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Estimation of fatty acids methyl esters FAME of castor oil in biodiesel samples using refractive index, density, and dynamic viscosity measurements

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Abstract

The content of fatty acid methyl esters (FAME) is a crucial property of biodiesel and serves as an indicator of the transesterification reaction that converts vegetable oil into biodiesel. To facilitate the rapid estimation of FAME quickly in the biodiesel industries, simple methods such as measuring refractive index, density, and dynamic viscosity measurements were employed to monitor the progress of transesterification of castor oil to biodiesel. Blends of castor oil and biodiesel were mixed at different weights to find correlations between FAME content and refractive index, density, and dynamic viscosity measurements. The transesterification of castor oil with methanol was carried out to examine how the refractive index, density, and dynamic viscosity properties change in relation to the conversion ratio. The results show that the optimal conditions for the transesterification of castor oil with methanol involve using a methanolic solution of KOH at 45 °C and 150 min with 18:1 mole ratio. This study demonstrated that these measurements were simple, fast, and suitable for monitoring the conversion of castor oil to its methyl and determining the % FAME content in castor biodiesel prepared.

Keywords: Biodiesel; Castor oil; Fatty Acids; Transesterification.

تقدير أسترات المثيل للأحماض الشحمية لزيت الخروع في عينات الوقود الحيوي باستخدام قياسات معامل الإنكسارو الكثافة واللزوجة

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الخلاصة :

يعد محتوى استرات ميثيل الأحماض الدهنية خاصية هامة للوقود الحيوي ويعمل كمؤشر على تفاعل الأسترة المتبادلة الذي يحول الزيت النباتي إلى وقود ديزل حيوي.تم تقدير محتوى استرات ميثيل الأحماض الدهنية بطرق سريعة وسهلة في صناعات وقود الديزل الحيوي مثل قياس معامل الانكسار والكثافة وقياسات

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اللزوجة الديناميكية لمراقبة تقدم الأسترالمتبادلة لزيت الخروع إلى وقود الديزل الحيوي. تم خلط مزيج من زيت الخروع ووقود الديزل الحيوي بنسب مختلفة لإيجاد العلاقة بين محتوى استرات مثيل الاحماض الدهنية ومعامل الانكسار والكثافة وقياسات اللزوجة الديناميكية. تم إجراء الأسترة المتبادلة لزيت الخروع بالميثانول لفحص تغير معامل الانكسار والكثافة وخصائص اللزوجة الديناميكية فيما يتعلق بنسبة التحويل. أظهرت النتائج أن الظروف المثلى لأسترة زيت الخروع بالميثانول تضمن استخدام كحول الميثانول عند 45 درجة مئوية و 150 دقيقة بنسبة مول 18: 1. أظهرت هذه الدراسة أن هذه القياسات كانت بسيطة وسريعة ومناسبة لمتابعة تحول زيت الخروع إلى الميثيل وتحديد النسبة المئوية لاسترات مثيل الاحماض الدهنية في وقود الديزل الحيوي المحضر من الخروع.

1 Introduction

Castor oil is obtained from the Ricinus communis L. plant, which is a member of the Euphorbiaceae family and grows in large areas around the world. The studies revealed that the main constituents of castor oil are Ricinoliec Triacylglycerol and other acids like Stearic, Linoleic, oleic, and Dihydroxystearic.

Castor oil and some derivatives are important products used to manufacture lubricants, grease, soaps, paints, plasticizers, and other materials [1, 2].The primary application of Castor oil is the production of biodiesel through a transesterification reaction with alcohols and an appropriate catalyst. This process yields an alternative fuel that offers several advantages when compared with classical fuel, such as renewable, biodegradable, and reduction of pollution [3-5].

In the production of biodiesel, it is essential to rapidly monitor the fatty acid methyl ester and FAME content in the transesterification reaction. Various methods are employed to estimate the FAME content in biodiesel samples, such as gas chromatography, HPLC, NMR, and laser spectroscopy [6, 7]. However, most of these methods necessitate sample pretreatment, expensive equipment, and a longer time.

