

## Sensing Method and Fiber Optic Capillary Sensor for Testing the Quality of Biodiesel Fuel

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**Abstract:** There are many fuel quality standards introduced by national organizations and fuel producers. Usual techniques for measuring the quality of fuel, as for example cetane index, fraction composition and flash point, require relatively complex and expensive laboratory equipment. Therefore, testing of fuel is not rapid and can be costly. On the fuel user side, fast and low cost sensing of useful state biodiesel fuel is important. For this purpose, we have investigated the sensing method and sensor head that could be cheap in instrumentation as well as in fuel examination, and lead itself to automation. The method presented in this paper is based on fiber optic capillaries with local heating. We have investigated the construction of the sensor that imitates the fuel injection process and of the local heating element, the two critical elements for biodiesel fuel testing. We propose a new capillary optrode construction that enables measuring of time of vapor phase creation. We examine fuels that are mixtures of characterized components of petrodiesel fuel and bio-esters as well as edible rapeseed oil. We show that useful state of biodiesel fuel can be determined from the time of local heating that is required for vapor phase creation and the local time of vapor bubble formation.

**Keywords:** *biodiesel fuel, fuel quality, useful state of fuel, fiber optic capillaries, fiber optic sensors, capillary sensors.*

### I. INTRODUCTION

Nowadays, the useful state of diesel fuel is defined by producers by several parameters: cetane number (min 51.0), density (860 to 890 kg/m<sup>3</sup>), and distillation temperature T<sub>90</sub> (max. 360°C), kinematic viscosity at 40°C (3.5 to 5.0 mm<sup>2</sup>/s), etc. Other diesel fuel parameters characterize its operability: carbon residue, water and sediment, cloud point, conductivity at 20°C, oxidation stability, acidity, copper corrosion, flashpoint, lubricity, appearance, and color [1]. For the ordinary fuel user such collection of parameters is often too complex for practical use because it requires special laboratory equipment. Therefore, fuel examination is not rapid and can be costly. Moreover, the introduction of biodiesel fuel increases the number of parameters connected

with bio component content. In this situation the user requires the simplest possible answer to a question: Is that fuel useful for my engine?

Sensing of useful state of biodiesel fuel is exceptionally important for car fleet owners and farmers. Car fleet owners are interested because of legal regulations and of the possibility of buying poor quality fuel. Farmers are interested because they can produce bio-fuel components for their own use. But the parameters of these components in pure form are not optimal. For example, rapeseed and canola oil have too small cetane numbers and too high viscosities. However, the viscosities of oils decrease with the increase of temperature. For these reasons, in tropical countries the potential of using biodiesel fuels is larger. The use of mixtures of lychee fruit oil with petrodiesel fuel with component shares of 10%, 20%, 30% and 40% are discussed in [2]. It turned out that, despite significant differences in fuel viscosity and flash point performance the observed engine parameters with the prepared mixtures were very similar [3].

In a European study, it was observed that using first generation of biodiesel fuel at low environment temperatures can lead to degeneration of engine parameters [4]. Therefore, production standards for biodiesel fuel were introduced: density at 15°C (ISO3675) and temperature of fluidity for the transitional periods of season and winter (DIN EN 116). The disadvantages of biodiesel fuel can be overcome by fuel processing [5-8] or by using biopetrodiesel fuel mixtures [9].

One of the reasons of low biodiesel fuel mixtures usage by farmers is the absence of low cost device to evaluate its useful state.

Our starting point was to consider the critical points of fuel conversion into energy. The first is the injector of atomized fuel into the combustion chamber by forcibly pumping it through a small nozzle. The second critical point is the exhaust of gases filtered with the diesel particulate filter (DPF). Periodically, the DPF has to be taken up to high temperatures to burn off the matter it has collected [10], which is realized by contact of DPF with a part of fuel vapor, [11].

