



Optical Analyzer for Continuous Monitoring of Dissolved Oxygen in Aviation Fuel and Other Non-aqueous Media

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ABSTRACT

A special sensitive element based on the novel composite material was created. The sensor has a linear calibration and is resistant to aliphatic hydrocarbons, in particular aviation fuel. A low cost and easy-to-implement method for calibrating the sensor was proposed. Temperature dependence of the oxygen mass transfer coefficient k_L was measured for aviation fuel TS-1 (Russian equivalent of Jet A-1) in the temperature range of 10 to 40 °C as a demonstration of the sensor capabilities. The dependence found obeys the Arrhenius equation with the parameters $E_A = 21.7 \pm 1.5$ kJ/mol, $k_{L0} = 1080 \pm 90$ m·h⁻¹. The resistance to mechanical action is one of the important advantages over the sensor made of a sol-gel matrix with a protective coating. Damage to some part of the surface does not change the properties of the entire sensor, since the composite material contains a large number of isolated particles with a dye.

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1. INTRODUCTION

At present, it becomes increasingly important to control the dissolved oxygen (DO) content in non-aqueous organic media. For example, dissolved oxygen may lead to jet fuel deposition and coking in the conditions of an aircraft engine. This process clogs fuel pipes and leads to the need for periodic labor-intensive maintenance or even failure of the unit [1-4]. On the other hand, ethanol-blended gasoline can produce lower levels of CO and HC than unleaded gasoline on SI engines, due to the higher oxygen content [5]. In addition, a large number of technological processes, including biotechnological, are associated with the use of organic solvents or complex media, and requires a method for continuous control of the DO content [6-7].

However, the practical implementation of measurements in such environments is a complex task. Titration, manometric determination, photochemical methods, ESR and chromatography are among the

approaches tested previously [8], but none of them can provide continuous measurement. This type of operation could be provided by the amperometric Clark sensor, but only a few papers report its use in nonpolar organic solvents or alcohol solutions [9]. The main problem of this sensor type is that the membrane used in the Clark electrode is usually made of a polymer that swells when exposed to organic solvents, which leads to errors in determination of the DO. Relatively recently, it was reported on the use of optical oxygen sensors [10-11]. The principle of the measurement is based on the indicator dye phosphorescence quenching by molecular oxygen.

The sensing element represents a dye distributed in a gas permeable matrix. The matrix may be made of a mesoporous structure obtained by the sol-gel process, silicone rubber, polystyrene or other polymers. In this case, unlike the amperometric Clark sensor, the optical sensor is not poisoned by hydrogen sulfide and other sulfur-containing compounds, that is extremely important for measurements, for example, in aviation fuel Jet A-1 (or its Russian equivalent - TS-1) derived

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from the sulfur-containing oil. Sustainability of the sensing element to the pressure and the ability to measure through a transparent porthole should be mentioned among the other advantages of the method. However, most of the existing optical sensors are also not suitable for measurements in organic solvents due to swelling or dissolution of the polymer matrix material.

Various types of resistant to organic solvents sensors were presented [12-14], but all the sensing elements used have non-linear calibration dependencies that complicate practical operation.

In this paper we used a composite material combining good oxygen permeability of fluorinated polymers [15] with linear calibration dependence, which is exhibited by mesoporous materials [16].

2. EXPERIMENTAL

2.1. Equipment and Reagents The experimental setup is shown in Figure 1. Measurements of the dye excited state lifetimes were performed using a commercially available analyzer with an optical sensor "Expert-009" (Econix-Expert Ltd, RF) connected to a personal computer. The device design and operation principles were described previously [17]. The specialized proprietary software was used for automated data collection and processing. Liquid thermostat of model TJ modification TS-01(100) was used for temperature control, the temperature control range from 0 to 100 °C with an accuracy of the temperature maintenance ± 0.1 °C. It was connected with a glass jacketed cell (nominal volume 250 cm³) equipped with a sealed cover with a fixed oxygen sensor, gas purging system and a liquid inlet port. The temperature in the measuring cell was monitored by means of a digital thermometer TCM-9410/M2, the measurement range from -50 to +200 °C, error $\pm(0.05\div 0.15)$ °C. Oxygen removal was carried out with high purity nitrogen gas (99.9995% purity) obtained from NII KM (www.niikm.ru). The atmospheric pressure was measured with an aneroid barometer M-67, the measurement range from 80 to 106.67 kPa, an error of ± 0.11 kPa.

