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The Characteristics of Fatty Raw Materials and their Influence on the Properties of Ethyl Biodiesel

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ABSTRACT

Various sources of fat ingredients may be used in the production of biodiesel thus allowing the agricultural potential to be vastly explored. However, in order to do so, the specific characteristics of the biodiesel produced from various raw materials may be known. In this research work, the biodiesel produced by the ethyl transesterification reaction of ten fatty raw materials was characterized according to its acidity index, kinetic viscosity, specific weight and iodine value. The experimental data were used to develop the correlation between each biodiesel property and the characteristics of the fat sources. Biodiesel properties are strongly influenced by the specific fatty esters composition in the raw material. A good correlation was observed between the values estimated by the equations obtained from the multiple linear regression and the experimental values, with exception of the iodine value.
Keywords: Biodiesel, Statistical Analysis, Physical Chemistry Properties

I. INTRODUCTION

The search for alternative solutions to crude oil and the growing concern with environmental pollution have been pushing up the production of biofuels all over the world. As a pioneer in the use of biofuels, Brazil has reached a position desired by many countries that search for renewable sources of energy as strategic alternatives to fossil fuels. In Brazil, the two main biofuels are the ethanol from sugar cane and the biodiesel produced from vegetable oils or animal fats [1].

Different processes are currently available and have been adopted for the production of biodiesel fuel, but the most commonly used method for converting oils to biodiesel is through the transesterification of vegetable oils or animal fats [2-3]. Vegetable oils, with various compositions of fatty acids, are the usual fatty raw materials used for biodiesel production, since they are renewable and can be produced on a large scale. Due to this flexibility, there is a much lower risk of fuel shortage and this diversity also avoids a production concentration similar to the one that occurs for fossil fuels. However, no matter the source of raw material used, the biodiesel produced should meet the specifications defined by each country´s Regulatory Oil Agency for the product commercialization. In Brazil, ANP (Agência Nacional do Petróleo, Gás Natural e Biocombustíveis) is the Regulatory Agency for Petroleum, Natural Gas and Biofuels. Therefore it becomes important to know the specific characteristics of the biodiesel produced from the different fatty raw materials.

The fatty acids used to produce the biodiesel differ from each other by three main characteristics: length of hydrocarbon chain, degree of unsaturation and presence of functional groups [4]. There are studies in the literature about biodiesel production which use various raw materials such as animal fats [5-6], microalgae [7-8] or vegetable oils. Among the vegetable oils studied the most common are soybean [9-11], rapeseed [12-14], palm [15-16] and sunflower oils [17-18]. However, there are few research works that analyze the biodiesel properties and correlate them to some parameter related to the source of fatty acid. Moreover, those studies have been limited to methyl biodiesel. According to Knothe [4], some methyl biodiesel properties, such as lubricity, viscosity, heat of combustion and oxidative stability, become higher as the ester chain gets longer and decrease as the degree of unsaturation in the chain increases. Ramos et al [19] studied the influence of the

composition of ten raw materials on the methylic biodiesel properties. The authors verified an inverse relationship between the cetane number and the concentration of linoleic and linolenic esters, and a direct correlation between the iodine value and the degree of unsaturation of the starting oil. The negative effect of the degree of unsaturation over the oxidative stability and the fluid properties has also been reported in the literature [19-22].

The purpose of this work was to produce ethyl biodiesel using ten different oilseed sources and to verify the influence of their specific characteristics over the biofuel properties.

II. METHODS AND MATERIAL

1. Reaction

The biodiesel was produced by the homogeneous ethyl transesterification reaction of ten fat sources: refined soybean, rice, canola, sunflower, corn and olive oils, Cyclus® (a commercial blend of canola, corn and sunflower oils); unrefined castor bean and murumuru oils; and frying residue. The fat source/alcohol molar ratio was 1/6 and the catalytic agent used was potassium ethanoate at a 1% ratio to the mass of fat. After 1h, at 343 K, the product was purified by decantation and vacuum evaporation.

