Capillary Rise Multiparametric Sensor for Testing of Diesel and Biodiesel Fuel

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Abstract-**There are many fuel quality standards introduced by national organizations and fuel producers. Usual techniques for measuring fuel parameters like viscosity, density, cetane index, fraction composition and flash point, require relatively complex and expensive laboratory equipment. On the fuel user side, fast and low cost sensing of useful state of biodiesel fuel is important. The main parameters of diesel fuel compatibility are: density and viscosity. Diesel fuel surface tension is one of the main properties for characterization of the quality of the fuel atomization process that affect fuel quality. The present paper concentrates on the construction of the sensor which enables the examination of the mentioned parameters in one arrangement. Results of development of a capillary photonic sensor working on the principle of multipoint monitoring of capillary rise of fuel in inclined capillary are presented. We discuss the principle of the sensor's operation, the construction of the head, and the experimental results of testing biodiesel fuels for their parameters. We conclude that the proposed construction may be in future the base of low cost commercially marketable instruments.**

Keywords-biodiesel fuel; diesel fuel quality; surface tension of diesel fuel; multiparametric sensor; capillary sensor.

I.   INTRODUCTION

The paper consists of 5 sections. First section was the introduction where the diesel fuel, diesel engine properties as well as known sensors for diesel fuel testing were discussed and the aim of work is presented. Second section describes the proposed idea of multiparametric sensor for diesel fuel testing. Developed sensor construction presented in section three. In the section four the discussion of obtained results of characterized diesel fuels examination in developed sensor is presented. The method of fuels classification is proposed. The short conclusion is gathered in section five.

Classical diesel fuels are made from distilled products of crude oil with addition of improvers. Biodiesel fuel is a mixture of classical diesel fuel and bio-components.

One of the most important diesel fuel quality parameters is ignition quality. The ignition quality depends on the molecular composition of the fuel and is characterized by the ignition delay time, which is the time between the start of injection and the start of combustion. For standardization reasons, measurements of ignition quality of fuel (CN) have to be carried out in the Cooperative Fuel Research engine (CFR-5) or ignition quality tester (IQT™). The basic disadvantage of such approach to fuel quality measurements is the high cost of the measurement device and the complexity of the procedure.

Nowadays, producers define the useful state of diesel fuel by several parameters: cetane number, density, and distillation parameters, kinematic viscosity. Other diesel fuel parameters characterize its operability: amount of carbon residue, water and sediment, cloud point, conductivity, oxidation stability, acidity, copper corrosion, flashpoint, lubricity, appearance, and color [1]. For the ordinary fuel user, such collection of parameters is often too complex. In this situation the user requires the simplest possible answer to a question: is that fuel useful for my engine?

A.   Diesel fuel and engine

The challenge for diesel engine designers is to match the combustion chamber size with the diesel fuel injection characteristics [2]. The diesel fuel must be introduced into the combustion chamber, vaporize and react with oxygen at an assumed speed. The fuel properties that have greatest effect on injection process are viscosity, density and surface tension. If the injection is made at a constant pressure, the viscosity affects the fuel spray formation by limiting the speed of fuel flow. If the injection system is designed to

meter the volume of injected fuel, the density of fuel defines the fuel injected mass that is linked with the useable portion of energy. The surface tension is the one of main factors that describes the fuel tendency to form drops, known also as the fuel atomization process or spray forming.

It was postulated that the surface tension of most liquid hydrocarbons is very similar. For example, for HydroCal 300 - a hydrotreated naphthenic medium grade lubricant oil and IFO-120, an intermediate fuel oil, the measured surface tensions at 25°C are the same and equal 31.8 mN/m, while their viscosities differ significantly: 162mPa·for HydroCal and 487mPa for IFO-120 [3]. It should be noted that contemporary diesel fuels components are not only pure hydrocarbons. Present-day diesel fuel may include some biodiesel which is obtained from vegetal oils or the bio-mass, and other additives. These supplements may significantly change surface tension and other diesel fuel parameters [4].

