European Journal of Emerging Technology and Discoveries
ISSN (E): 2938-3617
Volume 1, Issue 9, December, 2023.

ANALYTICAL CHEMISTRY ZOL-GEL IS PRODUCED ON THE BASIS OF PROCESSES AMMONIA USES NANOMATERIALS CREATION OF SELECTIVE GAS SENSORS

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Abstract
Today, the use of selective methods and sensitive sensors is rapidly developing in industry, in solving environmental problems, especially in solving the problem of atmospheric air monitoring, which has arisen worldwide as a result of the rapid development of chemistry, oil and gas. chemistry. Semiconductor sensors (SPDs) are sensitive devices, and their main advantages are ease of use, small size, long working life, high accuracy and speed. Semiconductor gas sensors based on nanomaterials obtained using sol-gel processes, sol-gel technology for the manufacture of ammonia semiconductor sensors, and studies of the hydrolytic polycondensation of TEOS during the synthesis of gas-sensitive nanocomposite films have been studied.

Keywords: semiconductor sensor, catalyst, selectivity, sensing elements, iron oxide, titanium oxide, ammonia, tetraethoxylane.

Introduction

Over the years of independence, a number of new industrial enterprises have been launched in our country, based on the introduction of modern technologies in various industries, modernization and production of new types of products based on them. Gas-sensitive materials based on SnO_2, TiO_2, ZnO, In_2O_3, MoO_3, Fe_2O_3, WO_3 and V_2O_5 are important in the development of semiconductor sensors, which are widely used to monitor gas mixtures in these plants. A special place among them is occupied by gas-sensitive materials (GCM) obtained using sol-gel technology based on TiO_2 and Fe_2O_3, which are very important in terms of their characteristics. In accordance with the Action Strategy for the Further Development of the Republic of Uzbekistan, the creation of selective semiconductor gas sensors based on sol-gel technologies is important in the development of the chemical industry.

The need for environmental monitoring of ambient air is growing all over the world, especially in sectors of the economy, especially in transport, energy and industry. In particular, the improvement of existing analytical methods and tools, the creation of new semiconductor highly sensitive sensors, and the scientific substantiation of the processes of selective gas-sensitive materials. Particular attention is paid to the search and development of high-performance semiconductor sensors that determine their metrological, analytical and operational characteristics.

Objective. Study of the formation of thin-film sensor elements containing SiO_2 / TiO_2 and the creation of selective semiconductor gas sensors that determine the concentration of ammonia using nanomaterials based on sol-gel processes.

Objectives of the study: to determine the regularities of the process of synthesis of a gas-sensitive film based on tetraethoxysilane, the composition of the initial components of the process, the ratio and temperature-time regime;

development of the technological sequence of the process of formation of gas-sensitive material, production of gas-sensitive material containing SiO_2: TiO_2, and creation of sensor elements on its basis;

Analytical analysis of the data presented in the literature shows that the number of studies devoted to ensuring the selectivity of the process for determining ammonia from a mixture of gases is limited. Existing sensors in the field of ammonia detection make it possible to determine their high pre-explosion concentration. Optical, electrochemical and thermal conductometric sensors and analytical methods for gas detection are now widely studied. However, these sensors have a number of drawbacks, the most important of which is that they are not selective enough, the signal size depends on external factors. Based on the above, it can be said that the development of new, improved and modern semiconductor methods and sensors for detecting toxic and explosive components of gas mixtures remains one of the most pressing problems in the field of environmental safety.

Studies of the influence of the composition of gas-sensitive material (GCM) on the speed, sensitivity and selectivity of semiconductor sensors (PPS) have been systematized and theoretical aspects have been analyzed. The principle of operation of semiconductor sensors, their main characteristics, advantages and disadvantages are considered. The works of foreign

ISSN (E): 2938-3617
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and domestic researchers in the field of the results of the use of catalysts in increasing the selectivity of semiconductor sensors are analyzed. The analysis of the cited works in the literature made it possible to choose the goal, objective and object of research of this work.