2. Characterization of biodiesel and raw materials

The following tests were done in triplicate, both for the biodiesel samples and for the specific fat source.

The C16-C18 esters composition was obtained by gas chromatography, using a Varian CP3800 equipment and a CPWAX52CB column. The main esters content derived from the fatty acids were measured: C16:0 (palmitic acid), C18:0 (stearic acid), C18:1 (oleic acid), C18:2 (linoleic acid) and C18:3 (linolenic acid).

The acidity index was determined by titration using a KOH alcoholic solution and phenolphthalein as the indicator. The ANP Resolution no. 45/2014 establishes that the acidity index should have a maximum value of 0.50 mg KOH/g of sample [23].

The kinematic viscosity was determined using a calibrated glass capillary viscometer immersed in a thermostatic bath at a stable temperature of 313 K. The ANP Resolution no. 45/2014 establishes that the viscosity at 313 K should be between 3.0 and 6.0 $mm^2 s^{-1}$ [23].

The specific weights of the biodiesel samples and of their respective raw materials were determined at 293 K using a calibrated pycnometer. The ANP Resolution no. 45/2014 establishes that the specific weight at 293 K should be within the range of 850-900 kg m^{-3} [23].

The iodine value was obtained through the Hulb method: a certain amount of sample is mixed with CCl_4, Hulb reactant (I_2 + $HgCl_2$) and KI. After stirring and letting the mixture rest for 16 hours, it was titrated with a $Na_2S_2O_3$ solution. The ANP Resolution no. 45/2014 suggests that the iodine value should be reported, but does not define a limit value.

3. Statistical Analysis

Initially, an exploratory analysis of the data obtained from the determination of the oil and biodiesel properties was conducted using principal components analysis (PCA). The PCA is a method for multivariate data analysis, by reducing the dimensionality of the original data in low dimensionality subspaces defined by few principal components [24], through the general equation (1):

$$X = \sum_{a=1}^{A} t_a p_a^T + E = TP^T + E \quad (1)$$

where P (K x A) is the matrix that contains vectors pa of loadings or principal components; T is the score matrix which contains the orthogonal projection location for the original observations at a latent subspace, ta are the vectors of the scores matrix T, which represent the new principal components with variances given by their respective auto values (λa) and the matrix E (m x K) contains the residues, i.e., the part that is not explained by the model [25].

The experimental results obtained were normalized so that they had zero average and standard deviations equal to one. The normalization was done by subtracting the value determined experimentally from the average of the results and dividing this value by the standard deviation. The purpose of working with normalized values is to reduce the influence of a set of very high values (e.g.:

specific weight) over another set of very low values (e.g.: acidity index). The correlations to estimate the biodiesel properties were obtained by using multiple linear regression analysis (MLR). For both cases, data were obtained using the Statistica 7.0 and Matlab 7.0 programs.

III. RESULTS AND DISCUSSION

A. Esters Composition

The raw material composition data obtained by chromatography are shown in Table I. The biodiesel samples showed different chemical compositions, including the high content of monounsaturated fats in olive (81.6%) and canola oils (65.7%), the high content of the ester derived from linoleic acid in soybean (57.4%) and sunflower oils (58.1%), and the low content of C16-C18 fatty esters in the murumuru sample (23.6%).

TABLE I
C16-C18 Fatty Esters Composition Obtained from Gas Chromatography

Sample	C16:0 (%)	C18:0 (%)	C18:1 (%)	C18:2 (%)	C18:3 (%)
Rice	18.2	1.8	42.9	35.5	0.88
Olive	9.7	2.4	81.6	3.9	0.42
Canola	4.9	1.7	65.7	18.2	7.8
Cyclus®	7.8	2.4	48.8	34.4	4.4
Frying residue	11.9	3.3	29.9	48.2	6.7
Sunflower	5.2	2.7	33.0	58.1	0.06
Castor	3.8	3.1	12.7	28.9	3.5
Corn	11.1	1.8	35.6	50.3	0.49
Murumuru	8.9	3.3	9.1	2.1	0.11
Soybean	10.1	2.4	22.2	57.4	7.3

The results regarding the average values from the properties determined for the biodiesel samples and the respective fat sources are shown in Tables II and III. Each one of these properties will be discussed separately.

