Full Length Article

Application assessment of UV–vis and NIR spectroscopy for the quantification of fuel dilution problems on used engine oils

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A B S T R A C T

Fuel dilution in engine oil is a frequent problem in internal combustion engines (ICE); its main consequence is the reduction of the oil viscosity, decreasing lubrication film strength, and causing a negative impact on friction and wear. The standard and more precise methods for assessing fuel content in oil are based on chromatographic analysis (e.g., ASTM D3524, ASTM D7593), requiring high-cost equipment and highly qualified personnel. This work performed a study to propose an alternative method for quantifying diesel fuel dilution in used engine oil by UV–vis and NIR spectroscopy. The samples for the study were prepared from used oil from six different vehicles with various mileages. According to the results obtained in this study, NIR spectroscopy proved to be the most suitable method for the quantification of diesel fuel in used engine oils. Furthermore, the use of NIR spectroscopy combined with multivariate calibration methods could predict the fuel concentration of the samples used for validating the model. The best predictive model for the quantification was obtained by Partial Least Squares Regression, which achieved a Root Mean Squared Error of prediction of 0.436% and a coefficient of determination of 0.9435. In comparison, the parameters for Principal Component Regression were 1.049% and 0.8441, respectively.

1. Introduction

Fuel dilution in engine oil is a common problem for both diesel and gasoline internal combustion engines (ICE): when the fuel contaminates the engine oil, it modifies its properties, mainly viscosity, reducing lubrication film strength and causing a negative impact on friction and wear. The implementation of active regeneration of particulate filters by delayed fuel post-injection and the move towards hybrid electric vehicles and vehicles equipped with stop-start systems will lead to increased fuel dilution [1–3]. This will be of more concern in diesel engines since significant fuel dilution could persist at the usual sump oil temperatures of 100–150 °C. In contrast, in gasoline engines, the more volatile gasoline fuel will have substantially evaporated at these temperatures [3].

Consequently, the quantification of fuel in oil (FiO) is an essential parameter in the ICE's condition monitoring, as it allows performance prediction and to take proper actions at the right time to avoid damage to the engine. The most common standard methods for assessing fuel content in oil, such as ASTM D3524 and ASTM D7593 [4,5], provide precise results but require high-cost equipment, including gas chromatographs with flame ionization detectors and temperature

programmable ovens, appropriate solvents, carrier gas for the procedure, highly qualified personnel, and considerable time for calibration. Another standard method proposed by ASTM requires a surface acoustic wave sensing fuel dilution apparatus; the process is detailed on the ASTM D8004; this method is simple but requires specific equipment only for that application and frequent recalibrations [6]. Other options, such as viscosity analysis and flash point testing, suggest a possible dilution problem but do not offer a direct quantification of fuel in oil since dilution is not the only factor that modifies these properties [3].

Some investigations have been done to propose alternative methods for quantification. For example, the work of Sejkorov presented a methodology for the construction of the FTIR-PLS regression model for the determination of contamination of mineral engine oil by pure diesel [7]; the study was done only in samples of fresh oil and presented satisfactory results. A different method was proposed by Neupane for rapid in situ measurement of the fuel dilution of oil in a diesel engine by Laser-Induced Fluorescence (LIF) spectroscopy to monitor the oil [8]. Another inline monitoring strategy was developed by Niedermayer, a resonant sensor system that based its dilution determination on the measurement of the viscosity of the oil tested [9].

In recent years, Near-Infrared (NIR) and UV–Visible spectroscopy have proven to provide spectral information that can support the development of quantitative analytical methods, with significant applications in the field of Oil Condition Monitoring (OCM), for example, the procedure to quantify soot in engine oil by ultraviolet–visible spectroscopy [10]. UV–Visible spectroscopy with univariate methods has been used to quantify biodiesel in diesel–biodiesel blends. In alliance with Partial Least Squares regression (PLS) have been able to quantify biodiesel in samples adulterated with vegetable oil [11]. The NIR technique combined with chemometrics has been used to discriminate crude oils and derivates and quantify them in blends [12]. Another study evaluated the use of visible and near-infrared (NIR) to determine the biodiesel content in biodiesel/diesel blends using Multiple Linear Regression (MLR) and variable selection by Successive Projections Algorithm (SPA), concluding that both regions have the potential for developing a rapid non-destructive method to quantify the biodiesel [13]. In recent years Liu et al. developed a compact moisture-content monitoring system based on the visible-near-infrared (Vis-NIR) spectroscopy technology capable of determining the moisture content in lubricating oil of high-speed rail gearbox in a fast, simple, and accurate way [14].