Iodometric titration was carried out on the automatic titrator "Titron" (Econix-Expert Ltd, RF). The platinum electrode ERP-103 (www.izmteh.ru) was used as the working electrode, a special chloride-silver electrode ESN-1 for non-aqueous media based on the standard electrode EVL-1M4 (www.zipgomel.by/en/) was used as a reference electrode.

The oxygen sensitive layer was made of a composite material, which consists of amorphous fluorinated polymer with dispersed nanostructured microparticles with an adsorbed dye [18]. Pt (II) complex of 5,10,15,20- tetrakis (2, 3, 4, 5, 6- pentafluorophenyl)-

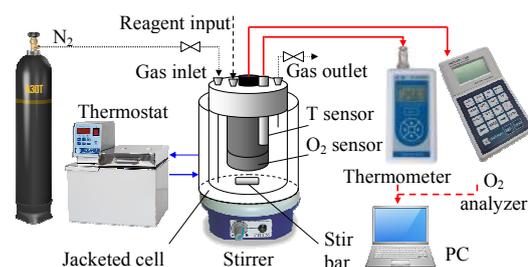


Figure 1. The experimental setup for the calibration dependence determination in non-aqueous media and for the measurement of the oxygen saturation kinetics

porphyrin (Pt-TFPP) was used as an indicator (Frontier Scientific, www.frontiersci.com). Merck silica gel 60 (pore size 52–73 Å, BET surface area 480–540 m²/g, particle size 40–63 μm) was used for sorption of the dye. Fluoroplastic 42 (F42, similar to Kynar 7200) was used as the carrier polymer (purchased from HaloPolymer, www.halopolymer.ru).

All solvents and other reagents were purchased from Chimmed (www.chimmed.ru/en/) and used without further purification. Aviation fuel TS-1 (national equivalent of Jet A-1) produced in accordance with GOST 10227-86 (national standard) was provided by Central Institute of Aviation Motors.

2.2. Sensor Fabrication A detailed study to determine the optimal conditions for the sensor fabrication was carried out earlier [19]. The sorption of the dye was carried out by mixing a known mass of dry microparticles with a solution of indicator in chloroform. The dye content of 5.5 μmol per gramm of SiO₂ was chosen as the optimal one. The mixture was kept in an ultrasonic bath for 20 minutes to ensure even distribution of the dye. The drying process was carried out on a rotary evaporator with bath temperature 50 °C, 80 rpm.

Then the particles were successively immersed in 0.1% aqueous solutions of cationic and (after drying) anionic surfactants – 3 [[heptadecafluorooctyl)sulfonyl] amino]-N,N,N-trimethyl iodide (FC-135) and tetraethylammonium perfluorooctanesulfonate (FT-248). This treatment ensures the formation of surface salts, thereby preventing the dye leaching at the next stage.

Further the microparticles with shell were added to the 10% polymer solution in acetone. The mixture was kept in an ultrasonic bath for 10 minutes to uniformly distribute the particles by volume. The mass ratio of polymer:microparticles equal to 5:1 provides longer sensor lifetime. A higher content of microparticles leads to partial crumbling after drying. The knife-coating device with 200 μm gap was used for applying suspensions on matted glass slides. The sensing layer after solvent evaporation was finally dried at 80°C overnight.

2. 3. Calibration of the Sensor in Aviation Fuel Using the Modified Winkler Method

We decided to apply a third-party method for determining the oxygen content in aviation fuel to establish the optical sensor calibration dependence. Currently ISO 5813:1983 [20] is the reference procedure and is applicable to all types of water having concentrations greater than 0.2 mg/l. We adapted it for non-aqueous media and automated the analysis procedure using the Titration titrator (Econix-Expert Ltd., RF) with a potentiometric measurement system.