Typically, fuel is injected into the cylinders just after the vapor fires and the exhaust valve opens. At injection point, the fuel vaporizes and a part of vapor moves down the exhaust to the DPF and cleans it in a precisely controlled injection scheme [12]. Because biodiesel fuel has a higher distillation temperature than petrodiesel fuel, it does not vaporize as fast. Some of the biodiesel fuel can end up adhering to the injector, the cylinder wall or runs past the rings, diluting the engine oil and diluting DPF deposits instead of cleaning it.

Therefore, the examination of vapor creation parameters of biodiesel fuels is critical to evaluate its useful state regardless of the composition of fuel. The methods of spray forming observation in diesel engine have been used [13], but are not good for integration into a sensor device. In this work we present new developments and new applications of on capillary photonic sensors working on the principle of monitoring optical intensity changes in dynamically forced measurement cycles, first postulated in [14]. The sensors use fiber optic capillaries in which the phase of the filling liquid changes locally to gas when forced by local heating, while the propagation of light in the capillary is monitored. Therefore, the sensors examine simultaneously many liquid parameters.

In this paper are presented the idea of the sensor head, the construction of the head, the experimental results of testing biodiesel fuels for their quality for use, and conclusions.

## II. IDEA OF SENSOR HEAD

We intend to imitate and examine fuel vaporization in conditions that are close to reality. The fuel injector nozzle diameters are from 50 to 200 $\mu\text{m}$ , [15]. Typical temperatures inside the fuel injection nozzle are from 235 to 275 $^{\circ}\text{C}$ , the maximum not exceeding 300 $^{\circ}\text{C}$  (see Fig. 1) [16]. Since the flame temperatures in the cylinders are about 1500 $^{\circ}\text{C}$  and the wall temperatures are under 350 $^{\circ}\text{C}$ , we can't replicate the flame temperatures in the sensor device. We have to create a set-up allowing the examination of partial evaporation of fuel which take place in the nozzle and can move fuel into orifice of few hundreds micrometers diameter. Such nozzle can be modeled with two glass capillaries that would allow observation of the direct optical fuel phases and their movement. The capillary with smaller outer diameter can be positioned inside the bigger capillary using glue forming a single-use replaceable optrode [17]. The inner temperature that is needed to create the bubble of vapor can be achieved with a local heater positioned near the capillary. With one end of capillary closed, the local heater can acts as a fuel pump by producing a vapor pressure (see Fig. 2). The creation and movement of the bubble depends on the type of liquid and vapor parameters as well as on the geometry and thermo dynamical conditions.

The faster is the bubble creation from liquid phase, the more probable is the turbulent flow of fuel in the nozzle. Therefore, we have to distinguish two stages of the bubble creation: the time of liquid fuel heating and the time of phase change from liquid to vapor that forms the bubble filling the full cross section of the capillary.

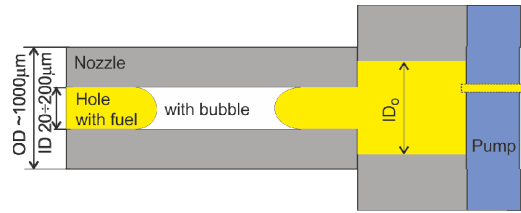


Figure 1. Schematic construction of the nozzle.

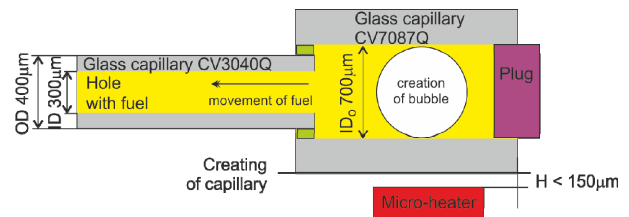


Figure 2. Schematic construction of model of the nozzle.

## III. HEAD CONSTRUCTION

The sensor's head consists of two functional blocks: the base and the optrode [18]. The base is used to integrate the microheater, the optical path and for positioning the optrode. The optrode is the replaceable part of the head that imitates the fuel nozzle and enables monitoring of creation of the vapor bubble.

