Rapid Measurement of Diesel Engine Oil Quality By Near Infrared Spectroscopy (NIRS)

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Abstract: A near infrared spectroscopy (NIRS) rapid measurement was applied to diesel engine oil quality. 100℃ kinematic viscosity change rate and flash point of CD 15W lubricant were built by the means of interval and combination partial least (icPLS). The calibration correlation coefficient ($R^2_{validation}$) and least prediction standard error (SEP) of these models were 0.8954, 2.24% and 0.7729, 2°C respectively. A t-test (α=0.05) showed that the NIR model had an accuracy equivalent to that of the standard method in the analysis for both 100℃ kinematic viscosity and flash point. It was indicated that NIRS method was a new analysis means, which could take place of traditional method, for rapid measurement of diesel engine oil quality.

1. INTRODUCTION

Lubricants play a key role in extending the working life of moving machines. Lubricant monitoring is an effective safeguard technology for the working state monitoring and maintenance of engine and transmission. Periodic monitoring of the chemical condition of lubricant is necessary when it has degraded to a point to which it no longer performs its proper function and may also be an indicator for abnormal operation of the engine. Highly degraded oils have the potential to damage lubricated system components and shorten the life expectancy of the engine or transmission. Then lubricant monitoring provides the foundation of maintenance decision. Some lubricant or equipment producers will recommend some oil-change criterion in order to protect equipment. Nowadays China have built multi national standard of oil-change criterion for lubricant and engine oil [1]. That's to say, lubricant quality is the basic safeguard for normal working of all movable components and the key of ascertain oil-change time [2].

Heavy duty vehicle always works in the quite bad environment such as high dust and high moisture. So that its engine and transmission system wear seriously. The lubricant quality monitoring of heavy duty vehicle has a rather important applying value. Among the monitoring criterion, 100℃ kinematic viscosity change rate and flash point are the direct reflection of the engine deterioration. And they are the main criterions in the GB/T 7607 oil-change criterion of diesel engine oil [3]. The traditional methods to determine the above criterion, ASTM D445 and ASTM D92, are very time-consuming, requiring

sophisticated equipment and specific instrument. Besides, the using engine oil which is the black ropy liquid is very difficult to test. The test error is rather large otherwise the analyst is a senior operator. The traditional methods can't determine these two criterions in line further more [4]. In short, it does not meet the need of a rapid, easy to use, selective, and sensitive analytical technique.

Near-infrared spectroscopy (NIRS) is an easy, rapid and nondestructive technique applied in many areas such as food, pharmacy and petroleum [5-10]. The study of determination kinematic viscosity and water of lubricant by NIRS have been reported [11, 12]. Therefore, NIRS should be a selective method to determine these two criterions of engine oil.

The aim of this paper is to obtain the mathematic relationship and regression equation of NIR spectrum absorption and 100℃ kinematic viscosity change rate or flash point by NIRS. Then a rapid determination method of diesel engine oil quality is to be developed.

2. MATERIALS AND METHODS

2.1. Instrumentation

All samples were tested for 100℃ kinematic viscosity on a kinematic viscosity instrument (Model HCR 1043, Haicr instruments and meters Co. Ltd., Zhen Zhou, China) and for flash point on a open cup flash point instrument (Model SHD-3K, Reshang instruments Co. Ltd., Da Qing, China). Then 100℃ kinematic viscosity change rate is calculated from equation (1):

$$\eta = \frac{v_1 - v_2}{v_2} \times 100 \tag{1}$$

Where v_1 is the determination value of used oil, v_2 is the determination value of new oil.

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Table 1.   Oil Laboratory Reference Data of CD 15W Engine for NIR Model Building

Sample NO.	100℃ kinematic viscosity change rate, %	Flash point, ℃	Remark	Sample NO.	100℃ kinematic viscosity change rate, %	Flash point, ℃	Remark
1	0	233	New oil	16	25.70	229	Used oil
2	19.52	229	Used oil	17	26.87	229	Used oil
3	17.18	233	Used oil	18	20.89	229	Used oil
4	10.17	233	Used oil	19	24.47	229	Used oil
5	18.97	230	Used oil	20	19.38	231	Used oil
6	21.79	231	Used oil	21	25.77	229	Used oil
7	14.50	229	Used oil	22	23.51	229	Used oil
8	20.14	238	Used oil	23	23.99	241	Used oil
9	16.49	231	Used oil	24	30.31	223	Used oil
10	9.76	233	Used oil	25	20.89	229	Used oil
11	15.33	237	Used oil	26	24.47	229	Used oil
12	23.99	241	Used oil	27	25.70	229	Used oil
13	25.70	234	Used oil	28	26.87	229	Used oil
14	27.22	223	Used oil	29	19.38	231	Used oil
15	30.31	229	Used oil	30	25.77	229	Used oil

These were the laboratory reference methods for analysis of samples employed as standards for NIRS calibration development.

