

## Capillary Sensors with Two Coupled LEDs for UV-Forced Degradation and Fluorescence Reading of Chemical Stability of Diesel Fuels

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**Abstract-** The stability of the diesel fuel is related to the fuel composition that evolved to the modern fuels from the historical ones. The instability of modern diesel fuel is mainly due to the presence of unsaturated components and components with oxygen. Standard laboratory techniques for measuring fuel stability are relatively complex and time consuming. The time of examination of initial stages of degradation is in the rank of a dozen of hours. The fast methods of diesel fuel stability pointing with single ultraviolet (UV) wavelength forced degradation and fluorescence reading have been proposed. The present paper concentrates on the possibility of further reduction of time of examination of the diesel fuel that enables initial and advanced stages of fuel instability indicating. The reduction of time of degradation is expected as two light emitting diodes (LED) working at different wavelengths are implemented in sensor head. The long-wave ultraviolet (UVA) - 365 nm high power light emitted diode and the short-wave ultraviolet (UVC) - 265 nm light emitted diode are selected for examination. The procedure of degradation and examination with signal switching of LEDs has been proposed. Experimental results are presented for the dual LED degradation system in comparison of to the single 265 nm LED degradation system. Obtained characteristics show fluorescence emitted signal at selected wavelength bands dependency of initial, medium and advanced effects of fuel degradation. The results show that using dual LED system increases fuel degradation speed as an advanced effects of fuel instability arise in the time less than one hour.

**Keywords-** biodiesel fuel stability; diesel fuel instability; unsaturated hydrocarbons; capillary sensor; LED excited fluorescence; UV degradation.

### I. INTRODUCTION

Capillary measurement systems are popular in many chemical laboratories as they enable measurement of complex solutions. Capillary electrophoresis (CE) is one of the highest efficiency small volume separation methods available and laser induced fluorescence (LIF) detection is the most sensitive CE detection method [1]. It should be pointed that, CE is appropriate to examination of conductive solutions. Therefore, CE is not applicable to direct inspection of oils [2][3]. The LIF detecting units are still expensive and dangerous to eyes. Thus, implementation of LIF in sensor is the subject of investigations [4]. Capillary measurement systems are also made with the use of liquid core waveguide (LCW) and LED fluorescence detection unit. A Teflon AF coated fused silica capillary usually serves as the LCW [5]. Therefore, examination of oils is quite difficult. But, implementation of low cost unit for fluorescence examination with dual LED system was proposed. This unit, consisting of one high power LED worked at 365 nm and other worked at 405 nm, seems very interesting and fit for in sensing device application.

A dedicated sensor of diesel fuels stability using a disposable capillary optrode and UV radiation as the fluorescence excitation factor and fuel degradation initiator was investigated [6]. The preliminary results of an examination, using a single wavelength configuration of the sensor, show that it is possible to obtain the first signs of fuel instability in a time less than one hour [7]. Therefore, advances of two wavelength examinations using two LEDs working at UVC and UVA in sensor dedicated to

examination of diesel fuel stability are presented in this paper.

#### A. Modern diesel fuels

There are many types of diesel fuels including petrodiesel, syn-diesel (synthetic) and bio-diesel. The fuel types can be further classified in groups as for example petrodiesels include classical petro-diesel and modern petrodiesel groups, while bio-diesels include Fatty-Acid Methyl ester (FAME) or Hydrogenated Vegetable Oil (HVO) components. The syn-diesel main groups cover liquid fuel components. Liquid syn-diesel is produced from any carbonaceous material that was gasified, purified and converted in the Fischer–Tropsch process. Gas syn-diesel is Dimethyl Ether (DME).

Classical petro-diesel fuels are made from stable components of straight run distillate products of crude oil (alkanes) with an addition of improvers [8]. Modern petrodiesel fuels include stable and unstable components.