Numerous studies have explored the use of various physical properties of biodiesel to study the conversion of vegetable oil to its ester using refractive index measurements to monitor the conversion of soybean oil to their methyl and ethyl esters, a good agreement between the results obtained from GC and proposed methods [8]. Borges et al. [9] used dynamic viscosity to estimate FAME content in biodiesel samples produced from sunflower, fried, and jatropha oils trans esterified. The results showed complete agreement with the results obtained from NMR and proposed methods.

Density, refractive index, and viscosity measurements were used to monitor FAME and acylglycerols content in soybean oil biodiesel. The results proved that the proposed methods can be used as initial tools to determine the triglycerides and methyl ester of soybean oil [10]. This study investigates a simple, fast, and inexpensive method to estimate the content of FAME in castor biodiesel via refractive index, density, and dynamic viscosity.

2 Experimental

2.1 Materials

Methanol and potassium hydroxide with purity greater than 99.95% were supplied from Sigma company. Castor oil was purchased from Mom company India, which has a dynamic viscosity of 183.939 cp at 40°C, refractive index of 1.4877 at 20°C, and density of 0.954 at 20°C.

2.2 Synthesis of Castor oil methyl ester blends

To establish a relationship between the FAME content and refractive index, density, and dynamic viscosity, various weight percentages of castor oil and its methyl ester were prepared. The biodiesel used in this mixture was prepared by using 18/1 mol ratio of methanol to castor oil in transesterification reaction with 1% KOH as a methanolic alkaline catalyst at constant mixing speed of 700 rpm at 45 °C temperatures and 150 min of reaction. At the end of the reaction, glycerin was separated and leaved the top ester phase in a separating funnel. The crude methyl ester of castor oil was then neutralized using 0.1% HCl, washed with D.W to remove the acid residue and finally dried via calcium chloride as drying material and analysis [11]. The properties of biodiesel were tested and are found in the Table 1.

Table 1 : castor oil and biodiesel properties

sample	Refractive index at 20 °C	Density at 20 °C (g/cm^3)	Dynamic viscosity at 40 °C (cp)
Castor oil	1.4877	0.954	183.939
Biodiesel	1.4676	0.906	9.856

In the second step, castor oil and its methyl ester blends were prepared in quantities specified in Table 2. The mixtures were prepared by weight to ensure consistency despite variations in temperature.

Table 2 : Blends of castor oil and its methyl ester

Sample	% Castor oil	% Biodiesel
1	100	0
2	90	10
3	70	30
4	50	50
5	30	70
6	10	90
7	0	100

The refractive index, density, and dynamic viscosity were measured for this mixture, and found the correlative equations with FAME content were used to evaluate these methods.

2.3 Methyl ester analysis via GC-MS

GC-MS was used to characterize the biodiesel sample produced from the castor oil transesterification reaction. The specification of GC-MS system was illustrated as follows:

Shimadzu Gas Chromatography GC 2010 Plus, Japan equipped with Inert Cap pure wax capillary column (30 m x 0.25 mm x 0.25 µm film thickness) and Helium (99.999%) as a carrier gas at a column flow rate of 17.0 ml/min and 1 µL injection volume with Shimadzu sample introduction (AOC 20i, Japan) via Split mode (15:1) at 240 °C injector temperature. Oven temperature programming of 100 °C, held 3 min, raised to 200 °C at a heating rate of 10 °C/min, and then kept at 200 °C, held 1 min, raised to 300 °C and maintained at 300 °C for 4 min. at about 25 min run time.

QPMS 2010 Ultra Shimadzu, Japan Mass Spectrometer with Positive ionization mode at Mass Range (m/z) of 50- 600 amu at source temperature of 200 °C and transfer line temperature of 280 °C with electron energy of 70.