In a European study, it was observed that using the biodiesel fuel at low environment temperatures can lead to the degeneration of engine [5]. Therefore, production standards for biodiesel fuel were introduced: density at 15°C (ISO3675) and low-temperature fluidity for the transitional seasonal periods and winter (DIN EN 116). Important disadvantages of biodiesel fuel can be overcome by fuel processing. A new generation of biodiesel fuel can be made with bio-component isomerization or hydrogenation [6].

It has been proven that surface tension is one of the most important of inhomogeneous fluid properties. It reflects the range of interactions in a fluid more directly than the bulk properties do [7]. The relationship between the surface tensions of different real vegetable oils and their fatty acid composition was postulated in [8]. Predicting the surface tension of biodiesel fuels from their fatty acid composition showed that the differences in surface tension between biodiesel types are not the only cause of the reported differences in engine tests [9].

There are few basic methods of surface tension measurements: the capillary rise method, the drop weight method, the ring or plate method, the maximum bubble pressure method, the method of analyzing the shape of the hanging liquid drop, and the dynamic methods [10].

The capillary rise method is the oldest method used for surface tension determination. For the surface tension measurement a capillary is dipped into the tested liquid. If the interaction forces of the liquid with the capillary walls are stronger than those between the liquid molecules, the liquid wets the walls and rises in the capillary to a defined level. If the cross-section area of the capillary is circular and its radius is sufficiently small, then the meniscus is semispherical. When the capillary is filled in the vertical position the surface tension may be calculated with following equation:

$$\gamma \cong \frac{1}{2\cos(\theta)} r \cdot g \cdot \left(h + \frac{r}{3}\right) \cdot (\rho_l - \rho_v), \qquad (1)$$

where: γ is the surface tension, θ is the contact angle, r is the inner radius of capillary tube, g is the gravity constant, h is the height of meniscus, ρ_l is the density of liquids, and ρ_v is

the density of vapor [11]. For standard temperatures, the density of liquid is much greater than of the density of vapor, therefore presence of the vapor phase is ignored. The contact angle may have different values in the static or dynamic situations. Such situations for a capillary being filled are presented in Figure 1.
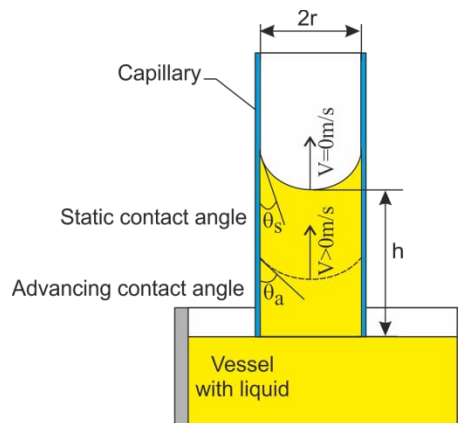

Figure 1.   Static and advancing contact angle in capillary flow.

Since a film of oil remains on the inner surface of the capillary after the receding phase, representing complete wetting, the static contact angle was considered to be equal to 0° [12].

Advancing contact angles between a glass slide and different hydrocarbon oils differ significantly depending on the temperature, the speed of liquid creep and the type of liquid hydrocarbon. For example, measured at 25°C and 264μm/s the advancing angle of HydroCal 300 is 36° while of IFO-120 is 54° [3].

The simplest technique for measuring the capillary rise is using a ruler with an optical reading, but its automation requires a camera, a mechanical device as lift or a liquid injector coupled with an additional light source to improve the optical reading of the meniscus position.

Because of its relative simplicity, the drop weight method, is one of the most common methods used for surface tension automated measurements. In this method drops formed at the tip of the glass capillary are weighted and counted. The pendant drop at the tip starts to detach when its weight reaches the value balancing the surface tension of the liquid. The advantage of the method is the possibility to measure surface tensions between liquids and other than optically transparent materials [13]. The disadvantage of this method is the necessity to calculate the corrections for the capillary tip parameters and the volume of the drop, which are characteristic of a given device.

The ring method and the Wilhelmy plate method are similar. In these methods an object is moved perpendicularly into or out of the liquid. The plate is moved towards the surface until the meniscus connects with it, or the submerged ring is pulled out of the liquid. The additional force acting on the plate or ring due to its wetting by liquid is measured.

