

Comparative Analysis of Derivative Parameters of Chemoresistive Sensor Signals for Gas Concentration Estimation

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Abstract: Signals from resistive gas sensors based on zirconium dioxide and silicon–carbon films have been extensively investigated to estimate gas concentration. In this study, the change in the normalized resistance of the sensor’s response under NO₂ exposure is shown and the analysis of the first and second derivatives of the response curves were carried out. A signal-processing scheme, reducing the effect of noise and signal drift, is proposed. The extreme of the second derivative of the sensor response, the initial reaction rate, and the slope of the curve of the approximating line in the coordinates of the Elovich equation are proposed as calibration dependencies. The calibration curves built from the values of the maximum second derivative turned out to be the most stable, with the lowest relative error in estimating gas concentration compared to the traditional fixed-time point method.

Keywords: gas sensor; sensor response; signal processing; Elovich equation



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1. Introduction

The detection of air pollutants, including nitrogen dioxide, continues to be relevant [1–3]. The efficiency of miniature resistive semiconductor gas sensors mainly depends on the type of gas-sensitive material, the simplicity of the design, and the possibility of integration into information systems used for air monitoring [4–6].

Nitrogen dioxide sensors can vary in the operating range of the detected concentrations depending on the purpose of monitoring. This can be direct monitoring at the emission source with high gas concentrations [7] or ambient air monitoring with low gas concentrations [8].

The deposition of gas molecules on the adsorption centers leads to a change in the concentration of the charge carriers of gas-sensitive material; the result is a change in resistance. Active centers on the semiconductor surface can interact with molecules of many gases and make a significant contribution to the change in resistance; therefore, the question of the selectivity of sensors remains open [9].

The use of signal processing methods can provide greater selectivity of the gas sensor. In [10,11], time-frequency and transient analyses were used for dynamic signal processing. Key signal characteristics can be viewed and analyzed to identify or quantify detected gases by these methods. In addition, empirical expansions in modes and the Hilbert spectra were used to analyze the dynamic response signals of gas sensors [11].

Artificial neural networks, discriminant analysis [12], principal component analysis [13], and other artificial intelligence methods were used to recognize gas and to detect its’ concentration by an array of sensors [14–16].

In addition, the analysis of the sensor response data allows us to solve the problem of signal drift, which occurs due to prolonged operation or external influence [17] and can be used to reduce the measurement error and improve the stability and selectivity of the sensor [18–20].

The problem of signal noise is it must be solved when processing the sensor signal [20,21]. Changes in temperature and gas flow during measurements also affect the detection of the gas concentration, and they should be considered when processing the signal [22].

In many cases, the main characteristics of a gas sensor are calibration curve, selectivity, dynamic measurement range, basic error, sensor resistance to overloads [23,24].

For slow-rate sensors with high gas response values in the presence of signal drift, the commonly used dependence of the change in absolute resistance on concentration is inconvenient to use since the time to reach the saturation state is long. Adam L. Washburn et al. [25,26] showed that when the sensor response is approximated by an exponential dependence, the calibration curve built from the initial slopes of these dependencies produces a reduction in the standard deviation compared to the standard deviations of multiple measurements using a fixed time point of about 3–6 times. The initial slope-based method also worked well when arrays of silicon nanowires were used as a gas-sensitive material [27].

The first-order time derivative can be used as an additional signal for gas sensors with low internal noise while maintaining high sensor response in applications where it is necessary to track abrupt changes in concentrations. To some extent, this allows exposure and relaxation to achieve time independence [28]. For the sensors that do not recover well after exposure to gases, the use of the approximation line slope to build the calibration dependence, based on the Elovich equation [29], and the first and second derivatives of the gas sensor response [30], showed good results.

In [28], the applicability of the first and second derivatives from the position of the Langmuir theory was substantiated. However, for sensitive elements of complex composition, the adsorption mechanisms may differ substantially from this theory. In particular, the assumption that the values of the square roots of the second derivative extrema, but not the extrema themselves, can be used as the calibration dependence. The possibility of using the extrema values of the second derivative, themselves as a measure of the degree of curvature of the response curve, has not been studied before, nor has the stability of the calibration dependence been fully explored. Such a possibility is investigated in the present work since it is the section near the second derivative's maximum, which can be the most stable and informative in the case of complex processes and specific adsorption processes.

This work aimed to analyze the semiconductor gas sensor's signal using several parameters that simultaneously characterized the dynamics of its response to gas exposure. The stability of two NO₂ sensors, operating in different concentration ranges, and comparison and applicability of various calibration dependencies, based on this analysis, were investigated.

The gas sensor based on silicon and zirconium oxides, described in [2,31], showed a stable response to nitrogen dioxide. The SiO₂/ZrO₂ gas-sensitive films were obtained by sol-gel technology using an alcohol solution based on tetraethoxysilane [2,32]. The gas sensors based on SiO₂/ZrO₂ can also be used to detect gases at low working temperatures [31]. Additionally, the response of a sensor based on a silicon–carbon film doped with copper was also investigated.

2. Materials and Methods

We deposited V/Cu/Cr layers, serving as the contact electrodes, via an interdigitated shadow mask onto the surface of the SiO₂/ZrO₂ film templates of the gas sensor studied for nitrogen dioxide (Type 1). Regarding the sensor (Type 2) described in [33–35], the possibility of depositing silicon–carbon films from organic solutions with gas-sensitive properties by the electrochemical method was shown.

In this work, the response of the sensor based on a silicon–carbon film doped with copper on a polycor substrate was investigated. For the vacuum thermal evaporation technique, applied for conductive layer formation, we used a copper layer (with a thickness of 2 µm) surrounded by chromium sublayers (with a thickness of 15 nm). Then, interdigitated

electrodes were formed by the laser demetallization technique; an area of 1 cm^2 was formed, with a step between the electrodes of $50\text{ }\mu\text{m}$ with a width of $50\text{ }\mu\text{m}$.

Further, as reported in [35], the sample was placed with a graphite electrode in a galvanic cell with a distance of 1 cm. Methanol, with a hexamethyldisilazane solution in a ratio of 9:1, was used as the electrolyte. Added to form the copper-doped film was 0.01% of $\text{Cu}(\text{CH}_3\text{COO})_2$. The deposition was carried out in three stages. In the first stage, a “pure” silicon–carbon film was deposited as an adhesive layer for 40 min. In the second stage, the copper-containing film was deposited for several minutes. In the third stage, a “pure” silicon–carbon film was deposited again. In the end, the resulting film annealed at a temperature of $500\text{ }^\circ\text{C}$.