We used a specialized experimental setup (Figure 1) to determine the calibration dependence of the optical sensor. The calibration procedure is as follows. The cell is filled with aviation fuel to the top, the exact volume of the sample is determined gravimetrically. Next, 1 cm³ of an alkaline solution (1 M) of potassium iodide (0.1 M) is added, and the cell is placed into a thermostat. The nitrogen purge begins after temperature stabilization and it continues until the certain lifetime of the dye excited state is reached according to the optical sensor readings. Then the oxygen fixation procedure is carried out by introducing 1 cm³ of the 2 M manganese sulfate solution. The cell is placed on a magnetic stirrer and the stirring is performed for 10 minutes in a dark place. Further the stirrer is switched off and the precipitate settles in the cell for 5 minutes. At the end of the time 5 cm³ of hydrochloric acid solution (25% by weight) are injected into the cell, and after a short stirring, the contents are transferred to the automatic titrator cell. Titration is carried out with a standard solution of sodium thiosulfate (0.010 mEq/dm³). Intensive turbulent stirring of the heterogeneous system is a necessary condition for obtaining reliable results. During titration the organic layer, initially colored in a dark orange color, becomes colorless, and the titration curve has a typical S-shape. The sequence of operations in the blank experiment is the same but the nitrogen purge continues for 30 minutes.

The mass concentration of oxygen in the analyzed sample is found by equation:

$$\chi = 8.0 \cdot c_T \cdot (V_T - V_B) \cdot 1000 / (V - V_1) \quad (1)$$

where χ – mass concentration of dissolved oxygen in the analyzed sample, mg/dm³; c_T – concentration of sodium thiosulfate solution ($c_T = 0.005$ mEq/dm³ was used); V_T – volume of sodium thiosulfate solution used for titration, cm³; V_B – volume of sodium thiosulfate solution used for blank titration, cm³; V – cell volume, cm³; V_1 – the total volume of manganese sulfate and potassium iodide added to the cell during dissolved oxygen fixation, cm³; 8.0 – oxygen millimole-equivalent mass, mg/mmol.

2. 4. Measurement of the Oxygen Mass Transfer Coefficient

The measurement principle is described

in detail by Garcia-Ochoa et al. [21]. 100 cm³ of examined liquid is placed into a glass jacketed cell (Figure 1). The oxygen sensor is located in the central part of the liquid phase. The magnetic stir bar is located right under the sensor. The rotation speed is minimal (120 rpm) to prevent distortion of the liquid surface. The system is brought to the required temperature, then it is purged with nitrogen until the optical sensor readings are stabilized (zero oxygen content). Subsequently, the nitrogen flow is stopped and the cell is opened to communicate with the atmosphere. The oxygen saturation dynamics is measured until a stable reading of the optical sensor is obtained (saturated solution).

3. RESULTS AND DISCUSSION

Detailed description of the measurement process principle is presented in [17]. The main parameters in optical oxygen sensing are the phosphorescence intensity (I) and the lifetime of the dye excited state (τ). They are related to the concentration of oxygen by the well-known Stern-Volmer equation, by means of which the concentration of the quencher can be calculated:

$$I_0 / I = 1 + K_{S-V} \cdot pO_2 \quad \text{or} \quad \tau_0 / \tau = 1 + K_{S-V} \cdot pO_2, \quad (2)$$

where I_0 and τ_0 are the intensity and the lifetime in the absence of oxygen, $K_{S-V} = k_q \cdot \tau_0$ is the quenching constant (Stern-Volmer constant), k_q is the bimolecular rate constant of quenching. Virtually the process does not consume oxygen and dye, because both return to the ground state in a while after the excitation is over. The photodegradation of the dye due to side reactions leads to a gradual decrease in intensity, but does not affect the lifetime τ . Therefore, the lifetime of the excited dye is usually measured. It is convenient to calculate it from the phase shift of the modulated signal [22]:

$$\tau = \tan(\varphi) / (2\pi f) \quad (3)$$

where f is the modulation frequency, φ is the phase of the modulation signal.

Since the sensor material has a linear calibration [19], the Equation (2) is sufficient for pO_2 calculation.

3. 1. Investigation of the Sensor Properties in Aviation Fuel

First of all, we tested the stability of the composite material in aviation fuel. Holding it for two weeks did not lead to dye leaching (solution phosphorescence is not observed). The sensitive element made of polystyrene [17] was tested under the same conditions. The first evidence of dye leaching was observed within a few hours, and a week later the film became almost transparent, but did not dissolve.

The calibration dependence in the Stern-Volmer coordinates, obtained for TS-1 fuel at 20.0 °C and 1 atm

is shown in Figure 2. It is linear, and the relative standard deviation of the measurements taken at all points of the calibration curve (DO concentration range from 0 to 70 mgO₂/dm³) does not exceed the error of the measuring instrument [17]. Thus, it is sufficient to carry out a two-point calibration (usually a saturated solution and an oxygen-free medium). The value found in air-saturated fuel was compared with values found by other method [23] to verify the correctness of the iodometric titration results. The relative error was less than 1%.