2.4 Physicochemical properties measurements of castor oil and biodiesel blends and prepared biodiesel samples

The refractive index was measured using ABBE Refractometer at 20 °C to an accuracy of 10^{-4}, the temperature was controlled by using a thermostatically bath with ISO 6320, while the density and dynamic viscosity were measured according to D4052 and D445, respectively [12] . Each sample was measured three times, and the average of the results was calculated.

2.5 Synthesis of biodiesel samples to evaluate the proposed methods

Several samples were prepared to find real data from castor oil transesterification progression. Various mole ratios of methanol to castor oil were applied in a transesterification reaction with 1% KOH as a methanolic alkaline catalyst at a constant mixing speed of 700 rpm at different temperatures and times of reaction, as shown in Table 3. At the conclusion of the reaction, the produced glycerin was separated and left the top ester phase at the separating funnel. The crude methyl ester of castor oil was then neutralized using 0.1% HCl, washed with D.W to remove the acid residue, and finally dried via calcium chloride as drying material and analysis [11, 12].

Table 3: Experimental parameters for transesterification reaction

Sample	mole ratio Methanol: oil	Temperature °C	Reaction time min
1	6/1	60	360
2	12/1	60	360
3	18/1	60	360
4	24/1	60	360
5	24/1	30	360
6	24/1	40	360
7	24/1	50	360
8	24/1	60	360
9	24/1	60	60
10	24/1	60	150
11	24/1	60	240
12	24/1	60	360
13	18/1	45	150

2.6 Physicochemical properties measurements of prepared biodiesel samples

The prepared biodiesel samples were tested using refractive index, density, and dynamic viscosity devices and calculated the FAME content according to equation 1,2,3, respectively.

3 Results and Discussion

3.1 Mechanism of castor oil biodiesel preparation

The first step in the preparation of castor oil methyl ester involves creating a methanolic solution of KOH to ensure proper mixing for produced biodiesel. Following the transesterification reaction, aliquot of water is added to reaction content to ensure the separation of glycerol and remove the excess methanol. Acidic solution of dil. HCl was added to the mixture to remove the excess KOH and the formed soap. Finally, the FAME is washed with water and dried with calcium chloride [13].

In the transesterification reaction of castor oil, triglycerides react with methanol in the presence of potassium hydroxide as the catalyst to produce diglycerides [14] which react with methanol to produce monoglycerides, then the latest react with methanol to produce the product and glycerol as byproduct [15].

3.2 Analysis of castor oil biodiesel using GC-MS technique

The peaks in the GC-MS chromatograph were identified by comparing them with the NIST mass spectra library (National Institute of Standards and Technology). The primary peak corresponding to methyl ester formation is ricinoleic acid, which is the main compound of castor oil, as shown in Figure 1.

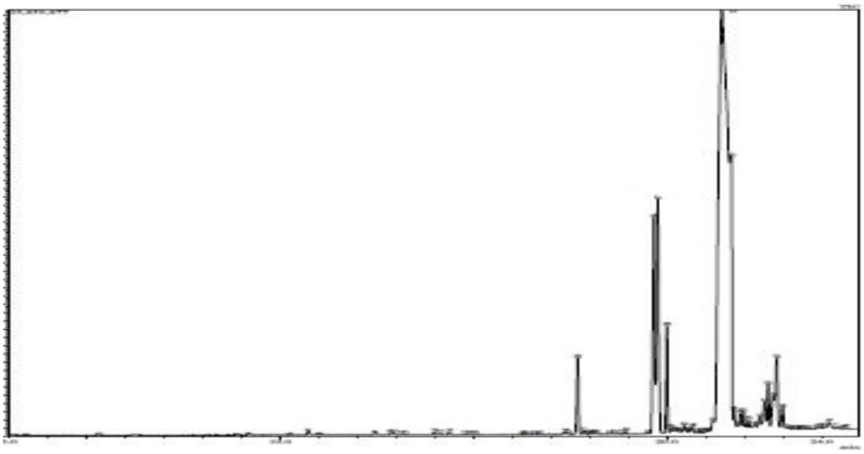

Figure 1: GC-MS ion chromatogram of castor oil methyl ester at optimal experimental preparation conditions, sample 13.

