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Application of Multivariate Exploratory Techniques to Predict Kinematic Viscosity of Biodiesel from Vegetable and Algae Oils

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As biodiesel is basically composed by fatty acid esters that can be methyl or ethyl or propyl or butyl (FAME or FAEE. FAPE or FABE, respectively). Its kinematic viscosity (KV) is usually higher than the fossil diesel. Current diesel vehicle engines are not adapted to use pure biodiesel with higher KV since it may cause deposits in engines and diminish efficiency during fuel combustion due to its poor atomization. Therefore, monitoring and predicting biodiesel KV is crucial to meet specifications defined by regulatory agencies for final diesel-biodiesel blends. One cheap and fast manner to obtain properties of fuels is applying predictive methods considering largely available data and related parameters such as composition, degree of unsaturation, density, and/or temperature. Therefore, the present study applied multivariate exploratory techniques such as Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA) to investigate the influence of esters composition from vegetable oils and algae oils on KV. As a result, some saturated esters and one type of unsaturated ester were related to KV. Therefore, they were used as variables for a new method to predict KV at 40 °C. It presented satisfactory accuracy with deviation AAD = 0.25 mm²/s and %AAD = 5.28 considering all biodiesel types. However, a different performance of KV prediction was observed between vegetable and algae biodiesel.
Keywords: PCA; HCA; Ester composition profile; viscosity prediction methods

1. Introduction

Recently, environmental concerns have been increasing worldwide due to greenhouse effect and its related climate change. Therefore, countries have been compromised to reduce petroleum dependence and replace fossil fuels with biofuels (Böhringer 2003; Horowitz 2016; UNFCCC 2021). In this sense, government programs, such as the RENOVABIO in Brazil, were launched to provide decarbonization credit for the use of biofuels. One of the most applicable biofuel is biodiesel to replace partially the fossil diesel (Salina, Almeida, and Bittencourt 2020). In addition to the similarity of performance of biodiesel compared to diesel, it is nontoxic and emits less pollutant (Mizik and Gyarmati 2021; Varanda, Pinto, and Martins 2011). Besides that, biodiesel can be obtained from several feedstock types, such as soybean, palm oil, jatropha oil, and algae oil. Despite being similar to diesel, varying the feedstock to produce biodiesel might implicate in properties variations of the final product. Therefore, some crucial properties such as Kinematic Viscosity (KV) must be monitored and controlled through experimental analysis or by predictions methods. The higher KV of biodiesel also may affect fuel atomization, causing incomplete combustion, deposit formation and consequently low engine efficiency.
In the literature, it was found three models that can be applied to predict KV at 40°C of biodiesel according to its composition, such as Sue et al.'s method based on weighted-average number of carbon atoms and weighted-average number of double bonds of the biodiesel (Su *et al.*, 2011); Verduzco et al.'s method based on number of double bonds (*db*) and molecular weight of saturated esters (Ramírez-Verduzco, Rodríguez-Rodríguez, and Jaramillo-Jacob 2012); and Calixto's method for algae oil based on the sum of the product of molar or mass fraction and *db* of each ester (Calixto et al. 2017). Another predictive methods were found but suitable for specific biodiesel, such as biodiesel from fish, coconut, jatropha and waste oil (Joshi and Pegg 2007) and others for diesel-biodiesel blends (Ramírez-Verduzco et al. 2011; Santos, Maciel, and Fregolente 2021).

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1.1 Exploratory Analyses

To calculate KV, specific properties must be set as variable of the method, such as double bonds, number of carbons and ester composition. To better choose the most suitable variable, exploratory analyses have been applied in some studies to associate them to a specific property (Baird and Oja 2016; Vrtiška and Šimáček 2016; Islam et al. 2015; Jahirul et al. 2021; Wu et al. 2016; Rashid et al. 2021).
Exploratory analyses are described as multivariate techniques based on chemical properties obtained at laboratory such as compositions and statistical calculations. Some usual techniques are Principal component analysis (PCA) and Hierarchical Component Analysis (HCA).
Through PCA analysis, which is obtained from linear transformation for an orthogonal projection of factors, a pattern is obtained considering data variance (Li Vigni, Durante, and Cocchi 2013; I. Jolliffe 2005; I. T. Jolliffe and Cadima 2016). The number of factors can be set following Kaiser's approach, considering eigenvalues higher than the unit (Kaiser 1960). The HCA analysis is based on clusters formed from branches associated to low distance between variables (Cecil C. Bridges 2016). This statistical technique can be performed through a specific linkage method, such as simple, complete, and Ward, and distance calculation method, such as Euclidian and Manhattan (Li Vigni, Durante, and Cocchi 2013).Therefore, this study assembled several biodiesel regarding vegetable and algae as feedstock to apply exploratory analysis to identify suitable esters, developed a new method for KV prediction and compared performances of existing methods.

