THERMOPHYSICAL PROPERTIES OF BIODIESEL OBTAINED FROM VEGETABLE OILS: CORN, SOY, CANOLA AND SUNFLOWER

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ABSTRACT

Biodiesel is biodegradable, renewable and obeys to the carbon cycle, being defined as mono-alkyl ester of fatty acids derived from renewable sources, such as vegetable oils and animal fat. For this work biodiesel was produced through transesterification reaction (methylic route) from sunflower, corn, soy and canola vegetable oils. From experimental data, it was possible to observe an inverse relation among temperature and viscosity/density. Among the four kinds of biodiesel (sunflower, corn, soy and canola), the one which has presented greater viscosity was canola, however all of them presented very similar behavior. Mathematic polynomial model used adjusted well to experimental data. Vegetable oils (sunflower, corn, soy and canola) are advantageous for biodiesel production. Experimental data were inside limits established by American Society for Testing and Materials, European Norm and Brazilian Agency of the Oil.


PROPRIEDADES TERMOFÍSICAS DO BIODIESEL OBTIDO A PARTIR DE ÓLEOS VEGETAIS DE MILHO, SOJA, CANOLA E GIRASSOL

RESUMO

No presente trabalho o biodiesel foi produzido através da reação de transesterificação (rota metílica) a partir de óleos vegetais de girassol, milho, soja e canola. A partir de dados experimentais, foi possível observar uma relação inversa entre a temperatura e a viscosidade / densidade. Entre os quatro tipos de biodiesel (girassol, milho, soja e canola), o que apresentou maior viscosidade foi o biodiesel obtido de óleo de canola, porém todos apresentaram comportamento muito semelhante. O Modelo polinomial utilizado se ajustou bem aos dados experimentais.

1. INTRODUCTION

Concerns about international oil price rise associated to climatic change questions has lead humanity to a more rational use of energetic sources available nowadays, together with the development of energy production by renewable sources (ITURRA, 2003). In this scenario is highlighted biofuel, which bring incentives to agriculture by elevated demand of raw material, mainly present in ethanol production in Brazil, USA and E.U. – European Union biodiesel.

Brazil produces and has a consolidate market for ethanol; however the country is in the initial phase of biodiesel production, fostered by law requirement from the beginning of 2008 to add 2% biodiesel to mineral diesel. Even when adding only 1% biodiesel to diesel oil, it is possible to reduce greatly harmful effects of its combustion. Since January 1, 2010, biodiesel sold in Brazil contains 5% biodiesel (ANP, 2012). This little addition represents an important economy in oil importation, generating employments and profit in agricultural sector. It is expected that 150 million hectares agricultural area and diversity of edafo-climatic conditions may help Brazil to become a worldwide leader in biodiesel production (BORGES, 2008; HOLANDA, 2004).

Brazilian cultures potential for biodiesel production overcomes 200 species, involving annual cultivations, such as castor beans, sunflower, canola, cotton and peanut and also the perennial ones such as palm, babassu and other palm trees. Completing raw material diversity for biodiesel there is also the use of tallow and residues from frying and domestic sewage, besides residues from crude vegetable oil refining. Brazil has potential to answer daily internal demand of 460,000 biodiesel barrels from oilseed species cultivation in 6 million ha, or else, only 18% of its deforested area (BODDEY, 1993).

Biodiesel is a generic term which refer to several fatty acid monoesters which may be used as diesel fuel. It is produced from the conversion of triglyceride (vegetable oil or animal fat) trough several sterification processes. There are at least three preliminary ways to produce biodiesel: microemulsions, thermic cracking (pyrolysis) and transesterification. Most used method is vegetable oil and animal fat transesterification (FANGRUI, 1999). Not all vegetable oils and animal fat are appropriate for being used in biodiesel production, once they may present high amount of viscosity or a great number of instaurations in fatty acid carbonic chain which may constitute a biofuel (KNOTHE, 2005). Among several oilseed which are known in literature, the ones which present a high amount of oil in the seed, are advantageous for biodiesel production. Among these we may highlight oilseeds soy, peanut, sunflower, babassu, corn, colza, castor bean and cotton (VARGAS et al, 1999).

