

## SPIN-COATING FOR OPTICAL-OXYGEN-SENSOR PREPARATION

### UPORABA SPINSKEGA NANOSA PRI IZDELAVI OPTIČNIH SENZORJEV ZA KISIK

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Thin-film oxygen sensors were prepared using the spin-coating technique, where a tris (4,7-diphenyl-1,10-phenanthroline) ruthenium(II) dichloride complex (RuDPP) in various solvents and silicones deposited on different substrates was used for the sensor production. By changing the spin-coating set-up parameters, homogeneous sensor coatings and the optimum sensor response to oxygen were studied – the sensors were exposed to various concentrations of oxygen within the range from 0 % to 100 %. During the presented study, the optimum results were obtained when a 150  $\mu$ L of sensor solution was applied to a Dataline foil using silicone E4 and a chloroform solvent. A spin coater with the following three rotation stages was used: 750/700 r/min for 3 s, 300 r/min for 3 s and 150 r/min for 4 s. The spin-coating technique has several benefits: it is fast, easy to use and appropriate for low-volume operations. It allows modifications and preparations of several sensor series using the minimum reagent consumption. However, the disadvantage of this technique also has to be mentioned, namely, an uneven film thickness in the radial direction. The film thickness mainly depends on the experimental set-up (volume, rotation time and speed, solvent viscosity and evaporation). Spin coating as an alternative and very flexible technique for an oxygen-sensor preparation is suggested for the laboratory-scale work, where the majority of experimental data could be used when other new coating methods are also researched and implemented.

**Keywords:** tris (4,7-diphenyl-1,10-phenanthroline) ruthenium(II) dichloride complex, spin coating, optical oxygen sensor, oxygen

Izdelani so bili tankoplastni optični senzorji za kisik s tehniko spinskega nanosa. Pri tem so bile uporabljene različne koncentracije tris (4,7-difenil-1,10-fenantrolin) rutenij(II) diklorid kompleksa (RuDPP), različna topila, polimerni nosilci, silikoni in parametri spinskega prekritja. Naš namen je bil pripraviti najbolj homogen nanos senzorske raztopine in tako dobiti najbolj optimalne lastnosti senzorjev. Preučevali smo tudi vpliv hitrosti in časa vrtenja spinske naprave za prekrivanje na odziv senzorjev, saj so bili le-ti po izdelavi izpostavljeni različnim koncentracijam kisika v območju od 0 % do 100 %. Najboljši nanos senzorske raztopine smo dobili s senzorsko raztopino v kloroformu 150  $\mu$ L z uporabo silikona E4 z nanosom na folijo Dataline. Pri tem smo uporabili tri različne stopnje vrtenja: 3 s pri 750/700 r/min, 3 s pri 300 r/min in 4 s pri 150 r/min. Prednost uporabe spinskega prekrivanja je, da je ta tehnika zelo hitra, enostavna za uporabo in je primerna za nanos majhnih prostornin. Omogoča izdelavo več serij senzorjev z različnimi lastnostmi ob minimalni porabi reagentov. Nanos senzorske raztopine na polimernem nosilcu v radialni smeri je v veliki meri odvisen od eksperimentalnih razmer: prostornine nanosa, hitrosti vrtenja, viskoznosti in hlapnosti topil. Metoda spinskega prekritja se je izkazala kot učinkovita metoda za nanos senzorskih raztopin v laboratorijskem merilu, vendar je po celotni senzorski površini težko pripraviti popolnoma homogen nanos, zato je za pripravo večjih količin identičnih senzorjev – po optimiranju vseh drugih eksperimentalnih razmer – smiselno preučiti še alternativne metode nanašanja.