The abundance of fatty acid methyl ester showed that it resulted to 98.98%, while the other compounds not detected as methyl ester resulted in 1.02%.

3.3 Study the physical properties of blends.

3.3.1 Refractive index and ester content

The refractive index has garnered significant attention for its application in estimating the FAME content in biodiesel samples since the compounds that formed in the transesterification reaction, like mono-, di- and triglyceride of methyl ester, exhibit different refractive index values [16], as shown

% Castor oil	% Biodiesel	refractive index at 20 C
100	0	1.4877
90	10	1.486
70	30	1.4822
50	50	1.478
30	70	1.474
10	90	1.4696
0	100	1.4676

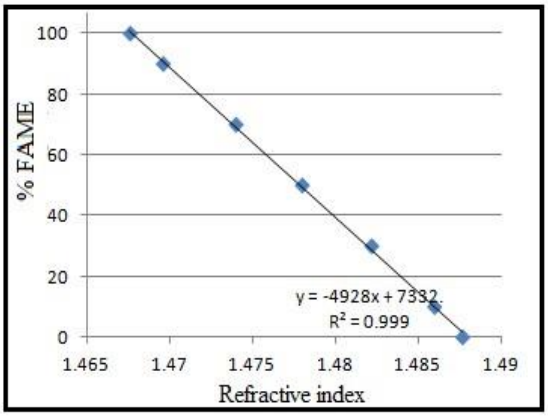

Figure 2. $Y = 4929X + 7332.9$

% Castor oil	% Biodiesel	refractive index at 20 C
100	0	1.4877
90	10	1.486
70	30	1.4822
50	50	1.478
30	70	1.474
10	90	1.4696
0	100	1.4676

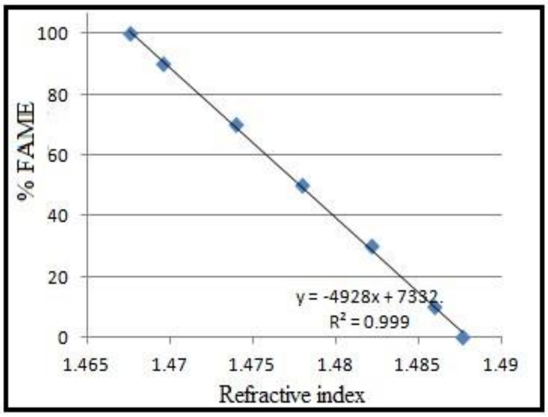

Figure 2: Refractive index of a mixture of castor oil and its methyl ester versus the percentage of FAME

3.3.2 Density and ester content

In a same manner, the density values were plotted against the %FAME in prepared samples. A nonlinear correlation was observed between methyl ester of castor oil content in mixture and its density values, Equation 2 applied to predict the % FAME in transesterification reaction, as shown in Figure 3.

$$Y = 29157X^2 - 56243X + 27120 \qquad (2)$$

% Castor oil	% Biodiesel	Density (g/cm^3)at 20 C
100	0	0.954
90	10	0.945
70	30	0.9325
50	50	0.9215
30	70	0.914
10	90	0.908
0	100	0.906

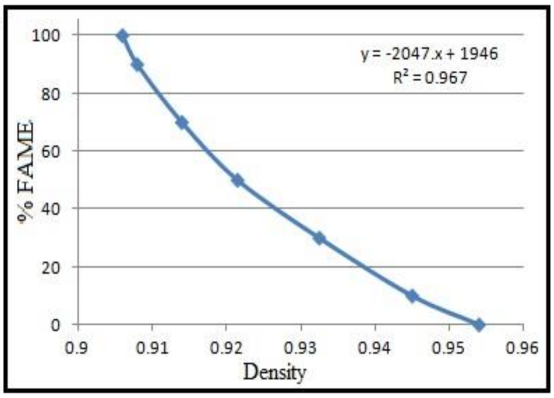

Figure 3: Density of mixture of castor oil and its methyl ester versus % FAME

3.3.3 Dynamic viscosity and ester content

Based on the variations in viscosity of castor oil and its methyl esters, viscosity measurements of biodiesel produced from castor oil was applied for the determination of the ester content, showing good agreement with results obtained from gas chromatography analysis [17]. The viscosity equation between dynamic viscosity and %FAME was applied to determine FAME content is used to monitor castor oil transesterification [18,19], as shown in Figure 4