TABLE III
Average Values Determined for Biodiesel Samples

Sample	Acidity Index[a]	Kinematic Viscosity[b]	Specific Weight[c]	Iodine value[d]
Rice	0.198	4.30	878.6	91
Olive	0.375	4.03	867.5	60
Canola	0.149	4.15	873.8	98
Cyclus®	0.209	4.43	877.9	108
Frying residue	0.412	4.55	885.8	90
Sunflower	0.155	3.71	873.8	108
Castor	0.432	10.90	910.9	53
Corn	0.182	3.65	875.2	100
Murumuru	2.709	2.84	863.4	7
Soybean	0.294	4.31	884.4	113

[a](mg KOH g^{-1}); [b](mm^2 s^{-1}); [c](kg m^{-3}); [d](g/100g)

TABLE IIIII
Average Values Determined for Fat Raw Materials

Sample	Acidity Index[a]	Kinematic Viscosity[b]	Specific Weight[c]	Iodine value[d]
Rice	1.102	35.76	917.0	73
Olive	4.310	37.78	910.6	63
Canola	0.288	32.32	916.8	99
Cyclus®	1.884	33.07	915.6	81
Frying residue	1.809	33.06	923.7	119
Sunflower	0.289	32.27	916.7	90
Castor	0.561	256.04	955.4	71
Corn	0.284	33.21	917.1	106
Murumuru	5.110	29.27	904.0	13
Soybean	0.546	30.89	921.2	122

[a](mg KOH g^{-1}); [b](mm^2 s^{-1}); [c](kg m^{-3}); [d](g/100g)

B. Acidity Index

According to Tables II and III, it is possible to observe that the acidity index values for biodiesel are lower than those for the respective raw material. It is worth noting that the acidity indexes for the biodiesel samples are in compliance with the ANP specifications, except for the murumuru biodiesel. This exception may be explained by the high acidity of the raw material and by the fact that the raw material was not refined.

C. Kinematic Viscosity

As shown in Tables II and III, the viscosities of the fat sources are quite higher than that for the biodiesel obtained, being considered as an indication of their conversion into ethyl esters.

In general, the viscosity values for the biodiesel samples are in accordance to the ANP specifications. One of the exceptions is the murumuru biodiesel whose viscosity value, though very close to the minimum value established, is still below the specification. The murumuru oil viscosity is the lowest of all the raw materials tested. On the other hand, the viscosity of the castor oil biodiesel is well above the value defined by ANP and does not comply with the specifications. This is caused by the high viscosity value for the fat ingredient.

D. Specific Weight

In general, the increase in the specific weight of the biodiesel follows the increase trend for the raw material specific weight.

For all samples studied, the specific weight values are in accordance with those from the Brazilian Regulatory Agency for Petroleum, Natural Gas and Biofuels, except for the castor oil biodiesel. The specific weight for the castor oil biodiesel is above the value defined by ANP and, as for the viscosity, the explanation may be related to the presence of ricinoleic acid ($C_{18}H_{34}O_3$) in this oil. Similar results were obtained by Albuquerque et al [26].

E. Iodine Value

The biodiesel's iodine values are lower than those for their respective raw materials (Tables II and III). However, the increase in iodine values for the biodiesel samples is not directly correlated to the increasing values for the respective oils.

F. Exploratory Data Analysis

The data matrix was built with 30 samples and 15 variables, previously described. The data were preprocessed using the autoscale. Therefore, the training set for the PCA model was built with 30 biodiesel samples, and the validation was carried out using the cross validation procedure. This internal validation is based on the removal of one sample at a time from the training set, which is, then, tested in the model created, making it a more robust model since all samples are tested.