**Ključne besede:** tris (4,7-difenil-1,10-fenantrolin) rutenij(II) diklorid kompleks, spinsko prekritje, optični kisikov senzor, kisik

## 1 INTRODUCTION

Oxygen ( $O_2$ ) is considered to be one of the more important gases in our environment. The determination of  $O_2$  concentrations in the air, especially at low levels, plays an important role in different areas ranging from environmental, biological, analytical and industrial monitoring. These are the reasons why there is still a growing interest in the construction and development of oxygen sensors.<sup>1-4</sup>

There has been a trend in the development of optical oxygen sensors over the last few decades because these sensors are more attractive than conventional amperometric sensors. Optical oxygen sensors have a lot of advantages such as: a faster response time, a high sensi-

tivity and selectivity, no  $O_2$  consumption, the inertness against sample flow rate or stirring speed, absence of poison, and no need for a reference electrode.<sup>5-15</sup> They are immune to exterior electromagnetic-field interference and can be produced as disposable sensors.<sup>16,17</sup> Optical oxygen sensors are cheap, easily miniaturized and simple to use; they mainly operate on the principle of oxygen quenching those dye molecules that have been entrapped within a porous support matrix. Ruthenium(II) complexes are, by far, the most widely used oxygen dyes, because they have relatively long fluorescent lifetimes determined by the metal-to-ligand charge-transfer (MLCT) excited state, fast response time, strong visible absorption, large Stokes shift, and high photochemical stability.<sup>2,3,11,12,14,18-23</sup> Ru(II) complexes exhibit a high

sensibility to luminescence quenching and the positions of their absorption and emission spectra permit an application of low-cost, solid-state optoelectronics for the detection of luminescence intensity. The dyes can be excited with blue or even blue-green light-emitting diodes (LEDs) exhibiting a large Stokes shift and resulting in the emission of orange-red light.<sup>24</sup>

The basic operational principle of a fluorescent optical sensor for measuring oxygen is based on reducing the intensity of the fluorescence (quenching) due to the involvement of oxygen within the dye structure. The calibration of the most luminescence quenching-based optical sensors relies, in essence, on the Stern-Volmer equation.

The immobilization of the Ru(II) complexes in sol-gel matrices has been recently investigated.<sup>4,25–27</sup> There have also been reports on optical oxygen sensors based on the luminescence change of the ruthenium(II) complex immobilized in organic and inorganic polymers (polystyrene, silicone polymer, sol-gel glass, etc.) and zeolite matrix.<sup>28–30</sup> The sol-gel process has been so far the most widely used method for the preparation of oxygen sensors.<sup>5,31,32</sup> It is an efficient immobilization technique due to its many desirable properties such as high thermal stability, good photostability and optical transparency within the visible region.<sup>5</sup>

The spin-coating technique is also used for the sensor preparation. Spin coating has been used for several decades for the application of thin films.<sup>33,34</sup> This is a technique that uses centrifugal forces created by a spinning substrate for spreading a coating solution evenly over a surface.<sup>35</sup> It can be controlled with a few parameters in order to yield a well-defined coating coverage.<sup>36,37</sup> The flow is governed by a balance between the centrifugal force against the viscosity and surface tension. It has been shown that the non-uniform distribution in the initial film profile tends to become uniform during spinning. Spin coating has been mainly used in the photoresist coating process because of its simplicity of operation, its uniformity and the thinness of the coated layers. The spin-coating process involves depositing a small puddle of fluid onto the center of a substrate, and then spinning the substrate at a high speed (typically around 3000 r/min). The centripetal acceleration then causes the solution to spread towards, and eventually off, the edge of the substrate leaving a thin film on the surface. The coat thickness is controlled with the rotational speed of the substrate; faster rotations result in thinner coating layers.

The spin-coating process needs to be reshaped and optimized because of the changes in the operational parameters and the wafer size.<sup>38</sup> There is scientific literature describing the spin-coating process, emphasizing the importance of rotational speed, time, acceleration, periods, liquid viscosity, density, polymer, temperature and humidity for the film thickness.<sup>39</sup>

There are four distinct stages in the spin-coating process (**Figure 1**):

- Deposition of a coating fluid onto a wafer or substrate.
- Acceleration of the substrate up to its final desired rotational speed.
- Spinning of the substrate at a constant rate; fluid viscous forces dominate the fluid thinning behavior.
- Spinning of the substrate at a constant rate; the solvent evaporation dominates the coating thinning behavior.