The regularities of the formation of gas-sensitive films of metal-oxide compounds have been studied. The convenience of LDPEs lies in their small size and high sensitivity. However, for some components of the mixture, their selectivity is not high enough. In practice, the selectivity of LDPE is achieved by adding a catalyst to the composition of the HCM at the stage of its preparation and selecting the optimal temperature of the sensor. To select a catalyst that ensures the selectivity of the PM, the activity of metal oxides in the the process of oxidation of ammonia by oxygen in the air. According to the results obtained, the decrease in the activity of the studied oxides in the oxidation of ammonia corresponds to the following order: Fe2O3> MnO2> CoO> Cr2O3> NiO> V2O5> CuO> MoO3 (experimental temperature 350 $^{0C. Fe2O3,}$ MnO2, CoO and Cr2O3 are among the catalysts with high activity in this process. Subsequent experiments were carried out in the presence of binary mixtures of these oxides with titanium oxide. These studies have shown that that Fe2O3 and TiO2 have the highest activity and selectivity in the ammonia oxidation process.

Subsequent experiments investigated the relationship between the components and the effect of temperature on the activity and selectivity of the mixture of Fe2O3 and TiO2 in the process of oxidation of combustible gases.

The relative ratio of Fe2O3 and TiO2 was studied in the range from 1:99 to 10:90 depending on the capabilities of the sol-gel technological method of PM formation. Based on the results obtained, it was determined that the optimal values of temperature and GSM in the process of ammonia determination correspond to 350 0S and 10Fe2O3 + 90TiO2, and under these conditions almost complete (99.8%) oxidation of ammonia is observed. From the experiments carried out, the following material was selected as a HCM for selective LDPE, containing 10Fe2O3 + 90TiO2. A PM with this content provides a high selectivity in the ammonia detection process. In this work, the design of a LDPE sensing element consisting of a heater in the form of a cylindrical spring and a part transmitting the signal passing through it is proposed and used in practice. The appearance of the developed sensor consists of a reaction chamber (1) and a gas-permeable mesh (2) covered by its apex (Fig. 1).

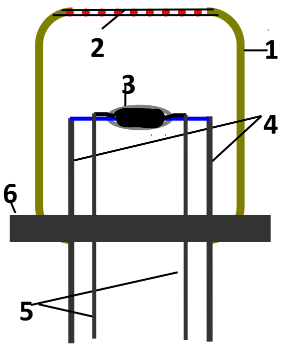

Figure 1. Diagram of a semiconductor ammonia sensor. 1st Reaction Chamber, 2nd Gas Pipeline Grid, 3rd Gas Sensing Element, 4th Signal and Transmission Contact, 5th Heater Live Contacts, 6th Housing.

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The gas-sensing element of the sensor (3) is made of platinum glass wire in the form of a microtube (12-ring spring) and the surface is coated with GSM.

The minimum dimensions of the sensing element ensure its resistance to mechanical stress. The platinum wire gas sensing element with glass coating uses a current of 50-70mW to detect ammonia at a constant temperature.

This, in turn, allows the LDPEs to be used in small, battery-powered, stand-alone devices. A gas-sensitive layer consisting of 90% TiO2-10% Fe2O3 was applied to the surface of the heating coil using sol-gel technology. The design used increases the stability and reproducibility of the ammonia-detecting LDPE signal. Platinum fiber coated with glass fiber (TU 610664-018) was used in the manufacture of the heating coil for the sensor sensing element. The appearance of the developed sensor (b) and the prepared LDPE samples (b) is shown in Figure 2. This compact design allows for easy gas exchange of the reaction chamber with the external environment, allowing the sensor to be placed in the gas intake channel of the gas analyzer.

a

B

Figure 2. Appearance of the developed sensor (a) and prepared LDPE samples (b).

When the voltage is supplied from an alternating current source, the signal of the sensor (Uoutput) is equal to the difference (Uout=Ur- Uv) of the signals of the sensing element in the fresh air medium (Uv) and in the gas. medium mixture (your). The technology of preparing a gas-sensitive coating consists of three stages.

First of all, you need to prepare an alcohol solution of TEOS. The second stage is the preparation of an aqueous solution of the dopant modifier, which is part of the sol. To do this, an appropriate amount of titanium salt is dissolved in distilled water and the required amount of 30% hydrochloric acid is added to this solution. The third step is to prepare the sol with all the components. To do this, the solution containing the second additive is gradually added to the first solution by stirring. Coating with a gas-sensitive layer and a catalyst is carried out in a special device. In this process, the uniformity of the structure and the thickness of the titanium oxide coating are controlled.