The gas sensor was placed in a test chamber equipped with a heating element, as shown in Figure 1.

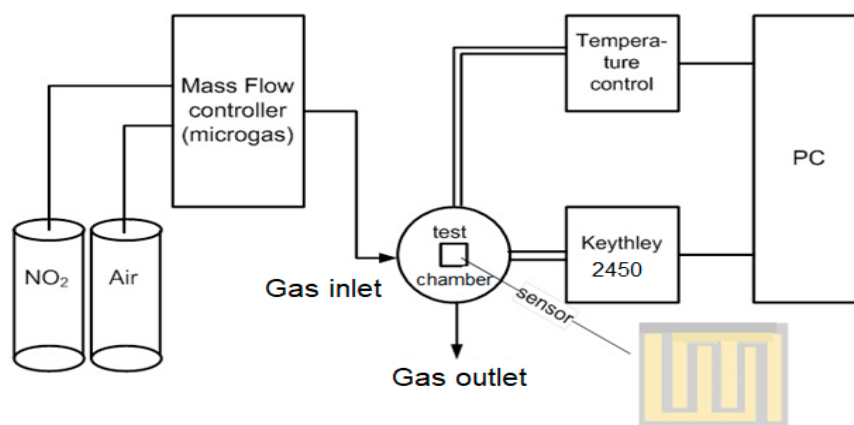


Figure 1. Schematic diagram of the measurement setup.

A cylindrical fluoropolymer chamber with a volume of 8 cm^3 has a gas inlet and outlet. At the bottom of the chamber, there is a plate with a heating element for setting the sample.

The gas sensor was heated by a direct current source with the intent of maintaining the required temperature of $\pm 1\text{ }^\circ\text{C}$. The measurements were performed by injecting an air mixture, with various gas concentrations, into the test chamber. The gas mixture was generated by mixing test gas (NO_2) with the synthetic air (Microgaz-FM02 installation (Russia)). The test gas met the requirements of ISO 6142-1: 2015. A Keithley 2450 Source Meter, connected to a computer, was used as a signal receiver. The gas mixture was supplied forcibly.

The gas exposure reversibly changed the film resistance. After purging the test chamber with the synthetic air, the film resistance returned to the initial value. The output parameters were recorded in the PC memory in the real-time mode.

The original signal—a data sequence of resistance versus time—comes from the sensor. In this case, the initial time of the gas exposure was recorded, as well as the time of the completion of the exposure and air purging. A series of experiments were carried out with different gas concentrations (Figure 2).

The data obtained during the measurements consists of the useful signal sum, random noise, and errors obtained during the measurements. Noises arise in electronic recording systems, when environmental conditions change, etc. Preliminary smoothing of the initial data was performed to increase the reproducibility of the analysis’ results.

Signal filtering in the data processing system was carried out using three well-known algorithms:

- Exponential smoothing;
- Smoothing by the Savitsky–Golay method;
- Smoothing after the discrete Fourier transform.

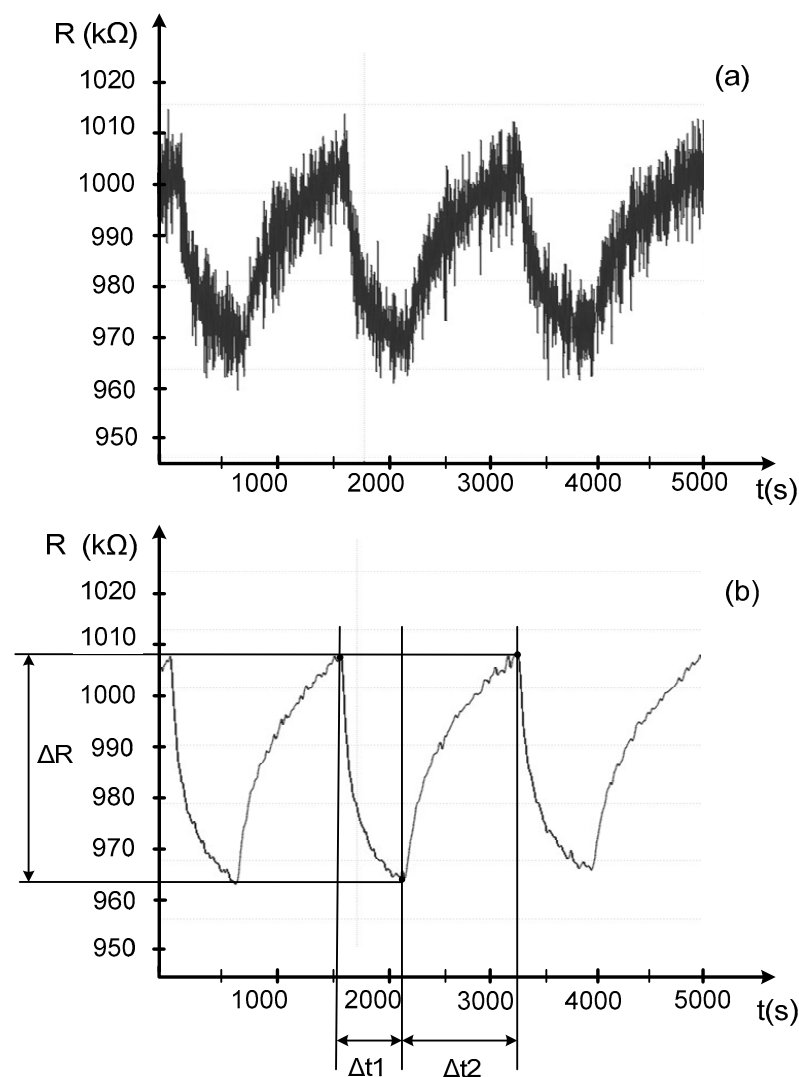


Figure 2. The graphs of (a) the original signal and (b) the filtered signal (sensor working temperature, 80 °C).

Next, local extrema, corresponding to the minimum and maximum points of a separate measurement, were determined. In this segment, the signal amplitude, when exposed to gas (ΔR), the response time to gas (Δt_1), and the recovery time (Δt_2), were determined, as shown in Figure 2b.