The final film thickness and other properties depend on the nature of the used polymer (viscosity, drying rate, percent of solids, surface tension) and the parameters chosen for the spin process (final rotational speed, acceleration). One of the more important factors in spin coating is the repeatability. Subtle variations in these parameters defining the spin process can result in drastic variations in the coated films.

The presented goal was a preparation of thin-film oxygen sensors using the spin-coating technique. In this work a spin coater was used for spreading different sensor solutions onto various polymer substrates. The substrates (polymer solid layers – foils) were optically transparent films. The most important function of the substrate was to act as a strong mechanical carrier with a high transparency, physical strength, and chemical resistance. Different amounts of RuDPP in various solvents were used for the sensor production and various transparent polymer substrates were used as the carriers. The goal was to obtain the most homogeneous sensor coating with the spin coater by changing the set-up parameters. After the sensor preparation the sensors were exposed to various concentrations of oxygen, ranging from 0 % to 100 %.

## 2 EXPERIMENTAL WORK

### 2.1 Chemicals and solutions

All the chemicals used were of analytical purity grade. All the solutions were prepared with deionized



**Figure 1:** Scheme of the spin-coating sensor preparation

**Slika 1:** Shematičen prikaz postopka spinskega nanosa pri izdelavi senzorjev

water. Silicon (Elastosil E4, Elastosil E41, Wacker), a polymer layer (foil DATALINE 57170, Dataline, EU; foil PLASTIBOR TOP COD 12530 12950, Lazertechas, UAB; foil ESSELTE 509700, Esselte, EU), a tris (4,7-diphenyl-1,10-phenanthroline) ruthenium(II) dichloride complex (Sigma-Aldrich), toluene (Sigma-Aldrich), chloroform (CHLO) (Sigma-Aldrich) and methyl ethyl ketone (ME) (Sigma-Aldrich) were used for the sensor preparation.

The following gases from Messer, d. o. o., Slovenia, were used for testing an optical oxygen sensor: nitrogen ( $N_2$ , 99.999 %) and oxygen ( $O_2$ , 99.9999 %).

## 2.2 Apparatus

Optical measurements were studied using an EOM- $O_2$  micro electro-optical module (PreSens) controlled by the EOM- $O_2$ \_v1\_3\_exe software, a gas-mixing device (Echo, d. o. o.) and a flow cell (Echo, d. o. o.). Additional equipment included: an AB54-S balance (Mettler Toledo), a spin-coater (Polos), a MST digital magnetic stirrer (Ika) and a SUPRA 35 VP (Carl Zeiss) scanning-electron microscope (SEM).

## 2.3 Preparation of RuDPP optical oxygen sensors

Different amounts of RuDPP within the range of 20 mg to 80 mg were diluted using different solvents (toluene, chloroform and methyl ethyl ketone).

An appropriate amount of RuDPP was weighted in a 10 mL flask and diluted using an appropriate solvent. The prepared sensor solution was then being stirred with a magnetic stirrer for 10 min. The sensor solution was then filtered through filter paper and a 4 mL sensor solution was added to 2 g of silicone. This sensor solution was mixed on a magnetic stirrer for about 1 h to become homogeneous and viscous. The sensor solution was protected from the external light with an aluminum foil, and was applied to the solid layers using the spin-coating technique. Different polymer solid layers (foils) were used for the substrate (ESSELTE, DATALINE and PLASTIBOR). Different amounts ((100, 150 and 200)  $\mu$ L) of the sensor solutions were applied using the spin-coating technique. The effects of changing the rotation speeds and times of spinning were also studied; the details are given in **Table 1**.

**Table 1:** Rotation speed and spinning time

**Tabela 1:** Hitrost vrtenja in čas vrtenja naprave za spinsko prekrivanje

Stage	Number of turns (r/min)	Time of spinning (s)
1 <sup>st</sup> step	500 to 900	1–10
2 <sup>nd</sup> step	300 to 700	1–10
3 <sup>rd</sup> step	100 to 150	1–10

The sensor solution was mounted on a rotating platform. The substrate was rotated according to the selected rotation speed/spinning time and the sensor

solution was dispensed directly onto it. The high-speed rotation threw off most of the solution, leaving behind a thin, uniform coating.

