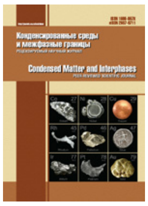

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Detection of acetone by a sensor based on clinoptilolite

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Abstract

Objectives: The active development of bioreactors used in a wide range of biotechnological, medical, agricultural, and environmental applications requires improvements in their components, including gas-sensitive sensors for various metabolites. Particular preference is given to sensors that are capable of recognizing the composition of complex gas mixtures without the use of bulky and expensive structures.

Experimental: Zeolite-based sensors are a promising technology. The ion conductivity relaxation curves of zeolites under the application of step voltage pulses are considered. The power dependence of the ion conductivity current over time is shown with parameters depending on the sorption of acetone vapors.

Conclusions: The results obtained demonstrate the possibility of using gas-sensitive sensors based on clinoptilolite for the detection of acetone, which is important for their use in microbioreactors.

Keywords: Clinoptilolite, Zeolite, Acetone, Gas-sensitive sensor, Microbioreactor, Ionic conductivity

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1. Introduction

Currently, there is active development of microbioreactor components that control and optimize the growth conditions of microorganisms, cells, and tissues. An important component of microbioreactors [1–4] are gas-sensitive sensors that allow the registration of microbial waste products [5, 6]. A distinctive feature of such sensors is their single-use nature, as well as increased requirements for miniaturization and cost.

Sensors based on natural zeolites meet these requirements [7–13]. The presence of a system of channels with a diameter of up to 1 nm in the crystal lattice provides sorption processes that affect the electrophysical properties of zeolites. The prevalence of zeolites in nature ensures their low cost. Of particular interest is the possibility of using a single sensor to detect a mixture of gases. This is especially important for microbioreactors, in which space is extremely limited in replaceable units with growth chambers, where it is difficult to place multisensors with a large number of sensitive elements. The most promising zeolite appears to be the most common natural zeolite, clinoptilolite, which has fairly large channels with a size of ~0.8 nm.

Impedance spectroscopy is most often used to record the response of zeolite-based sensors [14–17]. However, it is impossible to record a number of long-term processes associated with ion drift in zeolite channels [18]. The solution is to record the response to a step voltage function. Previously, it was shown that it is possible to isolate the response to ammonia vapor sorption against the background of water vapor sorption [19]. When using sensors in microbioreactors, it is also important to detect metabolites such as acetone or ethyl alcohol. They are more difficult to detect because they have large molecules that participate weakly in ion transport in zeolite channels.

The aim of this work was to find the response parameters of clinoptilolite-based sensors that enable the detection of acetone vapors for use in microbioreactors.

2. Experimental

A system of opposing pin electrodes (40×20 mm, electrode pitch 2 mm) with a layer of clinoptilolite was used as a sensor. The gas-sensitive layer was formed by precipitation from an aqueous suspension of zeolite without the use of additional binders (since the sensors in microbioreactors are disposable and do not require mechanical strength and durability).

The experimental design corresponded to that used previously in [19] for detecting ammonia vapor. To observe the ion conductivity relaxation process, a series of stepped pulses of constant voltage with variable polarity was applied to the electrodes. The pulse duration was 30 s, and the applied voltage amplitude was 5 V. To measure the current flowing through the zeolite layer, the voltage drop across a measuring resistor connected in series with the sensor was measured. The voltage drop across the resistor was first amplified by a precision instrument amplifier and then digitized using a 24-bit sigma-delta ADC with a sampling frequency of 20 Hz, which allowed for hardware-based suppression of the 50 Hz mains interference. The obtained data was smoothed using a 5-point moving average and transmitted to a computer. As a result, the noise amplitude in the obtained data did not exceed 0.1 nA.

The sensor was placed in a desiccator, the humidity inside which was set by a saturated salt solution (NaCl – to obtain a relative humidity of 75 % and KCl to obtain a relative humidity of 84 %). The relative humidity and temperature were additionally controlled by sensors with an accuracy of 1 % and 1 °C, respectively. Before being placed in the desiccator, the sensor was heated to a temperature of ~80 °C for desorption. The efficiency of desorption was monitored by measuring ion conductivity. After placing the sensor in the desiccator, it was kept at a constant temperature (20 °C or 25 °C) for 24 hours to achieve equilibrium. At the beginning of the experiment, the results were recorded for 1 hour without adding acetone vapors, after which a solution of acetone was fed into the desiccator, providing a vapor concentration of ~1000 ppm (which corresponds to the threshold of acetone perception by the human sense of smell) or ~50000 ppm (which roughly corresponds to the average lethal concentration of acetone for animals). The results were recorded for ~4 hours.

3. Results and discussion

An example of measuring ion conductivity current when applying pulses of variable polarity is shown in Fig. 1a. According to the results presented, the experiment can be divided into four stages (marked in Fig. 1a with Roman numerals I-IV). In the first stage, before the introduction of the acetone solution into the desiccator, the amplitude of the response to step pulses is constant. In the second stage, after the introduction of acetone, the amplitude of the response begins to decrease. In the third stage, the amplitude of the response begins to increase again, and in the fourth stage of the experiment, it reaches saturation (at this point, the amplitude exceeds the initial amplitude corresponding to the absence of acetone vapors).

Fig. 1b shows a fragment of the time series corresponding to the second stage of the experiment, and Fig. 1c shows a fragment of the relaxation of ionic conductivity in double logarithmic coordinates. The classical Curie-von Schweidler power law [18] is observed, with a change in the exponent at a certain point in time, which is typical for zeolites, in which the main contribution is made by ion conductivity in the crystal lattice channel system [18, 20].

