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Analysis of Empirical Models of Refractive Index for Biodiesel+Ultra Low Sulfur Diesel Blends

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***Abstract*—Biodiesel refers to a vegetable oil-based fuel consisting of long-chain alkyl esters. Although it can be used in its pure form, the most common use is seen as blends with petroleum fuel, being 20% biodiesel blend (B20) an international standard applied in unmodified diesel engines. Different scientific studies about blends fluid properties are disposable into open literature, but a relative gap exists on the use of refractive index as a reliable property to predict other core magnitudes. This paper analyses empirical equations of refractive index for biodiesel+petrodiesel blends and models for density estimation from this magnitude**

Keywords— Biodiesel, Petrodiesel, Density, Refractive index, Modeling

I. INTRODUCTION

The actual global warming and rising worldwide environment pollution make increasingly necessary to develop alternative green and renewable energy policies. Global biodiesel market is continuously growing as a consequence of the alternative fuels benefits if compared to traditional fossil fuels: safer to handle, sustainable character, diversified origin of raw chemicals, biodegradable nature and lower potential environment pollution [1-6]. The biodiesel concept refers to a vegetable/animal oil or fat-based diesel fuel consisting of a mixture of long-chain alkyl esters (C12-C22). It is obtained by a simple transesterification process, reacting triglycerides and alcohol to produce monoesters and glycerin as by-product. The majority of biodiesel today is produced by methanolic alkali-catalyzed transesterification, however, the oil and alcohol must be substantially anhydrous and have a low free fatty acid content, because the presence of water or free fatty acid or both promotes soap formation. Due to the nature of the process and raw materials, biodiesel is, conceptually, renewable and biodegradable. The most common biodiesel sources from oils are castor, coconut, cotton, jartropha curcas, jojoba, moringa oleifera, neem, palm, peanut, rapeseed, sunflower oils and animal tallow [6]. Actually, biodiesel is commonly accepted as fuel additive for diesel (so-called petrodiesel) in internal combustion engines. Many industrial manufacturers cover a percentage of biodiesel usage in the warranty agreement of the vehicle, in fact biodiesel could also extend the life of engine because it naturally lubricates the moving parts. Biodiesel is biodegradable meaning it will have no harmful effect on the environment in its liquid state. Blends of biodiesel and conventional hydrocarbon-based diesel are products most commonly distributed for use in the diesel fuel marketplace. Diesel fuel blends with up to 20% biodiesel (so-called B20) can be used with no, or only minor modifications in diesel equipment. Despite their potential benefits as additive into fuels, biodiesel has several problems if compared with traditional diesel fuel as higher particulate emissions and, until now, higher production costs. The later problem will be solved if waste edible oil is applied as raw material and if by-product glycerol is transformed into any valuable product [7-9]. There are different key properties which need to be known before using this kind of blends in a diesel engine, as kinematic viscosity, density, range of distillation temperatures, cloud point, pour point, and cloud filter plugging point which are strongly related to potential problems into the combustion system (incomplete combustion, solid deposits or inappropriate droplet size of fuel). In the last few years, a wide number of studies regarding biodiesel+petrodiesel fuel blends properties are presented in the literature [4, 8, 11-24] but only a few bring forward the possibility of use empirical methods for estimation of core thermodynamic properties as density of fuel blends by other mixing properties [25-28]. Among the different thermodynamic properties of solvents, volumetric and optical magnitudes have proved particularly informative in elucidating molecular interaction into liquid media, being these values of main interest for direct industrial applications. The aim of this study is to analyze the use of empirical equations for refractive calculations of these blends based on the pure values and the application of mathematical relations for density blends estimation from values of refractive index of the mixtures. To this end, open literature data of refractive index and density of diesel, biodiesel of different nature and different blends were compared with the computed values by different refractive index empirical rules [29-34]. Estimation of the density of blends was also evaluated using a modified Heller equation [35-36] and other models recently proposed [24, 37-38]. An analysis of the obtained predictions as a function of temperature by different methods with experimental values of density was made. Attending to the obtained results, it should be concluded that the tested empirical relations offer accurate results for refractive index on mixing for biodiesel+petrodiesel blends. The modified Heller equation tested showed an adequate capability of prediction of density at different temperatures, with higher accuracy that recently proposed correlation equations.