In the maximum bubble pressure method an air or gas bubble is blown at constant rate through a capillary submerged in the tested liquid. The maximum measured

pressure that is required to insert the bubble of gas into the liquid may be used for surface tension determination.

The shape analyzing methods of liquid drop is based on the effect of the liquid's deformation caused by the gravitation force action. The surface area of a drop is proportional to its squared radius and the gravitational deformation depends on its volume, which is proportional to the third power of the radius [14].

One of dynamic methods for surface tension determination is based on the analysis of the shape of an oscillating liquid jet. The jet flows out from an elliptic orifice and therefore it oscillates. Mathematical analysis of such a jet was given for the first time by Lord Rayleigh in 1879. One can conclude from these considerations that the surface tension may directly affect fuel injection characteristics.

B. Sensors for diesel fuel testing

Nowadays, optical sensors are introduced into the fuel testing market. Some known optical sensors enable the examination of a set of fuel parameters, but the possibility of easy measurement comes with a high price of the devices [15]. On the other hand, some proposed multiparametric sensors may be low cost, but using them requires trained operating personnel. The possibility of diesel fuel quality testing using an optoelectronic set-up implementing a multi-parametric method was shown in [16] [17].

II. IDEA OF MULTIPARAMETRIC SENSOR FOR DIESEL FUEL TESTING

The idea of the sensor was inspired by the need to come up with a measurement method of a set of diesel fuel parameters in one system that would require the minimum of automated mechanical elements. The aimed at set of diesel fuel parameters includes: density, viscosity, surface tension and wetting represented by the contact angle between fuel and glass. Such a set of parameters may be observed using the classical capillary rise method (Figure 1) with three capillaries of different inner diameters. The data obtained in the measurement enable solving of a three equation system derived from (1). To obtain the additional viscosity measurement data one needs to measure the capillary rise speed. An increase of the liquid movement distance in the capillary to improve the precision of the measurement is possible by the inclination of the capillary. When capillary's axis is inclined at the α angle to the horizontal, a liquid is drawn in by capillary forces according to equation (2).

$$\frac{dl}{dt} = \frac{\frac{2\cdot\gamma\cdot cos(\theta)}{r}-g\cdot l\cdot\rho_l\cdot sin(\alpha)}{8\cdot\eta\cdot l}\cdot r^2, \qquad (2)$$

where: γ is the surface tension, θ is the contact angle, r is the inner radius of the capillary tube, g is the gravity constant, l is the length coordinate of meniscus at capillary axis, t is the time of measurement, α is the angle of inclination, ρ_l is the density of liquid, and η is its viscosity. Eq. 2 may be solved for the sensor application in two ways.

The first solution is for local speed measurement at a set length coordinate of meniscus with the formula:

$$\frac{\Delta l_s}{\Delta t_s} = \frac{\frac{2\cdot\gamma\cdot cos(\theta)}{r}-g\cdot l_s\cdot\rho_l\cdot sin(\alpha)}{8\cdot\eta\cdot l_s}r^2, \qquad (3)$$

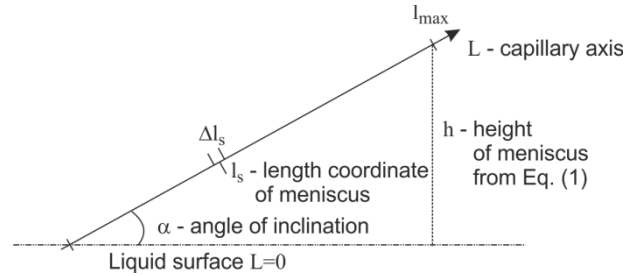where: l_s and Δl_s are presented in Fig 2.

Figure 2.   Parameters reading of local speed determination for (3).

