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Viscosity measurement of synthetic oils by means of laser induced fluorescence

B. R. WIESENT*, D. G. DORIGO, F. JÜLICH, Ö. ŞIMŞEK, A. W. KOCH
Institute for Measurement Systems and Sensor Technology, Theresienstr. 90 / N5, 80333, Munich, Germany

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The viscosity is one of the most important properties of lubricants. This parameter has to be adopted to the specific operation conditions where the lubricant is used. We used laser induced fluorescence (LIF) with deep UV excitation at 248 nm and a fibre optic probe to measure viscosity levels of fresh synthetic oil samples. Three types of synthetic oils each with seven different viscosity levels in the range of 32 to 320 mm²/s were measured. A small low cost fibre coupled grating spectrometer was used as interrogator. The different viscosities can be distinguished by their fluorescence intensity distribution in the spectral range 320 to 550 nm. The measured fluorescence spectra of all three oils show characteristic features which allow a clear classification of the fresh oil viscosity levels. To predict fresh oil viscosity levels in a linear model, a partial least squares regression (PLS1) was applied to the gained spectral data set. We reached a good model quality of viscosity levels of all three types of fresh oil. The used setup can also be constructed in a robust and very cost effective manner using a deep UV 250 nm LED and a low resolution VIS grating spectrometer. In-situ measurement of oil viscosity in production processes is possible as well.

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1. Introduction

Spectroscopic techniques for analysing and characterising oil are often based on mid- and near infrared (MIR & NIR) methods. In the MIR range most information on oil condition is contained in the so called fingerprint region at wave numbers lower than 1500 cm^{-1}. A band limited, low resolution LVF infrared miniature spectrometer [1] operating in the wave number range 1818 – 909 cm^{-1} was shown to be capable of detecting oil aging in condition monitoring applications [2]. Such a spectroscopic approach allows the detection of multiple condition indicating parameters like e.g. the total acid number (TAN), oxidation or additive depletion. If only one single parameter of interest has to be measured, it is possible to use a non-dispersive remote sensing approach [3]. However, measuring viscosity levels by means of infrared spectroscopy gives prediction models of rather poor quality. Pure oils can be divided into mineral and synthetic oils. Especially for high quality applications synthetic oils are more and more accepted despite of their higher price. During the hydrocracking step at the lube refining process, the hydrocarbon chains length is adjusted to the specific viscosity of the designed synthetic lubricants [4]. The most common unit for kinematic viscosity is reported in centistokes (cSt) or as SI unit square millimetres per second (mm²/s) [5]. As the viscosity of oil is temperature dependent it is generally given at two distinct temperatures, 40°C and 100°C.

For a number of applications like e.g. oil exploration, process control applications or environmental protection, a quick, non destructive and easy to use analysis method is essential. [6] and [7] demonstrate methods based on LIF to fingerprint crude oils. They used time resolved fluorescence methods to attain increased information.

The objectives of our approach were to develop an easy to handle and low cost method to measure oil viscosity levels of synthetic fresh oil.

2. Materials and methods

We use laser induced fluorescence (LIF) with deep UV excitation and a fibre optic probe to measure viscosity levels of fresh oil samples. A steady state excimer laser at 248 nm wavelength is coupled to a 200 µm core diameter low-OH fibre. The excitation light propagates via a 3 dB coupler and a measurement probe into the oil sample. The deep UV excitation induces fluorescence directly in front of the fibre tip. This characteristic fluorescence spectrum is captured by the same fibre tip probe and guided back to the VIS grating spectrometer where it is interrogated. We use the ultra compact, low cost mini spectrometer module C11007MA (RC-VIS-MOS) from *Hamamatsu* with 256 pixels. The spectral range is 326 to 792 nm. For all measurements an integration time of 1000 ms at low amplification gain setting is used and a mean over 100 spectra is calculated. The fibre tip is polished to be round and cleaned with isopropanol after each run. The fluorescence background spectrum of the fibre is measured before each shot and subtracted from the sample spectrum. To suppress stray light influences the cuvette filled with oil is placed in a sealed dark box. This setup allows a quick sample handling and a simple cleaning process for the re-usable probe. Only the tip of the probe has to be cleaned after each run. The cuvette can be of any oil resistant material. In our case we use a simple plastic container. The LIF is generated directly in front of the fibre tip. During the measurement, the fibre tip has to be placed in the middle of the container in order not to measure the plastic wall. Compared to methods based on IR-spectroscopy we require much less instrumentation, handling and cleaning effort.