Stable components are alkanes and cycloalkanes. Unstable components include acyclic and cyclic unsaturated hydrocarbons. Acyclic unsaturated components may include alkynes, alkadienes and alkatrienes, while cyclic unsaturated components may include monocyclic benzene (MAH – Monocyclic Aromatic Hydrocarbon) and Polycyclic Aromatic Hydrocarbons (PAHs). MAH is represented by benzene, a natural liquid constituent of crude oil and one of the elementary petrochemicals. The simplest PAHs are naphthalene (white crystalline solid), having two aromatic rings and the three-ring compounds anthracene (colorless solid) and phenanthrene (white powder). Diesel fuel is expected to contain PAHs particles ranging from three to five ring systems [9], although the harmfulness of PAHs for human health is a known issue. Thus, the volume of PAHs with two or higher number of rings is limited in Europe by the Directive 2003/17/EC to 11 wt%. But, for the defined in Sweden class 1 diesel fuel the limit value for PAHs with three or more rings is 0.02 vol%.

It was historically postulated that classical petro-diesel fuel may be kept in storage for prolonged periods, but also that synthetic diesel and HVO components are generally first class fuels.

On the other hand, modern petro-diesel and FAME are unstable and their degradation is accelerated with oxygen presence, temperature and sunlight, which include ultraviolet radiation [10]-[12]. The results of diesel fuel instability are sediments and gum formation. The results of bio-diesel degradation with polymer and soluble waxes formation is the increase in the diesel fuel viscosity [13]. The changes in molecular composition of the fuel affect its ignition characteristics, which is one of the most important diesel fuel quality parameters. The minimum cetane number of diesel fuel differs by region. For example, in Europe the minimum value is 51, but in some regions of United States it is 40. It should be noted, that obtaining fuel with high cetane number is not simple task.

There are two methods to increase the cetane number. The first method is to reduce the unsaturated content of fuel by hydrogenation - this is usually not done because of a

high cost. The second method is using cetane improving additives – this method is preferred due to its low cost [14]. The 2-Ethyl Hexyl Nitrate (2-EHN) is the most popular improver, but it can be an additional cause of the fuel instability. Thus, the extended period diesel fuel storage conditions have to be described with the use of parameters including exposure to air and light with a specified environment temperature.

#### B. Optoelectronic measurement systems and sensors for diesel fuel testing

There are many standards of fuel testing procedures, but their applications are limited to petrochemical laboratories. New methods and adaptation of examination standards to ordinary laboratory conditions and in-situ examinations are in development. The most popular directions of investigation include fuel type and fuel fit for use classifications [15]-[17]. For such measurement set-ups and corresponding measurement procedures dedicated electronic components [18]-[20] and units [21]-[23] as well as methods of data analysis [24][25] are under development.

Spectrophotometric methods consisting of examinations of light absorption, scattering and fluorescence are popular for diesel fuel testing. Light absorption in infra-red is used in screening analysis to detect specific types of adulteration in diesel/biodiesel blends [26]. Biodiesel content determination in diesel fuel is also possible with the use of fluorescence examination [27].

The Time-Resolved Laser-Induced Fluorescence Spectroscopy (TRLIFS) enables the characterization and identification of different petroleum diesel fuels, biodiesel from different feed stocks and their blends. A typical TRLIFS set-up consists of an ultraviolet excitation pulsed laser working at 266 nm wavelength, an acquisition system measuring the dynamic response with a specific time gate width, and software that compares obtained results as: recorded impulse shape, frequency behavior and decay behavior, maximal intensities and the wavelength of the maximal fluorescence intensity to a previously created data base [28]. Similar created databases used with measurement data of Synchronous Scanning Fluorescence Spectroscopy (SSFS) that are processed with multivariate analysis enable the determination of quality parameters of diesel blends [29]. The sample measurement set-up of SSFS includes a fluorescence spectrometer, for example Shimadzu RF-5301, that is equipped with a high power xenon discharge light source that converts about 150 W of power into radiation spectrum for sample excitation with wavelength width equal 1.5 nm scanned from 220 nm to 750 nm, while the recorded wavelength interval is 50 nm.