The second solution of (2) is based on integration:

$$t_2 - t_1 = \int_{l_1}^{l_2} \frac{8\cdot\eta\cdot l}{r^2\left[\frac{2\cdot\gamma\cdot cos(\theta)}{r}-g\cdot l\cdot\rho_l\cdot sin(\alpha)\right]}dl, \qquad (4)$$

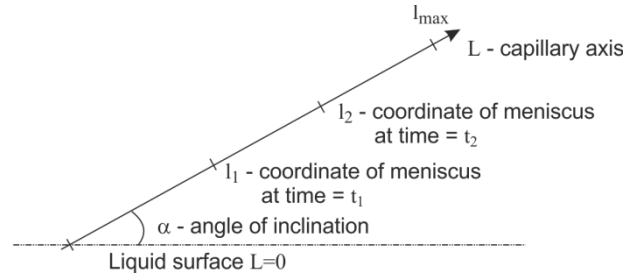where: l_1 and l_2 are presented in Figure 3.

Figure 3.   Parameters reading for integral solving of (3).

For the sensor application, (4) may be solved numerically by inputting in the measured time of rise t_2-t_1 at corresponding coordinates of meniscus.

III. SENSOR CONSTRUCTION

A. Sensor head

The sensor's head consists of two functional blocks: the base and the optrode, shown on Figure 4.
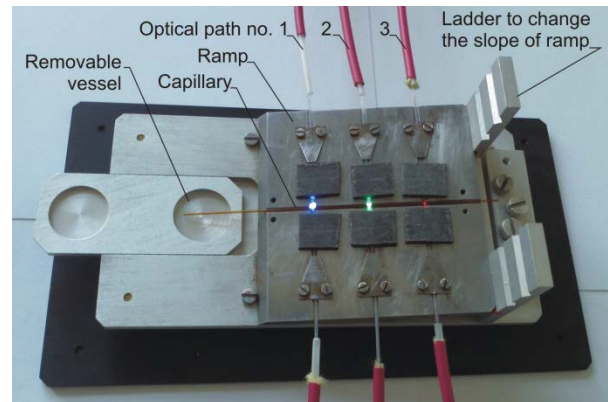
Figure 4.   View of the ramp sensor head.

The base is used to integrate the removable vessel for the examined liquid, the three optical paths of the source and the receiver, as well as for positioning the optrode at one of the three possible inclination angles.
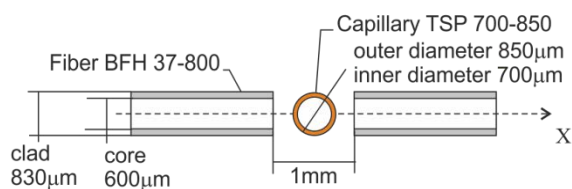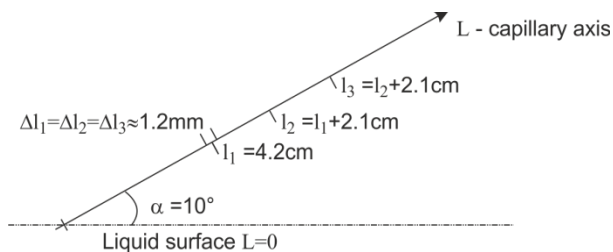

Figure 5.   Optical path geometry.

Figure 6.   Ramp geometry

The replaceable optrode is made from 15cm sections cut from the TSP 700-850 capillary from Polymicro Inc. In the optical paths large core optical fibers BFH 37-800 from Thorlabs were used. They are characterized by the core radius of 800µm and the hard clad outer diameter of 830µm, which is similar to the capillary's outer diameter. The tips of the fibers are positioned at the distance of 1mm, where in the middle of the distance between the tips is positioned the capillary (Figure 5). The geometry of the optical path provides the value of Δls, required in equation (3). As the optical paths are the same, Δls=Δl1=Δl2=Δl3$\approx$1.2mm, the experiment ramp geometry may be presented as in Figure 6.

B.   Optoelectronic signal processing

As light source, three fiber coupled LEDs with three different emission wavelengths were used. The lowest-power LEDs were selected from Thorlabs list of high power devices: M490F2, blue with the 490 nm wavelength; M565F1, green/yellow - 565 nm wavelength; and M625F1, red - 625 nm wavelength. The diodes were connected to DC2100 controllers operating in the light modulation mode. The receiving fibers were inserted into the optoelectronic detection unit of our own design, presented previously in [18]. The optoelectronic unit was connected to a PC by an analog input of IOtech Personal daq 3000 data acquisition system. That system was also used to monitor the ambient temperature with two LM35DT circuits and control it with a radiant heater at 25°C.