2. Methodology

In this study, biodiesel composition data were applied for assessment of predictive methods of KV at 40 °C. To analyze KV of biodiesel, 123 data with different range of ester composition (Table 1) and several biodiesel types were obtained from literature, which are consisted by 17 data for algae biodiesel (Yaşar 2020a; de Jesus et al. 2020; Song et al. 2013) composed by Fatty Acid Methyl Esters (FAME) and 106 data for vegetable biodiesel data (Bukkarapu and Krishnasamy 2018; Shrivastava et al. 2020; A. Sarin et al. 2010; Kim et al. 2012; Yaşar 2020b; A. Sarin et al. 2009; Yehliu, Boehman, and Armas 2010; Huang et al. 2020; Kapilan and Reddy 2008; Dunn 2011; Ramos et al. 2009; R. Sarin et al. 2007; Jain and Sharma 2011; Zuleta, Rios, and Benjumea 2012; Ramírez-Verduzco, Rodríguez-Rodríguez, and Jaramillo-Jacob 2012; Allen et al. 1999; Agarwal, Singh, and Chaurasia 2010; Lang et al. 2001; Chotwichien, Luengnaruemitchai, and Jai-In 2009), which include 90 data of FAME biodiesel, 8 data of Fatty Acid Ethyl Esters (FAEE), 5 data of Fatty Acid Propyl Esters (FAPE) and 3 data of Fatty Acid Butyl Esters (FABE).

Table 1: Range of content of specific esters and KV at 40°C of vegetable and algae biodiesel

C14:0 (%)	C16:0 (%)	C18:0 (%)	C18:1 (n9) (%)	C18:2 (n9,12) (%)	C18:3 (n9,12,15) (%)	C20:0 (%)	C20:1 (n9) (%)	KV@40 °C (mm²/s)
From 0.01 to 1.87	From 2 to 53.33	From 0.85 to 25.80	From 8.09 to 79.30	From 7.6 to 79.00	From 0.03 to 62.90	From 0.06 to 6.90	From 0.1 to 9.30	From 3.9 to 6.05

2.1 Exploratory analyses

The PCA and HCA analyses were carried out at Statistica software (Statsoft version 7 for Windows). For PCA analysis and Kaiser's index was applied so that only factors with eigenvalues higher than the unit was considered. HCA analysis was performed considering Euclidian distance and Ward's linkage method. The selection of dendrogram orientation was for variables.

2.2 KV prediction

After exploratory analyses, new method based on selected esters was developed through multiple regressions to predict KV (ν) at 40°C. The new method was compared with the existing ones found in the literature. The deviations of calculated KV from experimental values were used to assess the accuracy of the prediction methods. The most suitable method was the one with minor average of absolute deviation (AAD) (Eq. 1) and its percentage (%AAD) (Eq. 2).

$$AAD = \frac{1}{N}\sum_{i=1}^{N}\left|\nu_{\,calculated,i} - \nu_{\,experimental,i}\right| \quad (1)$$

$$\%AAD = \frac{100 * 1}{N}\sum_{i=1}^{N}\left|\frac{\nu_{\,calculated,i} - \nu_{\,experimental,i}}{\nu_{\,experimental,i}}\right| \quad (2)$$

3. Results and Discussion

3.1 Exploratory analyses

Applying PCA analysis with the data composed by vegetable and algae biodiesel, it was possible to visualize that KV might be related to mass fraction of specific esters such as C14:0, C16:0 and C18:1 (n9) (Figure 1-A, B, C). Those results were based on 4 principal components with 63.85% of accumulated explained variance, considering Kaiser's Index with eigenvalue higher than the unit. With HCA analysis a different ester was associated to KV due to minor distance, which was C18:0 (Figure 1-D). Therefore, no polyunsaturated esters were associated to KV but only saturated and monounsaturated esters.