The literature review may lead to the conclusion that spectrophotometric methods require costly equipment dedicated to a chemical laboratory environment. But fortunately, diesel fuel stability may be considered as a function of most unstable components presence and concentration. These unstable components include multi-unsaturated hydrocarbons are known as fluorophores [30]. For example, anthracene absorbs radiation in the range of 200 nm to 380 nm and emits light mainly in the range of

380 nm to 600 nm with characteristic multi peak spectrum with three major peaks with maximum at 430 nm [31]. For optical detection of such particles presence, the optoelectronic components are available. Such components may be the base of prototyping of rapid in examination and low-cost in use sensors for reliable determination of fuel chemical stability.

The rest of this paper is organized as follows. Section II describes the sensor configuration including the sensor head with the optrode as well as the optoelectronics system set-up. Section III addresses the experimental results of the fluorescent signal analysis. Section IV goes into short conclusions.

## II. SENSOR CONFIGURATION

The idea of the sensor was inspired by the need to come up with a low-cost measurement method for a set of diesel fuels utilizing forced UV degradation effects in one system that would require the minimum of automated mechanical elements.

### A. Sensor head

The sensor's head consists of two functional units: the head base with fixed optical fibers and the capillary optrode, as shown in Figure 1.

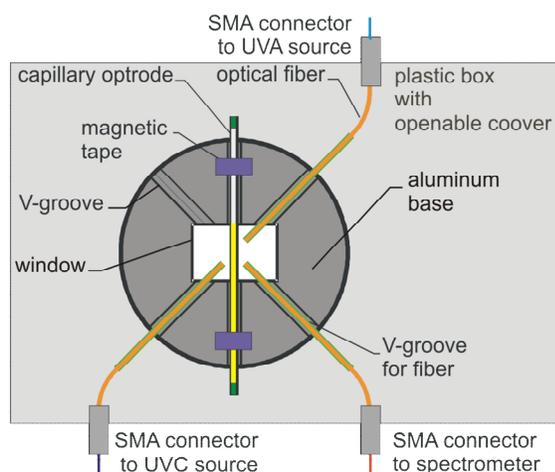


Figure 1. Head of the capillary sensor.

The use of capillary reduces significantly the costs of measurement in a few ways:

First, the measurement of diesel fuel degradation results in adhesive gums generation inside the vessel, thus a disposable vessel is a must. Moreover, diesel and biodiesel fuels affect plastic optical cuvettes made from poly(methyl methacrylate). Therefore, using a capillary glass optrode reduces costs versus classic cuvettes.

Second, the capillary optrode introduces a smaller fluorescent aperture and radiation path compared to classic cuvette holders. Thus, the optical power used for degradation and excitation of fluorescence in the proposed head may be significantly lower (100 times) than when a commercial spectrophotometer is used. Therefore, radiation

sources with lower power can be used in the construction of the sensors, for example light emitted diodes instead of laser diodes.

Third, the capillary optrode is filled with the use of capillary force; therefore, pipettes are not used contrary classic set-up.

Thus, the capillary optrode CV7087Q is used here as a replaceable and disposable vessel.

### B. Sensor scheme and measurement procedure

The set of parameters of interest may be observed using forced UV degradation realized simultaneously at UVC 265 nm and UVA range 366 nm wavelengths. Light emitted diodes can be used as a cost/performance balanced radiation source at these ranges of wavelengths. Commercial LEDs powers at UVA wavelengths are relatively high, as 15 mW at 365 nm are available. What should be noted is that the powers of LEDs in mentioned wavelengths increase with current technological progress. For example, single chip M365F1 LEDs, that represent the state of the art of the last few years, has the maximum power of about 4 mW, while this year's M365FP1e Thorlabs' element's maximum power is about 15 mW. LEDs at UVC and UVB ranges are characterized by significantly lower emitted powers than those for UVA [32]. In the UVC range the single chip LEDs maximum power is about 1.5 mW. Maximum power increase of UVC LEDs is realized with the multi-chip construction as for example in the UVLUX250-HL-5 element. Unfortunately, such construction does not fit well with the fiber optics sensor technology.

