Int J Thermophys (2009) 30:529–541
DOI 10.1007/s10765-009-0569-3

Density of *Jatropha curcas* Seed Oil and its Methyl Esters: Measurement and Estimations

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Received: 21 July 2008 / Accepted: 4 February 2009 / Published online: 24 February 2009

Abstract Density data as a function of temperature have been measured for *Jatropha curcas* seed oil, as well as biodiesel jatropha methyl esters at temperatures from above their melting points to 90 °C. The data obtained were used to validate the method proposed by Spencer and Danner using a modified Rackett equation. The experimental and estimated density values using the modified Rackett equation gave almost identical values with average absolute percent deviations less than 0.03 % for the jatropha oil and 0.04 % for the jatropha methyl esters. The Janarthanan empirical equation was also employed to predict jatropha biodiesel densities. This equation performed equally well with average absolute percent deviations within 0.05 %. Two simple linear equations for densities of jatropha oil and its methyl esters are also proposed in this study.

Keywords Biodiesel · Density · *Jatropha curcas* seed oil · Transesterification

1 Introduction

Biodiesel is a renewable fuel produced from biological oils and fats, which has many characteristics of a promising alternative energy resource. The most common process for making biodiesel is known as transesterification. It has properties similar to ordinary diesel fuel made from crude oil and can be used in conventional diesel engines. This process involves combining any natural oil (vegetable or animal) with alcohol and a catalyst.

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Rapeseed, soybean, and palm oils are the most commonly used oils to produce biodiesel, although other crops such as corn, mustard, hemp, jatropha, ricebran, and even algae show great potential as a source of raw materials. To compete economically with petroleum-based diesel fuel, biodiesel production costs can be reduced using less expensive vegetable oils including non-edible vegetable oils.

Currently, the demand for edible oils such as soybean, corn, and palm oil for biodiesel is growing fast and their prices are rising. Rising food prices as a result of growing demand is a concern for global food security. Development of non-edible oil-based biodiesel production can decrease the risk of food security.

Jatropha (*Jatropha curcas*) is one of such non-edible vegetable oils, which has great production potential; it can be grown in very poor soils or idle lands and it has seeds with 30 % to 40 % approximate oil content. The jatropha oil may be the key to addressing the problems of energy and food self-sufficiency. Cultivating the jatropha plant does not only provide biodiesel but can also ensure that agricultural lands devoted to food production will not be diverted to fuel crops.

Density or specific gravity data are important in numerous chemical engineering unit operations. In the oleochemical industries, lipid density data is needed to design reactors for splitting of fatty acids or conversion of fatty acids to their derivatives, for distillation units for separation of fatty acids, for storage tanks, and for process piping [1]. Biodiesel density data as a function of temperature are needed to model the combustion processes and other applications. The engine injection systems (the pump and the injectors) are set to deliver a predetermined volume of fuel, whereas, in the combustion chamber, the determining parameter is the air:fuel mass ratio [2].

The density of a methyl ester depends on its molar mass, free fatty acid content, water content, and temperature. As jatropha oil biodiesel is getting popular as a fuel, a similar procedure for correcting measured density and specific gravity data will be needed. Results of measurements and predictions of specific biodiesel properties have been reported recently but jatropha oil biodiesel density measurements and predictions were rarely (or possibly never) done.

The densities of fatty acids and biodiesel as a function of temperature were obtained by Liew et al. [3], Tate et al. [4], Tat and Van Garpen [5], and Noureddini et al. [1]. Yuan et al. [6] predicted the density of biodiesel using the modified Rackett equation. The density of palm oil methyl ester biodiesel was measured and predicted by Baroutian et al. [7]. Demirbas [8] obtained density data for the ethyl esters of vegetable oils produced by a non-catalytic supercritical ethanol method. Mathematical relationships were also derived for several properties. In another study, Demirbas [9] studied the density of ethyl esters obtained from cottonseed oil through transesterification using a non-catalytic supercritical fluid. Benjumea et al. [10] studied the density of palm oil methyl ester-diesel blends. Demirbas [11] studied the relationship between density and other physical properties of various biodiesel fuels. The density of rapeseed oil methyl esters was measured by Dzida and Prusakiewicz [12]. Aparicio et al. [13] determined the rapeseed oil methyl ester density at high pressure.