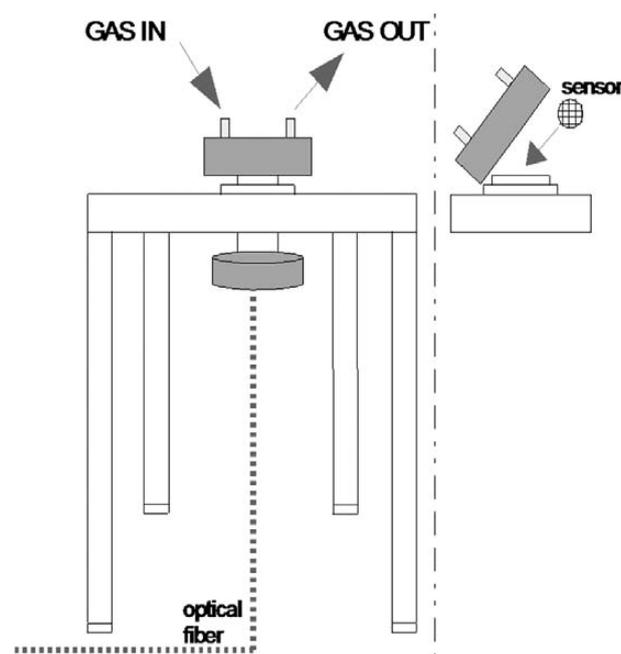
The prepared optical sensors were then dried; they were usually left to dry out for 24–48 h at a room temperature of  $(20 \pm 2) ^\circ C$ . After drying, the optical sensors were cut to the diameter dimensions of  $1.75 \text{ cm}^2/15 \text{ mm}$ . The sensors were stored in a dark and dry place before use.

## 2.4 Measurement procedures

The optical oxygen sensors were tested in a flow cell (**Figure 2**). They were excited with a blue LED and measured with an optical detector from PreSens. The gas mixtures ( $N_2/O_2$ ) passed the active sensor surfaces at a constant flow rate of 1 L/min.

The changes of the signal were measured for different concentrations of oxygen. The gas mixtures were prepared with a gas mixing device (Echo, d. o. o.).<sup>4</sup> During the constant flow of the carrier gas, various concentrations of oxygen were added to obtain different concentrations within the range of  $1 \cdot 10^{-6}$  to  $1000 \cdot 10^{-6}$ . The accuracy of the concentrations was  $\pm 0.7 \cdot 10^{-6}$ . The gas-mixing device provided a repeatability of  $\pm 0.15 \%$  and, within the full-scale mode, the temperature range was from  $15 ^\circ C$  to  $25 ^\circ C$  and the pressure varied from 70 kPa to 400 kPa. **Figure 3** schematically presents the system used for the optical measures. The measuring system consisted of: a gas-mixing device, a flow cell, an electro-optical module and a computer.

Surface analyses of the sensors were performed with a scanning electron microscope (SEM), Supra 35 VP



**Figure 2:** Scheme of the flow cell (left) and sensor positioning (right)  
**Slika 2:** Shema pretočne celice (levo) in namestitev senzorja (desno)

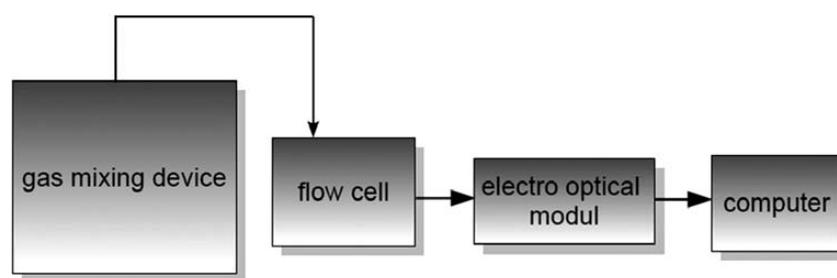


Figure 3: Scheme of the measuring system

Slika 3: Shema merilnega sistema

Carl Zeiss. All the pictures were recorded using a 30  $\mu\text{m}$  scan window at the 1 kV electronic potential.