% Castor oil	% Biodiesel	Dynamic viscosity, Cp
100	0	183.639
90	10	132.588
70	30	74.752
50	50	37.372
30	70	21.011
10	90	12.316
0	100	9.098

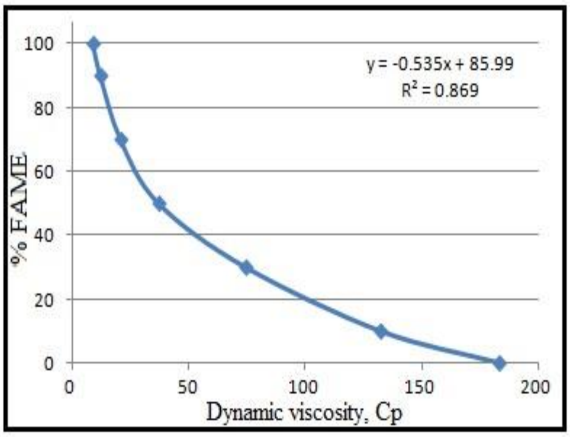

Figure 4 : Dynamic viscosity of castor oil and its methyl ester mixture versus the percentage of FAM

From studying the dynamic viscosity of the mixture, a nonlinear exponential correlation was achieved between methyl ester of castor oil content in the mixture and its dynamic viscosity values, Equation 3.

$$Y = 33.24\ln(x) + 172.48 \qquad (3)$$

The results show that the viscosity of castor oil is higher than other vegetable oils due to the presence of a hydroxyl group and unsaturated fatty acid, which signify Ricinoleic acid.

3.4 Study the best conditions of the transesterification reaction via refractive index, density and dynamic viscosity

The effect of special parameters; mole ratio, temperature and reaction time on the transesterification reaction of castor oil with methanol was studied. % FAME content was estimated according to the refractive index, density, and dynamic viscosity values achieved as physical properties using Equations 1, 2, and 3, respectively, Table 4.

Table 4 : Experimental: %FAME (Ri), % FAME (Den) , % FAME(Visc) values for samples obtained in transesterification reaction using the equations 1, 2 and 3

Sample	Refractive index at 20 °C	%FAME (Ri)	Density at 20 °C (g/cm^3)	%FAME (Den)	Dynamic viscosity at 40 °C (cp)	%FAME (Visc)
1	1.4688	94.650	0.917	62.969	15.609	81.967
2	1.4676	100.567	0.906	96.957	8.162	102.709
3	1.4680	98.596	0.908	90.252	11.700	90.742
4	1.4687	95.146	0.910	83.781	15.232	81.169
5	1.4732	72.970	0.927	38.194	30.261	59.164
6	1.4680	98.596	0.923	47.404	25.564	64.749
7	1.4676	100.567	0.906	96.957	9.856	96.426
8	1.4677	100.074	0.908	90.252	15.266	81.901
9	1.4681	98.103	0.912	77.543	15.550	81.269
10	1.4676	100.567	0.906	96.957	9.497	97.690
11	1.4688	94.650	0.917	62.969	10.339	94.864
12	1.4685	96.132	0.914	71.539	15.557	81.269
13	1.4676	100.567	0.906	96.957	9.856	96.426

3.4.1 Effect of reaction time

The %FAME content was estimated at various reaction times (60, 150, 240, and 360 min). Based on the results of refractive index, density, and dynamic viscosity, it was observed that 150 min is the optimal reaction time, yielding the highest % FAME content, as shown in Figure 5. This finding aligns with the results reported by Sanchez et.al. [20]. Although there are differences between the results of %FAME content, but they were carried out in the same pattern, that is all of them gave the highest content of FAME content at 150 min of reaction time.