The specific objective of the research presented here was to measure the densities of *Jatropha curcas* oil and its methyl ester biodiesel as a function of temperature and describe a method of estimating the density based on the chemical composition and structure.

2 Experimental

2.1 Materials

Jatropha curcas oil was purchased from PT. Kreatif Energi Indonesia (KEI); absolute methanol was obtained from Sigma-Aldrich, Malaysia. Pure potassium hydroxide and sulfuric acid were used as catalysts and obtained from Sigma-Aldrich, Malaysia. Reference standards such as methyl oleate, methyl palmitate, methyl linoleate, and methyl stearate of $>99\,\%$ purity were supplied from Sigma-Aldrich, Malaysia.

2.2 Biodiesel Preparation

Transesterification reactions were performed in a batch reactor. As can be seen in Fig. 1, the reaction setup included a 0.5 L jacketed glass vessel equipped with a thermometer and water-cooled condenser. A mechanical stirrer (Kika® Werke) fitted with a stainless-steel propeller provided the mixing requirement. The reaction temperature was set and controlled by hot water circulation using an RCS and RC6 refrigerated circulator (Lauda).

A two-step transesterification process was used to convert *Jatropha curcas* oil to its methyl esters. The two-step process was used because of the high free fatty acid content of jatropha oil which causes fatty acid salts (soap) formation during base-catalyzed transesterification. The soap could prevent separation of the methyl ester layer from the glycerin fraction.

The first step was acid esterification and pretreatment for removing free fatty acids in the oil, which is mainly a pretreatment process. The process was followed by base catalyst transesterification to obtain complete conversion.

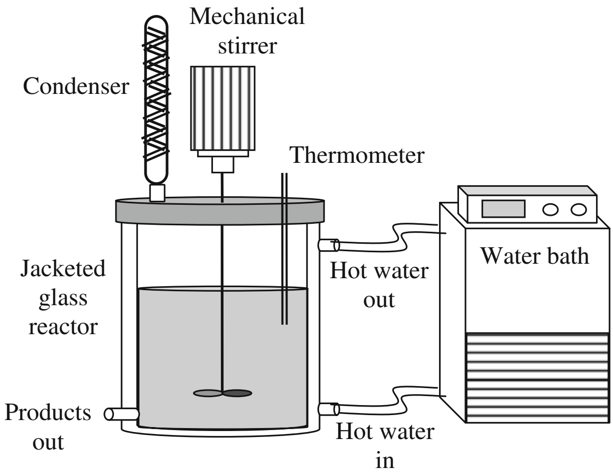

Fig. 1 Jatropha biodiesel production experimental setup

2.2.1 Pretreatment—First Step

In this step, free fatty acids were first converted to esters in a pretreatment process with methanol using an acid catalyst (H_2SO_4). The reactor was filled with 60 g jatropha oil. The catalyst, sulfuric acid (99 %), was dissolved in methanol and then added to the reactor at a reaction temperature of 70 °C. Agitation was set at a constant speed (700 rpm) throughout the experiment. The reaction was carried out using an oil:methanol:catalyst molar ratio of 1:100:3.8. After 3.5 h of reaction, the mixture was allowed to settle for 12 h and the methanol–water fraction at the top layer was removed.

2.2.2 Transesterification—Second Step

In the second step, the product of the pretreatment step was poured into the reactor and heated at 50 °C. The catalyst, potassium hydroxide (2 %), was dissolved in methanol and then added to the reactor. The reaction was carried out using 100 % excess methanol, i.e., the molar ratio of methanol to oil is 6:1. After the end of the reaction (1.5 h), the mixture was cooled to room temperature and transferred to a separatory funnel. The two layers were separated by sedimentation. The methyl ester phase was washed with hot distilled water. In order to avoid an emulsion during the washing step, 0.1 % aqueous tannic acid was used as a washing solution. The excess methanol was removed on a rotary evaporator at atmospheric pressure.