### A. Micro heater

The microheater has to supply sufficient heat for the biodiesel fuel to reach 300 $^{\circ}\text{C}$ . We examined experimentally and numerically the map of temperatures in the model of nozzle. We used Coventor software, a R300 NEC thermovision camera, and InfReC analyzer software. The results for a 4mm $\times$ 4mm planar micro heater positioned at 50 $\mu\text{m}$  under the capillary and dissipating 5W in 30 seconds are presented in Fig. 3. The temperature of surface reached 327 $^{\circ}\text{C}$  while the temperature inside the capillary reached 247 $^{\circ}\text{C}$ . Sequential simulation showed that the microheater temperature has to be at least 350 $^{\circ}\text{C}$  for the assumed distance between the capillary and microheater surface, which is more than can withstand the planar resistors e.g., Vishay High Power Thin Film Wraparound Chip Resistor in 2512 packaging [19].

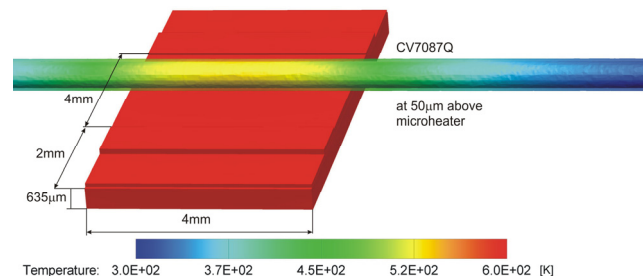


Figure 3. Temperature map in [K] at 30s of heating for a glass capillary CV7087Q filled with diesel fuel.

Wire heaters can easily work at such temperatures, but such constructions does not provide a constant and repeatable distance between the microheater and the capillary, and they can't replace the planar structures. The most favorable shape of planar microheaters is rectangular with side length from 2mm to 4mm, and the recommended power of heating is 5÷7W. The power density is  $1.75\text{W}/\text{mm}^2$ , which is also too high for classical hybrid resistors. For the heater current supply the recommended value of resistance is between  $10\div 50\Omega$  even current dividers from Vishay Current Sensing Bondable Chip Resistors type S.C. [19], are not optimal for the application. We have built different versions of planar microheaters using hybrid technology. With an optimized technology, the parameters of the microheater were stable for temperature shocks from  $30^\circ\text{C}$  to  $200^\circ\text{C}$  – the resistance changes were low, within  $1.5\Omega$  at  $30\Omega$  of nominal resistance. The microheater reached  $350^\circ\text{C}$  after 30 seconds, dissipating 6W of power, but it should not be powered for more than 60 seconds, because of the possibility of breaking into two symmetrical parts. The next heating cycle with maximum power was safe, when the heater was allowed to cool down to room temperature. In normal condition it required about 2 minutes.

**B. Path of optical signal**

The bubble creation can be observed from outside or inside of capillary with the use of optical fibers [20]. The bubble position can vary in the area of local heating due to variation of fuel composition. Therefore, the observation from outside is not optimal for measuring the bubble creation time. Observation of the bubble creation with two fibers inside the capillary is not optimal for a replaceable optrode set-up, and also complicates the fuel flow. To overcome those problems, we used a modified capillary optrode with a phosphor layer to convert radiation (see Fig. 4). In the presented optrode, the phosphor converts the light from 460nm wavelength of the high power light emitting diodes, to 562nm. Only part of the light radiated in the full angle extent propagates in the inner capillary to the area of examination. The efficiency of light conversion is low.