According to the "Study of hydrolytic polycondensation of TEOS in the synthesis of a gas-

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sensitive nanocomposite film", the regularities of the process of formation of a gas-sensitive material based on TEOS and TiO2 for ammonia LDPE were studied. The initial solution, which forms a nanocomposite film based on TiO2, consists of TEOS, water, an organic solvent, a catalyst and the corresponding metal salt. Important parameters in GSM synthesis include the concentration of the starting material, temperature, pH, and the method of mixing the components. Therefore, in experiments based on TEOS, an alloying additive was added and the effect of the above parameters on the properties of the solution without a dopant impurity was studied. Ratio of raw materials in the optimization of the sol-gel process of gas-sensitive material synthesis technology: Si (OC2H5)4: H2O: ROH: HX = (1-4) :(1-40) :(1 -45) :(0,01-0, 3). In this case, ROH is simple alcohols, HX is acid. TEOPs and aliphatic alcohols (ethanol, propanol-2 and isobutanol) were used as organic solvents, which are good solvents for most salts. An increase in the amount of alcohol in the solution, regardless of the solvent content in the test range, leads to a decrease in its viscosity and density. The dependence of the kinetics of the viscosity change of the ethanol-containing solution on the duration of the gelation process is shown in Figure 3.

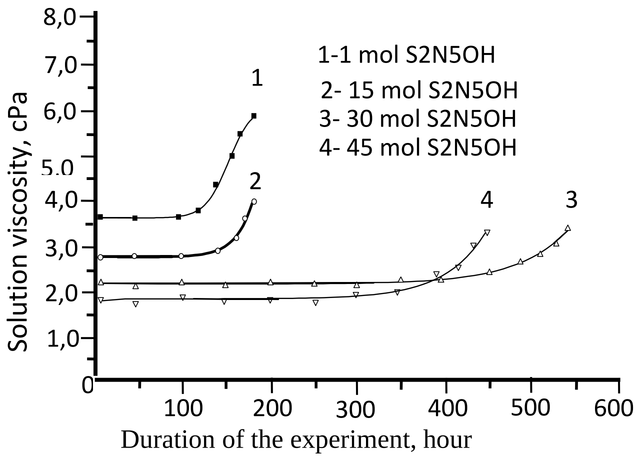

Figure 3. Graph of the viscosity of the solution at different C2H5OH/TEOS ratios on the duration of the experiment. Quantity in solution (mol): TEOS-1; water-20; HCl-0.05.

The maximum stability of the solution in the ethanol environment corresponds to the C2H5OH/FEOS ratio of 30. At this ratio, the solution remains stable for 450 hours, during which it can be used in the manufacture of a gas-sensing element of nuclear fuel. The results of the study of the effect of the amount of water in the solution on its properties are given in Table 1.

Table 1. Influence of water content on the properties of the solution of the mixture TEOS-H2O-HCl-ethanol forming a gas-sensitive film

T/R	Solution composition, moles				Mortar properties		
	TEOS	H2 O	HCl	i-Butanol	Density g/cm3	Electricity conductivity, mSm.	Viscosity spa
1	1	1	0,05	30	0,8248	9,6	1,7
2	1	10	0,05	30	0,8365	10,4	2,1
3	1	20	0,05	30	0,8578	16,5	2,3
4	1	30	0,05	30	0,8631	18,5	2,4
5	1	40	0,05	30	0,8684	20,0	2,6

The maximum stability of the solution is 445 hours, which corresponds to the ratio of H2O / THEOS = 20. The results of the study of the effect of the amount of TEOS in the solution on the stability of the sol in the range of 1-4 mol showed that the synthesis of HCM at a low concentration of TEOS in the solution, equal to 1 mol, provides high stability of the solution. Such a concentration of TEOS in solution makes it possible to obtain a homogeneous gel with high stability, without signs of sedimentation.