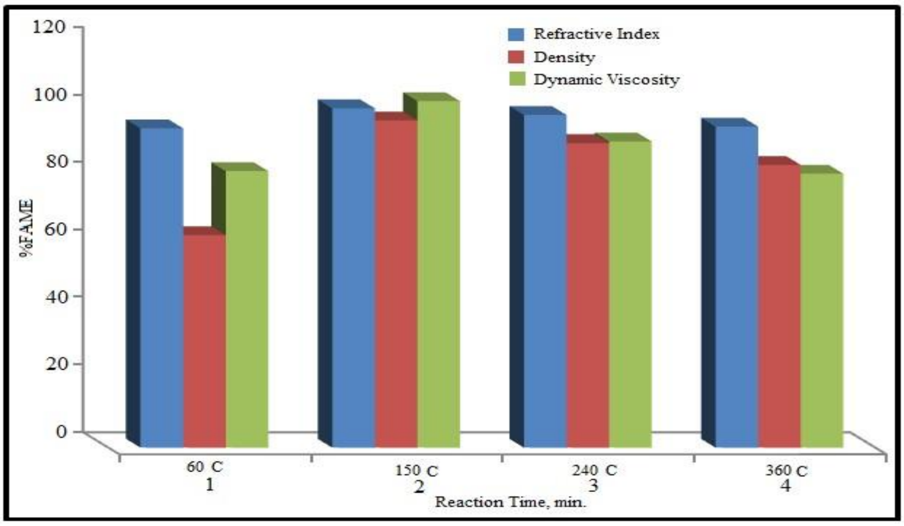

Figure 5: %FAME content with reaction time of castor oil transesterification.

3.4.2 Effect of mole ratio

The transesterification reaction requires 1 mole of castor oil and 3 moles of methanol to produce 3 moles of ester and 1 mole of glycerol but to shift the reaction to the right, it must use an excess of methanol, so a started methanol to castor oil mole ratio was 6:1 up to 24:1, Figure 6. The ester content was found to increase with increasing molar ratio until 18:1, and the ester will decrease. This may be attributed to the accumulation of methanol in the product. Additionally, it was evident that there were differences in the results obtained from refractive index, density, and dynamic viscosity measurements when calculating the ester content. This may be affected by the nature of the components that produce in reaction and have diverse values. However, the best mole ratio is 18:1, which is consistent with the value that obtained by Sanchez et.al. [21].