This work presents an approach to quantifying fuel dilution in used engine oil by UV–vis and NIR spectroscopy, considering them as techniques with simple operation and reduced cost, able to produce accurate and reliable results rapidly once the calibration model for the quantification of the particular analyte is done. The techniques will be used separately, and part of the study is to determine which one is more suitable for the quantification of diesel fuel in used engine oil. The work will be focused on diesel fuel dilution attending to the increasing problem of fuel dilution in these engines, its market importance, and being just the typical engines where condition monitoring is applied.

2. Methodology

The first step of this work was to record the fuel and the oil spectra in both spectrometers, UV–vis and NIR, and do a spectrum screening to compare them with the purpose of identifying regions for potential quantification where the absorbance of the fuel was higher than the absorbance of the oil.

The next phase consisted of analyzing prepared fresh oil samples with added diesel fuel in known concentrations. This phase aimed to evaluate the capabilities of both methods while avoiding interference in the spectra from contaminants, such as soot and other typical impurities that can be found on used engine oil, streamlining the analysis. Subsequently, each set of results (UV–vis and NIR spectra) was analyzed with univariate methods to determine a correlation between the signal and the fuel concentration. Comparing both methods would allow determining which spectrometric technique offers better quantification results to extend the study for used oil samples dopped with diesel fuel.

The final phase of the work was to use the spectroscopic method that provided the best results in the previous step to analyze the samples prepared from used oil with univariate methods and chemometric techniques, such as Principal Components Regression (PCR) and Partial Least Squares (PLS), which led to a calibration model that related the concentration of the diesel fuel to the spectral data collected from the sample.

2.1. Samples

Three types of samples were prepared. Group A consisted of 9 samples prepared using fresh engine oil with added diesel fuel in a concentration range from 0.0 to 10.0 % (w/w), see Table A1. The fresh engine oil used was a commercial SAE 5 W-30 fulfilling quality specs API SM/CF and ACEA A5/B5, *C*2.

Group B was prepared using the same fresh oil with added diesel fuel, but for this group, the fuel was dyed with a commercial UV fluorescent fuel dye at a concentration of 600 ppm by mass. The UV dye, Tracerline TP-3400, fluoresces a yellow/green color. This group of 6 samples was also prepared in a concentration range of 0.0–10.0 % (w/w); see Table A2; they were used only in the study performed on the UV–vis spectrometer.

Group C was prepared with used engine oil contaminated by diesel fuel. The oil was sampled from diesel engines working on vehicles from an urban bus fleet, and samples from six vehicles using the same commercial fresh oil SAE 5 W-30 were used. The soot concentration in the original samples varied from 0.067 to 0.282 %. In this set, 47 samples were prepared with different concentrations of diesel fuel between the range of 0.0–10.0 % (w/w), see Table A3. This group was later divided into two sets, one of 38 samples for creating the calibration model and the other 9 representative samples for evaluating the model, gathering low, intermediate, and maximum values in the evaluated fuel concentration range, which means that approximately 80 % of the samples were used for constructing the model and 20 % for evaluation. The samples used for validation are identified in Table A3 as Test_1-9.

Dilution of the samples of group C was necessary. The samples were too dark because of soot content, which caused noise in all the spectral range. Two non-polar solvents were tested, heptane and toluene, to decide the most suitable one for the subsequent studies.

Comparing the results obtained with each solvent, samples diluted with toluene showed noise in the area surrounding 4600 cm^{-1} which was relevant to the study - as will be demonstrated in Section 3.1.2.; consequently, this solvent was discarded, and heptane was the chosen solvent since it was able to dissolve the samples more proficiently for this application. Heptane has the additional benefit of being less toxic than toluene.

Part of the study was to select the appropriate dilution ratio attending to the soot content on the used oil. Fig. A1 shows three tested dilution ratios obtained for a sample from group C that contained 0.067 % of soot and 0.000 % of diesel fuel; Table A4 indicates the used ratios. The selected ratio for this case was 3.4 g of heptane per gram of sample since it produced a clean enough spectrum to work on the analysis stage. The dilution ratios used for the rest of the samples according to the soot content of the used oil were chosen following a similar testing procedure, aiming to obtain a dilution that has similar behavior for all the samples with 0.000 % of diesel fuel. The selected dilution ratios for each concentration of soot used in the study are shown in Table A5.

2.2. Instruments

The UV–vis spectral data were acquired in a Perkin Elmer UV–vis Lambda 365 spectrophotometer equipped with a deuterium and tungsten halogen lamp with a double beam. The spectra were recorded over the wavelength range of 700 and 200 nm, or 14285.7 – 50000 cm^{-1} in terms of wavenumber, with a spectral resolution of 1 nm.