The effect of the pH value of the solution medium on its stability was studied at the ratio of TEOS:HCl in the range from 1:0.01 mol to 1:0.30 mol. The results of the experiment showed that the optimal ratio was when the amount of HCl corresponding to 1 mol of TEOS in solution was equal to 0.05 mol. When the acid concentration exceeds the optimal value, the stability of the solution decreases. The optimal condition for the production of HCl corresponds to the ratio of HCl: TEOS = 0.05, which ensures the highest 450-hour stability of the solution. The study of the influence of the composition and ratio of the components of the film-forming solution on the kinetics of the gelling process made it possible to determine the most optimal ratios that ensure the stability of the initial solution. In the experiments, the highest value was the stability of the solution at the ratio of the initial components of the FEAS: H2O: alcohol: HCl = 1: 20: 30: 0.05.

The introduction of TiO2 into the silicate matrix makes it possible to obtain a selective gas-sensitive nanocomposite material with high sensitivity to a semiconductor ammonia sensor. TiCl4 salt was used as a source of TiO2. In the course of the study, the effect of the dopant content on the viscosity and stability of the film-forming solution was investigated. The results showed that the dynamic viscosity of the dopant solution (2.6-3.8 cPa) was higher than the viscosity of the dopant solution (2.1 cPa). It has been observed that the stability of solutions containing doppant is low compared to the stability of a solution without an impurity of the same composition.

In the process of synthesis of a gas-sensitive film, the effect of solution temperature on its properties in the temperature range of 20-60 0C at atmospheric pressure was studied. The results showed that an increase in temperature from 20 to 40 °C leads to a 2.5-fold decrease in the stability of the solution.

In experiments, conductometry was used along with viscometry to control GSM synthesis. Conductometric observation of the kinetics of the maturation process of doped and undoped solutions has shown that its high stability can be ensured by changing the rate of hydrolysis and polycondensation of the film-forming solution as a result of changes in the composition and quantity of added components. . The inert surface was covered with a film by immersing the coated sample in a film-forming solution. The peculiarity of the TEOS-based film is that after the solvent evaporates, a xerogel film is formed on the surface of the inert substrate, consisting of a modified polysiloxane matrix of molecules of a modified compound consisting of Ti oxide. The film was formed by drying it at 20-1200 °C (for 60 minutes) and heat treating it at 370, 450 and 550 °C. The best results were obtained when the TiO2-based film was heat treated for 25-30 minutes at each temperature. An increase in the heat treatment time from it leads to a decrease in the degree of porosity of the film. In conclusion, on the basis of the studies carried out, it should be noted that the properties of materials obtained by the sol-gel method largely depend on the initial composition of the mixture and the conditions for obtaining the film.

Findings

1. The effect of the composition and ratio of the components of the initial solution on the kinetics of the sol-gel of the process of formation of a selective semiconductor gas-sensitive material based on tetraethoxylane (TEOS) in a wide range of concentrations and temperatures has been studied. TEOS Composition: H_2O: Ethanol: HCl = 1:20:30:0.05 The highest stability of the respective solution has been determined. The optimal temperature-time regime (450 °C and 30 min) for the synthesis of SiO_2-TiO_2-containing gas-sensitive films was chosen. Based on the results of the study, a method for controlling the sol-gel process of obtaining gas-sensitive material for a high-sensitivity NH_3 sensor was recommended.
2. A method for the formation of gas-sensitive materials based on titanium and iron oxides by the method of sol-gel technology has been developed. Thin-film samples containing TiO_2-Fe_2O_3 were obtained for the creation of selective NH_3 sensors. The structure of the film consists of a two-layer coating, the first layer is a TiO_2-based layer, the base of which is completely and continuously dependent on the composition of the original sol, and the second layer is a layer consisting mainly of the catalyst Fe_2O_3.
3. Taking into account the development trend and technical conditions of gas sensors, the sensor design of the NH_3 detector is a spiral of glass-coated platinum wire, the surface of the sensing element is covered with a gas-sensitive material based on Titanium and iron oxides are proposed and applied using sol-gel technology. The size of the sensing element and the low thermal conductivity of the platinum material transmitting the signal made it possible to reduce the voltage supplied to the sensor to 100 mW. This allows the sensors to be used in the production of small, battery-powered devices.

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