The following parameters were defined to evaluate the quality of the model: the root mean square error of calibration (RMSEC) and the root mean square error of validation (RMSECV). The last one is used to measure the model´s ability to predict new samples. The values obtained were 0.49 and 2.41, respectively.

Two principal components (PC) accounted for 74.39% of the data variance, with 43.16% for PC1 and 31.23% for PC2. By plotting PC1 versus PC2 scores, it was possible to observe that the castor oil biodiesel samples are completely different from all the others, thus producing a biodiesel with different characteristics (Fig. 1). This is supported by the fact that some of the castor oil biodiesel properties (the specific weight and the viscosity) are very different from those for the other samples. This result can be explained by the high content of hydroxyl groups and unsaturations due to the presence of ricinoleic acid in the unrefined castor oil.

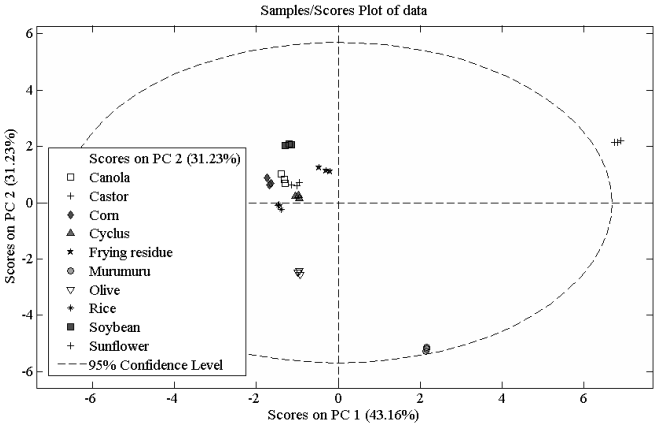

Figure 1: Scores PC2 versus PC1

It was also observed that the murumuru oil differs from the other oils and confirms the analytical data showing high acidity value and low iodine value for this biodiesel when compared to the other oils. The unrefined murumuru oil [27] also shows a distinct chemical composition, with low total C18 and low unsaturated values, since it is a rich source of fatty esters with a number of carbon atoms lower than 16, as shown in Table IV.

PCA confirmed the analytical results which indicated that the murumuru oil biodiesel samples (raw material with high acidity level) and the castor oil biodiesel (raw material with high viscosity) did not comply with some of the ANP specifications for the product.

TABLE IVV
Fatty Esters Composition of Murumuru Oil [26]

Fatty acid	**Content (%)**
Caproic (C6:0)	1.2
Capric (C10:0)	1.3
Lauric (C12:0)	47.8
Myristic (C14:0)	29.0
Palmitic (C16:0)	8.9
Estearic (C18:0)	3.1
Linolenic (C18:2)	6.3

G. Prediction of Biodiesel Properties

The data matrix (X) was built with 30 samples and 9 variables from the raw materials, as previously described. The matrix (Y) was built with the biodiesel property

measured for the 30 samples. For the two matrices (X and Y), the data were preprocessed using autoscale. The following biodiesel properties were predicted: acidity index (AI), kinematic viscosity (VS), specific weight (SW) and iodine value (IV). In all cases, multiple linear regression (MLR) was applied to predict the biodiesel properties. In the model´s equation only the regression coefficients with statistical significance are shown (α = 0.05), after the hypothesis test was performed.

The following figures of merit were evaluated to measure the quality of the model: RMSEC and RMSECV, both defined previously, and also the coefficients of determination of calibration (R^2_{Cal}) and validation (R^2_{Val}). These data are shown in Table V.

As can be seen in Table V, the figures of merit showed excellent results for all biodiesel properties under study, except for iodine value. Therefore, the proposed methodology for evaluation of acidity index, kinematic viscosity and specific weight for biodiesel samples can be used.