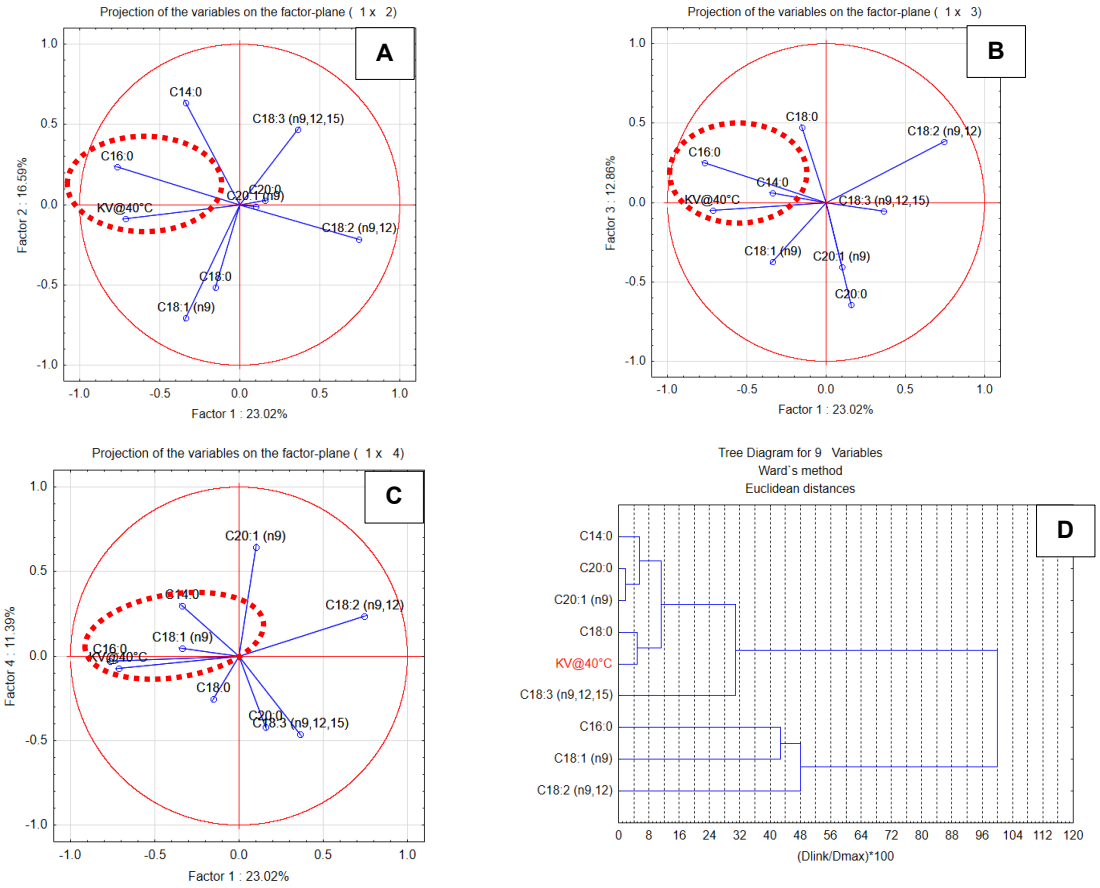

*Figure 1: Exploratory Analyses with associated esters (dashed red circle for PCA): depicturing 39.61%, 35.88% and 34.41%, respectively for PCA 1 and 2 (**A**), PCA 1-3 (**B**) and PCA 1 and 4 (**C**). Dendogram with vegetable and algae biodiesel data. Distance between KV and C18:0 = 5.05 (**D**)*