To operate the system, at the 0.1s sampling rate, a script in DasyLab 10 was designed [19]. The raw data collected for acetone is presented in shown on Figure 7. The initial values of signals for different paths are off set on Figure 7 for clarity of visualization. Moreover, the differences of the initial values do not matter, as the sensor operation is based on the differences of the measured time values. The time differences presented for acetone are in agreement with (3)

$\Delta t_1 < \Delta t_2 < \Delta t_3$, and time intervals are in agreement with (4) [$(t_2 - t_1)$=0.9s]<[$(t_3 - t_2)$=2.6s]. The t_1 measurement value is uncertain, as the filling of the vessel may not be repeatable when a hand held pipetor is used.
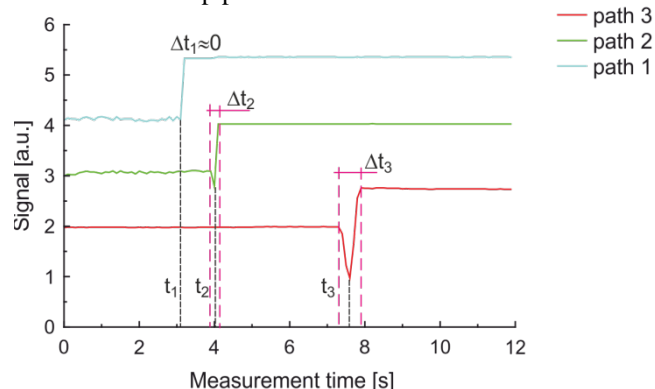

Figure 7.   Acetone characteristics.

IV.   EXPERIMENTAL RESULTS

In this section are presented the experimental procedure and the results of examination of different diesel and biodiesel fuels.

A.   Diesel and biodiesel fuels used for examination

The operation of the sensor was examined with fuels that are mixtures prepared from components according to the European Union standards. The fuels were fresh or stored in room condition for two years.

Base oil (BO) was prepared from crude oil distillation products. The petrodiesel (PD) was prepared with additives according to the EN-590 norm. Biodiesel fuels (BDx – x is the volume ratio of the biocomponent) were prepared from PD with addition of fatty acids methyl esters (FAME) and other additives according to the EU standard. The results of classical laboratory examination of prepared fuels are presented in Tab. 1. Density measurement accuracy was ±2.0 kg/m^3 while resolution was ±0.1 kg/m^3. Viscosity measurement accuracy was ±1 %.

TABLE I.   SELECTED PARAMETERS OF PREPARED FUELS

Fuel acronym	Parameter			
	FAME [%]	Density at 15ºC [kg/m^3]	Kinematic viscosity at 40ºC [mm^2/s]	CN
BO	0	805.0	1.581	49.8
PD	0	832.6	3.367	59.6
BD02	2	833.6	3.3825	58.4
BD04	4	834.5	3.394	58.3
BD06	5.8	835.4	3.401	58.6
BD08	7.8	836.4	3.413	57.3
BD10	9.7	837.4	3.432	57.3
BD30	28.8	847.0	3.595	54.9
BD50	48.9	857.4	3.813	53.6
BD70	68.6	867.3	4.058	53.7
BD100	100	883.2	4.509	51.2
EN-590*	0-7	820-845	2-4.5	>51

Abbreviations used: FAME – Fatty acids methyl esters (bio-component), CN – cetane number, EN-590 – diesel fuel standard.