Knowing thermophysical properties: density, thermic conductivity, as well as rheological behavior is of great importance for scaling equipments used for controlling processes which involve energy production. The lack of biodiesel thermophysical properties data makes it difficult dimensioning equipments and processes, usually assuming approximate values. Physic and thermic properties, such as density and viscosity are much used in projects for pumps, engines, ducts, biodiesel factories and several other equipment moved by biofuel.
The present work was proposed for collecting biodiesel thermophysical properties data obtained by means of soy, canola, corn and sunflower vegetable oil transesterification reaction.

2. MATERIALS AND METHODS

2.1 MATERIALS

Soy, corn, sunflower and canola vegetable oil (Bunge, Brazil) were purchased in the local market. For this experiment were used reagents of analytic degree and deionized water. Experimental data were analyzed using statistic package SAS®, (SAS, 1998).

2.2 METHODS

2.2.1 OBTAINING BIODIESEL

Biodiesel was obtained by means of transesterification reaction via methylic route, based in the methodology used by DANTAS (2006), and PEREIRA (2007). Initially potassium methoxide was produced mixing 40 mL methanol with 1 g KOH for each 100 mL vegetable oil into a beaker under shaking with the help of a magnetic shaker (Biomixer, Brazil) until the total dissolution of KOH. After that, potassium methoxide was added to respective vegetable oils.

The solution was submitted to a temperature of 45ºC under continuous agitation (180 rpm) with the help of an incubator shaker (Solab, Brazil), for making transesterification reaction for 60 minutes. After reaction, the mix was transferred to a decantation funnel, for phase separation. After 20 minutes it was possible to observe two distinct phases: one superior rich in methylic esters, less dense and clearer, and an inferior phase rich in glycerine, more dense and darker. After 24 hours resting, glycerine was collected into a beaker. Afterwards, the clearer one was submitted to washing process with the aim to neutralize basic excess, remove some undesired impurities and evaporate water residue.

2.2.2 DETERMINING DENSITY AND VISCOSITY

Kinematics viscosity and density were determined at different temperatures (283.15, 293.15, 303.15, 313.15 and 323.15) K. Biodiesel and ethanol mix were registered as follows: B100 – Pure Biodiesel; B75 – 75% Biodiesel + 25% Ethanol; B50 – 50% Biodiesel + 50% Ethanol; B25 – 25% Biodiesel + 75% Ethanol.

The density ($\rho$) was determined by using a standard volumetric pycnometer (~10 cm$^3$), with uncertainty of ± 0.01 Kg m$^{-3}$. The pycnometer was calibrated using double distilled water. Calibrated Cannon-Fenske glass capillary viscometer (sizes 50, 75, 100, and 150) was used to measure the kinematic viscosity ($\nu$) (Schott-Geräte, Germany) with an uncertainty of ± 0.01 mPa·s. Viscometers were placed into a thermostatic water bath (Schott-Geräte, CT 53 HT, Germany) for temperature control, with estimated uncertainty of 0.1 K. Variation coefficients for experimental measurements may be estimated as being not higher than 5.0 %.
3. RESULTS AND DISCUSSION

3.1 THERMOPHYSIC PROPERTIES

Tables 1 to 4 present experimental data for density and kinematic viscosity of sunflower, corn, soy and canola biodiesel samples in function of variables analyzed (temperature and biodiesel concentration).

**Table 1** – Density $\rho$, viscosity $\nu$ for sunflower biodiesel.

<table>
<thead>
<tr>
<th>T / °C</th>
<th>$w_1$</th>
<th>$w_2$</th>
<th>$\rho$ / Kg . m$^{-3}$</th>
<th>$\nu$ / mm$^2$ . s$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
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<td>6.471</td>
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<td>0.75</td>
<td>0.25</td>
<td>864.902</td>
<td>4.857</td>
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<td>0.25</td>
<td>860.647</td>
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<td>0.25</td>
<td>858.004</td>
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</tbody>
</table>

$w_1$ = Biodiesel (% v/v); $w_2$ = ethanol (% v/v).

