	0.833	3.77	1.4490	-1	-3	168

In Fig. 6 pour point of the biodiesel samples have been drawn as function of methyl ester content. This property increases by slope of around 0.1 and there are little differences among them depending on oil type. R-squared values are somewhat more than 0.9. The intercepts of these equations are about 0.5-1 degree less than pour point of pure oil. In the case of flash point, data and adapted trend lines have been presented in Fig. 7. This property decreases by increasing in methyl ester content in biodiesel samples by slope of near -0.3 and intercept are around pure oil flash point. R-squared values are slightly more than 0.95. In all cases, consistency criteria ( $R^2$ ) is more than 0.9 which shows a linear behavior of data point, especially in case of specific gravity, viscosity and refractive index. In these cases, in addition to excellent accordance, there is

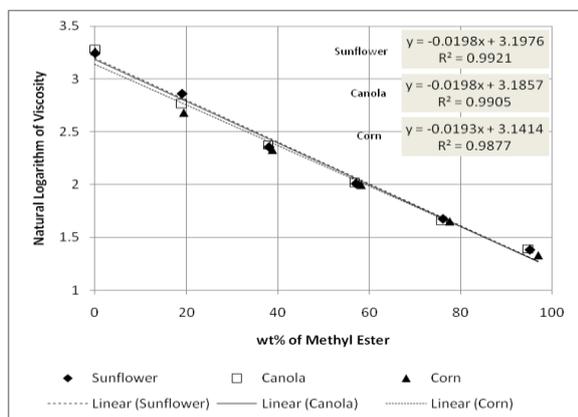
remarkable uniformity among model parameters in various oil types. Although, there are some differences in property levels in several oils, decreasing rates are exactly the same. On the other hand, the intercepts of model equations can be determined depending on fresh oil property. Hence, it is clear that these properties of oil decrease with fixed slope from oil property during transesterification reaction. It means that the rate of change in physical property is independent from oil type, however the intercept of each equation depends on oil type, completely. Although, this conclusion is approved also by the other properties including cloud point, pour point, and flash point, some irregularities are observed, too. It might be due to the test methods and unavoidable experimental errors or as a result of nonlinear behavior of variations.



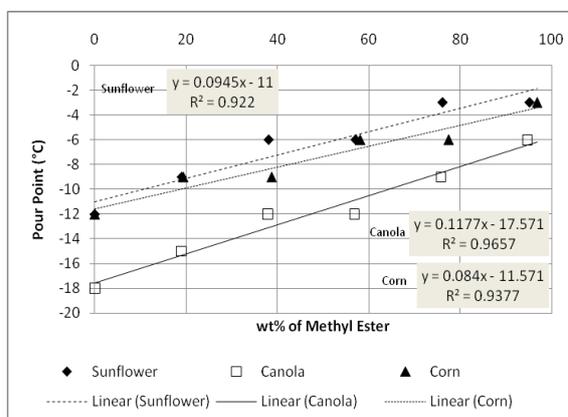
**Fig. 2.** Specific Geravity of biodiesel blends at 60°F versus wt% of Methyl Ester.



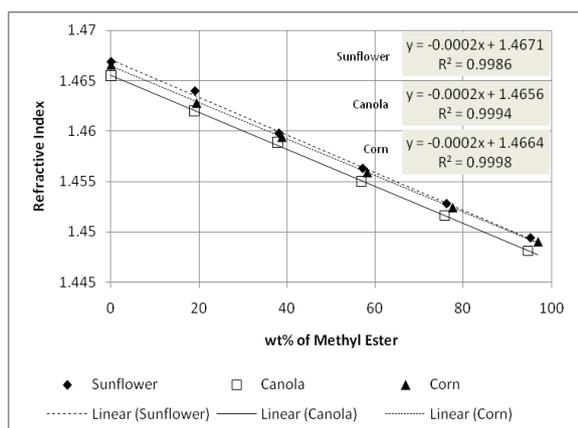
**Fig. 5.** Cloud point of biodiesel blends versus wt% of Methyl Ester.



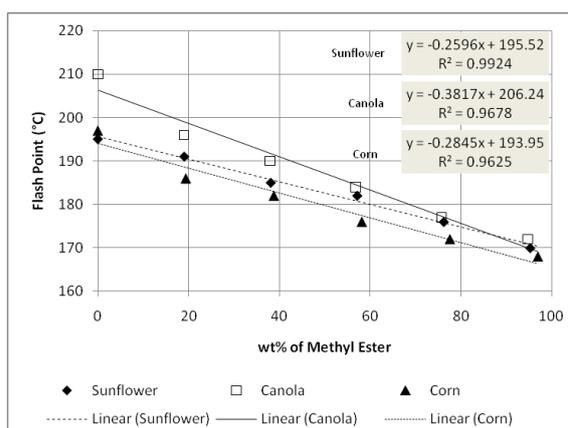
**Fig. 3.** Natural logarithm of viscosity of biodiesel blends at 40°C versus wt% of Methyl Ester.