### 3 RESULTS AND DISCUSSION

The influences of the dye concentration, different polymer solid layers (foils), silicones, film thickness and different solvents on the sensor sensitivity were studied.

#### 3.1 Influence of the RuDPP concentration vs. the sensor sensitivity

The Stern-Volmer equation describes the fluorescence intensity versus the measured concentration of oxygen.<sup>4</sup> A deviation from the linearity is connected with the heterogeneity of a polymer matrix; the fluorophore molecules are usually surrounded by voids and polymer particles, therefore, all the indicator molecules are non-equally accessible to oxygen. A decline in fluorescence is strongly dependent on the diffusion and adsorption of oxygen and on the dye solubility. The concentration of the indicator must be appropriately selected in order to obtain the optimum sensor sensitivity, and the dye concentration must be additionally optimized according to the measure range.

The sensors were prepared according to the procedure described in Section 2.3. The amounts of (20, 40, 60 and 80) mg of RuDPP were used for preparing the sensor solution (Figure 4), while the linearity ( $R^2$ ) was

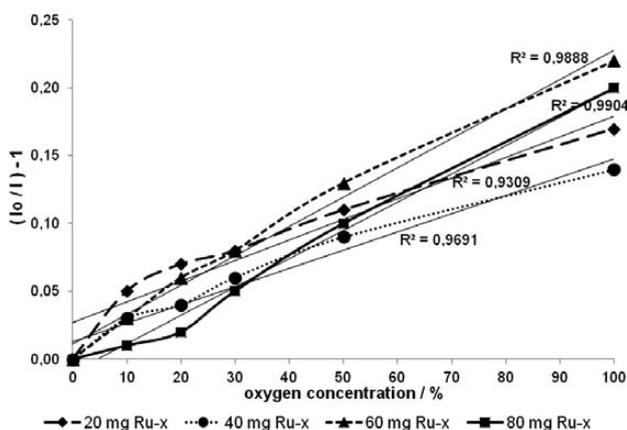


Figure 4: Influence of RuDPP concentration vs. sensor response

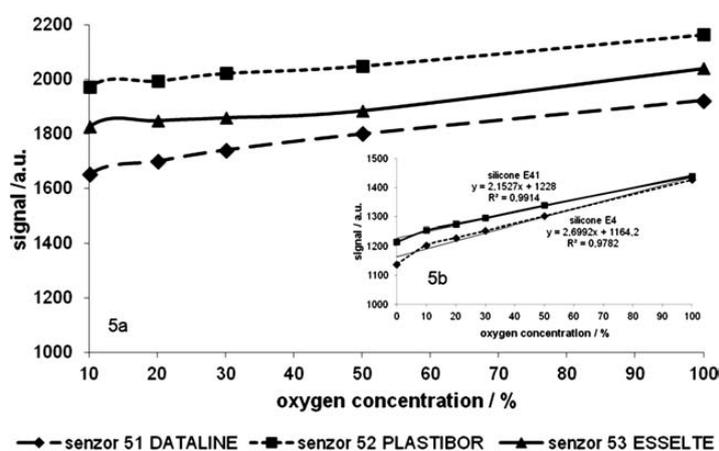
Slika 4: Vpliv koncentracije RuDPP na odziv senzorjev

tested within the range of 0 % to 100 % concentration of oxygen.