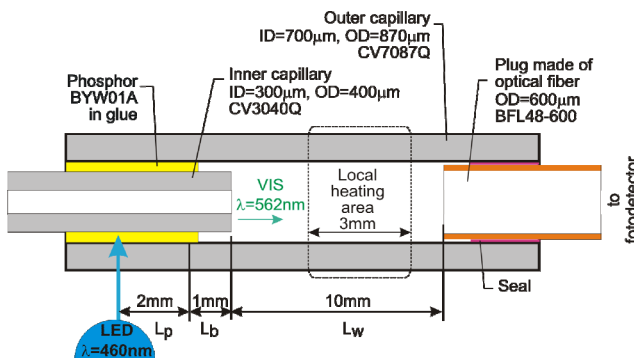


Figure 4. Optrode that uses phosphor to convert outer radiation into light inside capillary.

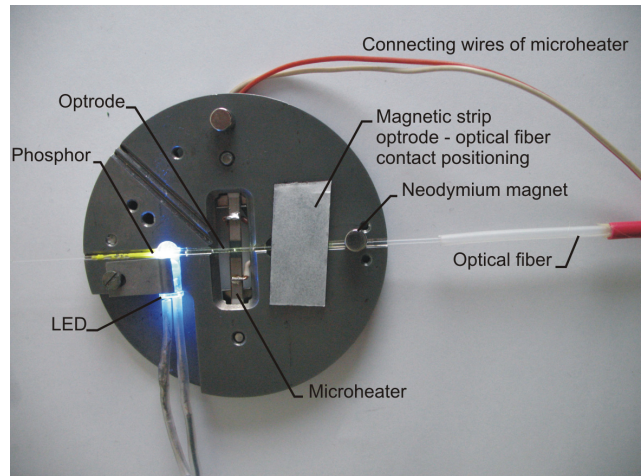


Figure 5. The head construction.

After optimizing the construction, from a L7113QBC-G LED operating at 20mW, we got at the end of the plug made from optical fiber, 111nW for an empty capillary and  $0.3\mu\text{W}$  for a capillary filled with biodiesel fuel. The uncertainty of low signal level in our construction was 10nW.

We optimized the optrode elements position:  $L_p$ ,  $L_b$  and  $L_w$ , as well as the method and parameters of phosphor deposition. The optrode was held in position with elastic magnetic strips, while the optical fiber was secured with miniature neodymium magnets. The head construction is presented in Fig. 5.

**C. Optoelectronic signal processing**

As light source driver we built an electronic device that enabled current modulation from DC to 50 kHz at selected frequencies, and was equipped with configurable current limiters to prevent accidental LED burning.

The optoelectronic detection unit of our own construction had an SMA fiber input and consisted of an integrated photo-amplifier and a band-pass filter with amplification and RMS detection. We used the S8745-01, AD8253, UAF42, AD536 and AD8250 components. The optoelectronic unit was connected to a personal computer through an analog input IOtech personal Daq 3000 16bit/1MHz USB data acquisition system. We fed the heater from a laboratory power supply Hameg HM8143 controlled by the analog output from Daq. The view of sensor hardware set-up is presented in Fig. 6.

We also used a Daq 3000 system to monitor the temperatures of the measuring head base and of the surrounding ambient with two LM35DT circuits connected by low pass filters. To operate the system, we designed a script in DASYLab with a 0.01s sampling rate. The script automates the measurements and automatically switched off the heater when the light signal dropped under a specified value corresponding to the point of vapor bubble creation. The script was programmed also to switch off the heater when the maximum heating time was reached, but the bubble did not form. The length of signal registration was 60s.