Three types of synthetic fresh oils, each with six different viscosity levels in the range of 32 to 320 mm²/s were measured. The used setup is illustrated in Fig. 1.

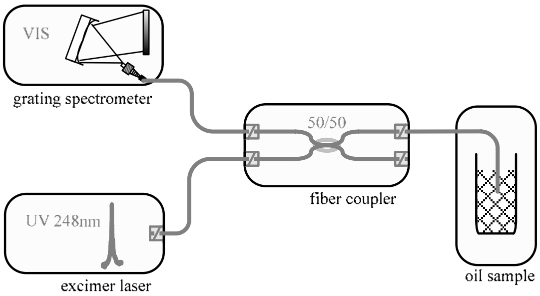

Fig. 1 Measurement setup. A VIS grating mini spectrometer and a steady state excimer laser at 248 nm are coupled to a 200 µm core diameter low-OH 3 dB fibre coupler. The sensing head consists of a polished low-OH 200 µm core diameter fibre coupled to the 3 dB fibre coupler.

The extraction of the viscosity levels out of the measured fluorescence spectra requires sophisticated data processing. We use a multivariate regression model to predict the viscosity level out of each LIF spectrum. As dependent variable the manufacturer information in mm²/s is used. This forms the **Y** matrix, in this case a vector. The **X** matrix consists of the corresponding measured spectra.

First a learn data set has to be composed. This data set includes the LIF spectra **X** as well as the dependent variable **Y** which covers the entire range of the viscosity level that will be predictable later. For each type of oil an individual model has to be built.

The goal of PLS1 regression is to predict **Y** from **X** [8]. PLS components are calculated to reduce the data room. A set of components from **X**, the so called latent vectors which perform a simultaneous decomposition of **X** and **Y** are searched. In this step as much of the covariance between **X** and **Y** as possible has to be explained. The next step performs a regression where the decomposition of **X** is used to predict **Y**. In the first component most of the variance of the **X** data set (LIF spectra) is explained. The higher the component number the less variance is contained. The challenge consists in the choice of the ideal amount of components. The maximum variance and thus the component with the lowest number do not imply the most contribution to the prediction model. In many cases the important contribution to the model comes from higher components and thus from smaller variations in the spectral data set which otherwise stay hidden [9]. However, the higher the component the more noise is modeled. The chosen components are summed up and are comprised in the so called regression coefficient **b** which is calculated in the calibration process.

In order to minimize the influence of modeled noise and thus to optimize the prediction performance of the model, we limited the LIF spectra to the spectral range 326 to 550 nm. In the range 550 to 792 nm the fluorescence intensities of all three types of oil are weak and instrument noise is dominant. The number of used PLS components was found by minimizing the root mean standard error of calibration (RMSEC) and maximizing the signal to noise ratio (SNR) of the regression coefficient **b**.

The prediction of the specific viscosity level is calculated using following equation:

$$y = \mathbf{x}^{T}\mathbf{b} + b_{0},$$

where y is the predicted dependent variable, $\mathbf{x}^{T}$ is the measured LIF spectrum and b_0 is the static offset of the regression line.

We use three different types of oil for all subsequent analysis steps. All three oils belong to the group of synthetic poly-alpha-olefin (PAO) oils and are further named Oil1, Oil2 and Oil3. Oil1 feature a variation in ester concentration over the sample set whereas Oil2 and Oil3 show a change in PAO mixture ratio.

3. Results and discussion

The first analyzed type of oil is the synthetic Oil1. The corresponding LIF spectra are shown in Fig. 2. Visual inspection allows the interpretation of two characteristic features in the spectrum. The LIF intensity at wavelength around 420 nm increases with falling viscosity levels whereas around 480 nm intensities seem to decrease.

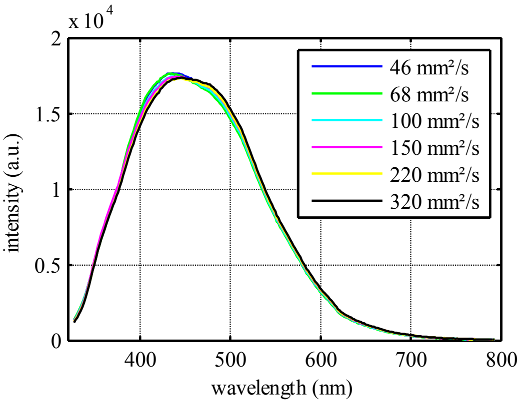

Fig. 2 LIF spectra of the synthetic Oil1 at different viscosity levels.