The signal-processing scheme is shown in the Supplementary Materials, Figure S1.

The data were normalized for deeper processing. The gas sensor response was calculated by the Formula (1):

$$S(t) = \frac{R(t)}{R_0}, \quad (1)$$

where R_0 is the film resistance before the gas exposure and $R(t)$ is the film resistance under the gas exposure at time t .

The parameters of the response dynamics of the sensor resistance, namely the rate of reaction of the sensor to the gas as the first derivative of the gas sensor response versus time (dS/dt) and the acceleration of the sensor response as the second derivative of the gas sensor response versus time ($(d(dS))/(dt^2)$), were calculated.

In order to obtain information on the mechanism of gas adsorption, the time dependences were also plotted as ΔS vs. $\ln(t)$ (Elovich equation (Equation (2))) [35–37]:

$$q = \frac{1}{\alpha} \ln(\alpha\alpha) + \frac{1}{\alpha} \ln(t) \quad (2)$$

where q is the amount of gas adsorbed during the time t ; α is the initial constant of the adsorption rate; α is a constant associated with the measure of the potential barrier for sequential adsorption.

In our case, the value of q is proportional to the value of S , $\Delta S = 1 - S(t)$.

For comparison, a fixed-time point method was used as a traditional one. Calibration dependencies were built for ΔS at the saturation moment of the normalized response [27].

3. Results and Discussion

The dependence of the sensor resistance on the temperature is shown in the Supplementary Materials, Figure S2.

The Type1 sensor under study was exposed to nitrogen dioxide in the concentration range from 5 to 50 ppm, while the Type 2 sensor was exposed to nitrogen dioxide in the concentration range from 0.5 to 5 ppm. The concentration range was attributed to the operating range of the sensors. Three series of experiments were carried out in 1-week intervals, with three measurements at each gas concentration in each series. Figure 3 shows the typical sensor response after noise elimination.

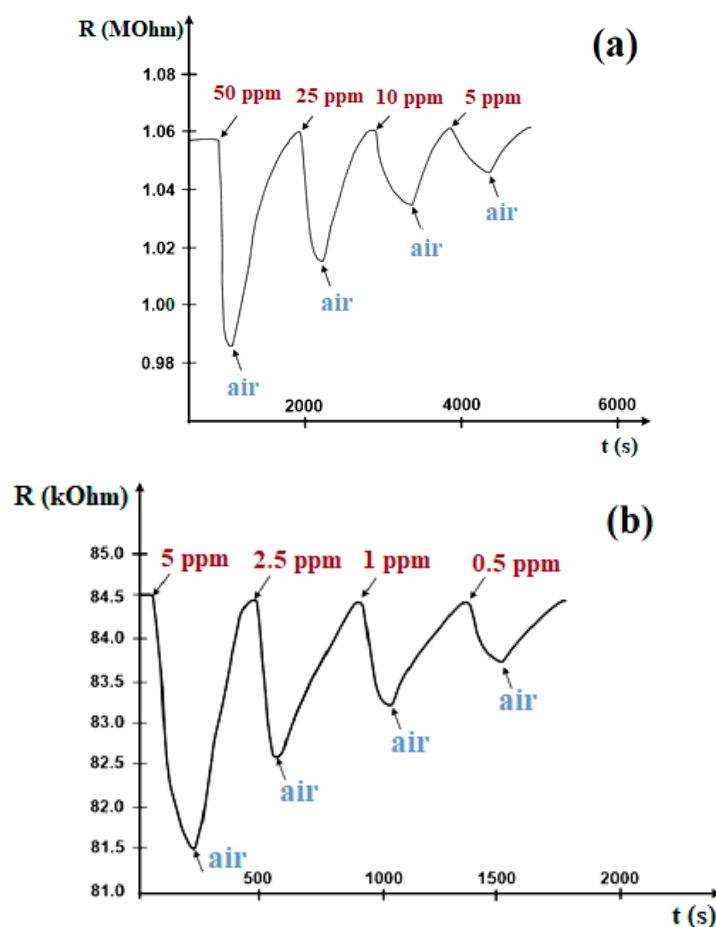


Figure 3. The real-time sensor response to various NO₂ concentrations: (a) Type 1 sensor (working temperature is 80 °C); (b) Type 2 sensor (working temperature is 22 °C).

The graphs of the response parameters for the initial section under the NO_2 exposure after normalization and reduction to a single origin are shown in Figure 4 (Type 1) and Figure 5 (Type 2).