TABLE V
Figures of Merit for MLR Models of Biodiesel Properties

	Acidity Index	**Kinematic Viscosity**	**Specific Weight**	**Iodine Value**
RMSEC	0.057[a]	0.06[b]	0.7[c]	4.8[d]
RMSECV	0.091[a]	0.08[b]	1.1[c]	7.3[d]
R^2_{Cal}	0.9983	0.9993	0.9967	0.9776
R^2_{CV}	0.9955	0.9986	0.9925	0.9478

[a](mg KOH g^{-1}); [b](mm^2 s^{-1}); [c](kg m^{-3}); [d](g/100g)

Biodiesel Acidity Index

The use of MLR provided the following equation to predict the acidity index for biodiesel, where the regression coefficients and the standard deviations are indicated between parentheses.

ACIDITY INDEX (biodiesel) = (0.008 ± 0.002)*VS + (0.006 ± 0.002)*IV - (0.036 ± 0.007)*C16: 0 - (0.038 ± 0.005)*C18:1 - (0.039 ± 0.003)*C18:2 - (0.064 ± 0.010)*C18:3 (2)

It can be inferred from equation (2) that the acidity index for biodiesel increases as the oil viscosity and the iodine value increase. On the other hand, an increase in C16 and C18 contents in the oil causes a reduction in the biodiesel acidity index.

Biodiesel Viscosity

The use of MLR provided the equation (3) to predict the kinematic viscosity for biodiesel.

VISCOSITY (biodiesel) = (0.038 ± 0.002)*VS - (0.011 ± 0.002)*IV + (0.061 ± 0.007)*C16:0 + (0.235 ± 0.076)* C18:0 + (0.023 ± 0.001)*C18:1 + (0.030 ± 0.003)*C18:2 + (0.161 ± 0.010)*C18:3 (3)

According to the aforementioned equation, the biodiesel viscosity increases with an increase in the raw material viscosity and a reduction of the iodine value for the oil. This last result is in accordance with the data obtained by Garcia [28], who also verified the reverse correlation between the iodine value for the oil source and the biodiesel viscosity. Besides that, the fatty acid that contributes the most for the increase in viscosity is the stearic (C18:0), followed by the linolenic acid (C18:3). Garcia [28] also observed a greater contribution from the stearic acid for the viscosity value for methylic esters, but it was followed by oleic (C18:1) and palmitic acids (C16:0).

Biodiesel Specific Weight

The use of MLR provided the equation (4) to predict the specific weight for biodiesel.

SPECIFIC WEIGHT (biodiesel) = (720.6 ± 117.8) + (0.158 ± 0.024)*VS - (0.054 ± 0.019)*IV + (0.663 ± 0.083)* C16:0 + (0.068 ± 0.018)*C18:1 + (0.281 ± 0.037)*C18:2 + (1.404 ± 0.130)*C18:3 (4)

The specific weight for biodiesel tends to increase with higher oil viscosity values. On the other hand, this same property increased with a reduced iodine value for the raw material, although to a less extent. Linolenic acid (C18:3) is the acid that contributes the most to the specific weight of biodiesel, followed by palmitic acid.

IV. CONCLUSION

Under the experimental conditions used, it was possible to obtain biodiesel through the homogeneous ethanolic

transesterification of various fat sources, which, in most of the cases, met the product's specifications for some properties. Statistical analysis confirmed the analytical results, which showed that biodiesel samples from castor oil (high viscosity and specific weight) and from murumuru oil (high acidity index and low viscosity) did not meet the specification defined for the product. The specific characteristics of the castor oil biodiesel may be explained by the high hydroxyl group content and unsaturated groups present in the raw material, and the murumuru's, by the high content of fatty acids with less than 16 carbon atoms. Multiple linear regression analysis allowed the prediction of the biodiesel properties from the characteristics of the fat source that were quite appropriate for use, as verified through the analysis of the merit parameters for the proposed models. The results obtained confirm that the characteristics of the fat raw material strongly influence the biodiesel physicochemical properties.

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