**Table 2** - Density $\rho$, viscosity $\nu$ for corn biodiesel.

<table>
<thead>
<tr>
<th>T / °C</th>
<th>$w_1$</th>
<th>$w_2$</th>
<th>$\rho$ / Kg . m$^{-3}$</th>
<th>$\nu$ / mm$^2$ . s$^{-1}$</th>
</tr>
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<tr>
<td>T / °C</td>
<td>$w_1$</td>
<td>$w_2$</td>
<td>$\rho$ / Kg $\cdot$ m$^{-3}$</td>
<td>$\nu$ / mm$^2$ $\cdot$ s$^{-1}$</td>
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<td>-------</td>
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<td>----------------</td>
</tr>
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<td>5.651</td>
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<td>0</td>
<td>873.945</td>
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<tr>
<td>323.15</td>
<td>1</td>
<td>0</td>
<td>868.716</td>
<td>3.655</td>
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</table>

$w_1 =$ Biodiesel (% v/v); $w_2 =$ ethanol (% v/v).

**Table 3** - Density $\rho$, viscosity $\nu$ for soy biodiesel.

<table>
<thead>
<tr>
<th>T / °C</th>
<th>$w_1$</th>
<th>$w_2$</th>
<th>$\rho$ / Kg $\cdot$ m$^{-3}$</th>
<th>$\nu$ / mm$^2$ $\cdot$ s$^{-1}$</th>
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</thead>
<tbody>
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<td>0.75</td>
<td>832.249</td>
<td>2.678</td>
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<td>303.15</td>
<td>0.25</td>
<td>0.75</td>
<td>828.011</td>
<td>2.136</td>
</tr>
</tbody>
</table>

$w_1 =$ Biodiesel (% v/v); $w_2 =$ ethanol (% v/v).

**Table 4** - Density $\rho$, viscosity $\nu$ for Canola biodiesel.
Density and viscosity experimental values presented an inverse relation with temperature. The same tendency was observed by BARIOUTIAN et al (2008) when analyzing palm tree biodiesel density, obtained to methylic route and by CERIANI et al (2008) when analyzing density and viscosity of vegetable oils babassu, buriti, brazil nuts, macadamia nut and grape seed. Highest cinematic viscosity values were noticed for canola biodiesel, however all samples presented values very close to one another. Density has presented a varied behavior, not revealing more dense biodiesel. BARIOUTIAN et al (2008) has compared palm biodiesel density obtained via methylic route, and has concluded that, methylic esthers density is higher than ethylic esthers. For the author this tendency may be attributed to higher molecular weight of ethylic esthers and fatty acids. Samples densities depend on the molecular weight. Hence, according to molecular weight reduction there happens also an increase in density.

PEREIRA (2007), analyzed cinematic viscosity at 313.15 K of Mabea fistulifera Mart (canudo-de-pito) biodiesel, canudo de pito vegetable oil, diesel fuel and mix between biodiesel and diesel viscosity and has observed that biodiesel viscosity was lower than the one of vegetable oil, getting closer to values registered to diesel oil (a little superior, justifying the great advantage of using transesterification process for obtaining biodiesel derived from vegetable oil. Viscosity of diesel sample used in the works of PEREIRA (2007) was 3.327 mm².s⁻¹. The present work has obtained pure biodiesels cinematic viscosity values at 313.15K inferior to 4.679 mm².s⁻¹. Viscosity is related to fuel optimization, as well as its flowing in the engine. For using biodiesel in diesel cycle engines (FELIZARDO et al, 2006), cinematic viscosity should be between 3.5 and 5.0 mm².s⁻¹. Brazilian Agency of the Oil (ANP), has determined as limit, interval between values ranging from 3.0 – 6.0 mm².s⁻¹ to 313.15 K. It is observed that great part of experimental data was inside this limit, except for some samples in

\[
\begin{array}{cccc}
313.15 & 0.25 & 0.75 & 823.277 & 1.747 \\
323.15 & 0.25 & 0.75 & 819.552 & 1.490 \\
283.15 & 0.5 & 0.5 & 847.431 & 4.768 \\
293.15 & 0.5 & 0.5 & 845.047 & 3.608 \\
303.15 & 0.5 & 0.5 & 841.294 & 2.945 \\
313.15 & 0.5 & 0.5 & 836.685 & 2.358 \\
323.15 & 0.5 & 0.5 & 833.835 & 1.968 \\
283.15 & 0.75 & 0.25 & 866.717 & 6.564 \\
293.15 & 0.75 & 0.25 & 866.209 & 4.983 \\
303.15 & 0.75 & 0.25 & 863.269 & 3.927 \\
313.15 & 0.75 & 0.25 & 859.241 & 3.188 \\
323.15 & 0.75 & 0.25 & 855.067 & 2.634 \\
283.15 & 1 & 0 & 886.431 & 10.317 \\
293.15 & 1 & 0 & 882.418 & 7.699 \\
303.15 & 1 & 0 & 878.945 & 5.835 \\
313.15 & 1 & 0 & 874.511 & 4.679 \\
323.15 & 1 & 0 & 871.732 & 3.890 \\
\end{array}
\]