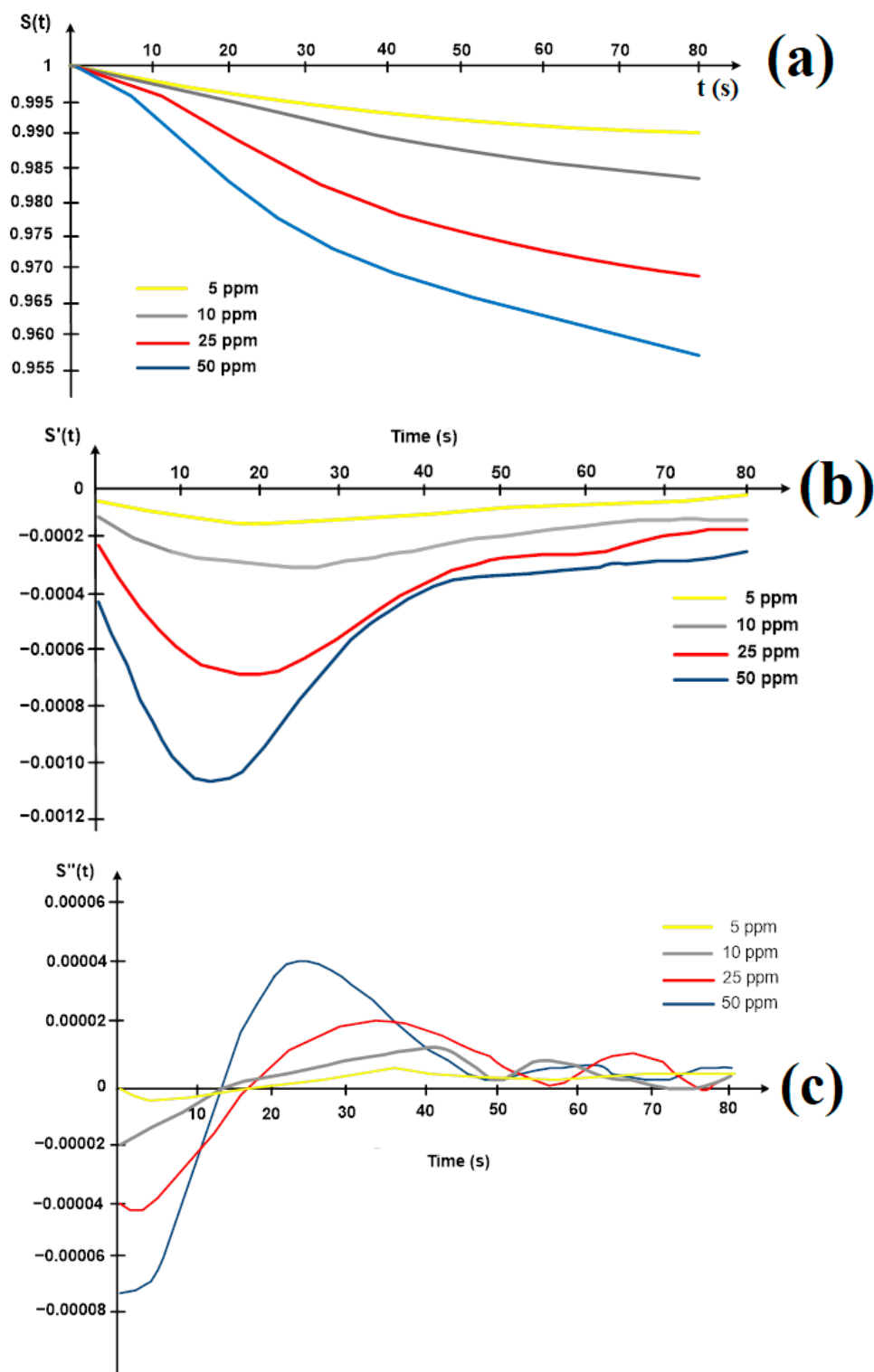


Figure 4. The kinetics (a) of the Type 1 gas sensor response $S(t)$; (b) of the first derivative $S'(t)$; (c) of the second derivative $S''(t)$ at different NO_2 concentrations.

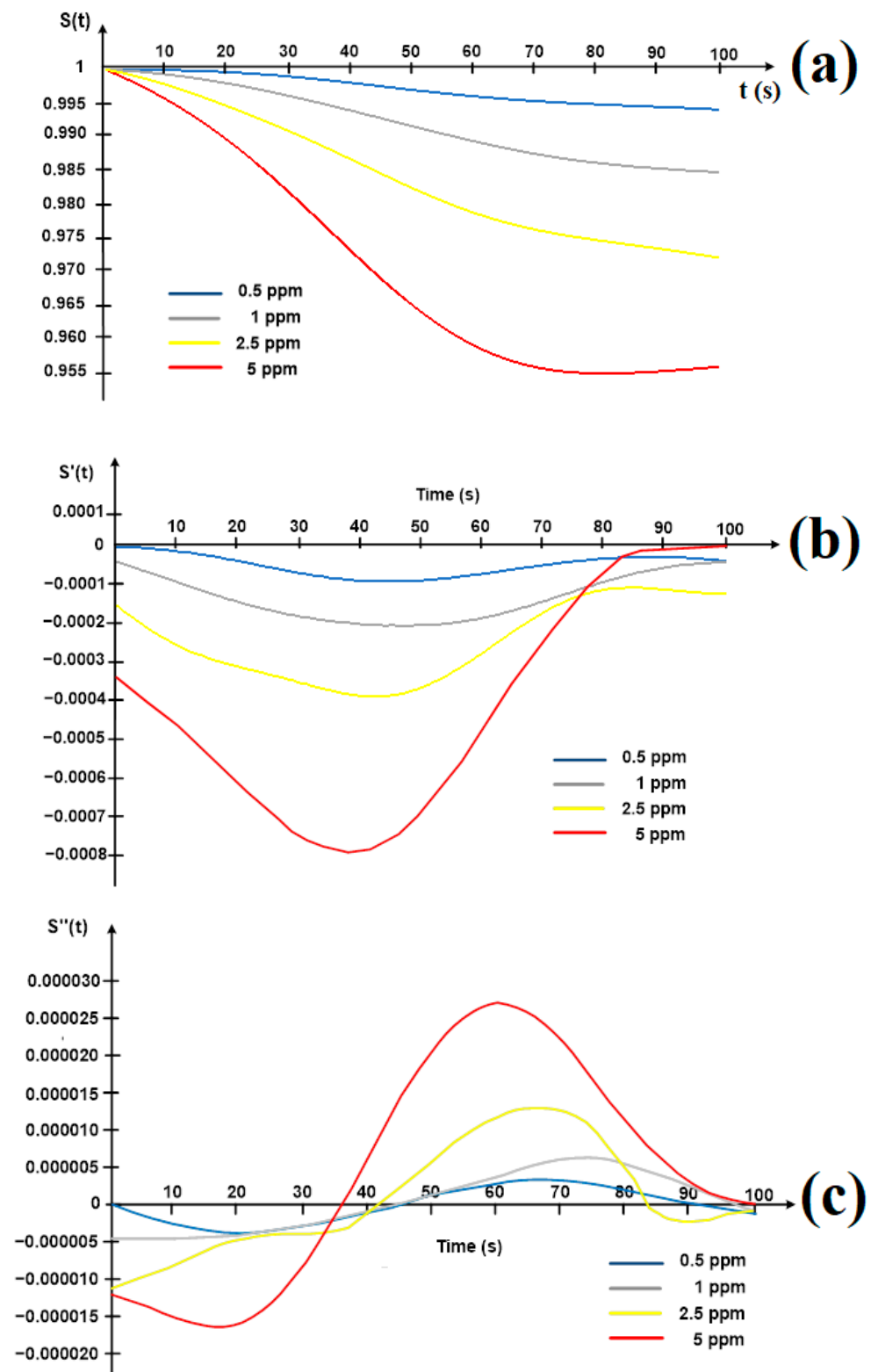


Figure 5. The kinetics (a) of the Type 2 gas sensor response $S(t)$; (b) of the first derivative $S'(t)$; (c) of the second derivative $S''(t)$ at different NO_2 concentrations.