The sensor scheme designed to realize simultaneous degradation at UVC and UVA and fluorescence reading with excitation at UVC is presented in Figure 2.

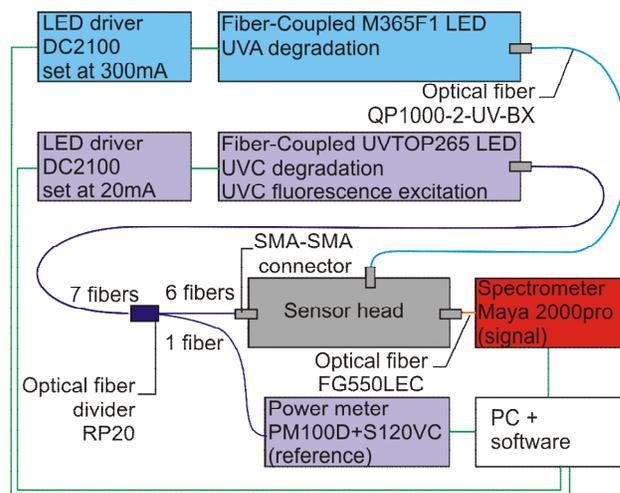


Figure 2. Scheme of the capillary sensor.

The optoelectronic set-up is divided into two parts, the UV radiation sources and the detection unit, both connected to a personal computer equipped with spectra acquisition software and system script designed for measurement automation.

As the UVA source, fiber-coupled LED Thorlabs M365F1 with the 365 nm dominant wavelength was used. As the UVC source, the UVTOP265 from Roithner LaserTechnik was implemented into Thorlabs SM1 micromechanical components to make a fiber-coupled unit. Diodes were connected to DC2100 controllers operating in the constant current mode. In the presented set-up the powers delivered from both sources are not perfect balanced. The source working at 366 nm delivers 1 mW of power, while the source working at 265 nm delivers 300 μW.

The detection unit consists of the power meter (PM100D+S120VC), the optical fiber divider (RP20) and the spectrometer (Maya 2000pro). The RP20 divider is used to divide the UVC signal between the head and the power meter. Thus, UVC radiation can be used for degradation and for fluorescence excitation with a precisely monitored value. This value is used for the calibration of the fluorescence emitted signals. The spectrometer is used for the acquisition of spectra signals. The software on the personal computer enables proper sequencing of the degradation measurement procedure.

Examination procedure resulted in better than 280 [a.u.] absolute measured signal accuracy when full scale range of analyzed signal is 64000 [a.u.].

### III. EXPERIMENTAL RESULTS

Experimental results have been obtained for two measurement procedures. The first procedure was with active UVC and UVA sources. The time stamp of first two steps of this procedure is presented in Figure 3.

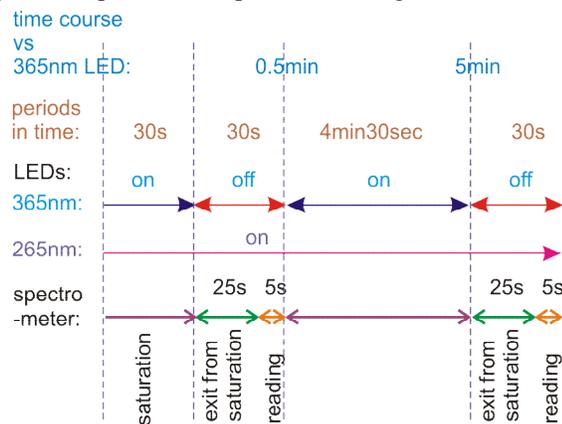


Figure 3. Initial steps of first measurement procedure with both UV sources used in fuel degradation.

