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Application of dielectric spectroscopy for engine lubricating oil degradation monitoring

L. Guan*, X.L. Feng, G. Xiong, J.A. Xie

Oil Application and Management Department, Logistical Engineering University, Shapingba District, Chongqing 401311, China

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ABSTRACT

In this paper, Dielectric Spectroscopy (DS) was employed to analyze the oxidation degradation process of engine lubricating oil qualitatively and quantitatively compared with Fourier Transform Infrared Spectroscopy (FTIR). It was found that both DS and FTIR can directly obtain the degradation features from the spectral data. With the combination of DS and multivariate calibration (Partial Least Square PLS), three main oil monitoring properties including Oxidation Duration (OD), Total Acid Number (TAN) and Insoluble Content (IC) can be determined quantitatively and accurately. It was proved that operating temperature had more influence on DS data than excitation amplitude. The results in the article show that DS can be developed into an effective oil monitoring/analysis method.

1. Introduction

Lubricating oil plays a key role in internal-combustion engines. It consists of complex mixtures of hydrocarbons and is a combination of base oils and additives [1]. Engine lubricants are used to reduce the frictions of the mobile components and to keep the different elements clean, being able to work as detergents and dispersant agents. The engine lubricating oil ageing process is a very complex process during which degradation of the base oil and depletion of its additives take place simultaneously. Oxidative high temperature degradation and contamination by water, ethylene glycol, fuel, soot, and wear metals are the main factors. Currently, the main methods for determining engine lubricating oil condition are the routine physical & chemical tests to assess the properties including kinematic viscosity, Total Acid Number (TAN), Total Base Number (TBN) and Insoluble Content (IC), which are always time-consuming, laborious and require specific equipment for the determination of each property of interest. In this context, kinds of efficient alternative methods have been developed. Ferrography [2–5] is currently the prevalent and effective method to evaluate the wear ferromagnetic particles. Analytical and direct reading (DR) ferrography are the two main types. As for determination of wear metals such as Na, Mg, Al, Ca, Ti, V, Cr, Cu, Zn, Mo, Ag, and Cd. Laser-induced breakdown spectroscopy (LIBS) [6,7], atomic spectroscopy [8,9], X-ray fluorescence spectroscopy [10] are already introduced respectively. Visible spectrophotometric detection in association with flow injection analysis (FIA-visible spectrometry) is proposed and employed to determinate the insoluble content [11]. In particular, middle infrared spectroscopy (MIR) has been largely applied [1,11–17], which offers several advantages for this type of application, such as nondestructive nature.

Recently, much attention has been paid to dielectric and electrochemical impedance properties of industrial lubricants. Lvovich and Schmiechowski [18] have discussed the relationship between chemical composition of lubricants and their electrochemical properties obtained by means of electrochemical impedance spectroscopy (EIS). The non-linear impedance analysis of industrial lubricants has also been performed by higher harmonic non-linear electrochemical impedance spectroscopy (NLEIS) [19]. EIS and NLEIS are able to offer the opportunities to characterize, evaluate and provide insights into chemical composition, changes and mechanisms of lubricants. Wang and Lee [20,21] have used a.c. impedance technique to detect glycol contamination in engine oil. And a new technique to detect minor antifreeze in engine oil by measuring the changes of engine oil resistance was introduced too. EIS has been used to estimate soot and diesel contamination in engine oil simultaneously [22]. Oil condition sensors [23–27] based on dielectric constant and conductivity measurement of the engine lubricating oil have been designed and fabricated. These sensors can detect the relative variation of lubricant degradation. Wang [28] has established a good correlation between TAN and this kind of sensor's output.

* Corresponding author. Tel.: +86 023 86730900.
E-mail address: gl_200122@163.com (L. Guan).