NIR spectra were acquired using the NIR spectrometer (Model SupNIR-2600, Juguang science and technology Co. Ltd., Hang Zhou, China) with a transmission InGaSe detector in a useable wavelength range of 1,000 nm -1,800 nm. All measurements were performed using air as reference.

2.2. Sample Preparation and Data Collection

The diesel engine oil model was CD 15W. The certain heavy duty vehicle using diesel was chosen to take samples. 29 samples with 500km interval mileage were obtained by tracking a same vehicle. Then the sample set with 30 samples was consistent of the above 29 samples and a new engine oil. 30 samples were divided into 22 samples as a calibration set and 8 samples as a validation set by K-S method.

The laboratory reference data (Table **1**) were obtained by the standard methods.

2.3. Spectra Collection

All measurements were performed using a 5 cm path-length quartz cuvette at sample temperature (25℃±0.5℃). Fig. (**1**) showed the NIR curve of diesel engine oil.

3. RESULTS AND DISCUSSION

3.1. Methodology

Partial least-squares (PLS) is among the most widely used for processing spectral data and relating spectroscopic signals (predictor variables) to target variables (analyte concentrations, usually). Interval and combination partial least (icPLS) [13] was applied to select the efficient spectral regions that provided the lowest prediction error. The actual modeling course was as follows. Firstly, spectra of calibration set were divided into n regions by a perfect window. PLS Model was built on every spectra region respectively. The optimal PLS model of every region was obtained by the means of cross validation standard error (SECV) and $R^2_{calibration}$. Secondly, the characteristic spectra regions were added from the region with the least SECV and $R^2_{calibration}$ in turn. Lastly, the spectra regions with least prediction standard error (SEP) were chosen to combine to built the prediction model by judging of SEP.

The accuracy of these two NIRS models was analyzed by the statistic analysis of *t*-test respectively. A set of data were built by selecting $z_i=y_i-x_i$ according to the requirement of *t*-test. The mathematical expectation value is 0. The *t* value was calculated from equation (2):

$$t = (\bar{z} - \mu)\frac{\sqrt{n}}{S} \tag{2}$$

Where x_i is the reference value of the No.*i* sample in validation set, y_i is the model prediction value of the No.*i* sample in validation set, z_i is the difference of the model prediction value and reference value of the No.*i* sample in validation set, *t* is the statistical probability at the 95% confidence, $\bar{z}$ is the average of *z*, μ is the mathematical expectation value of *z*, *n* is the sample number of validation set, *S* is the prediction standard error of model.
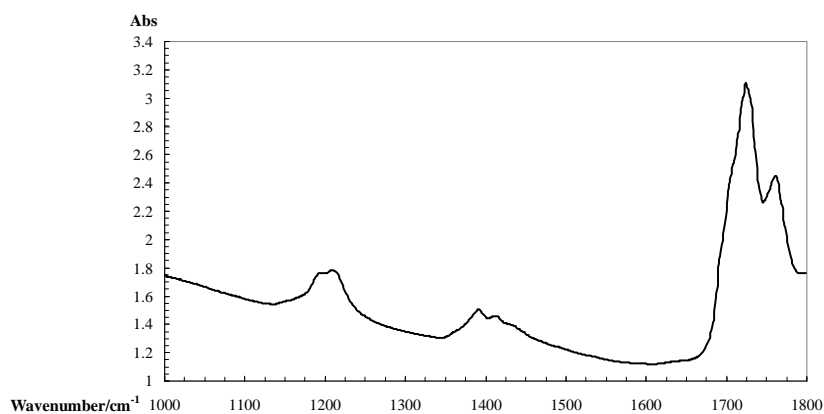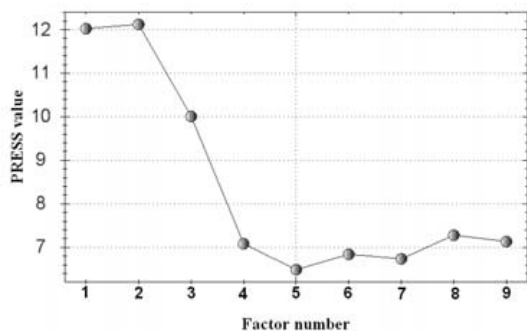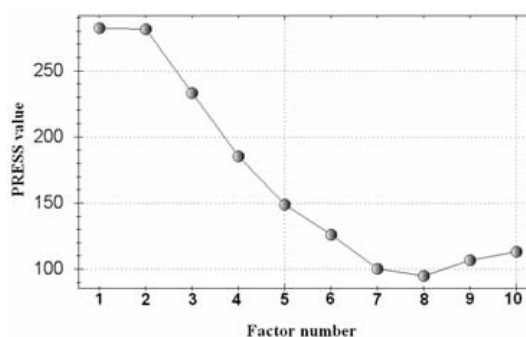
Fig. (1). CD 15W diesel engine oil NIR spectrum.