The NIR spectra were acquired in a Perkin Elmer FT-NIR Spectrum Two N equipped with a lithium tantalate (LiTaO$_3$) NIR detector. The spectra were recorded over the wavenumber range of 11500–4400 cm^{-1}, or 869.6–2272.7 nm in terms of wavelength, with 24 accumulative scans with a resolution of 4 cm^{-1}.

The measures in both spectrophotometers were made using a quartz cuvette with an optical path length of 10 mm. All the samples in the study were measured in triplicate to mitigate any errors that could affect the next modeling step, specifically the experimental errors inherent to the procedure. To record the spectra of Group C, the samples were diluted with heptane using the dilution ratios specified in Table A5; the reference cell was filled with the solvent.
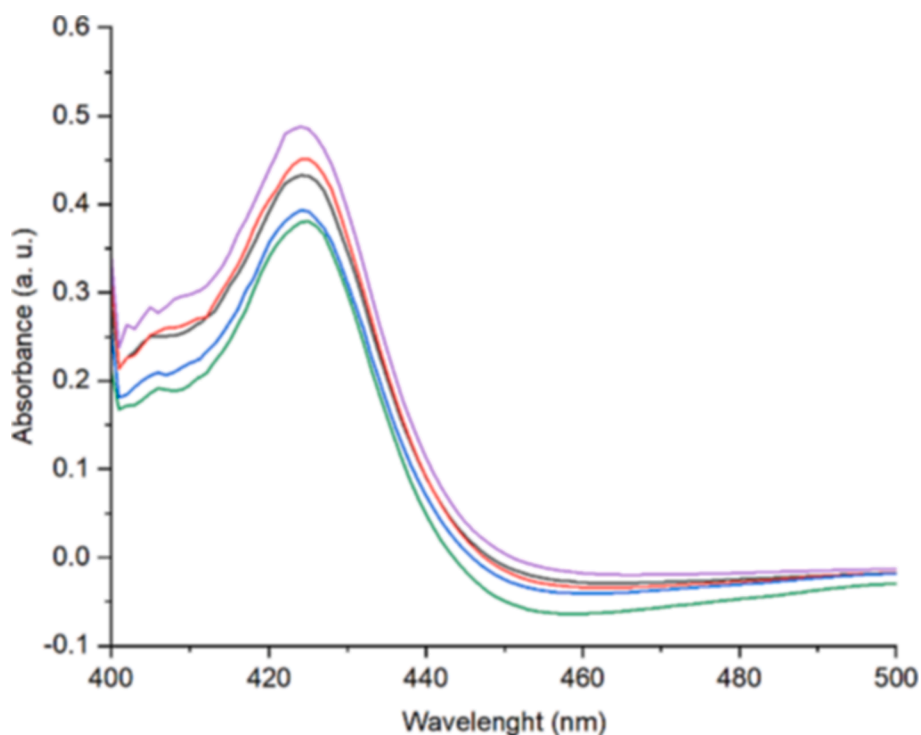
Fig. 1. Group B samples UV–vis spectra expansion between 400 and 500 nm.
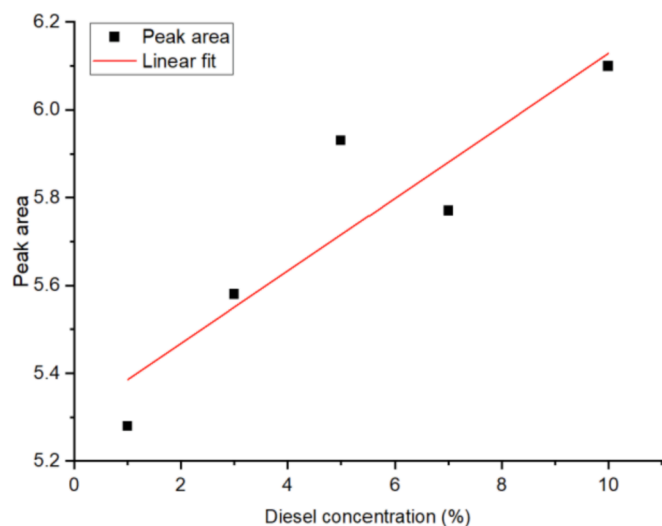

Fig. 2. Linear fitting for the fresh oil samples doped with dye diesel fuel and analyzed by UV–vis.

Fig. 3. Fresh engine oil and diesel fuel NIR spectra between 6000 and 4500 cm^{-1}.