The fuels BO, BD30 BD50, BD70 and BD100 do not meet the standards of density, viscosity and FAME ratio, but meet the quality test of CN.
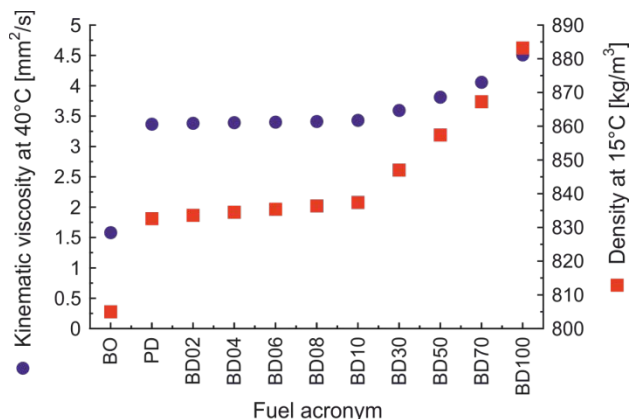

Figure 8. Viscosity and density of examined fresh fuels. ± 0.01 lb/ft3

The densities and the viscosities of the examined fuels change almost monotonically with their content of FAMEs, as presented in Figure 8.

B. Examination of biodiesel fuels using the developed sensor

We examined at least 3 times samples of each fuel, and on the following figures we present representative measurement signals. The results of measurement of fresh PD presented in Figure 9 show that, contrary to acetone, the Δt_s are measurable in each optical patch $\Delta t_1=0.2s$, $\Delta t_2=0.3s$, $\Delta t_3=0.7s$, and that the time intervals $(t_2-t_1)=6.5s$ and $(t_3-t_2)=13s$ are greater than for acetone, as fuel viscosity is much greater than that of acetone.
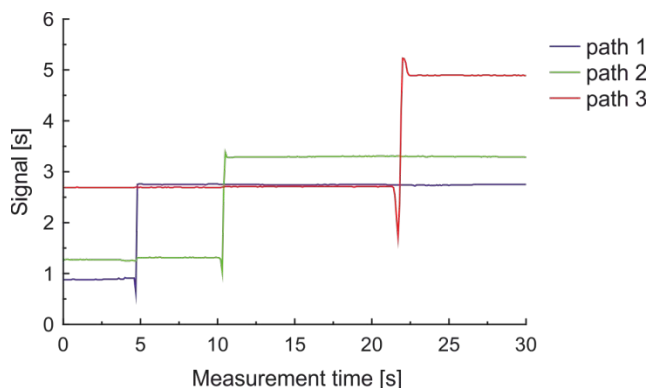

Figure 9. Measurement signals of fresh premium quality petrodiesel PD.

From our experimental results we saw that fresh BO, BD70 and BD100 fuels differ significantly from other fuels when the t3-t2 parameter is considered, as is shown on Figure 10. The difference in (t3-t2) times from one fuel to another varies much greater than the fuels densities or viscosities. Calculated values of surface tension of fuels, with the assumption that contact angle is 0°, are presented in Figure 11. These results show that the values of surface tensions are probable and that the advancing contact angles differ significantly for the analyzed fuels.

The next test performs the analysis of time interval values of fuels stored in a closed tank for two years in dark room conditions. The volumes of fuels seem to be constant, no presence of resins was observed, but the fuels seem to be more transparent. The results presented in Figure 12 in comparison to the results seen in Figure 10 indicate that the measured times intervals increase for stored fuels, but not monotonically.
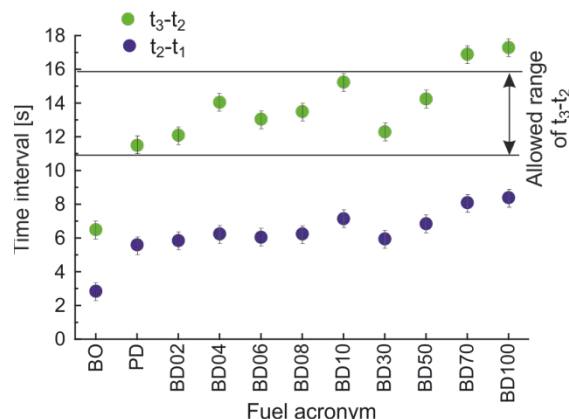