The work of [30] shows that the extrema of the first and second derivatives of $S(t)$ depend on the gas concentration; therefore, they can be used to build calibration curves. Figures 6 and 7 show the calibration curves of ΔS made at the saturation moment, as well as for the $S'(t)$ and $S''(t)$ extrema for the sensor under study.

Figures 8–10 show the confidence intervals calculated based on the found variance at the points of multiple measurements. The standard deviations of the measurements based on the second derivative turned out to be 2–5 times less than for the measurements based on the fixed-time point method. It can be noted that the correlation coefficient (R^2) for the second derivative is maximal for the Type 1 and 2 sensors.

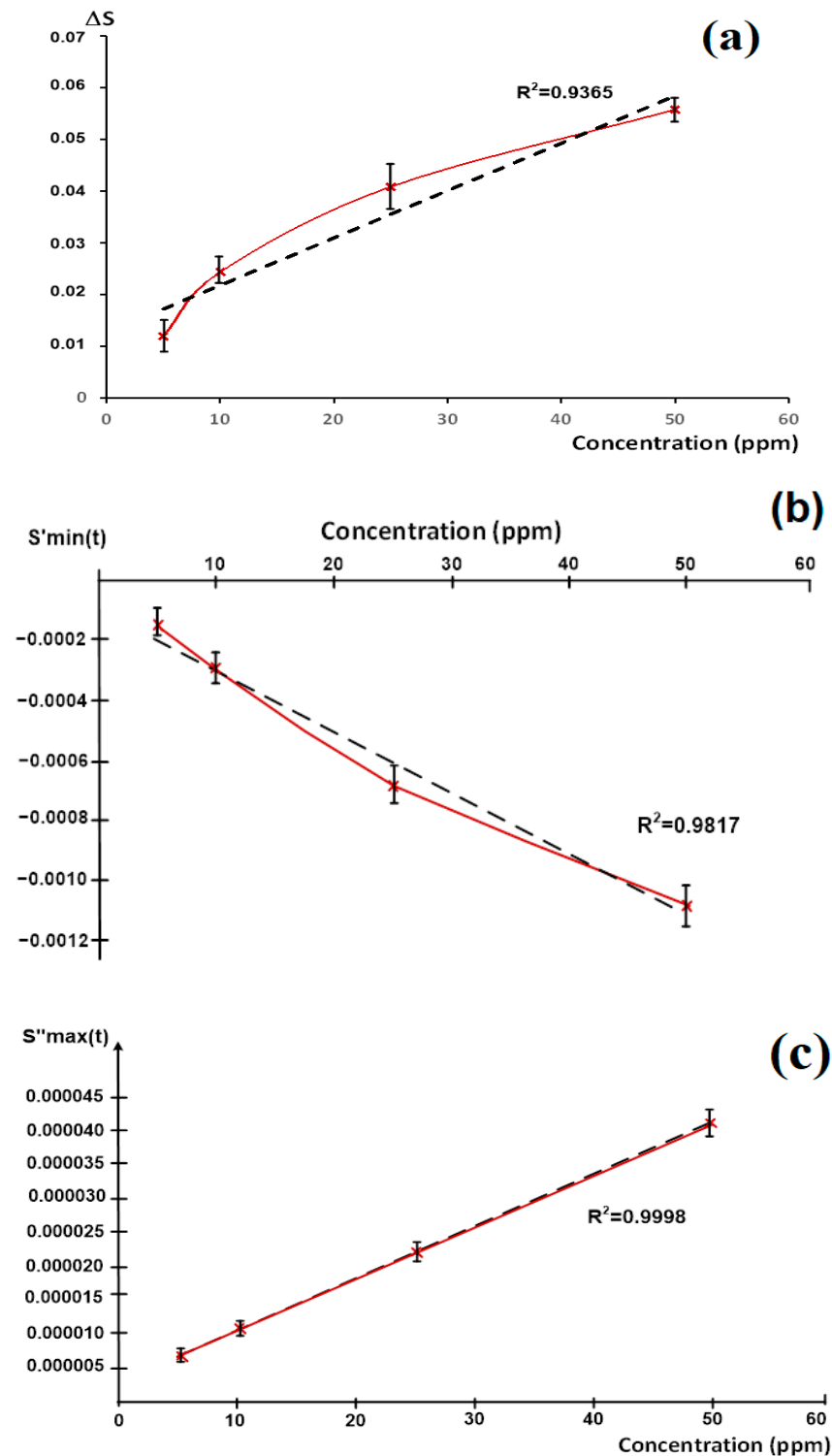


Figure 6. Calibration curves for NO₂ concentration estimation of Type 1 sensor (a) of the adsorption response ΔS at the saturation moment; (b) minimum of the first derivative $S'(t)$; (c) maximum of the second derivative $S''(t)$.