TABLE I: Standard specifications for petrodiesel and biodiesel

Property	ASTM method	Biodiesel	Petrodiesel
Density (15 ºC, gcm^{-3})	D941	0.88	0.850
Kinematic viscosity (40 ºC, mm^{2}s^{-1})	D445	1.9-6.0	1.9-4.1
Higher heating value (MJkg^{-1})	D2015	39-43.33	49.65

TABLE II: Characteristic properties of the used biodiesel and petrodiesel

Property	Biodiesel	Petrodiesel
Composition	C12-C22 FAME	C10 – C21 HC
Refractive index at 293.15 K		
Density at 293.15 K (gcm^{-3})	0.878789	0.834916
Distillation curve 90% (ºC)	351	317
Cloud point (ºC)	26	10
Cold filter plug point (ºC)	24	6
Pour point (ºC)	20	-20
Flash point (ºC)	310	130
Viscosity at 313.15 K (cSt)	4.03	2.55
Heat of combustion (BTU/lbm)	17272	19682
Ethylic esters of fatty acids (% w/w)	96.5	3.0
Cetane number	51.0	43.5
Sulfur content (mg/kg)	0.0	10.0

II. EXPERIMENTAL

A. Materials and measurement devices

In this study, biodiesel (Petrobahia SA-Usina Candeias-Petrobras, Brazil) was supplied by usual local providers. It was stored in sun light protected form and constant humidity and temperature and used immediately as received. It was synthesized by the industrial provider by transesterification of fats and oils (70% soy oil, and 30% animal tallow) with methanol as an alcohol and potassium hydroxide as catalyst. The molar ratio alcohol/oil was 6:1 and the catalyst was 1% by weight based on oil mass. The biodiesel was used as provided without additional processes, being stored in sun light protected form and constant humidity and temperature.

The petrodiesel used in this study (ultra low sulphur diesel) was supplied by a local distribution company (petrodiesel PETROBRAS S10, better than 10 ppm in sulphur). Both fuels were in accordance with ASTM specifications (Table I). The characteristic properties of the used biodiesel and petrodiesel are enclosed into Table II. Usual manipulation and experimental procedures in our laboratory are described into earlier papers [39-40]. Blends for measurements were prepared by weighing diluted stock solutions of corresponding biodiesel and petrodiesel in sealed vials. Each vial was weighted with a digital balance (accuracy better than ±10^{-4} g) (METTLER AT-200 Analytical Balance). All the solutions were kept tightly sealed to minimize absorption of atmospheric moisture. Measurements were performed immediately after preparation of solutions. The molar mass, experimental data and open literature data of density of biodiesel and petrodiesel [4, 8, 11-23] are gathered in Table II. Densities were measured with an Anton Paar DSA-5000M vibrational tube densimeter and sound analyser, with a resolution of 10^{-5} gcm^{-3} and 1 ms^{-1}. Apparatus calibration was performed periodically in accordance with vendor instructions using Millipore quality water and ambient air at each temperature. Accuracy in the measurement temperature was better than ±5 10^{-3} K by means of a temperature control device that applies Peltier´s principle to maintain isothermal conditions during the measurements. Open literature of density and experimental data for different temperatures are gathered into Table III.

TABLE III: Experimental and literature data of density (gcm^{-3}) (raw materials in brackets) for the studied biodiesel and petrodiesel at different temperatures

Compound	Exp. Dens.	Lit. Dens. (temperature) (used raw material)
BIODIESEL	0.875138	0.8836[a](293.15 K) (soybean oil) 0.885[a](293.15 K) (soysoapstock) 0.878[b](288.15 K) (fish oil) 0.87[c](288.15 K) (soybean oil) 0.8840[d](288.15 K) (sunflower oil) 0.8845[d](288.15 K) (soybean oil) 0.8840[d](288.15 K) (corn oil) 0.8828[d](288.15 K) (canola oil) 0.8845[d](288.15 K) (soybean oil) 0.8836[d](288.15 K) (cottonseed oil) 0.860[e](288.15 K) (mahua oil) 0.885435[f](293.15 K) (colza and coconut oil) 0.886575[f](293.15 K) (soybean and coconut oil) 0.88451[g](293.15 K) (cotton and babassu oil) 0.877[h](298.15 K) (shea butter) 0.8812[i](293.15 K) (vegetal oil) 0.8869[j](293.15 K) (peanut and sunflower oil) 0.884[k](293.15 K) (groundnut oil) 0.880[k](293.15 K) (soybean oil) 0.8689[l](293.15 K) (vegetal oil) 0.880[m](293.15 K) (jatropha curcas L.) 0.867[m](293.15 K) (chicken fat) 0.8774[m](293.15 K) (lard fat) 0.835[n](313.15 K) (rapeseed oil) 0.8253[o](288.75 K) (castor oil)
PETRODIESEL	0.831389	0.848[b](288.15 K) 0.8368[d](288.15 K) 0.840[e](288.15 K) 0.87019[f](293.15 K) 0.854[h](298.15 K) 0.8344[j](293.15 K) 0.859[m](293.15 K) 0.785[n](313.15 K) 0.8253[o](288.75 K)