2.3 Fatty Acid Composition Analysis

The composition of the esters was analyzed by gas chromatography using an HP 6890 series gas chromatograph system equipped with a flame ionization detector and automated split injector (Agilent 7683 automatic sampler). The column was a 60 m × 0.248 mm × 0.15 µm DB-23 capillary column (J & W Scientific, USA). According to the literature, the major fatty acids in jatropha oil are palmitic acid (14.1 % to 15.3 %), stearic acid (3.7 % to 9.8 %), oleic acid (34.3 % to 45.8 %), and linoleic acid (29 % to 44.2 %) [14–17].

2.4 Density Measurements

Density measurements were carried out using a DMA 4500 density/specific gravity meter (Anton Paar, Austria). The adjustment of the density meter was checked using degassed bi-distillated water; the measured value at 25 °C was compared with the corresponding value in the density tables, and the uncertainty was $0.00003\,\mathrm{g \cdot cm^{-3}}$. Jatropha oil and jatropha methyl ester densities were measured at temperatures ranging from 15 °C to 90 °C and measurements were done three times to obtain mean values for each temperature. Depending on the temperature, the density uncertainty was estimated to be between $0.00001\,\mathrm{g \cdot cm^{-3}}$ and $0.00002\,\mathrm{g \cdot cm^{-3}}$ and the temperature uncertainty was 0.01°C.

3 Estimations

3.1 Janarthanan Empirical Method

A simple method for estimating the density of methyl esters of fatty acids is to use the empirical relation developed by Janarthanan et al. [18];

$$\rho = a_i(t) + b_i \tag{1}$$

where ρ is the density ($\mathrm{g \cdot cm^{-3}}$), t is the temperature (°C), and the component-dependent empirical constants, a_i and b_i, are reported in Table 1.

The density of jatropha methyl ester which is a mixture of these components can be estimated using a simple linear mixing rule, as shown in Eq. 2.

$$\rho_m = \sum x_i(a_i(t) + b_i) \tag{2}$$

where x_i is the mole fraction of fatty acid methyl esters.

3.2 Spencer and Danner Method

The critical properties of fatty acids or biodiesel are the important starting points to estimate the liquid density. Reid et al. [19] recommended two different methods to compute the critical properties of each pure constituent and to achieve high accuracy in the estimation of the mixture properties.

The first method is Ambrose's method [20,21]. In this method the three critical properties, T_c (K), P_c (bar), and V_c ($\mathrm{cm^3 \cdot mol^{-1}}$), are estimated by the following relations:

$$T_c = T_b\left[1 + \left(1.242 + \sum \Delta_T\right)^{-1}\right] \tag{3}$$

$$P_c = M\left(0.339 + \sum \Delta_P\right)^{-2} \tag{4}$$

$$V_c = 40 + \sum \Delta_V \tag{5}$$

where, T_b (K) is the normal boiling point, M ($\mathrm{g \cdot mol^{-1}}$) is the molar mass, and Δ_T, Δ_P, and Δ_V are constants for Ambrose's method given in Table 2–1 in Reid et al. [19].

Table 1 Empirical constants of Eq. 1 used to estimate methyl ester densities

Methyl ester	a_i	b_i
Palmitate	-7.4608×10^{-4}	0.879094
Stearate	-6.9247×10^{-4}	0.877325
Oleate	-6.8563×10^{-4}	0.888357
Linoleate	-7.2226×10^{-4}	0.900981

The second method is the Joback modification of Lydersen's method [22]. The proposed relations for the critical properties are

$$T_c = T_b \left[0.584 + 0.965 \sum \Delta'_T - \left(\sum \Delta'_T \right)^2 \right]^{-1} \tag{6}$$

$$P_c = \left(0.113 + 0.0032 n_A - \sum \Delta'_P \right)^{-2} \tag{7}$$

$$V_c = 17.5 + \sum \Delta'_V \tag{8}$$

The Δ'_T, Δ'_P, and Δ'_V values are given in Table 2- 2 in Reid et al. [19].