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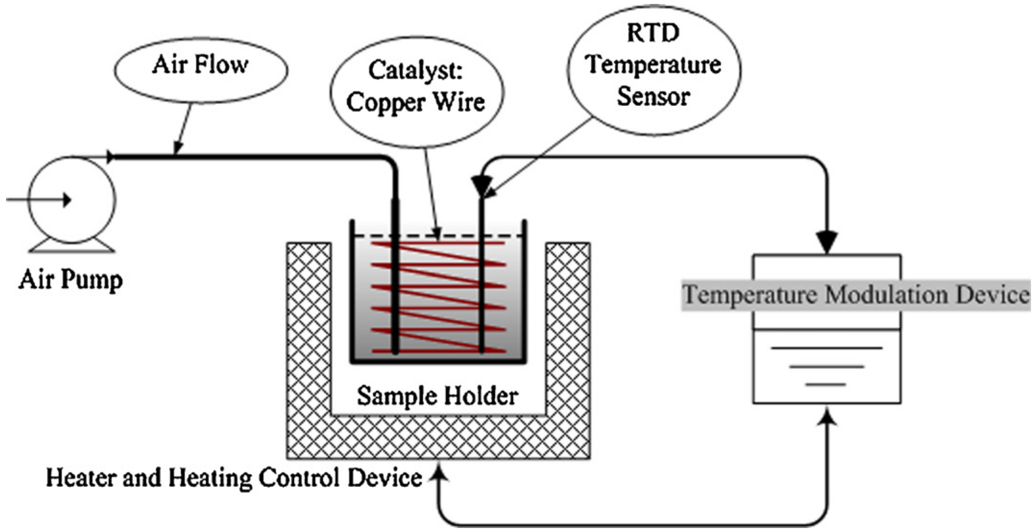

Fig. 1. Schematic diagram of sample preparation device.

Dielectric and electrochemical impedance analysis methods are relatively fast, simple, inexpensive and free from the difficulties associated with the current standard testing methods. Moreover, dielectric and electrochemical properties are related with the chemical composition and physical structures of engine lubricating oil.

Dielectric spectroscopy (DS) is an analytical technique on the interaction between dielectric material and electromagnetic energy in the radio-frequency and microwave range, which is a powerful structural detection technique for dielectric materials. What DS studies is the dependence of materials' dielectric properties on wavelength or frequency. The difference between DS and EIS: the main interest of DS is on the intrinsic electric material properties. The complex permittivity $\varepsilon^*(\omega)$ or conductivity $\sigma^*(\omega)$ spectra can be easily evaluated from $Z^*(\omega)$ with the help of sample dimensions; the focus of EIS is mostly on the properties of electrode/material interfaces and the materials under test are often electrolytes or ion conductors. Petroleum products including engine lubricating oil are all typical dielectric materials. So DS is more suitable than EIS for engine lubricating oil analysis.

At present, DS technique enables researchers to make sound contributions to contemporary problems in modern physics. DS has been employed to quality sensing application of agricultural product [29,30]. Our previous publications have investigated the correlations between DS data and petroleum products' composition and quality indexes by means of multivariate calibration, which include classification of virgin engine lubricating oils by SAE and source [31] and determination of clean gasoline octane numbers [32]. It is already proved that DS is a practical and effective analysis method to obtain rich composition and structure information of complex mixture systems. Especially, with the help of chemometrics multivariate calibration methods the direct relationships between DS data and quality properties can be established efficiently, which is more effective than the interpretation of EIS results by means of complex equivalent circuit (EC) models. The new analysis idea is explained in our previous publication too [32].

The monitoring sensor for oil condition, especially for engine lubricating oil should be easy to be cleaned and is able to extract sufficient dielectric information. Interdigitated comb capacitor sensor is a good and practical selection, which has been applied for oil analysis widely [27,31–33]. So the measuring sensor for engine lubricating oil in this article is a type of interdigitated comb capacitor sensor too.

In this article, DS is employed to examine the oxidative degradation of engine lubricating oil and to determine the Oxidation Duration (OD), Total Acid Number (TAN) and Insoluble Content (IC) properties. All the DS analysis results are discussed compared with the Fourier Transform Infrared spectroscopy (FTIR).

2. Experimental

2.1. Samples

All the samples were prepared with the device illustrated in Fig. 1. The main function of the device is to oxidize the virgin samples under given oxidation conditions. The air flow generated by the air pump and the copper wire catalyst are used to accelerate the oxidization process.

According to the sample preparation device, three types of virgin engine lubricating oils were employed to produce three series of samples with different degrees of degradation. The three virgin engine lubricating oils are listed in Table 1.

For each series of samples, three types of properties including Oxidation Duration (OD), Total Acid Number (TAN) and Insoluble Content (IC) were recorded. OD values were recorded according to sampling time. TAN and IC properties were respectively determined by TAN&TBN Analyzer and Insoluble Content Analyzer made by Beijing China Invent Instrument Technology Ltd. Company. The samples with three properties are shown in Tables 2–4.

Table 1
Information on three virgin engine lubricating oils.

Name	API	SAE	Company	Oxidation temperature (°C)
Set one	CD	15 W/40	KunLun (China)	150
Set two	CD-SE	50	Shell	150
Set three	SJ	15 W/40	Dalian Petroleum (China)	150