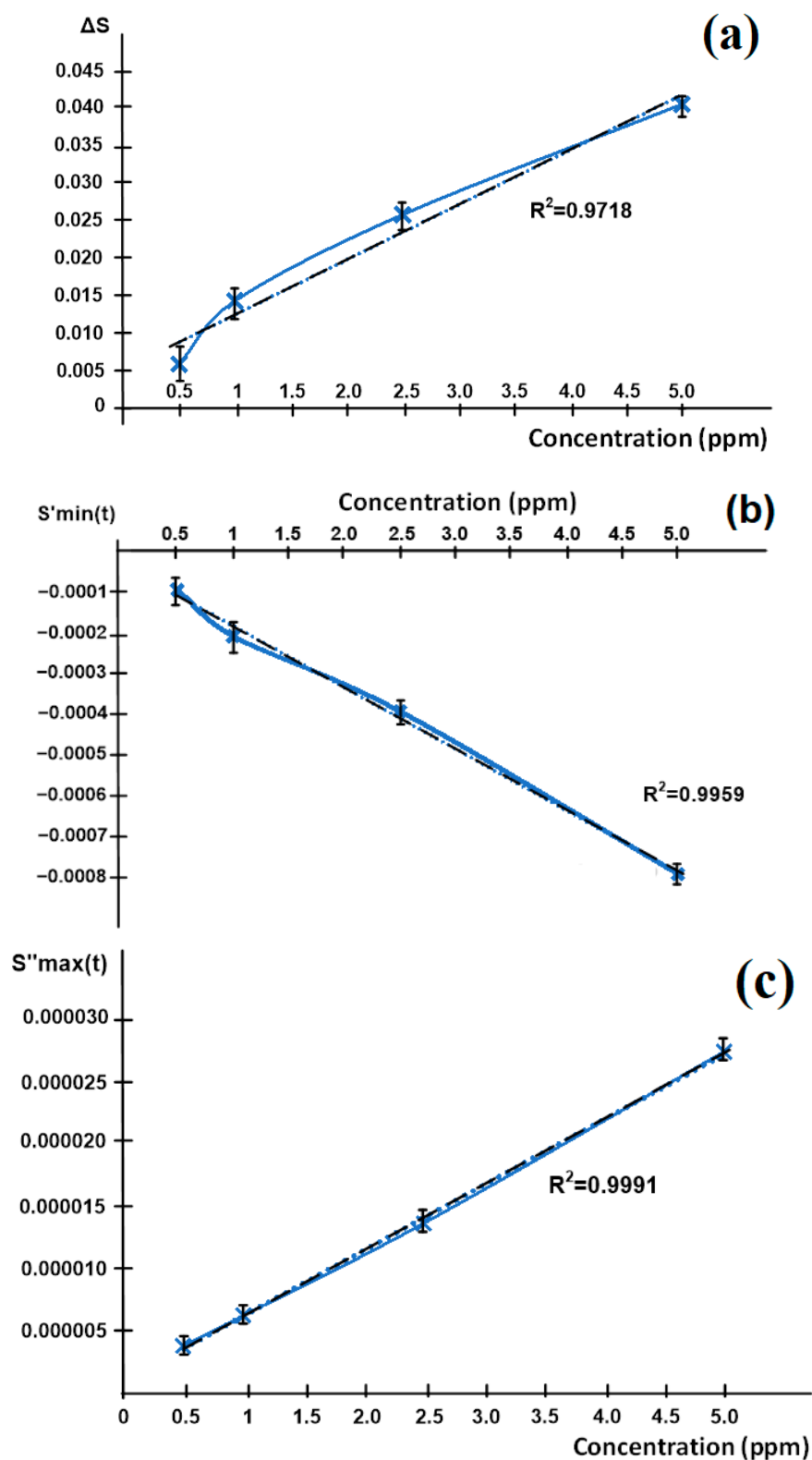


Figure 7. Calibration curves for NO_2 concentration estimation of the Type 2 sensor: (a) of the adsorption response ΔS at the saturation moment; (b) minimum of the first derivative $S'(t)$; (c) maximum of the second derivative $S''(t)$.

For example, the plotting of the calibration curves in ΔS vs. $\ln(t)$ or $\ln(\Delta S)$ vs. $\ln(t)$ does not lead to linearization over the entire concentration range since the adsorption mechanism can be different and vary depending on the concentration and composition of the gas mixture [38].

The monomolecular Langmuir adsorption mechanism prevails in the initial time; then, at higher concentrations, the adsorption mechanism can change to a polymolecular one, according to various scenarios, which, in any case, leads to the saturation and non-linearity of the $S(t)$ dependence. The reaction rate of the response at the initial time $S'_0(t)$ depends on the gas concentration [20], so it can serve as the sensor calibration curve. Figure 8 shows the calibration curves for $S'(t)$ at different time points. It is evident that linearity presents up towards the extremum for the $S'(t)$ function while the correlation coefficient of the approximation decreases.