**Fig. 6.** Pour point of biodiesel blends versus wt% of Methyl Ester.



**Fig. 4.** Refractive Index of biodiesel blends at 40°C versus wt% of Methyl Ester.



**Fig. 7.** Flash Point of biodiesel blends versus wt% of Methyl Ester.

**Table 4.** Physical properties correlation for the three type of vegetable oil.

Vegetable oil	Slope	Intercept	R-squared value	Property of pure vegetable oil	Equation number
	a	b	R <sup>2</sup>		
Specific Gravity Correlations : Sp.Gr = a× (wt% Methyl Ester) +b					
Sunflower	-0.0004	0.9225	0.997	0.922	2
Canola	-0.0004	0.9196	0.9989	0.920	3
Corn	-0.0004	0.9213	0.998	0.922	4
Viscosity Correlations : Ln(Visc.) = a× (wt% Methyl Ester) +b					
Sunflower	-0.0198	3.1976	0.9921	3.2434 <sup>#</sup>	5
Canola	-0.0198	3.1857	0.9905	3.2801 <sup>#</sup>	6
Corn	-0.0193	3.1414	0.9877	3.2612 <sup>#</sup>	7
Refractive Index Correlations : R.I. = a× (wt% Methyl Ester) +b					
Sunflower	-0.0002	1.4671	0.9986	1.4669	8
Canola	-0.0002	1.4656	0.9994	1.4655	9
Corn	-0.0002	1.4664	0.9998	1.4666	10
Cloud point Correlations : C.P. = a× (wt% Methyl Ester) +b					
Sunflower	0.108	-9.8095	0.9576	-11	11
Canola	0.092	-11.857	0.9245	-13	12
Corn	0.0855	-9.1429	0.9611	-10	13
Pour point Correlations : P.P. = a× (wt% Methyl Ester) +b					
Sunflower	0.0945	-11	0.922	-12	14
Canola	0.1177	-17.571	0.9657	-18	15
Corn	0.084	-11.571	0.9377	-12	16
Flash point Correlations : F.P. = a× (wt% Methyl Ester) +b					
Sunflower	-0.2596	195.52	0.9924	195	17
Canola	-0.3817	206.24	0.9678	210	18
Corn	-0.2845	193.95	0.9625	197	19

# Natural logarithm of viscosity at 40°C for different oils

All results indicated that the properties and variations for sunflower and corn oil are very close to each other. It is due to similar composition of these two oil types. Also, the properties of canola oil and biodiesel are different from that of corn and sunflower, which is due to differences in composition between canola and others.

**Conclusion**

In present study, we have tried to determine the correlation between some physical properties and methyl ester content for various vegetable oils and biodiesel blends, in order to introduce a simple and fast approach for estimation of methyl ester content (TG conversion to ME) in different unknown biodiesel products. Therefore, variation of specific

gravity, viscosity, refractive index, cloud point, pour point, and flash point were investigated in transesterification process and then related correlations were obtained and compared with each other for three different oils (sunflower, canola and corn oil). Depending on the results the following points have been concluded:

- In all cases, other than viscosity, variation is linear. In the case of viscosity, natural logarithm of this property changes linearly.
- Agreement between data point and model equation is surprisingly good for specific gravity, viscosity, and refractive index and these properties decrease, linearly, in compare with fresh oil properties with slope of -0.0004, -0.019, and -0.0002, respectively. Also, the variations have shown the same behavior for all mentioned types of oils.
- For three other properties, cloud point, pour point and flash point, agreement between data point and model equation is not as well as specific gravity, viscosity, and refractive index, but they have approved the above conclusion. Variation rate for these properties was around 0.1, 0.1, and -0.3, respectively.
- Comparison between different oils shows that the rate of change is independent from the type of oil, however, intercepts of all equations are related to oil type, completely.
- Refractive index, specific gravity, and viscosity are strongly recommended as reliable physical properties to determine methyl ester content of biodiesel products. Although other properties are in agreement with these properties, but they are only appropriate for estimation.
- By replacing this approach instead of an expensive and time-consuming quantitative analysis, it is possible to reduce analysis cost and time and also simplify the research programs.

#### Acknowledgements

The authors acknowledge the analytical support of Mahidasht agro-industry co. (Nazgol), especially Mr. Karami, Mr. Sharifi and Ms. Ghorbani.

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