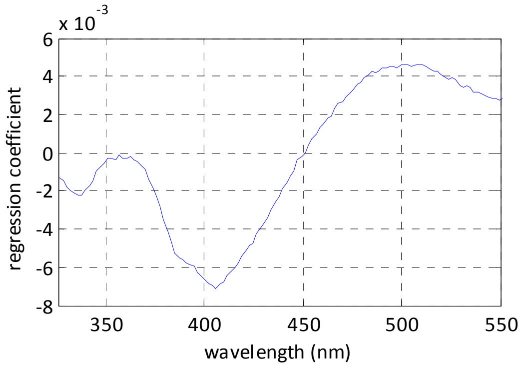

Fig. 3. Regression coefficient of synthetic Oil1. Two PLS components are used.

The regression coefficient clarifies the results of the visual inspection and further reveals a hidden feature in the wavelength range 320 to 350 nm. In addition, the angular point of LIF spectra shown in Fig. 2 corresponds to the zero crossing at 450 nm of the regression coefficient shown in

Fig. **3**. A positive contribution to predicted viscosity levels at higher wavelength and as expected a negative one at wavelength between 360 to 450 nm is evident.

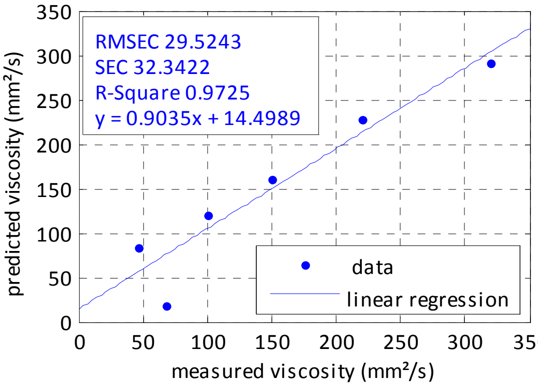

Fig. 4. PLS calibration model of synthetic Oil1. Two PLS components are used.

The predicted versus measured plot illustrated in Fig. 4 indicates a linear prediction model with a RMSEC of 29.524 mm²/s at a correlation R² of 0.9725. The slope of the regression line is 0.9035 at a static offset of 14.4989 mm²/s. One outlier at 68 mm²/s is noticeable. The slope and RMSEC values of the model indicate a good prediction quality but can be further optimized by reducing the impact of the outlier.

The LIF spectra of Oil2 are shown in Fig. 5. By visual inspection a clear classification of spectra with respect to the viscosity levels of the probe is possible. The LIF intensity in the range 400 to 550 nm provides the respective information.

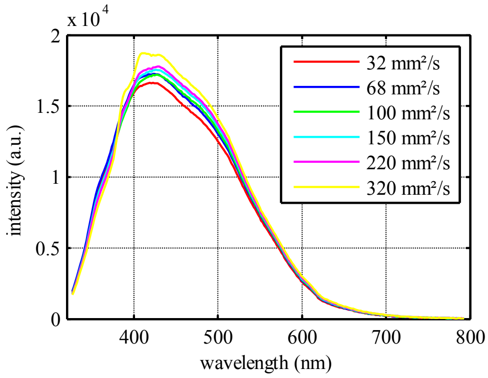

Fig. 5. LIF spectra of the synthetic Oil 2 at different viscosity levels.

The corresponding regression coefficient of Oil2 is shown in Fig. 6. It consists of three PLS components and reveals the range 320 to 380 nm to include most information, whereas the range 380 to 550 nm features a fine structure. This fine structure may only be determined using component analysis.

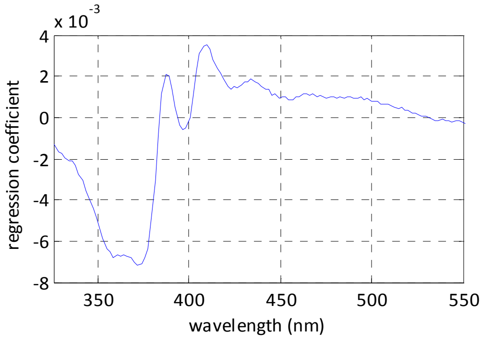

Fig. 6. Regression coefficient of synthetic Oil 2. Three PLS components are used.