Reid et al. [19] concluded that Ambrose's method was more accurate for estimation of the critical temperature and critical pressure while the Joback modification of Lydersen's method yielded higher accuracy in the estimation of critical volumes. For that reason, Ambrose's method was employed in this study to estimate T_c and P_c and the Joback modification of Lydersen's method was used for V_c.

To determine the mixture critical properties, Lee-Kesler mixing rules recommended by Knapp et al. were used [23]. The equations are

$$T_{\mathrm{cm}} = \frac{1}{V_{\mathrm{cm}}^{1/4}} \sum_i \sum_j x_i x_j V_{\mathrm{c}ij}^{1/4} T_{\mathrm{c}ij} \tag{9}$$

$$P_{\mathrm{cm}} = (0.29050 - 0.085\omega_m) R T_{\mathrm{cm}} / V_{\mathrm{cm}} \tag{10}$$

$$V_{\mathrm{cm}} = \sum_i \sum_j x_i x_j V_{\mathrm{c}ij} \tag{11}$$

where

$$T_{\mathrm{c}ij} = (T_{\mathrm{c}i} T_{\mathrm{c}j})^{1/2} k'_{ij} \tag{12}$$

$$\omega_{\mathrm{m}} = \sum_i x_i \omega_i \tag{13}$$

$$V_{\mathrm{c}ij} = \frac{1}{8} \left(V_{\mathrm{c}i}^{1/3} + V_{\mathrm{c}j}^{1/3} \right)^3 \tag{14}$$

In this study, binary parameters k'_{ij} were simplified to be unity because no published data are available. The pure component acentric factor (ω_i) was computed by [19]

$$\omega_i = \frac{\alpha_i}{\beta_i} \tag{15}$$

where

$$\alpha_i = -\ln P_{ci} - 5.97214 + 6.09648\theta_i^{-1} + 1.28862\ln\theta_i - 0.169347\theta_i^6 \tag{16}$$

$$\beta_i = 15.2518 - 15.6875\theta_i^{-1} - 13.4721\ln\theta_i + 0.43577\theta_i^6 \tag{17}$$

$$\theta_i = \frac{T_{bi}}{T_{ci}} \tag{18}$$

The Rackett equation modified by Spencer and Danner [24] to estimate liquid densities is expressed as

$$\rho = \frac{\rho_R}{Z_{RA}^{\phi}} \tag{19}$$

where

$$\phi = \left(1 - \frac{T}{T_c}\right)^{2/7} - \left(1 - \frac{T_R}{T_c}\right)^{2/7} \tag{20}$$

ρ_R and T_R in Eqs. 19 and 20 are the reference density and reference temperature, respectively. Z_{RA} is the Rackett compressibility factor and can be determined for jatropha oil and jatropha biodiesel using measured densities and applying Eq. 19.

4 Results and Discussion

4.1 Fatty Acid Composition

The compositions of esters were detected by GC-FID. The fatty acid composition of the methyl esters of jatropha oil is presented in Table 2.

4.2 Density Measurements

Densities were determined at temperatures ranging from 15 °C to 90 °C at 1 °C intervals, and the results are shown in Tables 3 and 4. The results show that the liquid

