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Instituto Politécnico de Bragança & Ordem dos Engenheiros

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Validation of a prototype of a miniaturized near infrared spectrometer on complex organic samples

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Fourier Transform Infrared (FTIR) spectroscopy focused on the near infrared (NIR) region has become crucial for quality control on diverse areas, from energy to biomedical applications, by enabling *in-situ* and in real time analysis of samples with complex organic compositions [1,2]. The development of portable and miniaturized NIR spectrometers (miniNIR) can further extend NIR spectroscopy applications [3,4], thus this work compares *in-situ* analysis based on a FT-NIR benchtop spectrometer with a miniNIR prototype to detect and quantify contaminants in biodiesel, such as vegetable oils, methanol, and glycerol. Good models based on principal component analysis-linear discriminant analysis of FT-NIR spectra were obtained, predicting contaminants with accuracies between 75 to 95%, while the miniNIR prototype's delivered models with accuracies between 66 to 86%, showing the device's potential for preliminary quality control of biodiesel, with the added advantages of low cost and portability.

Introduction

For a long time now, quality control of products across multiple industries has been performed with well-established techniques such as liquid and gas chromatography, which while efficient and adequate, are time-consuming, generate a large amount of toxic waste, and are usually operated off-line [5]. This has created a need for quick analytical procedures that operate which allow for in-situ monitoring of samples and processes, such as infrared spectroscopy.

Infrared (IR) spectroscopy is quickly becoming a popular technique for quality analysis, as the wavelength accuracy and spectral quality of the spectrometers can provide detailed information on complex organic samples [6]. For example, in the biodiesel industry, infrared spectroscopy focused on the near infrared (NIR) range has been used to monitor biodiesel synthesis and contaminant detection with great results.

The application of this technique has been further expanded thanks to the utilization of miniaturized and portable NIR spectrometers, pocket-size devices with lower utilization costs than regular spectrometers and that are used to perform at-line analysis of samples [7]. This work aimed to evaluate the performance of a prototype of a portable miniaturized NIR (miniNIR) spectrometer and compare it to the performance of a benchtop FT-NIR spectrometer with a transflectance probe, based on spectral regions focusing on part of the vibration combination and the first overtone regions, in the simultaneous *in-situ* analysis of vegetable oil, used cooking oil, methanol, and glycerol in biodiesel.

Materials and Methods

Biodiesel blends with one contaminant each (rapeseed oil, two different types of used cooking oil, methanol, and glycerol) were prepared and analyzed with a benchtop FT-NIR spectrometer, using a NIR transflection fiber optic probe and operating in the 9000 – 4000 cm^{-1} region of the spectrum, and with a prototype of a miniNIR spectrometer (pictured in Figure 1), with a matrix of six independent LED covering the 7700 – 4700 cm^{-1} region of the spectrum.

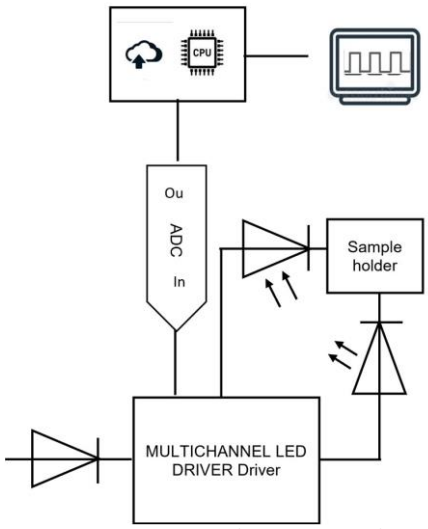

Figure 1. Schematic representation of the prototype of the miniaturized and portable NIR spectrometer used.

Different spectra preprocessing methods were used on the obtained data before conducting a principal component-linear discriminant analysis (PCA-LDA) to predict the presence of contaminants in biodiesel, and building the partial least square (PLS) regression models to quantify the contaminants in the samples.

Results and Discussion

The PCA-LDA models with the best accuracies obtained from FT-NIR spectral data and miniNIR data to predict the presence of contaminants in biodiesel are shown in Table 1.

Table 1. Accuracies of the best PCA-LDA analyses based on FT-NIR and miniNIR data

Target contaminants	Accuracy based on FT-NIR analysis/%	Accuracy based on miniNIR analysis/%
All	75	78
Rapeseed and used cooking oils	95	72
Rapeseed oil	84	86
Used cooking oil	79	74
Methanol	91	68
Glycerol	78	79

Although not as high as the models based on FT-NIR data, the accuracies of the PCA-LDA models based on miniNIR data are considered reasonable, showing the prototype's potential to detect different contaminants.

As for the PLS models built to quantify the contaminants in the biodiesel, the models based on the miniNIR prototype data showed poor prediction abilities, with low R-Squared values (between 0.50 and 0.85) and high values of root mean square error of prediction (RMSEP), while the models based on FT-NIR data showed very good predictive ability. The discrepancy in results can most likely be attributed to the LED set-up of the prototype, which is different from the miniaturized NIR devices already commercially available, and could potentially be corrected by using LEDs with a higher resolution.

Conclusions

Portable and miniaturized NIR spectrometers are a more economical alternative to benchtop spectrometer which allow for a quick *in-situ* analysis of complex samples, which could increase quality control. Compared to the benchtop FTIR spectrometer, the miniaturized and portable prototype showed good predictive ability of target contaminants, but didn't allow for their quantification. In the future, other LED set-ups could be evaluated to see how the performance of the prototype is affected. As it is now, thanks to its low-cost and small dimensions, the prototype could be used in primary assessments of organic samples, which could then be analyzed by more sophisticated techniques.

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