3. Results and discussion

3.1. Fuel, oil, and fresh oil samples analysis

3.1.1. Ultraviolet–visible analysis

After comparing the oil and the diesel fuel spectra, it was not possible to identify a prominent difference between the fuel and the oil spectra. As expected, the analysis of the UV–vis spectra of the group A samples could not provide results for the quantification of fuel in oil.

The next proposal was to study the use of a fuel dye to enable quantification in the UV–vis region, following a similar approach to the one done by Neupane et al. [8] when using laser-induced fluorescence spectroscopy. For this proposal, group B samples were used. It was noticed that the addition of the dye influences the absorption spectra, creating areas of higher absorption.
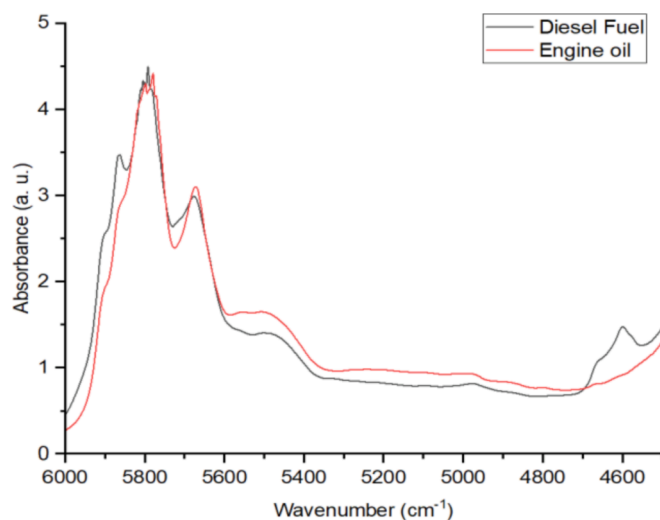
For the spectra of the group B samples, the use of the dye in the fuel allowed the identification of an area that changed as a function of the concentration of fuel in each sample, which could be used for quantification by applying a univariate method.

For each prepared sample, the absorbance spectrum of the fresh oil was subtracted from the absorbance spectrum of the sample; the area under peak centered at 424.4 nm was calculated on the equipment software *Perkin Elmer UV Win Lab DPV* considering the baselines at 405.0 nm and 445.0 nm. Fig. 1 displays an expansion to highlight the area used for quantification. Later, the spectral peak area calculated was correlated with the fuel concentration; the linear fit is displayed in Fig. 2. The determination coefficient obtained for this group of samples was $R^2 = 0.8240$.
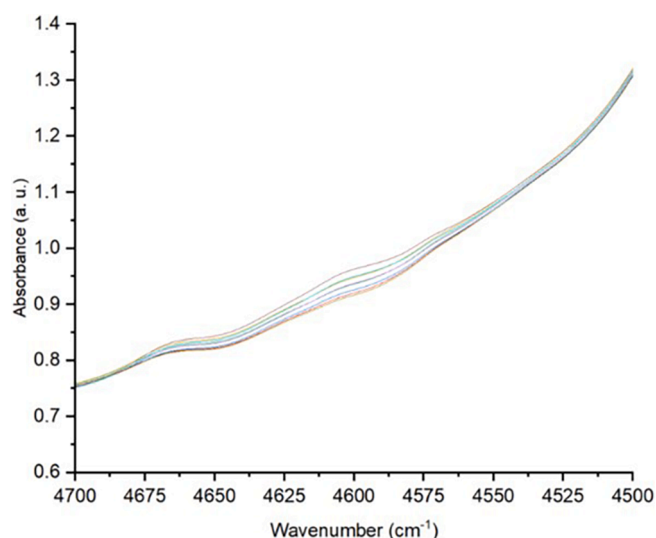
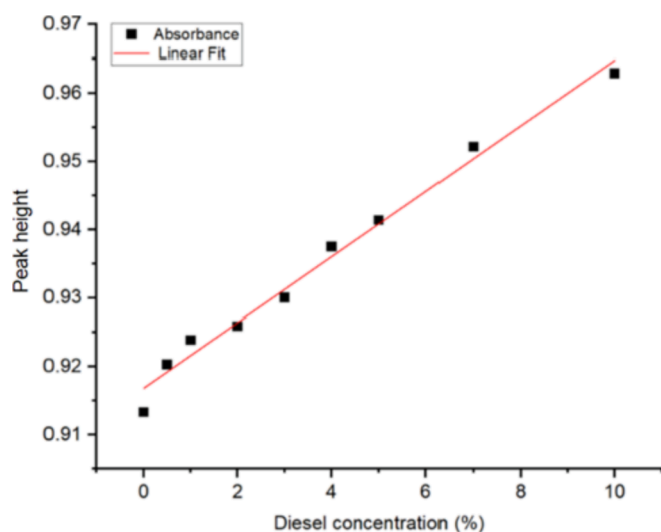**Fig. 4.** Group A samples NIR spectra between 4700 and 4500 cm^{-1}.