The linearity of the sensors prepared from 20 mg of RuDPP was 0.9309, for 40 mg of RuDPP it was 0.9691, for 60 mg of RuDPP it was 0.9888, and 0.9904 for 80 mg of RuDPP. The optimum sensor was the one with 80 mg of RuDPP, therefore, it can be concluded that the concentration of RuDPP strongly influences the sensor response. In general, with higher concentrations of RuDPP, the sensor linearity, accuracy and precision are improved. In addition, a strong fluorescence signal was obtained and no additional amplification of the measuring signal was used. The electronic-optical noise usually increased with a higher amplification rate, which can also be a reason for a nonlinear sensor response. On the other hand, due to the high cost of RuDPP, it is important to incorporate low dye concentrations. In order to prepare sensors with different properties, typical amounts of (40, 60 and 80) mg of RuDPP were used for further studies.

#### 3.2 Sensor preparation – modification of foils and silicones

In the next step different foils and silicones as the support matrices were tested. In order to optimize the sensors, different foils (Plastibor, Dataline, Esselte) and commercially available silicones (E4, E41) were used. The linearities and sensitivities ( $k$ ) of different sensors were tested; Table 2 presents all the major sensor characteristics. Figure 5a presents the changes in the measured signal versus the various concentrations of oxygen with different foils, and Figure 5b shows the change in the signal with different silicones. When using the Datalain foil a slightly better linear response was obtained, especially at low concentration ranges of oxygen, when compared to the Plastibor or Esselte foils, but the selection of the solid layers does not significantly improve or change the sensor properties. It is generally known that with the increasing roughness of a substrate foil the adhesion of the coatings on the surface is improved even in spin coating. Here, it is important to mention that rough surfaces cause a lower transparency with a significant back-scattering light effect, and for this reason we used low-roughness foils. Additionally, a compromise between the foil transparency and surface



**Figure 5:** a) Impact of a solid layer (a foil) on sensor response (signal/a.u. – arbitrary units vs. concentration of O<sub>2</sub> in %), b) comparison of different silicones vs. sensor response

**Slika 5:** a) Vpliv trdnega nosilca (folije) na odziv senzorjev (izmerjen signal (a.u. enote) vs. koncentracija O<sub>2</sub> v %), b) vpliv silikona na občutljivost senzorja

**Table 2:** Influence of the solid layers on the sensor characteristics

**Tabela 2:** Vpliv trdnih plasti na lastnosti senzorjev

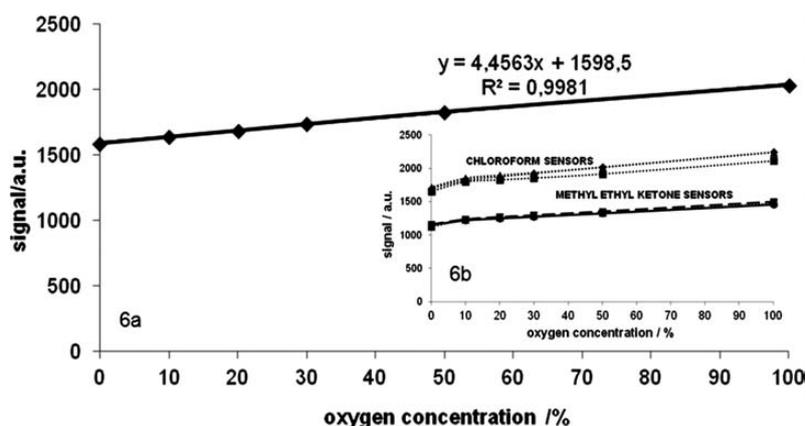
RuDPP/mg	FOIL	SILICONE	SOLVENT	V/ $\mu$ L	STAGES	k	R <sup>2</sup>
60	DATALINE	E4	ME	150	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.699	0.978
60	PLASTIBOR	E4	ME	150	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.464	0.960
60	ESSELTE	E4	ME	150	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.684	0.886
60	DATALINE	E41	CHLO	200	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	4.141	0.964
60	PLASTIBOR	E41	CHLO	200	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	4.516	0.974
60	ESSELTE	E41	CHLO	200	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	4.531	0.957
80	DATALINE	E41	ME	200	1300 r/min → 3 s 2200 r/min → 5 s 3100 r/min → 2 s	-2.727	0.361
80	ESSELTE	E41	ME	150	1300 r/min → 3 s 2200 r/min → 5 s 3100 r/min → 2 s	-8.761	0.992
80	PLASTIBOR	E41	ME	150	1300 r/min → 3 s 2200 r/min → 5 s 3100 r/min → 2 s	-5.709	0.834
80	DATALINE	E41	CHLO	150	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	5.106	0.975
80	PLASTIBOR	E41	CHLO	150	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	4.073	0.943
80	ESSELTE	E41	CHLO	150	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	4.787	0.966
20	DATALINE	E4	ME	150	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	4.277	0.735
20	PLASTIBOR	E4	ME	150	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	3.693	0.606