[a] Haas, 2005
[b] Joshi et al., 2007
[c] Park et al., 2008
[d] Alptekin and Canakci, 2008
[e] Bhale et al., 2009
[f] Feitosa et al., 2010
[g] Nogueira et al., 2010
[h] Enweremadu et al., 2011
[i] Atadashi et al., 2011
[j] Ramirez-Verduzco et al., 2011
[k] Aliyu et al., 2012
[l] Mosesane et al., 2015
[m] Gorji, 2015
[n] Kumbar and Skřivánek, 2015
[o] Amin et al., 2016

B. *Data treatment*

The measured physical properties were correlated as a function of composition and temperature using Eqs. 1 and 2:

$$P = \sum_{i=0}^{N} A_i x_1^i \qquad (1)$$

$$A_i = \sum_{j=0}^{M} B_{ij} T^j \qquad (2)$$

where P is density (gcm^{-3}), x_1 is the biodiesel molar fraction, T is absolute temperature in Kelvin and B_{ij} are fitting parameters. N and M stand for the extension of the mathematical series, optimized by means of the Bevington test. The fitting parameters were obtained by the unweighted least squared method applying a fitting Marquardt algorithm. The root mean square deviations were computed using Eq. 3, where z is the value of the property, and n_{DAT} is the number of experimental data.

$$\sigma = \left(\frac{\sum_{i=1}^{n_{DAT}} \left(z_{exp} - z_{pred} \right)^2}{n_{DAT}} \right)^{1/2} \qquad (3)$$

Fitting parameters of the Eqs. 1 and 2 and the root mean square deviations (Eq. 3) are gathered in Table IV. In Figure 1, the temperature trend of density of pure biodiesel, pure petrodiesel and their blend are gathered as a function of temperature.

TABLE IV: Parameters of Eqs. 2 and 3 for 288.15-323.15 K and the corresponding root mean square deviations in accordance with Eqs. 1 and 2.

B_{00}	1.034738	B_{10}	-0.010805
B_{01}	-6.583319 10^{-4}	B_{11}	-1.595091 10^{-4}
B_{02}	-7.881754 10^{-8}	B_{12}	3.132991 10^{-7}
B_{10}	0.082084	B_{20}	-0.012101
B_{11}	-6.483546 10^{-5}	B_{21}	1.454545 10^{-4}
B_{12}	3.964704 10^{-8}	B_{22}	-2.617266 10^{-7}
σ=1.754 10^{-7}			

This surface shows an almost linear diminution of density when temperature rises or biodiesel composition diminishes, due to a strong diminution of the packing efficiency of the molecular kinetics and steric hindrance effect.

As commented, a considerable gap of disposable information was observed when open literature of volumetric properties was looked forward. As example, in Figure 2 different available open literature density correlations data and our experimental measurements of the petrodiesel+biodiesel blend are gathered. We compared the correlation proposed by Tesfa et al., 2010, Ramirez-Verduzco et al. 2011, and Moradi et al., 2013 [24, 37-38], as well as the Kay's mixing rule in accordance with Eq. 4 and 5:

$$\rho = \sum_{i=1}^{N} \phi_i \cdot \rho_i \qquad (4)$$

where

$$\phi_i = \frac{\left[\frac{x_i M_i}{\rho_i} \right]}{\sum_{i=1}^{N} \left[\frac{x_i M_i}{\rho_i} \right]} \qquad (5)$$

where ρ is density (gcm^{-3}), ϕ_i is the volume fraction and N is the number of compounds into the blend.