Fig. 5. Linear fitting for the fresh oil samples dopped with diesel fuel and analyzed by NIR.

3.1.2. Near-infrared analysis

Comparing the fresh oil and the diesel fuel NIR spectra, it was possible to identify a specific peak at the wavenumber 4600 cm^{-1}, where the absorbance of the fuel was higher than the absorbance of the oil, as shown in Fig. 3. This peak could possibly be used to correlate the spectrum collected from a sample to the concentration of the diesel fuel in the sample.

After the collection of the spectra of the group A samples, it was confirmed that there was a differentiation around the located wavenumber on each sample, which is shown in Fig. 4. Therefore, the height at 4600 cm^{-1}, considering 0 as the base, was correlated with the concentration of diesel fuel. The correlation proved the existence of a linear relationship between the peak height at 4600 cm^{-1} and the concentration of diesel fuel in the sample; the fitting equation displayed a determination coefficient of R^2 = 0.9855 in the studied range, as shown in Fig. 5.

3.1.3. Comparison of both spectrometric techniques

Comparing the results of both methods, the NIR spectroscopy led to the obtention of significantly better outcomes for quantification using

the fresh oil samples doped with diesel fuel. This method showed a stronger correlation between the signal and the fuel concentration using a more straightforward procedure. In contrast, the correlation obtained by the UV–vis method was not considered strong enough to continue the study with used engine oil samples. Consequently, NIR spectroscopy was the chosen spectroscopic alternative to continue the analysis with the samples prepared from used oil from diesel engines.

3.2. Used oil samples analysis: Near-infrared analysis

3.2.1. Univariate analysis: Peak height and peak area analysis.

The first approach to obtain a model that related the fuel concentration in the sample to the NIR spectral data collected was to use univariate analysis in a particular group of samples from Group C; all prepared from used oil from the same engine.

For this initial part of the study, nine samples from group C were analyzed (samples C_8 – C_17, see Table A3). The soot content in the original sample was 0.067 %. To generate a calibration model for this data, two methods of univariate quantitative analysis were used, peak height and peak area analysis.

For the peak height analysis, the height at 4600 cm^{-1}, considering 0 as the base, was correlated with the concentration of diesel. The fitting displayed a determination coefficient of R^2 = 0.9646 in the studied range. The results are presented in Fig. A2.

For the spectral peak area analysis, the areas were measured considering the peak height at 4600 cm^{-1} and one baseline from 4635 to 4558 cm^{-1}. For the construction of the calibration model, the area corresponding to each sample was the difference after subtracting the area of the used oil without diesel added from the area of the prepared sample. The determination coefficient was R^2 = 0.9615, which indicates a strong correlation between the located peak area and the concentration of diesel fuel in the samples. Additionally, the similarity between the coefficients obtained by both univariate methods and the result for the fresh oil samples reinforces the hypothesis that it is possible to estimate the dilution for used oil samples from the measurements of molecular absorbance in the NIR region using the proposed dilution method. The results are presented in Fig. A3.

The same procedure was applied to another group of seven samples (samples C_41 – C_47, see Table A3), prepared from used oil from another engine (Vehicle_6), having a higher content of soot (0.115 %). The results obtained were equivalent to the previous ones; for this second group, the fitting equations presented a determination coefficient R^2 of 0.9715 and 0.9749, respectively, for peak height and peak area analysis.

These separate analyses on a reduced part of the samples were performed to test whether it was possible to correlate the obtained NIR spectra with the fuel content in the sample even after the dilution as a feasibility analysis. The use of samples prepared from the same source (vehicle) enhances the calibration; it is expected that including samples with different content of soot in the same calibration would lessen the coefficient of determination, considering the limitations of univariate methods.

3.2.2. Multivariate analysis

Although some studies have successfully used the spectral peak area analysis in the near-infrared region, as the work of Brülls [15], the most common practice for this type of spectroscopy is the use of multivariate regression methods like Principal Component Regression (PCR) and Partial Least Squares Regression (PLSR) [16], especially when dealing with complex matrixes like used engine oil. Multivariate methods allow the construction of a calibration model for more than one analyte. They can construct the model for the analyte in the presence of multiple spectral interferents.