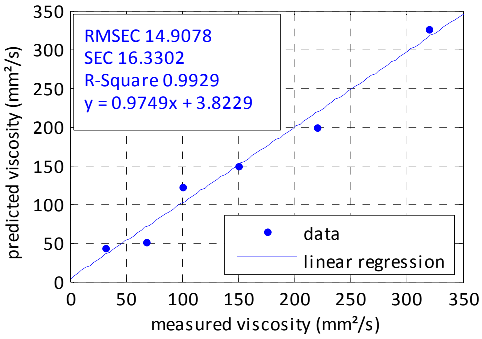

Fig. 7. PLS calibration model of synthetic Oil 2. Three PLS components are used.

The predicted versus measured plot of Oil2, illustrated in

Fig. ***7*** shows a very linear calibration. The RMSEC is at 14.9078 mm²/s at a slope of 0.9749. These parameters indicate a calibration model. Fig. 8 illustrates the LIF spectra of Oil3.

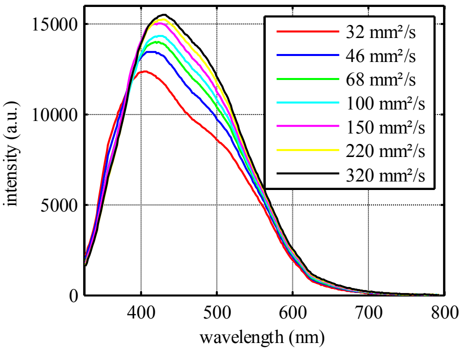

Fig. 8. LIF spectra of the synthetic Oil3 at different viscosity levels.

Visual inspection of LIF spectra of Oil3, plotted in Fig. 8 reveals a clear correlation of viscosity levels and corresponding spectra. LIF intensity in the range 400 to 550 nm features most pronounced variation of all three measured types of oil.

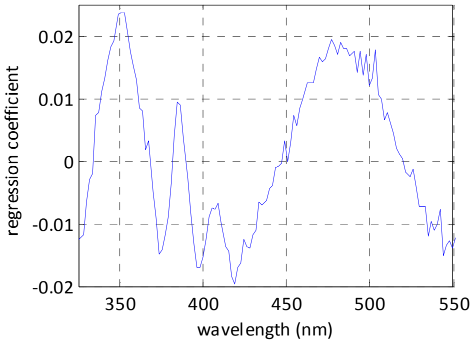

Fig. 9. Regression coefficient of synthetic Oil3. Four PLS components are used.

The regression coefficient of Oil3 was built by four PLS components and features sufficiently distinctive fine structure. In the range 320 to 400 nm several small changes in the measured LIF spectra have strong impact on the prediction. The range 400 to 550 nm features nearly the same contribution to the model as the previous wavelength section.

The predicted versus measured plot of Fig. 10 shows a very linear and steep model with a slope of 0.9955 and a low RMSEC of 6.3273 mm²/s. The prediction quality thus is best of all analyzed types of oil but shows noise appearing in the regression coefficient.

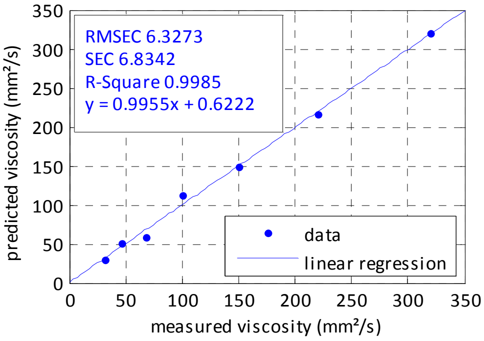

Fig. 10. PLS calibration model of synthetic Oil3. Four PLS components are used.

The prediction performance of all three models can be improved by using more samples to span the **Y** data room. In a larger **Y** data room a cross validation process would lead to a further improvement. Also the spectral SNR has an important impact on model quality. Here a longer integration time, an averaging over more spectra or a higher core diameter of the fiber would give more light and thus give better results.

4. Conclusions

We demonstrate a measurement setup based on LIF spectroscopy and a corresponding multivariate data processing method for measuring viscosity levels of fresh synthetic oils. The measurement system features a fast, easy to use setup with a very good linear measurement response over a wide range of industrial lubricant viscosity levels. The system performance is demonstrated by measuring three different synthetic types of fresh oil. Thereby the sample preparation and handling effort has been reduced to a minimum. This setup can also be constructed very cost effective using a deep UV 250 nm LED, substituting the excimer laser and a low resolution ultra-compact MEMS mini-spectrometer. The measurement system is potentially applicable in in-situ process control applications or in portable hand analyzers. In the next step a sample set of used oils will be analyzed and the performance of the measurement system to oil adulteration in condition monitoring applications is evaluated.

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[*] Corresponding author: b.wiesent@tum.de