Table 2. icPLS Model Parameter of CD 15W／40 Engine Oil

Model Name	Spectra Pretreatment	Optimal Factor	SECV	$R^2_{calibration}$
100℃ kinematic viscosity change rate	5 points smoothing, centering, first derivative (30) Orthogonal signal calibration (45)	5	2.11 %	0.916
Flash point	centering, Savitzky-Golay derivative	8	1.023 ℃	0.842

a . 100℃ kinematic viscosity change rate   b . Flash point

Fig. (2). Calibration model PRESS curves.

3.2. NIRS Model Built and Validation

The icPLS models parameters of 100℃ kinematic viscosity change rate and flash point were listed in Table **2**. And the PRESS curves were shown in Fig. (**2**). $R^2_{calibration}$ in Table **2** presented these two NIR models had good correlation with the criterions.

The 8 samples belonging to the validation set were used to test the accuracy of the calibration model. A comparison of the values reported by the NIR method with those of the laboratory reference method was presented in Table **3**. The correlation between the reference values and the predicted values received from the NIRS calibration model was shown in Fig. (**3**).

The correlation coefficient $R^2_{validation}$ of 100℃ kinematic viscosity change rate and flash point were 0.8954 and 0.7729 respectively. SEP of those were 2.24% and 2℃ respectively.

It was presented that these two NIRS models had a good precision.

The difference between NIRS and laboratory reference of these two criterions were statistically significant at the 95% confidence ($\alpha = 0.05$) level respectively because $t_{calculation}$ values of 100℃ kinematic viscosity change rate was 1.200 less than 2.365 and that of flash point was 1.194 less than 2.776. That's to say that NIRS method can replace the traditional method to analyze 100℃ kinematic viscosity change rate and flash point.

4. CONCLUSION

NIRS has the distinct advantages of being much faster— once initial calibration is done—requiring less or no chemicals at all and no sample preparation.Results here showed that NIRS has great potential to predict the physical criterions of engine oil. NIRS provided accurate estimation

Table 3.   Comparison of Results Determined by NIRS and Laboratory Reference Method

NO.	100°C Kinematic Viscosity Change Rate (%)			Flash Point (°C)		
	Reference	**NIR**	**Error**	**Reference**	**NIR**	**Error**
1	10.17	13.71	-3.54	233	230	3
2	18.97	16.54	2.43	230	227	3
3	21.79	19.97	1.82	231	231	0
4	20.22	22.29	-2.07	223	225	-2
5	30.31	31.70	-1.39	229	230	-1
6	19.38	18.94	0.44	231	231	0
7	24.77	22.46	2.31	229	227	2
8	9.87	11.12	-1.25	237	235	2
$t_{calculation}$			1.200			1.194
t_{table} (α=0.05)			2.365			2.776
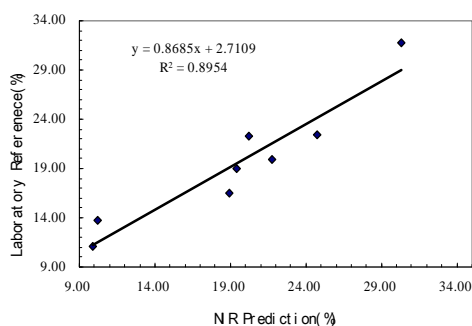
a . 100°C kinematic viscosity change rate
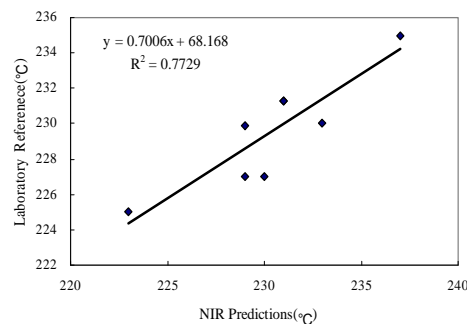
b . Flash point

Fig. (3). NIR prediction versus reference determination

of 100°C kinematic viscosity change rate and flash point. The results obtained encourage the study of these techniques to develop models to determine other physical and chemical properties of lubricant base oils.

CONFLICT OF INTEREST

Declared none.

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