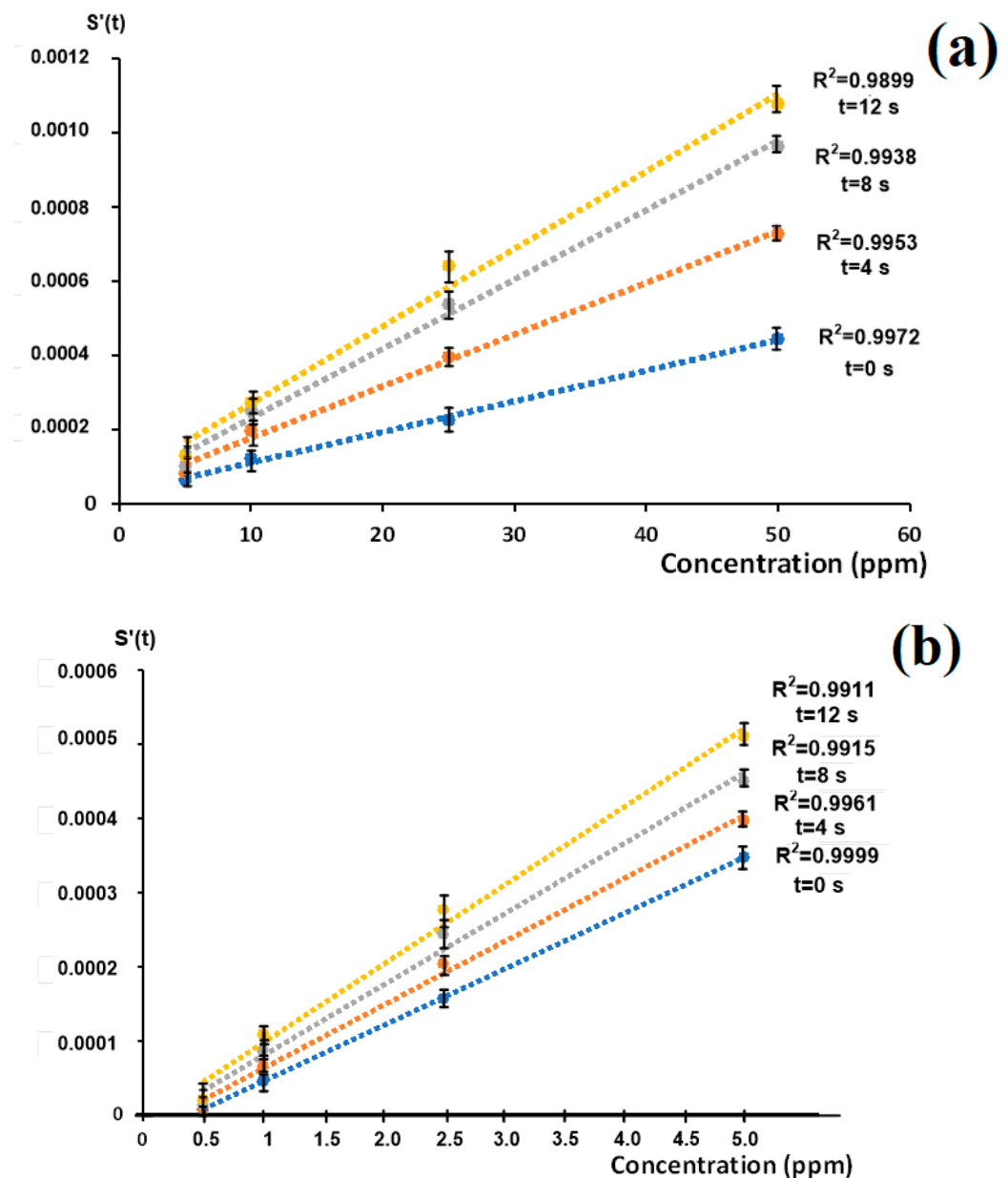


Figure 8. Calibration curves $S'(t)$ at different moments: (a) Type 1 sensor; (b) Type 2 sensor.

Figure 9 shows the graphs of the coordinates of the Elovich equation, ΔS vs. $\ln(t)$. At the initial moment, the dependence is not linear since the adsorption mechanism differs from the Elovich mechanism. It is possible to observe linear sections in the area of the extremum, $S'(t)$, with one slope and after $\ln(t) = 3.5$, with another.

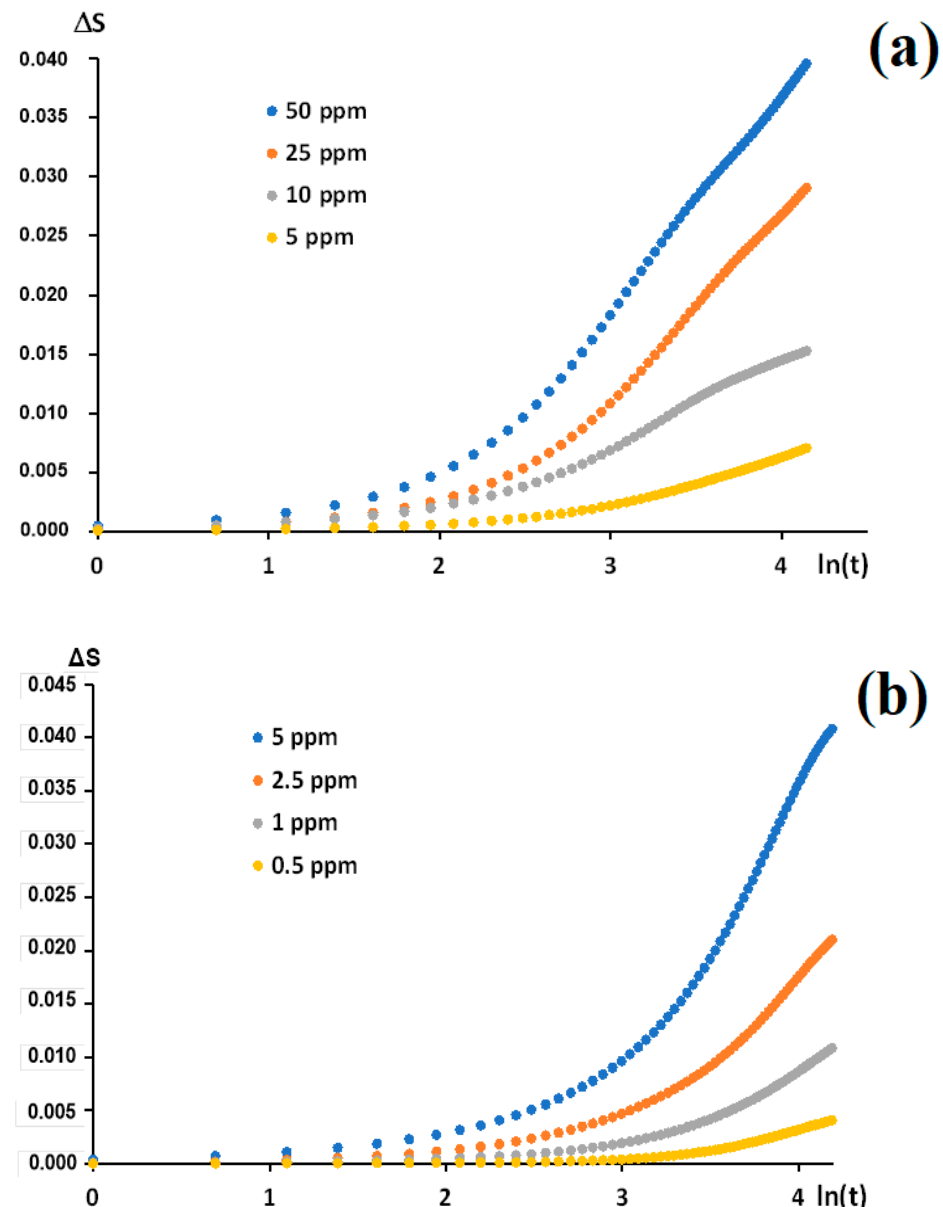


Figure 9. The dependences ΔS vs. $\ln(t)$: (a) Type 1 sensor; (b) Type 2 sensor.

The slope of the graph, ΔS vs. $\ln(t)$ in the area of the extrema, $S'(t)$, was taken to build the calibration curve. The resulting graph is shown in Figure 10.

Calibration dependences (Figures 8 and 10) evaluate the gas concentration by sensors before the saturation plateau of the response curve is reached, which significantly reduces the time of the response data reading and makes it possible to expand the upper limit of the sensor measurement range. Additionally, at the initial moment, the noise level is significantly lowered (in this work, it was lowered by up to 80%), and the error of noise elimination, at this segment of the response dynamics, is lowered [20].

Using various calibration curves, an experiment was conducted to check the error in estimating the gas concentration. The Type 1 sensor was exposed to 33 ppm of NO_2 , and

the Type 2 sensor was exposed to 1.5 ppm. The gas concentration was estimated by the response in various ways. The results are shown in Table 1.