These methods require a larger number of samples to create a robust model that allows the prediction of future unknown samples. The objective is to include in the model definition as many samples as
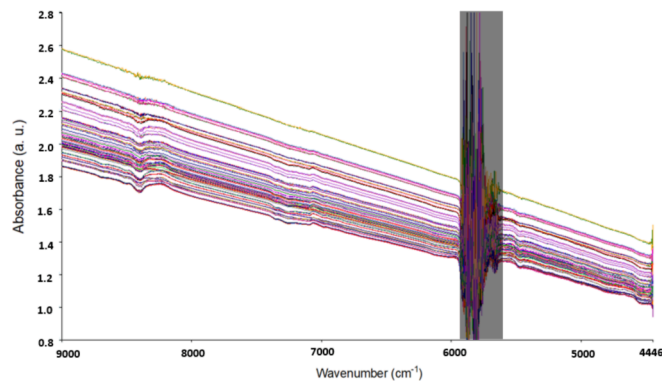
Fig. 6. Spectra of the set of samples included in the model.

possible that represent all the potential variations expected. This way, the model should be able to predict external samples with different characteristics. It is important to consider that a calibration set that includes a wide variation of sample types and a substantial component range will allow a calibration model where a more comprehensive range of samples may be analyzed, but with a resultant loss of accuracy [16].

For this analysis, all the samples of group C were included, 38 samples in the model definition and the other 9 samples for the validation process. Each sample was measured in triplicate, and all three spectra were included in the calibration.

The chemometric calculations were done on Spectrum Quant Quantitative Analysis Software by Perkin Elmer. For both methods, the preprocessing consisted of mean centering the spectral data, which means that the mean spectrum of the samples is subtracted from each standard, and excluding the regions from 11500 to 9000, 5950–5630; 4446–4400 cm^{-1} since they presented noise and do not have information that could contribute to the model, but instead would distortion it. This selection of the appropriate regions enhances the robustness of the model and diminishes the computational cost of the calculations. Fig. 6

shows all the spectra included in the model definition for both methods.

3.2.2.1. Principal component regression. The PCR algorithm was employed to build a quantitative calibration model to assess the concentration of diesel fuel in the prepared samples of used engine oil.

The model's performance was adjusted by K-fold cross-validation. The method is based on an iterative algorithm that removes one or more sample spectra from the data matrix, in this case, 3 samples at a time, and then a model is built based on the remaining samples [16]. The model is then used to estimate the previously removed samples; the process was repeated for all the samples in the calibration set. This allows calculating the error from the cross-validation. The number of principal components used by the model was determined as the number of components that provides a model with the smallest standard error of cross-validation (SECV) [17].

For this model, the number of principal components selected by the method was 7, which achieves a SECV of 1.771 %. Fig. 7 plots the calculated standard error of cross-validation (SECV) for each principal component in the PCR model.

The evaluation of the calibration was assessed by the determination coefficient (R_c^2) and the standard error of estimate (*SEE*), which can be described as the squared root of the residual variance divided by the degrees of freedom, where the degrees of freedom are equal to calibration samples (n_s) minus the number of principal components (n_f) minus one, attending that the data was mean-centered [17]. The parameters are calculated as in the following equations:

$$SEE = \sqrt{\frac{\sum_{i=1}^{n_s}(y_i - \widehat{y}_i)^2}{n_s - n_f - 1}} \qquad (1)$$

$$R_c^2 = 1 - \frac{\sum_{i=1}^{n}(\widehat{y}_i - y_i)^2}{\sum_{i=1}^{n}(\widehat{y}_i - \overline{y_i})^2} \qquad (2)$$

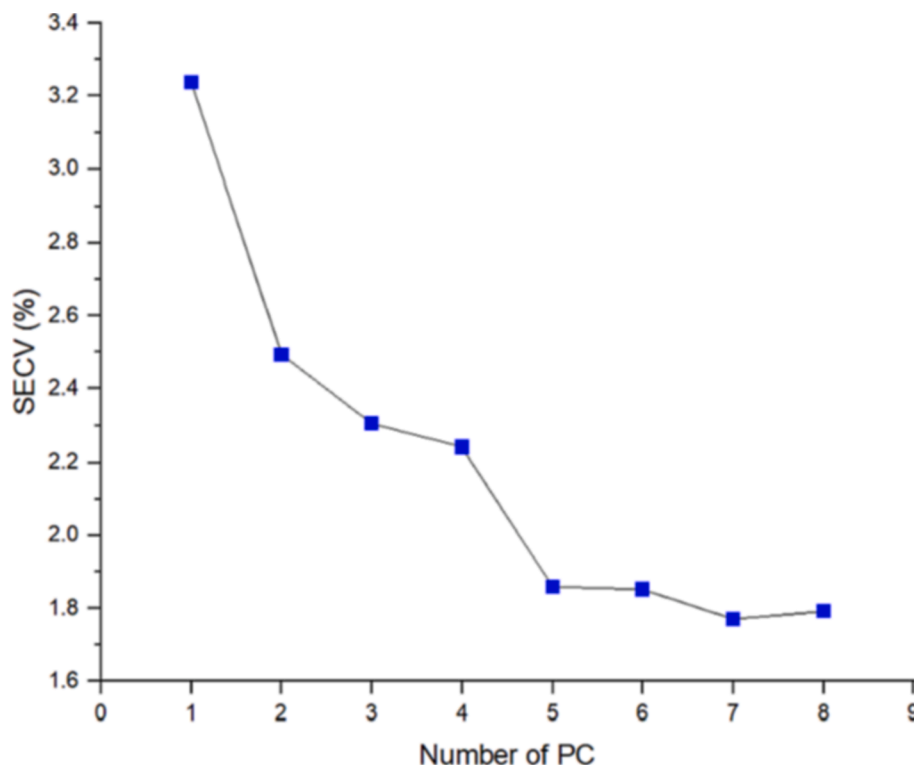where $y_i, \widehat{y}_i$, are respectively, the reference and the predicted value by