Table 2 Fatty acid mole fraction x of jatropha oil methyl esters

Fatty acid	x
C 16:0	0.16
C 18:0	0.07
C 18:1	0.45
C 18:2	0.32

Table 3 Density ρ of jatropha oil

t (°C)	ρ $(\text{g} \cdot \text{cm}^{-3})$	t (°C)	ρ $(\text{g} \cdot \text{cm}^{-3})$	t (°C)	ρ $(\text{g} \cdot \text{cm}^{-3})$
15	0.91725	40	0.90021	66	0.88263
16	0.91662	41	0.89953	67	0.88195
17	0.91593	42	0.89885	68	0.88127
18	0.91525	43	0.89817	69	0.88060
19	0.91456	44	0.89750	70	0.87993
20	0.91387	45	0.89681	71	0.87925
21	0.91316	46	0.89613	72	0.87858
22	0.91250	47	0.89545	73	0.87791
23	0.91182	48	0.89478	74	0.87723
24	0.91114	49	0.89410	75	0.87656
25	0.91042	50	0.89343	76	0.87589
26	0.90977	51	0.89275	77	0.87522
27	0.90909	52	0.89207	78	0.87455
28	0.90840	53	0.89140	79	0.87388
29	0.90772	54	0.89072	80	0.87321
30	0.90704	55	0.89005	81	0.87254
31	0.90636	56	0.88937	82	0.87187
32	0.90568	58	0.88870	83	0.87120
33	0.90500	59	0.88802	84	0.87053
34	0.90432	60	0.88734	85	0.86986
35	0.90363	61	0.88667	86	0.86919
36	0.90290	62	0.88599	87	0.86852
37	0.90225	63	0.88532	88	0.86785
38	0.90157	64	0.88465	89	0.86718
39	0.90087	65	0.88397	90	0.86648

densities of jatropha oil and jatropha biodiesel decrease linearly with an increase in temperature. The densities of jatropha oil are considerably higher than those of jatropha methyl esters. This can be attributed to the molar mass differences.

The accuracies of the density data were further evaluated by correlating them with temperature by means of Eq. 21;

$$\rho = n + m(t) \tag{21}$$

where ρ is the density $(\text{g} \cdot \text{cm}^{-3})$, t is the temperature (°C), and the values of n, m are given in Table 5. Good correlations were obtained, and the correlation coefficients are 1.

To compare the measured density of jatropha oil with other vegetable oils, density data of various oils (rapeseed, soybean, milkweed, lesquerella) from the study of Noureddini et al. [1] were employed. The comparisons are shown in Fig. 2.

The measured density of jatropha oil methyl ester was compared with the methyl esters of canola, soy, and fish oil from the study of Tate et al. [4] and palm oil methyl

Table 4 Density ρ of jatropha methyl ester

t (°C)	ρ ($g \cdot cm^{-3}$)	t (°C)	ρ ($g \cdot cm^{-3}$)	t (°C)	ρ ($g \cdot cm^{-3}$)
15	0.88032	40	0.86212	66	0.84314
16	0.87960	41	0.86140	67	0.84242
17	0.87887	42	0.86067	68	0.84169
18	0.87814	43	0.85983	69	0.84096
19	0.87742	44	0.85910	70	0.84024
20	0.87669	45	0.85838	71	0.83951
21	0.87596	46	0.85768	72	0.83878
22	0.87522	47	0.85695	73	0.83806
23	0.87450	48	0.85622	74	0.83733
24	0.87376	49	0.85550	75	0.83659
25	0.87304	50	0.85477	76	0.83588
26	0.87231	51	0.85404	77	0.83515
27	0.87157	52	0.85331	78	0.83443
28	0.87088	53	0.85259	79	0.83370
29	0.87012	54	0.85186	80	0.83297
30	0.86939	55	0.85113	81	0.83225
31	0.86867	56	0.85040	82	0.83152
32	0.86794	58	0.84968	83	0.83079
33	0.86721	59	0.84895	84	0.83004
34	0.86648	60	0.84820	85	0.82933
35	0.86575	61	0.84750	86	0.82860
36	0.86503	62	0.84677	87	0.82787
37	0.86430	63	0.84604	88	0.82714
38	0.86360	64	0.84532	89	0.82642
39	0.86286	65	0.84460	90	0.82567

Table 5 Linear correlations[a] for jatropha oil and jatropha methyl esters

n	m	Correlataion coeficient	
Jatropha oil	0.9273	–0.0007	1.0000
Jatropha methyl esters	0.8912	–0.0007	1.0000

[a] $\rho = n + m(t)$

esters from the study of Baroutian et al. [7]. The comparisons are shown in Fig. 3, and the density of jatropha oil methyl ester is higher than those for other methyl esters.