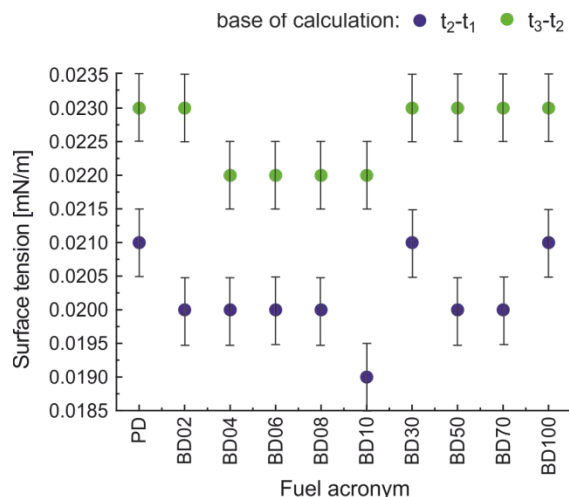Figure 10. Measurement data of fresh diesel fuels for solving (4).

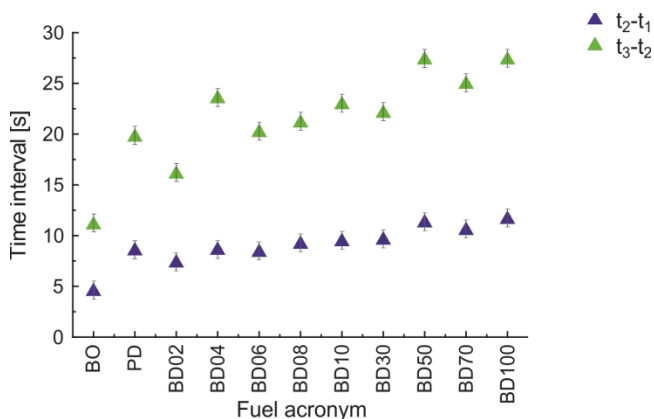Figure 11. Calculated surface tensions of fresh fuels.

Figure 12. Measurement data of diesel fuels stored for 2 years.

The results of examination of time differences Δt_3 for fresh and stored fuels are presented in Figure 13. The summarized results of the experiments led us to the conclusion that Δt_3 time measurement data differences and t_3-t_2 time intervals relate more clearly to the acceptable quality fuels than the set of surface tension, contact angle, viscosity and density data. On the base of data collected in the experiments we can set the parameters determining the useful state of biodiesel fuel as the limits of the time intervals t_3-t_2 and the limits of time differences Δt_3.
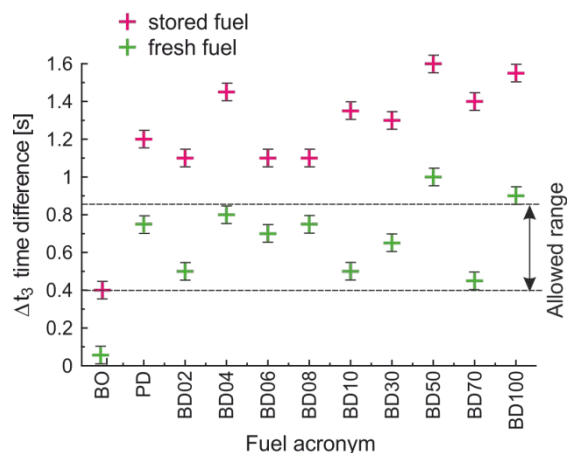

Figure 13. Measured time differences Δt_3 for fresh and stored fuels.

The analysis of data showed that in the presented method the useful state of diesel biodiesel fuel was not determined as precisely as presented in [20], but the construction of the head is much simpler and its use is easier.

V. CONCLUSIONS

We proposed a sensor working on the principle of optical examination of diesel fuel with capillary rise. The presented device has been tested with ground truth data and it shows great promise.

The analysis of the measured signals of diesel and biodiesel fuels showed the relationship of the times of fuel flow in the capillary with the useful state of diesel fuels. We showed that the information on useful state of diesel fuel could be presented in the form of recommended ranges of times of the fuel crossing sections of an inclined capillary during the capillary fill. It is not as accurate as other methods yet, but its intrinsic advantages of simplicity of design and cheap manufacture could make the technology viable in the near future.

The future work will consist of integration of the LEDs and photodiodes in a common unit with the ramp.

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