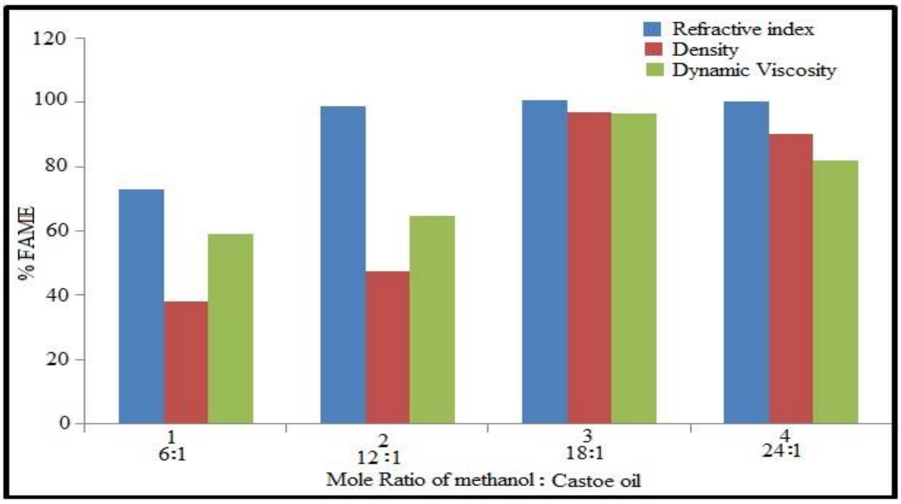

Figure 6: % FAME content with mole ratio of transesterification of castor oil

3.4.3 Effect of reaction temperature

The temperature of transesterification reaction is another important factor critical factor influencing biodiesel yield. The results indicate that the reaction reaches steady state quickly at a temperature 45 °C, due to the high polarity of Ricincolic acid and methanol which gave a complete miscibility at room temperature, Fig 7. It is observed that 45 °C is the best temperature to yield the higher content of % FAME according to the refractive index, density, and dynamic viscosity results, which agree entirely with the result revealed by Sanchez et.al. [21].

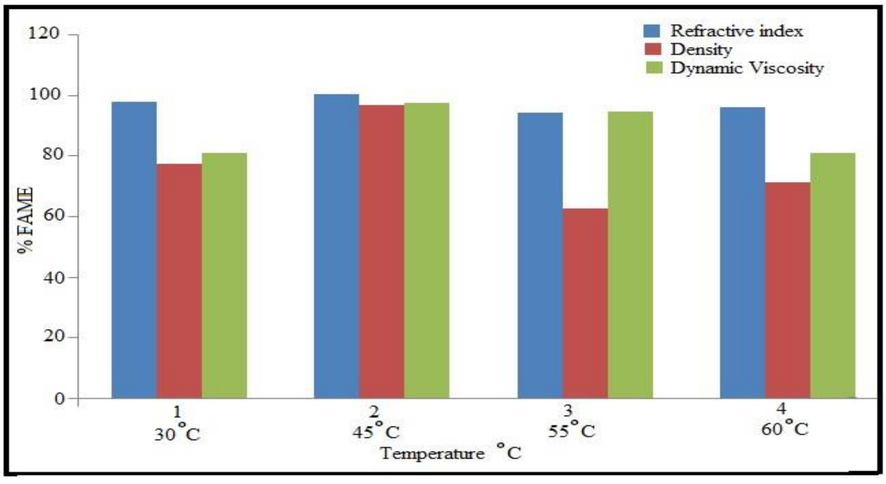

Figure 7: % FAME content with transesterification reaction temperature of castor oil

3.5 Comparison of the FAME content using different techniques

The optimal preparation condition of the biodiesel [22] sample was analyzed by GC-MS to determine the FAME and compare them with the proposed methods. The FAME analysis demonstrates strong correlation between the proposed methodologies, with error values falling within acceptable ranges. This means these methods are suitable for monitoring the conversion of castor oil to its methyl ester. Analysis further confirms that the sample consists mainly of just two components: triglycerides and their corresponding methyl esters, as shown in Table 5. The higher errors were obtained by dynamic viscosity and density methods, this may be referred to the presence of trace amounts of mono and diglycerides, which affect the values of the blend.

Table 5 : FAME% content measured by GC-MS and proposed method with their relative error

Method	%FAME	Equation	R^2	Error between GC-MS and proposed methods	
				Absolute	Relative (%)
GC-MS	98.980				
Refractive index	100.567	$Y= - 4928 X +7332.9$	0.9983	1.587	1.603
Density	96.957	$Y= 29157 X^2 - 56243 X+27120$	0.9976	2.023	2.043
Dynamic viscosity	96.426	$Y= -33.24 \ln (x) + 172.48$	0.9990	2.554	2.580

Conclusions

The relationship between % FAME content and refractive index demonstrated strong linearity while the relationships with density and dynamic viscosity produced exponential curves. A comparison between the result of %FAME content via GC-MS and the values calculated through these proposed correlations revealed excellent agreement between them. In summary, this study has demonstrated that simple, fast, and inexpensive methods such as refractive index, density, and dynamic viscosity can be effectively used to estimate the FAME% content in biodiesel samples.

Conflicts of interest

There are no conflicts to declare.

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