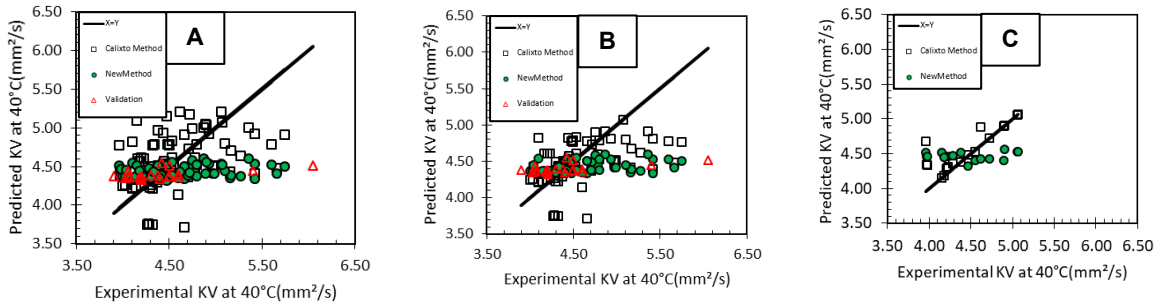

*Figure 2: KV prediction of vegetable and algae biodiesel (**A**); KV prediction of only vegetable biodiesel (**B**); KV prediction of only algae biodiesel (**C**), considering experimental values (—) and results from the prediction of Calixto's method (□), the new method (●) and validation of the new method (Δ).*

3.2 KV prediction

Considering results from PCA and HCA saturated esters, such as C14:0, C16:0 and C18:0, were more associated with KV than unsaturated ones. Only one monounsaturated ester, C18:1 (n9), was close to KV (Figure 1). Therefore, the mass fraction (w_i) of these esters were used as the independent variables to be adjusted for the new method to predict KV at 40ºC by multiple regression (Eq.3).

$$\nu = w_{C14:0} * 1.00 * 10^{-2} + w_{C16:0} * 5.35 * 10^{-1} + w_{C18:0} * 1.29 * 10^{-2} - w_{C18:1} * 2.43 * 10^{-4} + 4.32 \quad (3)$$

Considering all the collected data, the performance of the KV prediction was satisfactory with low deviation, AAD = 0.25 mm²/s and %AAD = 5.28. For vegetable biodiesel, the prediction of the new method was similar with AAD = 0.23 mm²/s and %AAD = 4.82. For algae biodiesel, the deviation was higher with AAD = 0.33 mm²/s and %AAD = 7.53. The validation of the new method was also carried out, presenting similar performance with slight increment of deviation, with AAD =0.29 mm²/s and %AAD = 6.18%. For that, 22 data (20% of database) was reserved and applied for validating the new method.

In addition, the three existing methods presented in the introduction section were tested. As a result, for all biodiesel types, Calixto's method presented higher accuracy with lower deviation, with AAD = 0.27 mm²/s and %AAD = 5.62. The Sue et al.'s method presented AAD = 0.57 mm²/s and %AAD = 12.07 and the Verduzco's method resulted in AAD = 0.56mm²/s and %AAD = 12.12. Considering separately vegetable and algae biodiesel, a different prediction was reported comparing the three existing models.

For vegetable biodiesel, Calixto's was the most precise with AAD =0.25 mm²/s and %AAD = 5.38, which was a similar performance to the new method. For the other two methods, similar performance was identified with Sue et al.'s method with AAD = 0.51 mm²/s and %AAD = 10.74 and Verdusco's method with AAD = 0.50 mm²/s and %AAD = 10.69. For algae biodiesel, Calixto's method presented the most satisfactory performance, with AAD = 0.15 mm²/s and %AAD = 3.75. The other two methods, no satisfactory prediction was obtained, presenting higher deviation, such as Sue et al.'s method with AAD = 0.94 mm²/s and %AAD = 19.91 and Verdusco's method with AAD = 1.04 and %AAD = 22.29.

Through the KV prediction and comparison among methods, it was possible to observe that Calixto's method and the new method were the most suitable approaches to predict KV for several biodiesel types, with similar performance (Figure 2), except for algae biodiesel. It might be explained by the fact that the new method is more related to saturated esters, differently of Calixto' one that require unsaturation factors (db), which is highly present in algae biodiesel. In addition, higher deviation was obtained for FABE and FAPE biodiesel and for biodiesel with higher KV, such as higher than 5.3 mm²/s from both methods.

4. Conclusion

With this study, it was possible to observe that exploratory analyses were techniques that could help to identify components in biodiesel that impact KV, which it is usually monitored and controlled. In addition, a new method was developed to predict KV at 40 °C of biodiesel with satisfactory performance for several biodiesel type including FAME, FAPE, FAEE and FABE, considering the mass fraction of some esters, such as C14:0, C16:0, C18:0 and C18:1 (n9). Analysing individually, the performance for vegetable and algae biodiesel were different. The most satisfactory performance was observed by applying the new method for vegetable biodiesel. For algae biodiesel, higher accuracy was obtained by using Calixto's method, indicating high influence of double bonds on KV of algae biodiesel.

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