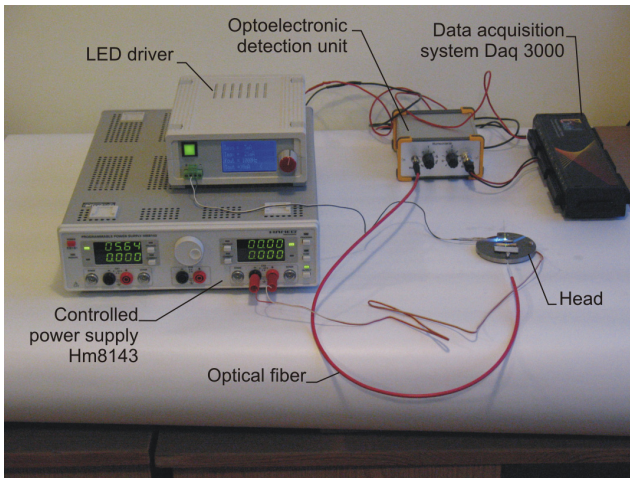


Figure 6. View of experimental set-up

IV. EXPERIMENT RESULTS

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

A. Experiment procedure

At the start of the experiment the part of the optrode consisting of the CV7087Q capillary was filled with fuel, after which its end was closed. When there were bubbles of gas observed at the initial state of experiment, the optrode and capillary had to be withdrawn [14]. When the capillaries were filled uniformly by the liquid, the initial levels of transmitted signals were measured and used as normalization levels. We normalized the initial signal level to 4 a.u.

As the fuel in the useful state is semitransparent, we expected initially high signal levels and low signal levels when the bubble would appear. The bubble directed the signal from the liquid to the capillary walls. When the transmitted signal decreased rapidly it gave the impulse to switch off the microheater. We terminated the heating when signal dropped under 2.5 a.u. Depending on the thermodynamical conditions; the vapor gas phase moved the fuel to the open end with a laminar or a turbid flow. The turbid flow could be detected optically after the experiment as a presence of series of small bubbles in the CV3040Q capillary. We also observed a repeatable presence of a small bubble that remained after heating in the center of the microheater, Fig. 7.

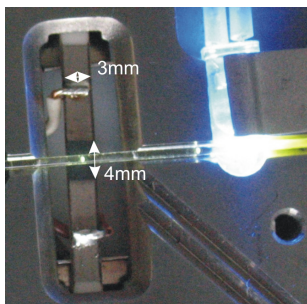


Figure 7. Small bubble remaining after heating in the center of the microheater

We thought that bubble appeared due to structural changes in fuel induced by heating of the components that were added after distillation and some bio-components decomposition, since the bubble was present also after examination of rapeseed oil.

B. Examination of biodiesel fuels

We examined 5 fuel mixtures prepared from the same components at different ratios, the commercial 100% biodiesel fuel, as well as edible rapeseed oil (RO). Selected parameters of prepared fuels are grouped in Table 1. The distillation of RO and its parameters can be found in [9].

TABLE I. SELECTED PARAMETERS OF PREPARED FUELS

Parameter	Fuel acronym				
	P2	P12	P14	P17	P21
Base oil [%]	100	90	70	40	0
FAME [%]	0	10	30	60	100
Density at 15°C [kg/m³]	832.6	837.4	847.0	862.3	883.2
Temp of flame [°C]	74	75.5	79.5	90	163
Kinematic viscosity at 40°C [mm²/s]	3.367	3.432	3.595	3.934	4.509
CI	54.9	57.7	57.5	56.8	*
CN	59.6	57.3	54.9	54.0	51.2
T <sub>0</sub> [°C]	188.6	195.6	196.7	200.2	*
T <sub>10</sub> [°C]	225.7	230.4	242.1	278.1	*
T <sub>90</sub> [°C]	345.5	343.6	344.1	345.3	*

Abbreviations used: FAME – Fatty acids methyl esters (bio-component); CI – cetane index, CN – cetane number, T<sub>0</sub> temperature of distillation start, T<sub>x</sub> – temperature of x% volume of distillation, \* - our lab equipment do to allow of such examination.

The laboratory fuel examination prior to the experiment showed that fuels P2-P17 were meeting the norms. P21 did not meet the distillation standards. We made first experiments with P2 fuel with two powers of heating 4W and 6W, (see Fig. 8 and Fig. 9). The examination results showed that increasing the power from 4W to 6W reduced the average time of heating  $\tau$  from 14.5 seconds to 9 seconds. The differences in time of heating were in agreement with the thermo dynamical properties of the evaluated mixtures.