As it should be observed, all the analyzed correlations showed strong deviations from our new experimental values. Only the Kay's mixing rule and the correlation proposed by Moradi et al., 2013 showed analogous values. In fact, Tesfa and Ramirez-Verduzco's correlations only gathered relative low deviations at high and low biodiesel compositions, respectively (Figure 2).

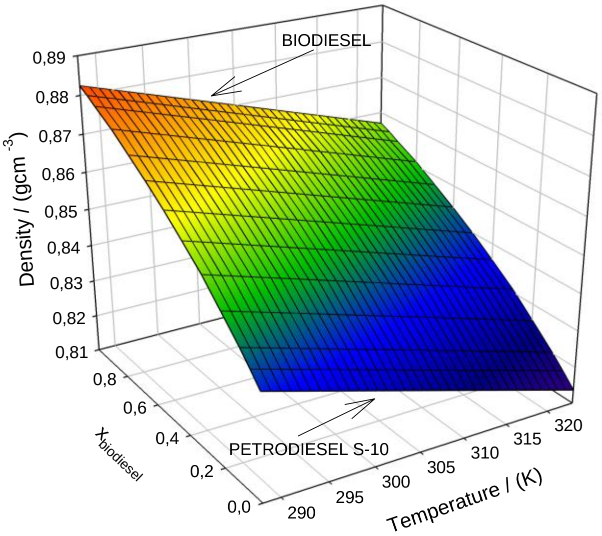

Figure 1 Temperature influence on density of biodiesel, petrodiesel and their blend

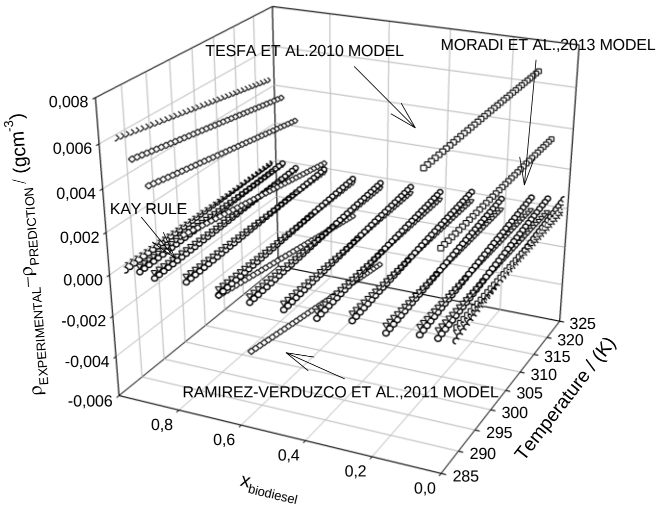

Figure 2 Comparison of disposavel density data correlations with experimental measurements of biodiesel, petrodiesel and their blend

C. *Isobaric expansibility*

A frequently applied derived quantity for industrial mixtures is the temperature dependence of volumetric properties, which is expressed as isobaric expansibility or thermal expansion coefficient (α). The data reported in the literature normally show only values of the thermal expansion coefficients both of pure compounds and its mixtures, showing the relative changes in density, calculated by means of (-Δρ/ρ) as a function of temperature, and assuming that α remains constant over the temperature range.

As in the case of pure chemicals, α can be computed at a fixed mole fraction by way of the expression:

$$\alpha = -\left(\frac{\partial \ln \rho}{\partial T}\right)_{P,x} \tag{6}$$

taking into account the strong temperature dependence of density. Attending to this relation, in Figure 3 is showed the isobaric expansibility of the mixture petrodiesel+biodiesel as a function of composition and temperature.

As observed, the isobaric expansibility diminishes for lower temperatures, as well as, for rising compositions of biodiesel. It is important to highlight as all the experimental points gather positive expansibility values at the studied ranges.

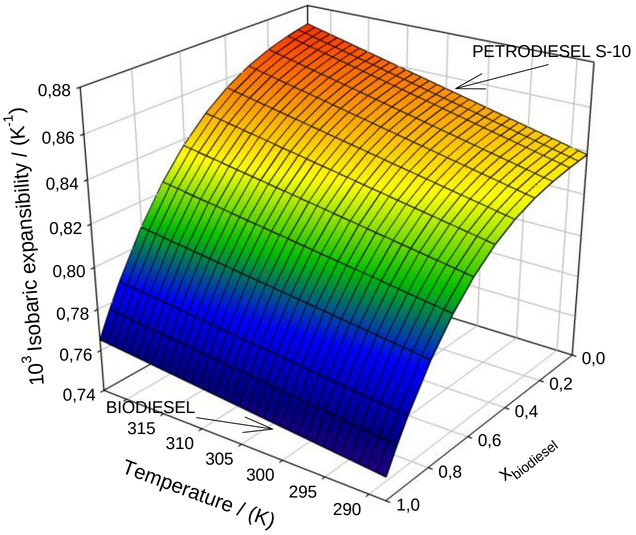

Figure 3 Isobaric expansibility as a function of composition blend at different temperatures