Comparison of the Stern-Volmer calibration in the form of $\tau_0/\tau-1$ vs pO_2 dependence with the results obtained in gas phase [19] showed almost complete identity. This confirms the absence of solvent influence on the properties of a sensitive element made of a composite material, and also makes it possible to simplify the calibration procedure, eliminating the time-consuming stage of titration, if the solubility of oxygen in the medium is known.

The photostability test showed decrease of phosphorescence intensity by 3% per day at constant illumination. It should be noted that the observed kinetics of degradation corresponds to many months of operation under conditions of practical use, since no more than 200 μ s is spent on one measurement.

3. 2. Measurement of the Oxygen Mass Transfer Coefficient in Aviation Fuel

The temperature dependence of the oxygen mass-transfer coefficient k_L was measured in aviation fuel to demonstrate the capabilities of the sensor material. The investigation was carried out in the temperature range from 10 to 40 °C. Each temperature point was measured twice by two different sensors to estimate the convergence, and the average was taken as the result. The calibration of the sensors was performed in the form of $\tau_0/\tau-1$ vs pO_2 dependence by two points measurement to simplify the experiment. The stable sensor readings in inert gas

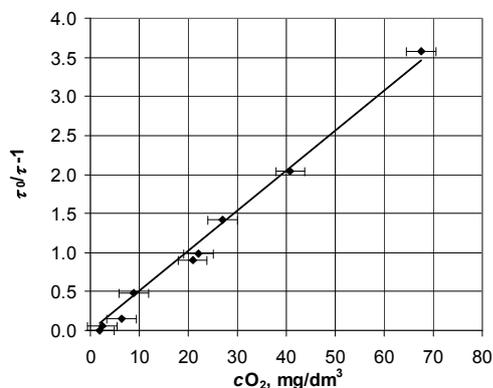


Figure 2. The optical sensor calibration plot in the Stern-Volmer coordinates for TS-1 aviation fuel at the temperature of 20.0 °C and 1 atm

flushed fuel were taken as τ_0 and the readings corresponding to the air saturated fuel were taken as the second point. The current atmospheric pressure and the percentage of oxygen in the air were taken into account. The minimal response time of the sensing element is critical for reliable results. Often t_{90} is measured representing the amount of time that it takes for the sensor to measure 90% of the real analyte level. The time t_{90} for the developed sensor did not exceed 5 s in gas phase. It is quite enough in the time scale of the experiment. Examples of the dependencies obtained for different temperatures are shown in Figure 3. It could be seen that parts representing gas purging are almost identical for all temperatures, and the observed rate of oxygen loss is greater than the saturation rate. Hence the sensor does not distort the measured dependences.

Since oxygen is not consumed during the optical measurement, the rate of oxygen mass transfer during saturation can be written as [12]:

$$d(cO_2)/d\tau = k_L \cdot a(cO_2^* - cO_2) \quad (4)$$

where $k_L \cdot a$ is the volumetric mass transfer coefficient, which is composed of the mass transfer coefficient k_L and a the specific interfacial area. cO_2^* and cO_2 are the saturation concentration of oxygen and the concentration of oxygen in the liquid phase at a given time, respectively.

According to Henry's Law, cO_2 is proportional to pO_2 :

$$cO_2 = k_H \cdot pO_2 \quad (5)$$

k_H – Henry's law solubility constant.

Substituting Equation (5) into Equation (4), we obtain:

$$d(k_H \cdot pO_2)/d\tau = k_L \cdot a(k_H \cdot pO_2^* - k_H \cdot pO_2) \quad (6)$$

Solving Equation (6) gives:

$$\ln(pO_2^* - pO_2) = -k_L \cdot a \cdot \tau + \ln(pO_2^*) \quad (7)$$

This dependence is linear in the coordinates of $\ln(pO_2^* - pO_2)$ vs τ , and it is possible to determine the $k_L \cdot a$ from the angular coefficient. The specific interfacial area a in our experiments was 26.38 m⁻¹ (cell diameter 6.5 cm, sensor area 6.8 cm², liquid volume 100 ml). The results are shown in Figure 4.

The dependence of the mass transfer coefficient obeys the Arrhenius equation:

$$k_L = k_{L0} \cdot \exp(-E_A / RT) \quad (8)$$

with the following parameters: $E_A = 21.6 \pm 1.5$ kJ/mol, $k_{L0} = 1010 \pm 90$ m·h⁻¹.