The time stamp is calculated versus time of activation of UVA source. The second procedure used for evaluation was performed with only one active UVC source. For the second procedure, the time stamp is provided with the same starting point as for the first procedure. The measurement procedure time events are triggered with 1 second accuracy. The experiments were performed for two winter diesel fuels marked as standard and premium, but both characterized with identical chemical stability according to EN 15751

standard equals 20 hours and 25 g/m<sup>3</sup> base of ISO 12205. Base of both fuels consist of hydrocarbon mixtures that include particles with carbon number from C9 up to C25. Both fuels are characterized by initial boiling points in 175-180°C range and solid state pollution 24 mg/kg according to EN 12662. Examined fuels differ in cetane number, which for premium one is 55 and for standard fuel is 51. The bio-component is not present in premium diesel fuel. The standard fuel includes 93% of petro-diesel and up to 7% of FAME including saturated and unsaturated components with carbon number from C16 to C18.

The premium winter diesel fuels examination with first procedure results are presented in Figure 4, while examination results with the second procedure are presented in Figure 5.

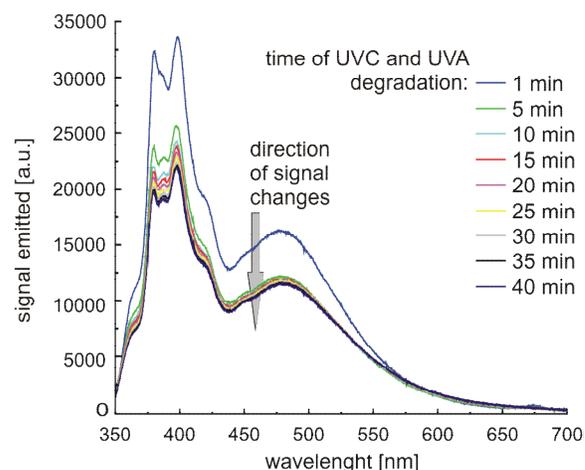


Figure 4. Spectra of first measurement procedure with UVA and UVC sources used for premium winter fuel degradation.

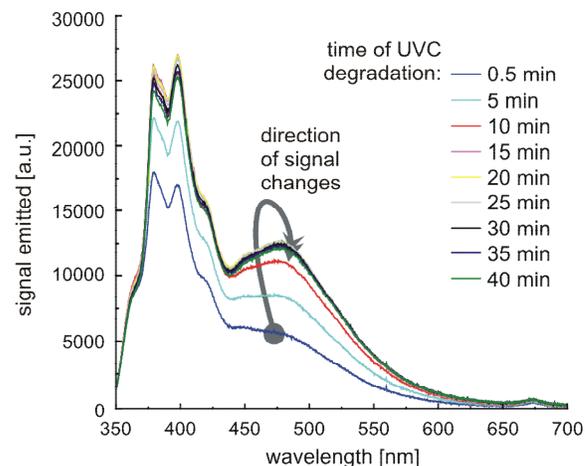


Figure 5. Spectra of second measurement procedure with one UVC source used for premium winter fuel degradation.

The results obtained for two types of winter fuel degradation look similar. The big difference is in the direction of signal changes during degradation. The signal decreases for two UVC and UVA sources degradation,

while it first increases and then slowly decreases for single UVC source degradation. Moreover, for two UVC and UVA sources degradation, the absolute emitted signal level is higher than for one UVC source. The shape of saddle between peaks at 380 and 405 nm is also different, pointing that there is a possibility of a new unsaturated hydrocarbon generation for two wavelengths degradation. The small peak of emission at 675 nm can be observed in Figure 4 and Figure 5. For degradation with first procedure the peak disappears while with second procedure the peak presence lasts.