20	ESSELTE	E4	ME	150	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	3.962	0.635
20	DATALINE	E4	CHLO	150	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.545	0.946
20	PLASTIBOR	E4	CHLO	150	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	1.609	0.585
20	ESSELTE	E4	CHLO	150	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	1.94	0.845
40	DATALINE	E41	CHLO	200	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	4.531	0.957
40	PLASTIBOR	E41	CHLO	200	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	3.857	0.959
40	ESSELTE	E41	CHLO	200	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	4.077	0.865
40	DATALINE	E4	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	6.468	0.528
40	PLASTIBOR	E4	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	6.654	0.524
40	ESSELTE	E4	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	7.003	0.524
40	DATALINE	E41	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.166	0.555
40	PLASTIBOR	E41	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.207	0.562
40	ESSELTE	E41	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	1.806	0.405

**Table 3:** Variation of silicones vs. sensor sensitivity

**Tabela 3:** Vpliv silikona na odzivnost senzorjev

RuDPP/mg	FOIL	SILICONE	SOLVENT	V/ $\mu$ L	STAGES	k	R <sup>2</sup>
40	DATALINE	E4	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	6.468	0.528
40	DATALINE	E41	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.166	0.555
40	PLASTIBOR	E4	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	6.654	0.524
40	PLASTIBOR	E41	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.207	0.562
40	ESSELTE	E4	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	7.003	0.524
40	ESSELTE	E41	CHLO	200	1750 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	1.806	0.405
60	DATALINE	E4	ME	150	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.699	0.978
60	DATALINE	E41	ME	150	1600 r/min → 3 s 2300 r/min → 3 s 3150 r/min → 4 s	2.152	0.991



**Figure 6:** a) Sensor response under optimum conditions (signal/a.u. – arbitrary units vs. concentration of O<sub>2</sub> in %), b) sensitivity of the sensor using different solvents (chloroform and methyl ethyl ketone)

**Slika 6:** a) Odziv senzorja pri optimalnih pogojih (izmerjen signal (a.u. enote) vs. koncentracija O<sub>2</sub> v %), b) vpliv topil (kloroform, metil-etil keton) na odziv senzorjev

roughness was made by adding inert silicones to the sensor solution. **Table 3** presents the conditions for preparing the sensor solutions using different silicones. The optimum linearity (0.9914) was obtained using silicone E41, foil Dataline and 60 mg of RuDPP. The silicone adhesion of the sensor solution to the foils was significantly improved, while preserving the optimum light transparency.

### 3.3 Spin coating and sensor thickness

A lot of factors participated in the spin-coating sensor preparation<sup>33,34</sup> – one of these was also the selection of a suitable solvent. The sensors were prepared using chloroform, toluene, and methyl ethyl ketone. Toluene as a solvent proved to be unsuitable because it partially dissolved the surfaces of the foils. Other solvents were constantly evaporating, also during the spin-coating period, but the sensors prepared with chloroform presented better characteristics (linearity and sensitivity) than the sensors prepared with methyl ethyl ketone (**Figure 6a**). Chloroform had a lower evaporating rate than methyl ethyl ketone and the sensors prepared with chloroform had a uniform film thickness. **Figure 6b** demonstrates that the sensor prepared with chloroform also had a substantially higher signal (by approximately 30 %), while the other parameters remained constant.