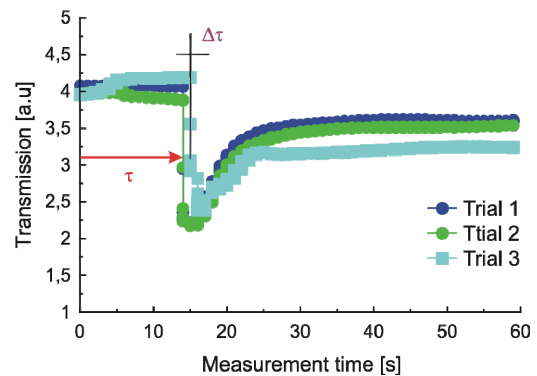


Figure 8. Measurement procedure signals of P2 heated with 4W.

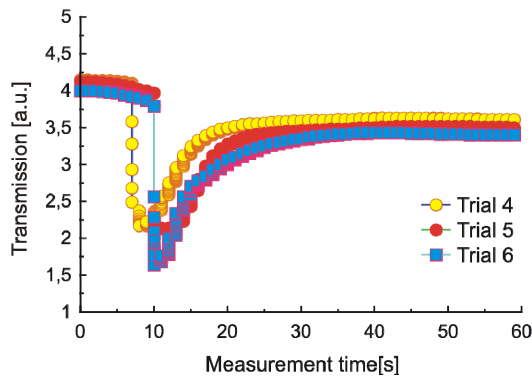


Figure 9. Measurement procedure signals of P2 heated with 6W.

We also observed low values of the times of bubble creation  $\Delta\tau$ , decreasing from 0.2 seconds for 4W, to 0.1 seconds for 6W. The achieved results were agreement with expectation. The next experiments were made with 6W heating power and their results are presented in Fig. 10 to Fig. 14 and summarized in Table 2.

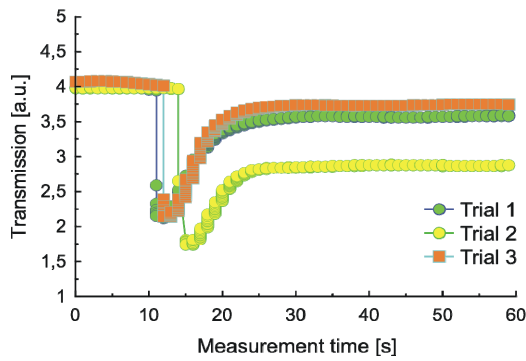


Figure 10. Measurement procedure signals of P12 heated with 6W.

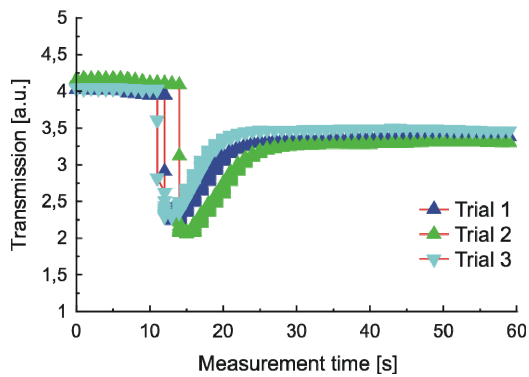


Figure 11. Measurement procedure signals of P14 heated with 6W.