4.3 Density Estimations

The estimated critical properties of fatty acids and fatty acid methyl esters are listed in Table 6. The results of applying critical properties T_{ci}, P_{ci}, and V_{ci} from Table 6 in

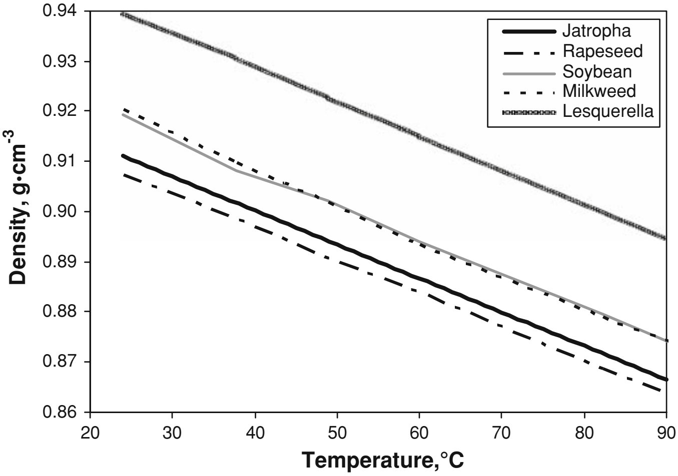

Fig. 2 Density comparisons of Jatropha oil with rapeseed, soybean, milkweed, and lesquerella oil [1]

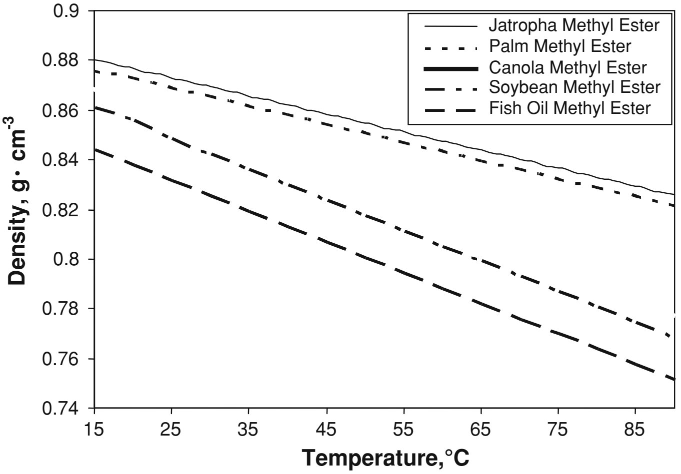

Fig. 3 Density comparisons of Jatropha methyl ester with methyl esters of palm, canola, soybean, and fish oil [4,7]

Table 6 Estimated critical properties of the fatty acids and fatty acid methyl esters; T_c and P_c obtained using Ambrose's method and V_c using Lydersen's method

Property	Fatty acids				Fatty acid methyl esters			
	C16:0	C18:0	C18:1	C18:2	C16:0	C18:0	C18:1	C18:2
T_c (K)	893.76	938.39	959.06	982.79	767.1	774.2	772.3	795.3
P_c (bar)	14.68	13.26	16.68	21.67	12.50	11.46	11.68	11.91
V_c ($\text{cm}^3 \cdot \text{mol}^{-1}$)	955.50	1067.50	1047.50	1027.50	1013.50	1125.50	1105.50	1085.50
M · W	256.43	284.48	282.46	280.44	270.46	298.50	296.49	294.47
T_b (K)	711	757	761	765	611	625	622	639

Table 7 Properties for use with the Rackett equation

Property	Jatropha oil	Jatropha methyl esters
T_c (K)	954.56	778.89
P_c (bar)	15.21	12.35
V_c $(\text{cm}^3 \cdot \text{mol}^{-1})$	1027.59	1085.02
Z_{RA}	0.14761	0.19898