Our further experimental work used different spin-coating stages (periods) and accelerations. The optimum results were obtained when a 150  $\mu$ L sensor solution in chloroform containing 80 mg of RuDPP with silicone E41 (foil Dataline) was applied (**Figure 6a**) under the following spin-coating conditions:

1<sup>st</sup> step: 750/700 r/min  $\rightarrow$  3 s

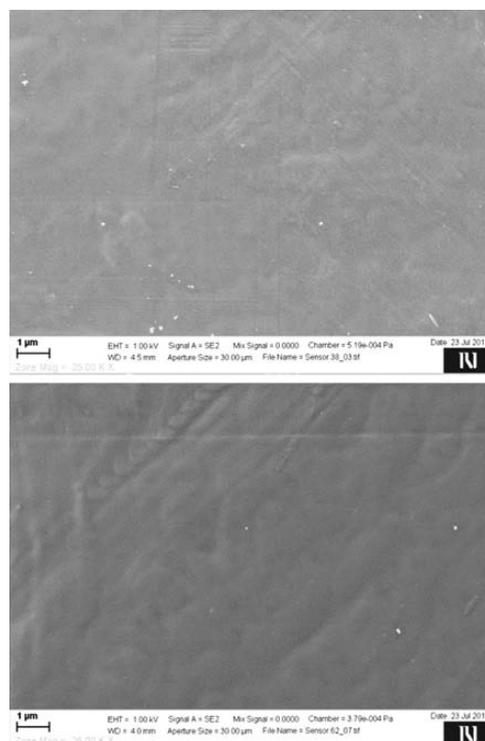
2<sup>nd</sup> step: 300 r/min  $\rightarrow$  3 s

3<sup>rd</sup> step: 150 r/min  $\rightarrow$  4 s

This spin-coating technique had several benefits including a fast process time (only a few seconds) when using low volumes of reagents. A modification of the spin speeds or an increase in the spin time allowed a thin-film preparation (below 1  $\mu$ m).

Using scanning electron microscopy, the irregularities in the sensor surface were searched. For the SEM analysis, the sensors with homogeneous surfaces (an optical selection) and optimum oxygen responses were selected. An optical selection means that the sensors with the most uniform coating and without any visible solid particles or air bubbles were scanned (**Figure 7**).

The coatings and thicknesses of the sensors were incompletely uniform throughout the sensor surfaces varying within the range of (3.5–5.0  $\pm$  0.5)  $\mu$ m. Air bubbles were visible on individual parts, probably captured in the sensors during the polymerization step. This



**Figure 7:** Optical-oxygen-sensor SEM images at 2500-times magnification  
**Slika 7:** SEM-posnetka površine optičnih senzorjev za kisik pri 2500-kratni povečavi

could be avoided, to some degree, by implementing a vacuum chamber over the treated surface. The main problem regarding the entrapped air bubbles was the fluctuation of the scattering light causing a lower fluorescence signal – the light was scattered in all directions. The entrapped air bubbles could also cause a longer response time – the measuring oxygen molecules can be trapped within the presented voids.

#### 4 CONCLUSION

The spin-coating technique was studied while used for the optical-sensor preparation when different parameters directly affected the film thickness and, therefore, also the sensor response to oxygen. This paper primarily focuses on the influences of the rotation speed and spinning time on the film thickness, in addition to the acceleration, temperature, humidity, viscosity, solvents, silicones, foils and RuDPP concentration studied. The optimum results were obtained when 80 mg of RuDPP was dissolved in chloroform, silicone E41 was added and a 150  $\mu$ L of sensor solution was applied to the Dataline foil under the following spin-coating conditions: 1<sup>st</sup> step: 750/700 r/min for 3 s, 2<sup>nd</sup> step: 300 r/min for 3 s, and 3<sup>rd</sup> step: 150 r/min for 4 s.

Spin coating is an alternative method for a sensor preparation. It is a very fast, simple method, consuming low volumes of reagents, but making it difficult to prepare completely homogeneous layers on the whole sensor surface. Therefore, it is suggested to be used for a laboratory-scale sensor preparation, where the majority of experimental data could be used later when new coating methods are researched.

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