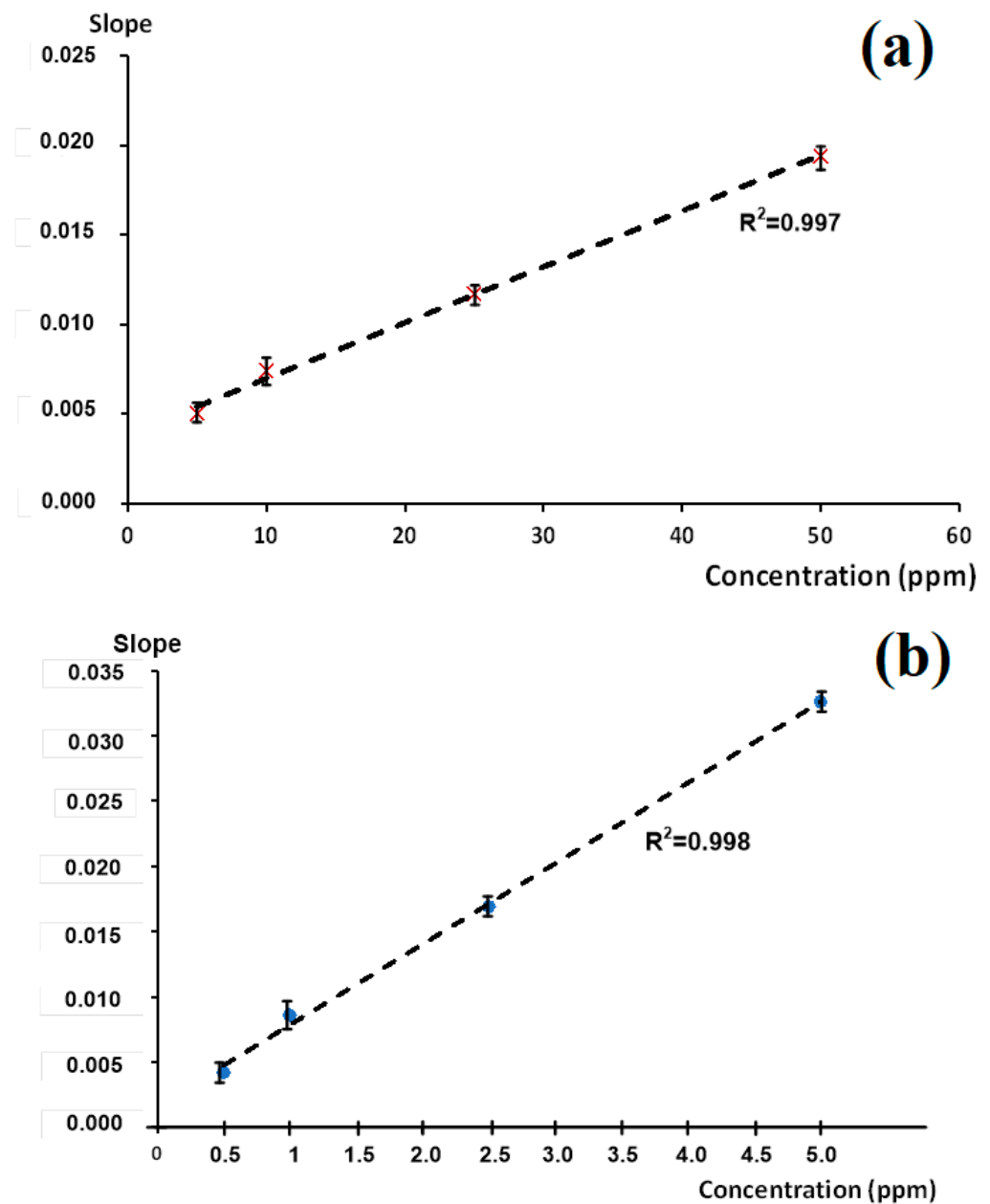


Figure 10. Calibration curve plotted from graphs ΔS vs. $\ln(t)$: (a) Type 1 sensor; (b) Type 2 sensor.

Table 1. The results of an experiment to verify the error in estimating the gas concentration using various calibration curves.

Calibration Method	by ΔS	by a Minimum of $S'(t)$	by a Maximum of $S''(t)$	by $S_0'(t)$	from Graphs ΔS vs. $\ln(t)$
NO ₂ concentration, ppm	36.1	34.8	33.3	32.5	33.5
Relative error, %	9.4	5.4	0.9	1.5	1.5
NO ₂ concentration, ppm	1.72	1.64	1.52	1.54	1.56
Relative error, %	14.6	9.3	1.3	2.6	4.0

The relative error was estimated. The smallest relative error was found when estimating the gas concentration by the calibration curve built from the maximum of the second derivative. In both cases, the worst result turned out to be for the traditional calibration

dependence built by ΔS at the moment of normalized response saturation; the relative error exceeded by about 10 times the smallest relative error obtained by using the calibration curve of the maximum of the second derivative. For the Type 2 sensor, the relative error was slightly higher than for the Type 1 sensor.

4. Conclusions

Such parameters as the extrema of the second derivative, $S''(t)$, the initial reaction rate to the gas exposure, $S_0'(t)$, the slope of the graph, ΔS vs. $\ln(t)$ in the area of the extrema, $S'(t)$, showed a high degree of linearity of the calibration curves and the lower standard deviations compared to the traditional fixed-time point method.

The proposed calibration curves at different sorption time intervals showed a high degree of linearity; at the initial moment, when mono-molecular adsorption takes place, this is the initial rate of reaction to the gas exposure. In the region of the first derivative maximum of the sensor response, the Elovich adsorption mechanism operates.

The calibration curves built from the values of the second derivative maxima turned out to be the most stable with the lowest relative error in estimating the gas concentration.

Interference can occur when implementing a measurement system in the gas sensors at any stage, so using the declared calibration methods will ensure the reliability of the system and reduce any error in determining the concentration of gas in the gas mixture.

The proposed signal processing scheme of the sensor and the calibration curves' plotting allow limited wait time for the onset of a steady-state mode when gas is supplied to estimate the gas concentration and reduce the analysis time. In the future, we will apply the methods based on the evaluation of several parameters of the sensor response dynamics for not only estimating the gas concentration, but also for recognizing the gas itself.

Supplementary Materials: The following are available online at <https://www.mdpi.com/article/10.3390/chemosensors10040126/s1>, Figure S1: The general signal processing scheme; Figure S2: The dependence of the Type 1 sensor resistance on the temperature (1) without exposure; (2) in the airflow; (3) in the nitrogen flow (the flow rate of air and nitrogen 110 mL/min).

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