The results of the eco winter fuels examination with the first procedure are presented in Figure 6, while the results for examination with second procedure in which one UVC source is active are present in Figure 7.

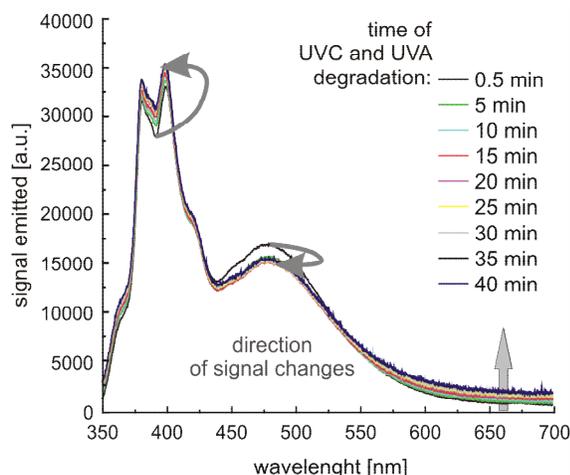


Figure 6. Spectra of first measurement procedure with UVA and UVC sources used for standard winter fuel degradation.

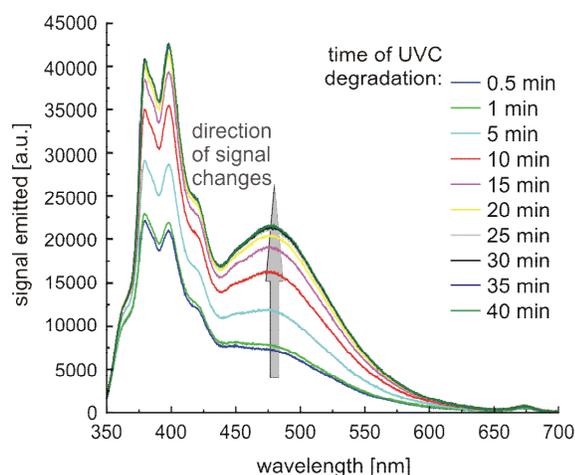


Figure 7. Spectra of second measurement procedure with one UVC source used for standard winter fuel degradation.

In the latter case, the spectra are also similar, but as previously changes of emitted radiation are in different directions. But, the spectral signal increases in range from 550 nm towards 700 nm in the case of UVA and UVC

degradation, presented in Figure 6. This is significant for detection of advanced fuel degradation products, as emitted signal presence in this range can be connected with resin development.

#### IV. CONCLUSIONS

We have examined a capillary sensor of diesel fuel chemical stability with forced UV degradation realized at 265 nm and 365 nm wavelengths with fluorescence reading with excitation at 265 nm. These observation points, that chemical instability of diesel fuel is a result of complex chemical reactions that follow one after the other. Obtained results show emitted signal dependency on degradation grades. The initial grade of degradation can be related to increase of emitted signal in 380 - 420 nm and 450 - 525 nm ranges. The medium grade of degradation can be related with signal saturation in mentioned above ranges as well as disappearance of 675 nm peak. The advance grade of degradation can be related to increase of signal in 550 - 700 nm range.

The analysis of the signals measured during fuel degradation with two radiation sources working at UVA and UVC shows that the emitted signal characteristics saturate faster than in the case of examination with one UVC source. Moreover, in the one-sensor set-up we have shown that depending on the used degradation regime the changes of emitted signal differ for different fuels. Interestingly, for different fuels characterized with identical chemical stability at 475 nm, the saturation time of changes is similar for examinations with two UV sources. Therefore, we conclude that the proposed sensor configuration may lead in future to the reduction of the time of examination of diesel fuel stability. Obtained results, for set-up with two LEDs sources, show the necessity of degradation procedure, parameters optimization as initial stages of fuel degradation are crossed a bit too fast.

#### ACKNOWLEDGMENT

This work was supported by the WUT grant no. 8956/E-365/S/2017, task 504/03115/1035/40.000105.

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