III. MODELING

A. *Modeling refractive index on mixing*

Refractive index is a very useful fluid characterization magnitude with widespread academic and industrial applications. It has been used for many years for accurate characterization of pure fluids and mixtures. Values are disposable into open literature for many pure liquids but really scarce data should be found for binary and multi-component mixtures. Thermophysical properties of blends containing petrodiesel and biodiesel of wide spectrum raw materials, are also of scientific and industrial interest but, as commented, only scarce collections of experimental data are disposable. Due to this fact, different empirical procedures have been proposed to estimate values of refractive index on mixing based on the values of pure compounds enclosed into the mixture [29-34]. The most commonly used mixing rules are the Lorentz-Lorenz, Dale-Gladstone, Arago-Biot, Eykman, Newton, Oster, Wiener, Heller and Edwards, as follows:

Lorentz-Lorenz

$$\frac{n_D^2 - 1}{n_D^2 + 2} = \sum_{i=1}^{N}\left[\phi_i\left(\frac{n_{Di}^2 - 1}{n_{Di}^2 + 2}\right)\right] \tag{7}$$

Dale-Gladstone

$$n_D - 1 = \sum_{i=1}^{N}\left[\phi_i\left(n_{Di} - 1\right)\right] \tag{8}$$

Arago-Biot

$$n_D = \sum_{i=1}^{N}\left(\phi_i n_{Di}\right) \tag{9}$$

Eykman

$$\frac{n_D^2-1}{n_D^2+0.4}=\sum_{i=1}^{N}\left[\phi_i\left(\frac{n_{Di}^2-1}{n_{Di}^2+0.4}\right)\right] \qquad (10)$$

Newton

$$n_D^2-1=\sum_{i=1}^{N}\left[\phi_i\left(n_{Di}^2-1\right)\right] \qquad (11)$$

Oster

$$\frac{(n_D^2-1)(2n_D^2+1)}{n_D^2}=\sum_{i=1}^{N}\left[\phi_i\left(\frac{(n_{Di}^2-1)(2n_{Di}^2+1)}{n_{Di}^2}\right)\right] \qquad (12)$$

Eyring-Jhon

$$n_D=n_{D1}\phi_1^2+n_{D2}\phi_2^2+2\left(n_{D1}^{1/2}n_{D2}^{1/2}\right)\phi_1\phi_2 \qquad (13)$$

Wiener

$$\frac{(n_D^2-n_{D,1}^2)}{(n_D^2+2n_{D,1}^2)}=x_2\frac{(n_{D,2}^2-n_{D,1}^2)}{(n_{D,2}^2+2n_{D,1}^2)} \qquad (14)$$

Heller

$$\frac{(n_D-n_{D,1})}{n_{D,1}}=\frac{3}{2}x_2\left[\frac{\left(\frac{n_{D,2}}{n_{D,1}}\right)^2-1}{\left(\frac{n_{D,2}}{n_{D,1}}\right)^2+2}\right] \qquad (15)$$

where n_D is the refractive index on mixing, n_{Di} is the refractive index of pure compounds into mixture, ϕ_i is the volumetric fraction, ρ_i is the density of pure compounds, M_i is the molecular mass of pure compounds and N the number of compounds into blend.

For example, Figure 4 shows the deviations of these equations from the experimental data of open literature containing biodiesel+petrodiesel at 298.15 K. As observed, all these equations gather low deviations in terms of prediction of refractive index on mixing of this blend.