The determined value $k_L \cdot a = 5.34$ h⁻¹ at 30.4 °C is in the middle between values for isooctane and water [12].

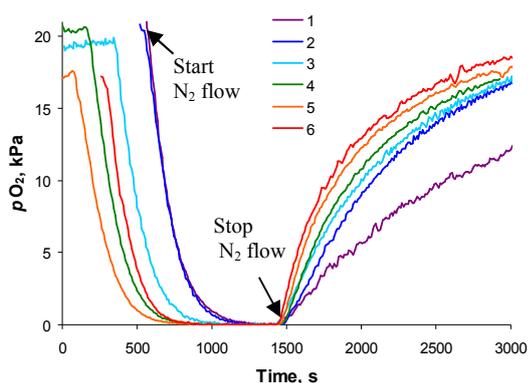


Figure 3. Kinetic curves of aviation fuel saturation by air oxygen at various temperatures: 1 – 10.2; 2 – 18.1; 3 – 25.2; 4 – 30.4; 5 – 35.4; 6 – 40.4 °C

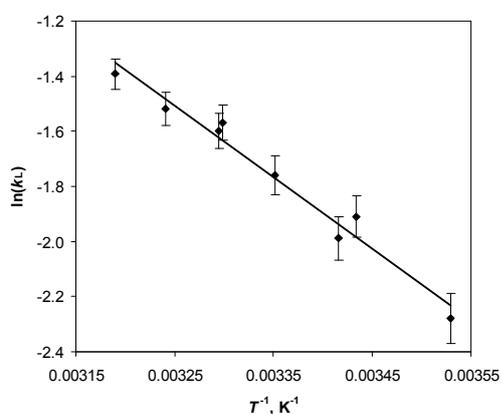


Figure 4. Dependence of oxygen mass transfer coefficient k_L on temperature in Arrhenius coordinates

4. CONCLUSION

A special sensitive element based on composite fluorinated material [18] with optimized composition showed excellent resistance to the action of aliphatic hydrocarbons, in particular aviation fuel. The results indicate not only that the sensor can be used to measure oxygen in organic solvent, but can also be used online in a dynamic method, thus facilitating the collection of oxygen transfer data. Such online measurements could be especially useful when working with a process in which the oxygen content control is very important [24-26]. The sensor has a linear calibration dependence, which facilitates exploitation compared to polymers based sensors.

Approbation was carried out on a real sample of aviation fuel TS-1 (Russian equivalent of Jet A-1). The measured value of the oxygen mass transfer coefficient in aviation fuel is more than twice as high as it is in water [12].

The created sensor has one important advantage over sensors made of sol-gel matrices with a protective coating. The damage to the protective coating of a conventional sensor can lead to its failure, since the entire membrane becomes accessible to the solvent. In contrast the composite material contains a great number of isolated particles, the mechanical damage to some part of the surface does not change the properties of the entire sensor.

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Optical Analyzer for Continuous Monitoring of Dissolved Oxygen in Aviation Fuel and Other Non-aqueous Media

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یک عنصر حساس ویژه بر اساس مواد کامپوزیتی جدید ابداع گردید. سنسور مورد نظر یک کالیبراسیون خطی دارد و به هیدروکربن های آلیفاتیک، مخصوصاً سوخت هواپیما، مقاوم است. یک روش کم هزینه و آسان برای پیاده سازی کالیبراسیون سنسور پیشنهاد شده است. وابستگی دما به ضریب انتقال جرم اکسیژن k_L برای سوخت هواپیما TS-1 معادل روسی (Jet A-1) در محدوده دما ۱۰ تا ۴۰ درجه سانتیگراد ظاهراً قابلیت های اندازه گیری سنسور را دارد. وابستگی مشخص شده مطابق معادله آرنینوس با پارامترهای $E_A = 21.7 \pm 1.5 \text{ kJ/mol}$, $k_{L0} = 1080 \pm 90 \text{ m} \cdot \text{h}^{-1}$ است. مقاومت در برابر فعالیت مکانیکی یکی از مزایای مهم در برابر حسگر ساخته شده از ماتریس sol-gel با پوشش محافظتی است. آسیب به برخی از قسمت های سطح خواص کل سنسور را تغییر نمی دهد، زیرا مواد کامپوزیت شامل تعداد زیادی ذرات جدا شده با رنگ را دارند.

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