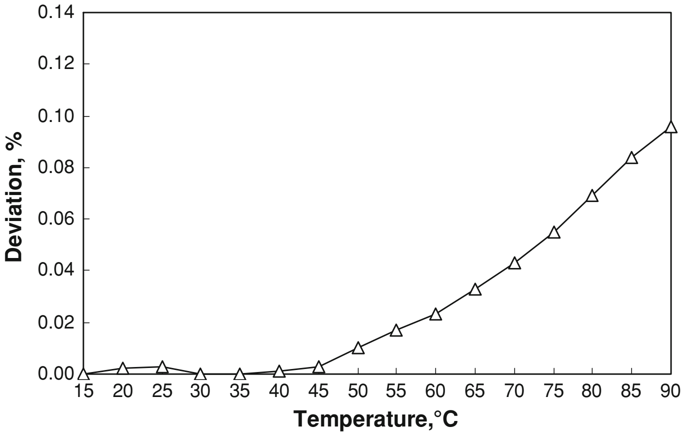

Fig. 4 Comparison of jatropha oil density estimation deviations from experimental data

the mixing rules of the Lee-Kesler equation to be used with the Rackett equation are shown in Table 7. These values were used in the predictions of jatropha oil and jatropha methyl ester densities. By selecting $T_R = 288.15\,\text{K}$ and corresponding densities and solving Eq. 19 for the experimental densities at $T = 303.15\,\text{K}$, the Rackett compressibility factor was determined (Table 7). The predicted densities of jatropha oil methyl ester jatropha biodiesel at temperatures ranging from 15 °C to 90 °C using the Rackett equation modified by Spencer and Danner are shown in Figs. 4 and 5. The average absolute percent deviations (AAPDs) between estimated values using the modified Rackett equation and experimental data were less than 0.03 % for the jatropha oil and 0.04 % for the jatropha methyl esters. The AAPD was calculated according to

$$\text{AAPD} = \frac{1}{N}\sum_{i=1}^{N}\left|\left(\frac{\text{Density}_{\text{measured}} - \text{Density}_{\text{estimated}}}{\text{Density}_{\text{measured}}}\right)_i\right| \times 100 \tag{22}$$

The results of using the empirical method proposed by Janarthanan to estimate methyl ester densities are shown in Fig. 5. It was found that the AAPDs between estimated values and experimental data were less than 0.05 %. As seen from Fig. 5, the accuracy of the estimates is reasonably good, but the Rackett equation method is more accurate, especially at lower temperatures.

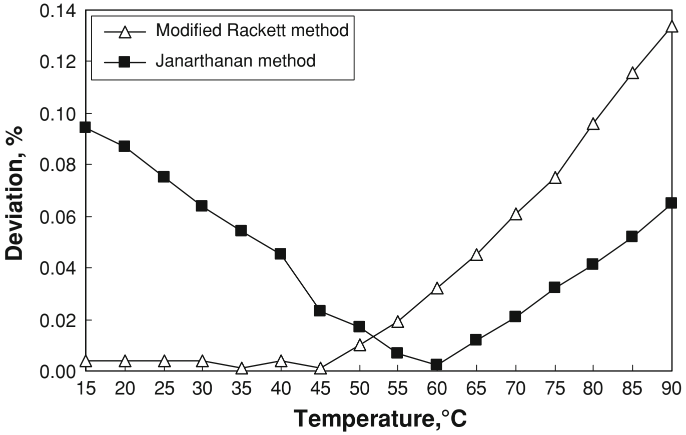

Fig. 5 Comparison of jatropha methyl esters density estimation deviations from experimental data

5 Conclusion

Jatropha methyl esters were produced using a two-step (acid/base catalyst) process. Densities of *Jatropha curcas* seed oil and jatropha methyl esters were measured as a function of temperature. The critical properties of a jatropha oil and jaropha oil-based methyl ester biodiesel were estimated using two widely utilized methods: Ambrose's method and the Joback modification of Lydersen's method.

The results of these two methods were employed to represent the properties of the pure components; and the mixing rules for the Lee-Kesler equation were employed to compute the critical properties of the mixture. The Rackett equation modified by Spencer and Danner was used to predict the density; the average absolute percent deviations, less than 0.03 % for the jatropha oil and 0.04 % for the jatropha methyl esters, showed very good agreement. An empirical estimation method was employed to predict biodiesel densities, and the results were good.

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