\[w_1 = \text{Biodiesel (\% v/v)}; \ w_2 = \text{ethanol (\% v/v)}.\]
certain temperatures. It is worth to observe that extension of reaction, as well as experimental circumstances used in biodiesel production has extremely influentiated fuel properties especially apparent viscosity.

CALAI e CLARK (2007), produced biodiesel from canola oil, obtaining cinemetic viscosity at 313.15K between 4 and 6 mm².s⁻¹ value closer to 4.679 mm².s⁻¹ obtained with this work. Table 5 presents cinemetic viscosity values at 313.15K biodiesel produced from different oilseed and also limits established by American Society for Testing and Materials (ASTM), European Norm (EN) and ANP.

Table 5 – Cinematic viscosity at 313.15K biodiesel from different raw materials.

<table>
<thead>
<tr>
<th>Raw materials</th>
<th>v (mm².s⁻¹)</th>
<th>Limits (mm².s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>USA</td>
<td>EU</td>
</tr>
<tr>
<td></td>
<td>ASTM</td>
<td>EN</td>
</tr>
<tr>
<td>Sunflower</td>
<td>4.571</td>
<td>1.9-6.0</td>
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<tr>
<td>Corn</td>
<td>4.329</td>
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<tr>
<td>Soy</td>
<td>4.440</td>
<td></td>
</tr>
<tr>
<td>Canola</td>
<td>4.679</td>
<td></td>
</tr>
</tbody>
</table>

DANTAS et al (2006) has studied biodiesel cinemetic viscosity obtained from corn vegetable oil as well as diesel at 313.15, and obtained value 5.110 mm².s⁻¹ and also noticed a great similarity between biodiesel corn viscosity and diesel fuel. In this work at 313.15 K it was obtained value of 4.329 mm².s⁻¹, which is a little inferior value that reveals biodiesel higher quality once viscosity reduction may reflect positively during injection and biofuel burning inside the engine combustion chamber (PEREIRA, 2007).

It was possible to identify through data analysis (Table 1 to 4), a direct relation between viscosity our density and biodiesel concentration. The same behavior was observed by PEREIRA (2007), when studying canudo de pito biodiesel density and diesel fuel at 293.15 K obtained 892.8 and 864.7 Kg.m⁻³, respectively. In this work, sunflower, corn, soy and canola biodiesels at 293.15 K were respectively: 883.410, 880.947, 882.993, 882.418 Kg.m⁻³, comparatively quite similar. Limits specified by ANP for specific biodiesel mass at 313.15 K are among 850-950 Kg.m⁻³. Only mix with 75 and 50% ethanol were lower than ANP specification; however this fact is explained by low ethanol density. Table 6 presents density values at 313.15K biodiesel produced from different oilseeds and also limits established by EN and ANP.

Table 6 – Density at 313.15 K biodiesel from different raw materials.

<table>
<thead>
<tr>
<th>Raw materials</th>
<th>ρ (Kg.m⁻³)</th>
<th>Limits (Kg.m⁻³)</th>
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<tbody>
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<td>EU</td>
<td>BRA</td>
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<tr>
<td></td>
<td>EN</td>
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<tr>
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<td>860-900</td>
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<td>Corn</td>
<td>873.728</td>
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<tr>
<td>Soy</td>
<td>873.945</td>
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</tr>
<tr>
<td>Canola</td>
<td>874.511</td>
<td></td>
</tr>
</tbody>
</table>
3.2 MATHEMATICAL MODELLING