Fig. 7. Calculated standard error of cross-validation (SECV) for each principal component in the PCR model.

Table 1
Parameters of PCR and PLS models to determine fuel dilution in used engine oil.

Algorithm	N. variables	R_c^2	SEE (% wt)	SECV (% wt)	RMSEP (% wt)	R_p^2
PCR	7	0.8441	1.353	1.771	1.049	0.8507
PLS	7	0.9435	0.814	1.518	0.436	0.9698

Table 2
Prediction results for PCR and PLS models.

Test samples		Diesel content estimation resulting from		Absolute error for	
Sample	Diesel content (% wt)	PCR (% wt)	PLS (% wt)	PCR (% wt)	PLS (% wt)
Test_1	4.00	4.47	3.97	0.47	0.03
Test_2	8.10	9.11	7.97	1.01	0.13
Test_3	7.50	7.17	8.18	0.33	0.68
Test_4	3.00	3.63	3.25	0.63	0.25
Test_5	3.08	5.05	3.81	1.97	0.73
Test_6	6.31	5.93	6.46	0.38	0.15
Test_7	8.97	8.00	8.72	0.97	0.25
Test_8	2.21	2.01	2.43	0.20	0.22
Test_9	5.03	4.14	4.46	0.89	0.57

the model and $\overline{y_i}$ is the average of the observation values by the model.

The coefficient of determination (R^2) for the whole model gives the proportion of the variability of the property that the model describes. It indicates the strength of the relationship between the property values and the scores.

The proposed PCR calibration model presented a coefficient of determination (R_c^2) of 0.8441 and a standard error of estimate (SEE) of 1.353 %. The standard error of cross-validation was 1.771 %.

To value the model's actual performance, the concentration of nine test samples was predicted by the model; the predicted concentration and the prepared concentration of the samples are shown in Table 2. To evaluate the performance of the predictions, the parameters used were Root Mean Squared Error of Prediction (*RMSEP*) and coefficient of

determination of the test set (R_p^2), that can be defined as follows:

$$RMSEP = \sqrt{\frac{\sum_{i=1}^{m} (y_i - \widehat{y}_i)^2}{m - 1}} \tag{3}$$

$$R_p^2 = 1 - \frac{\sum (\widehat{y}_i - y_i)^2}{\sum (\widehat{y}_i - \overline{y_i})^2} \tag{4}$$

where y_i, $\widehat{y}_i$, are respectively, the reference and the predicted value by the model and $\overline{y_i}$ is the average of the observation values by the model, and m is the number of samples in the test set. The results are shown in Table 1.

3.2.2.2. Partial least squares regression. A second chemometric approach for the fuel in oil quantification was made using Partial Least Squares Regression (PLS) to develop the quantitative calibration model. The spectral data were mean-centered. The performance of the model was adjusted by adjusted by K-fold cross-validation, the number of latent variables (LV) was optimized following the previous criteria and established at 7 latent variables.

Fig. 8 represents the standard error of cross validation (SECV) and the cumulative Y-variance achieved by adding each latent variable in the model.

The calibration was evaluated by the determination coefficient (R_c^2) and the standard error of estimate (*SEE*) where for this case, the degrees of freedom are equal to calibration samples minus the number of latent variables used in the model minus one.

The parameters for the model built by the PLS method were the following: a determination coefficient of 0.9435 and a standard error of estimate of 0.814 %; Table 1 summarizes the parameters for both models.