TABLE II. EXAMINED PARAMETERS OF FUELS HEATED WITH 6W

Parameter	Fuel acronym					
	P2	P12	P14	P17	P21	RO
Average $\tau$ [s]	9	12.5	13	21.6	22*	13
Average $\Delta\tau$ [s]	0.10	0.13	0.17	0.30	0.2*	0.6
Percent of samples with created bubble	100	100	100	100	33	100

\*- no existing the average value, RO - rapeseed edible oil from supermarket

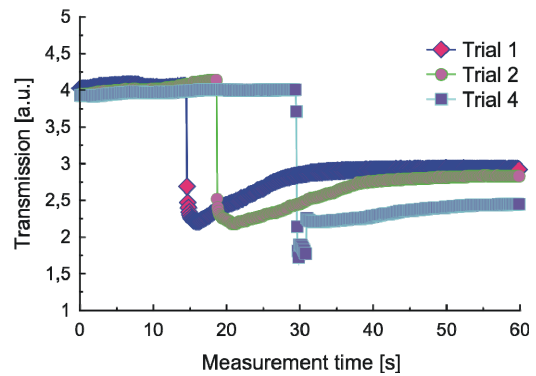


Figure 12. Measurement procedure signals of P17 heated with 6W.

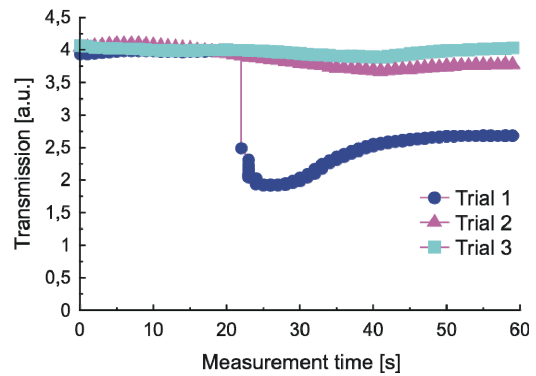


Figure 13. Measurement procedure signals of P21 heated with 6W.

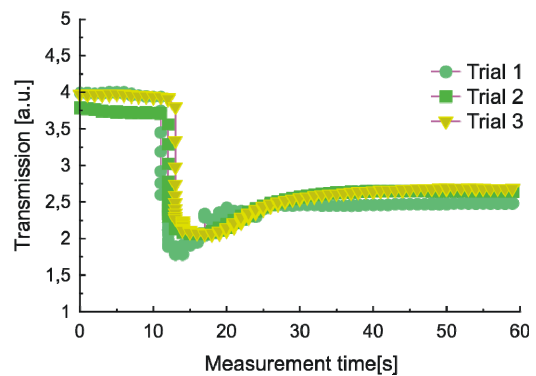


Figure 14. Measurement procedure signals of RO heated with 6W.

From our experiment results we saw that P2, P12 and P14 fuels did not differ significantly. The P17 fuel formed in our heating condition a vapor phase, but the mixture was characterized by very high dispersion of time of heating. Interestingly, the P21 seemed to be a worse fuel than RO, because it required a longer time of heating while occasionally showed a lower time of bubble creation. More over the RO had the lowest  $\tau$  dispersion, in agreement with its distillation parameters that were close to its boiling temperature, which is not a good property for a fuel.

Therefore, we may set for the parameters of useful state of biodiesel fuel the upper limits of average time of heating, the range of dispersion of time of heating and the upper limit of time of vapor phase creation. The data analysis showed

that in our method the useful state of biodiesel fuel was directly and firmly connected with the gas phase creation.

#### V. CONCLUSIONS

We proposed a sensor working on the principle optical examination of fuel under local heating. Our optoelectronic devices enabled conducting the experiment in lighting room conditions. The results of the measured signals analysis of biodiesel fuels showed the relationship of times of gas phase creation parameters with the useful state of fuel. We showed that the information on useful state of diesel fuel as well as biodiesel fuel could be presented in the form of recommended ranges and times of fuel heating and vapor creation. Because the heating was taking place in a closed capillary, the fuel did not ignite during experiments. We conclude that the proposed construction may be in future the base of commercially marketable instruments.

The future work will consist of optimization of the construction and of the data processing function. The sensor construction needs to be integrated into a complete portable instrument and be built more resistant for use in harsh environments outside of the laboratory.

#### ACKNOWLEDGMENT

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