The values of the degree indicators obtained in the experiment are shown in the table. The values of indicator n1 correspond to the initial stage of conductivity relaxation (~2.5 s from the moment of a step change in the applied voltage), while indicator n2 corresponds to the final stage of relaxation (time interval ~20÷30 s). As can be seen, the introduction of a high concentration of acetone vapors is accompanied by a decrease in the n1 index during all stages of the experiment. In contrast, the n2 index first decreases in value and then increases. At the same time, the ratio of n2/n1 always increases from the moment acetone is introduced. This behavior persists both when the temperature of the experiment is changed and when the relative humidity is changed.

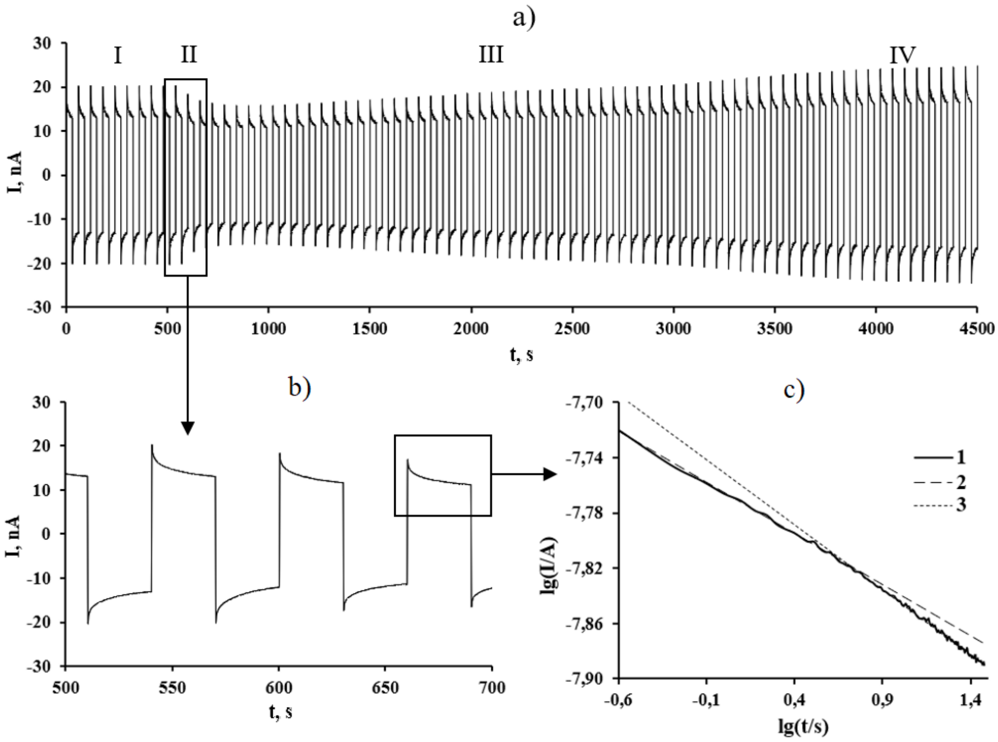

Fig. 1. Response of a clinoptilolite-based sensor to step voltage pulses in the absence of acetone vapors (I) and in the presence of acetone vapors (II–IV): a) time dependence of ion conductivity current; b) time dependence of ion conductivity current at the moment of acetone vapor introduction; c) power-law relaxation of ion conductivity: *1* – experimental curve in double logarithmic coordinates; *2* – approximating power-law dependence for the interval $t < 2.5$ s; *3* – approximating power-law dependence for the interval $20 < t < 30$ s

Table. Values of exponents for ion conductivity relaxation curves of clinoptilolite

Acetone vapor content, ppm	*T*, °C	%RH	Experiment stage	n1	n2	n2/n1
~50000	25	75	I	0.081	0.108	1.33
			II	0.070	0.095	1.36
			III	0.073	0.104	1.42
			IV	0.072	0.102	1.42
		84	I	0.088	0.124	1.41
			II	0.076	0.113	1.49
			III	0.075	0.114	1.52
			IV	0.075	0.125	1.67
	20	75	I	0.085	0.102	1.20
			II	0.073	0.088	1.21
			III	0.074	0.094	1.27
			IV	0.076	0.100	1.32
~1000	25	75	I	0.075	0.098	1.31
			II	0.076	0.101	1.33
			III	0.080	0.118	1.48
			IV	0.078	0.120	1.54

At low acetone concentrations, the ratio n2/n1 continues to increase with the introduction of acetone, but this is caused not by a decrease in the n1 index, but by an increase in the n2 index.

Compared to the significant change in degree indicators obtained earlier during ammonia vapor sorption [19], the change in degree indicators during acetone sorption is significantly less. This is due to the fact that ammonia molecules, firstly, have a significantly smaller diameter and, therefore, greater mobility in the pores of zeolite compared to acetone. Second, ammonium ions formed in the zeolite pores are directly involved in ionic conductivity. In the case of acetone, the effect on conductivity appears to be indirect (a change in the mobility of water and metal ions in the zeolite pores, as well as a change in the number of water molecules in the pores).

4. Conclusion

The detected change in the power indices of the clinoptilolite conductivity relaxation curve in the presence of acetone vapors allows us to consider zeolite-based sensors as being promising components of microbioreactors. It has been shown that the ratio of exponents during different stages of ion conductivity relaxation can be used as an important parameter for acetone detection.

Author contributions

All authors have made an equivalent contribution to the preparation of the publication.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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