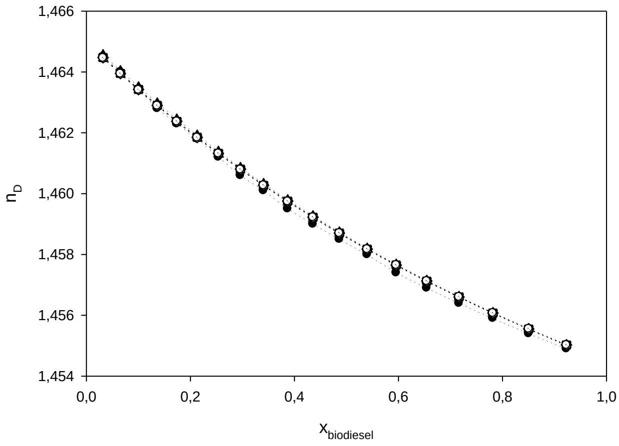

Figure 4 Comparison of open literature data [26] of refractive index for biodiesel+petrodiesel and estimation different mixing rules (Eqs. 7-15) at 298.15 K

B. *Estimation of density from refractive index on mixing*

In the last few years, prediction of density from refractive index mixing rules was suggested by some authors [30-31]. They have proposed that the mixing rules for refraction indices are functions of the volume fractions of the mixture, since it is possible to generalize them in a function of the density of the mixture and of the pure components (eq. 16):

$$\frac{f(n_D)}{\rho}=\sum_{i=1}^{N}\frac{w_i f(n_{Di})}{\rho_i} \qquad (16)$$

Then, in a more explicit polynomial:

$$\rho=\frac{f(n_D)}{\sum_{i=1}^{N}\frac{w_i f(n_{Di})}{\rho_i}} \qquad (17)$$

In this equation, ρ is the density of the mixture and ρ_i is the density of a component i in the mixture, as above indicated, w_i is the mass fraction and f is a mathematical function of the refractive indices of the mixture (n_D) and the refractive mixture of each component (n_{Di}).

In this paper, experimental new data and open literature data of density for biodiesel+petrodiesel blend [4, 8, 11-24] are compared with the estimation by eq. 17 applying Lorentz-Lorenz (eq. 18) refractive index mixing rule:

$$f(n_D)=\frac{(n_D^2-1)}{(n_D^2+2)} \qquad (18)$$

For example, Figure 5 shows the deviations of these estimations with our experimental data for this blend at 298.15 K. As observed, the estimation of the modified Heller equation with Lorenz-Lorentz mixing rule using previously estimated refractive index on mixing by the same rule, gathers accuracy lower than 0.03%, much better than estimations by other mixing rules previously published (greater than 0.75% [25-26]).

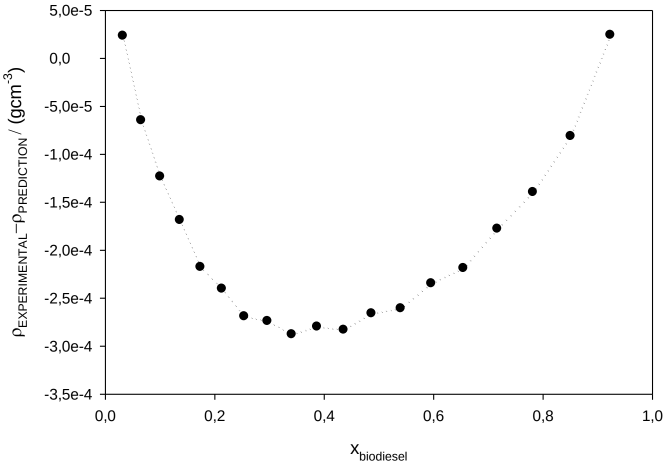

Figure 5 Comparison of our experimental data of density for biodiesel+petrodiesel blend at 298.15 K with the estimation of modified Heller equation with Lorenz-Lorentz mixing rule, using previously estimated refractive index on mixing by the same rule

IV. CONCLUSIONS

This paper contains the results of a new experimental study of density as a function of temperature of Brazilian commercial biodiesel (B100) (70% soybean oil, and 30% animal tallow)+petrodiesel blends. Different empirical equations were tested in terms of refractive index estimation of blends. Although consideration of volumetric additivity trend was made, the empirical equations gather low deviations and accurate behavior when estimations are computed at different temperatures.

In what is referred to density estimation by the modified Heller equation by means values of refractive index, accurate results were obtained too, despite the application of previously estimated refractive index by mixing rules.

The tested procedures showed accurate trend when estimations were tried at different temperatures, proving to be a useful tool for refractive index on mixing calculations, as well as, when density data are not available.

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