A polynomial model was used for relating viscosity data or density in function of temperature, biodiesel concentration and ethanol, respectively. General quadratic model, equation 1 was analyzed and non significant parameters were eliminated with base on $t$ test (Student) and $p>0.05$. Table 7 shows predictive models based in equation model 1 for each oil used.

$$\Phi = y_0 + a \cdot x_1 + b \cdot x_2 + c \cdot x_3 + d \cdot x_1^2 + e \cdot x_2^2 + f \cdot x_3^2 + g \cdot x_1 \cdot x_2 + h \cdot x_1 \cdot x_3 + i \cdot x_2 \cdot x_3$$

(1)

where $\Phi$ is viscosity (mm$^2$·s$^{-1}$) or density (kg·m$^3$), $x_i$ corresponding to temperature (K), $x_2$ at biodiesel concentration (%v/v), $x_3$ ethanol concentration (%v/v) e $a$, $b$, $c$, $d$, $e$, $f$, $g$, $h$ and $i$ correspond to parameters calculated by equation.

Table 7 – Model parameter (equation 1) employed for correlating a) sunflower, b) Corn, c) Soy, d) Canola biodiesel density and viscosity.

<table>
<thead>
<tr>
<th>Parâmetros</th>
<th>$\rho_a$</th>
<th>$\rho_b$</th>
<th>$\rho_c$</th>
<th>$\rho_d$</th>
<th>$V_a$</th>
<th>$V_b$</th>
<th>$V_c$</th>
<th>$V_d$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$y_0$</td>
<td>838.215</td>
<td>837.282</td>
<td>769.939</td>
<td>863.030</td>
<td>159.176</td>
<td>147.542</td>
<td>140.936</td>
<td>159.994</td>
</tr>
<tr>
<td>$a$</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>-1.035</td>
<td>-0.955</td>
<td>-0.904</td>
<td>-1.038</td>
</tr>
<tr>
<td>$b$</td>
<td>102.833</td>
<td>101.458</td>
<td>103.533</td>
<td>70.454</td>
<td>41.291</td>
<td>39.405</td>
<td>34.151</td>
<td>41.894</td>
</tr>
<tr>
<td>$c$</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
</tr>
<tr>
<td>$d$</td>
<td>-0.0006</td>
<td>-0.0006</td>
<td>*</td>
<td>-0.0005</td>
<td>0.0017</td>
<td>0.0015</td>
<td>0.0014</td>
<td>0.0017</td>
</tr>
<tr>
<td>$e$</td>
<td>*</td>
<td>*</td>
<td>23.856</td>
<td>*</td>
<td>4.864</td>
<td>4.051</td>
<td>5.496</td>
<td>5.591</td>
</tr>
<tr>
<td>$f$</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
</tr>
<tr>
<td>$g$</td>
<td>*</td>
<td>*</td>
<td>-0.2182</td>
<td>*</td>
<td>-0.138</td>
<td>-0.13</td>
<td>-0.119</td>
<td>5.591</td>
</tr>
<tr>
<td>$h$</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
</tr>
<tr>
<td>$i$</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
<td>*</td>
</tr>
<tr>
<td>$R^2$</td>
<td>0.99</td>
<td>0.99</td>
<td>0.99</td>
<td>0.99</td>
<td>0.99</td>
<td>0.99</td>
<td>0.99</td>
<td>0.98</td>
</tr>
</tbody>
</table>

* parâmetros não significativos ($p>0.05$)

In table 7 it is possible to observe that for viscosity, most significative variables ($p>0.05$) were temperature and biodiesel concentration in the mix. In case of density the variable which most significantly influenced ($p>0.05$) was biodiesel concentration in the mixture. In all cases studied correlation coefficient ($R^2$) was equal or superior to 0.98, demonstrating that model used adjusted well to behavior presented by viscosity and density experimental data.

4. CONCLUSION

Biodiesels density and viscosity biodiesel has presented an inverse relation with temperature. More viscous biodiesel was produced from canola vegetable oil; however all of them presented very similar behavior. Average biodiesel viscosity was closer to diesel fuel, demonstrating once more the relevance of
transesterification process. Samples with 25% biodiesel presented smaller viscosity and density, showing that there is a direct relation between biodiesel concentration and viscosity/density. Polynomial mathematic model used adjusted well to experimental data.

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REFERENCES