For both methods, the same samples were predicted, and the results are shown in Table 2. The parameters root mean squared error of prediction (RMSEP) and coefficient of determination of the test set (R_p^2) were calculated from the values of Table 2 and following Eqs. (3) and (4). The PCR model achieved a RMSEP of 1.049 %, while the PLS attained a RMSEP of 0.436 %; this is a magnitude of the expected error
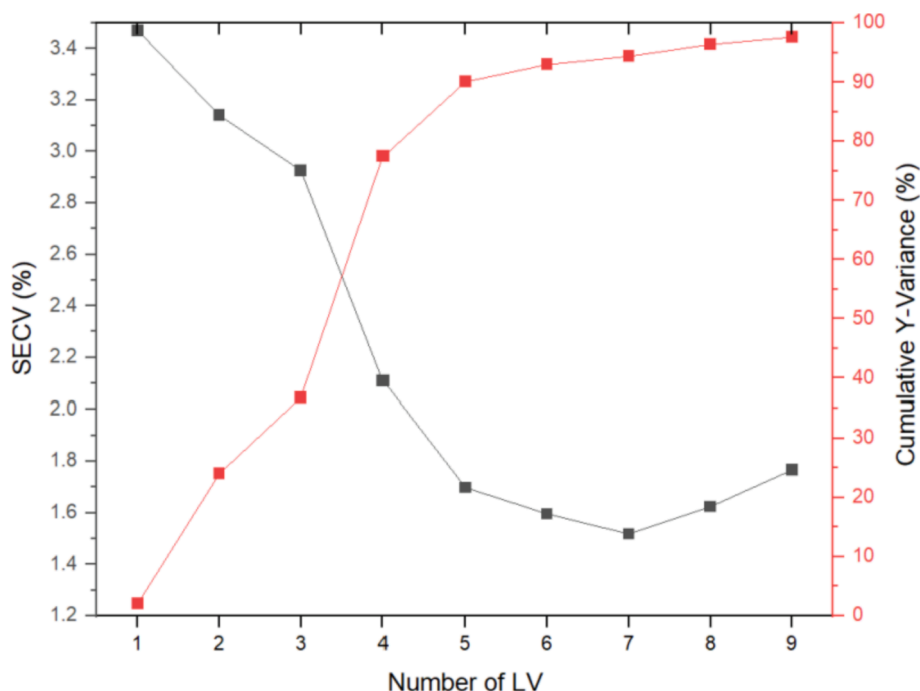

Fig. 8. Calculated standard error of cross validation (SECV) for each LV in the PLS model.

for future samples predicted by the model, which means that better prediction results would be obtained from the PLS method. This result proves that it was possible to obtain a model to access the concentration of diesel fuel in used oil samples using NIR spectroscopy, which could be used as an alternative technique in case chromatography is not available.

Although the results presented for the univariate methods presented a higher coefficient of determination, 0.9646 for the peak heigh analysis against 0.9435 for the PLS model, the PLS model has the advantage of including a larger variety of samples in the same range of concentrations of 0 to 10 % of diesel fuel. The univariate analysis was performed on a reduced group of samples prepared from the same source. In contrast, the multivariate analysis incorporates samples prepared from used oil from six different vehicles, with various mileages and content of soot.

4. Conclusion

This investigation proposes a method to quantify diesel fuel in used engine oil by NIR spectroscopy using a dilution procedure to enable the collection of the NIR spectra; it was verified that the dilution ratio must be proportional to the soot content of the engine oil. It was possible to correlate the fuel dilution with the NIR spectra of used oil samples using univariate methods, as the peak heigh at $4600 \ cm^{-1}$ or the peak area from 4635 to $4558 \ cm^{-1}$ achieving coefficients of determination of 0.9646 and 0.9615, respectively, on a study done on nine samples prepared from the same used oil.

The best predictive model for the quantification of fuel dilution in used oil samples applying chemometric analysis techniques to the spectral data was obtained by Partial Least Squares Regression, which achieved a root mean squared error of prediction of 0.436 % and a coefficient of determination of 0.9435. In comparison, the parameters for Principal Component Regression were 1.049 % and 0.8441, respectively. These multivariate models include samples prepared from used oil from six different vehicles, with various mileages and soot content.

CRediT authorship contribution statement

Vicente Macián: Resources, Funding acquisition, Project administration, Conceptualization. **Bernardo Tormos:** Conceptualization, Validation, Supervision, Writing – review & editing. **Antonio García-Barberá:** Methodology, Writing – review & editing. **Adbeel Balaguer:** Investigation, Formal analysis, Methodology, Validation, Writing – original draft.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fuel.2022.126350.

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