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MEMBRANE STRUCTURE OF ION-SELECTIVE ELECTRODE FOR DETERMINING CADMIUM ION, ANALYZED BY PHYSICOCHEMICAL METHODS

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Abstract. Currently, ion-selective electrodes are used to control the composition of process solutions in various industries, and to analyze ultrapure water. They have also found application in medicine, as they meet the requirements of biomedical research and can be used to control the quality of food products, such as milk and dairy products. However, ISEs are most widely used to analyze wastewater in various fields. Despite the diversity of existing electrodes, an urgent task is to study the composition and structure of membranes of new, more efficient, stable, sensitive, reliable, and durable electrodes.

Keywords: electrode active material, heavy metals, polyvinyl chloride.

Fizik-kimyoviy usullar yordamida kadmiy ionlarini aniqlovchi ion-selektiv elektrod membranasining tuzilishi

Annotatsiya. Hozirgi vaqtda ion-selektiv elektrodlar turli sohalarda texnologik eritmalar tarkibini nazorat qilish va toza suvni tahlil qilish uchun ishlatiladi. Ular tibbiyotda ham qo'llanilishini topdilar, chunki ular biotibbiyot tadqiqotlari talablariga javob beradi va sut va sut mahsulotlari kabi oziq-ovqat mahsulotlarining sifatini nazorat qilish uchun ishlatilishi mumkin. Biroq, ISElar turli sohalarda oqava suvlarni tahlil qilish uchun eng keng tarqalgan. Mavjud elektrodlarning xilma-xilligiga qaramasdan, yangi, samaraliroq, barqaror, sezgir, ishonchli va bardoshli elektrodlarning membranalarining tarkibi va tuzilishini o'rganish dolzarb vazifadir.

Kalit so‘zlar: elektrod faol moddasi, og'ir metallar, polivinilxlorid.

Структура мембраны ионселективного электрода для определения ионов кадмия физико-химическими методами

Аннотация. В настоящее время ионселективные электроды используются для контроля состава технологических растворов в различных отраслях промышленности, анализа сверхчистой воды. Они также нашли применение в медицине, так как отвечают требованиям биомедицинских исследований и могут использоваться для контроля качества пищевых продуктов, таких как молоко и молочные продукты. Однако наиболее широкое применение ИСЭ получили для анализа сточных вод в различных областях. Несмотря на многообразие существующих электродов, актуальной задачей является изучение состава и структуры мембран новых, более эффективных, стабильных, чувствительных, надежных и долговечных электродов.

Ключевые слова: активный материал электрода, тяжелые металлы, поливинилхлорид.

Introduction. Numerous studies are being conducted worldwide to create highly efficient ion-selective materials (ISM) for ion-selective electrodes (ISE) of medicinal substances. In the conditions of analysis outside the laboratory, potentiometry using ISE is an alternative to labor-intensive and expensive methods such as high-performance liquid chromatography, capillary electrophoresis and spectral analysis due to its speed, simplicity and availability. In this regard, the creation and study of new highly efficient ISE based on accessible and inexpensive electrode-active substances is one of the important tasks. Such electroactive substances include phosphomolybdic, phosphotungstic and other heteropoly acids and their derivatives, which are easily accessible and, in some cases, significantly exceed natural ionophores in selectivity [1-3].

Heavy metal toxicity is a clinically significant condition that, if not properly treated, can cause serious illness and death. Heavy metals can be ingested, inhaled, or absorbed through the skin. The extent of damage and severity depend on the type of metal, the route of entry, the patient's age, and the level of toxicity [4-6].

Ion-selective membrane electrodes (ISE) play an important role in chemical analysis due to their simplicity, speed and accuracy compared to other analytical methods. Their high selectivity allows them to be used to determine a variety of organic and inorganic compounds without prior separation [7].

In this study, PVC matrix-based ion-selective membrane electrodes were developed and characterized for the determination of Cd^{2+} cation [8].

In recent years, the need to determine heavy metals has increased due to the aggravation of environmental problems. Cadmium is known as one of the most toxic elements. Cadmium toxicity is due to its interaction with enzymes and nucleic acids, which leads to biochemical disturbances, which are often the source of physiological abnormalities [9-12]. Cadmium damages the kidneys, liver, and lungs of humans, and also causes high blood pressure and the destruction of red blood cells. Zinc is widely distributed in the environment and is vital. The human body contains about 2 g of zinc, but its maximum permissible daily dose is 1 mg / kg of body weight. Large doses of this metal can cause fever, chills, gastroenteritis, and pulmonary symptoms. In addition, zinc can cause anemia, nausea, vomiting, kidney failure, and damage to internal organs [13-15].

Method of research. Rapid and selective methods for the determination of such metals are needed. Potentiometric ion-selective electrodes (ISEs) are known to offer an affordable means for the selective, sensitive and rapid determination of various analytes in a wide range of applications. The use of ion-selective electrodes in environmental analysis and monitoring, as well as for clinical and industrial purposes, is constantly expanding. Electrodes with solid internal contact are a type of ISE in which the internal reference electrode is in direct contact with the electroactive membrane without the use of an internal solution [4]. These electrodes have certain advantages over traditional ones, such as compact size, low production cost and the ability to work under high-pressure conditions where traditional ISEs may be damaged.

Applications of ion-selective electrodes:

1. Determination of various ions in aqueous solutions.
2. Measuring the pH value of solutions.
3. Monitoring of pollutants in natural and waste waters by measuring CH–, F–, S–, Cl–, NO_3– and other ions.
4. Measurement of different types of ions in soil.
5. Determination of NO_3^- and NO_2^- ions in meat preservatives, as well as the salt content in meat, fish and fruit juices.
6. Detection of fluoride ions in drinking water and other beverages.
7. Determination of ions such as Ca^{2+}, K^+, Cl^- in body fluids including plasma, serum and sweat.
8. Use as chemical sensors for research purposes.

The presence of heavy metals in the environment is of great concern due to their toxicity to humans. Until now, the quality control of surface and groundwater has been based on reliable but expensive laboratory analytical measurements. Thus, the need for reliable, sensitive and inexpensive online monitoring systems remains relevant. Interest in the use of electrochemical sensors based on selective glass membranes for miniature devices has led to their extensive research in recent years. Chalcogenide glasses have proven their promise as materials for chemical sensors due to their flexible structure, great variability and virtually unlimited doping and alloying ability. In many cases, chalcogenide glasses demonstrate good chemical stability, high selectivity and reproducibility.

Experimental part. Ionic compound cadmium phosphomolybdate (Cd- PH MA): To prepare the compound, take 100 ml of 0.01 M phosphomolybdic acid solution and 100 ml of 0.03 M $Cd(NO_3)_2 \cdot 4H_2O$ solution. Mix both solutions in a 1:1 ratio in a 200 ml beaker. Leave the resulting precipitate for 8 hours, then filter through a Schott filter (pore size 40 nm).

After filtration, the precipitate is washed repeatedly with distilled water until the chloride ions are completely removed. It is then air-dried at room temperature for 24 hours. The final product is a powder.

These methods allow obtaining compounds with high purity and homogeneity, which ensures reproducibility in further research and application.

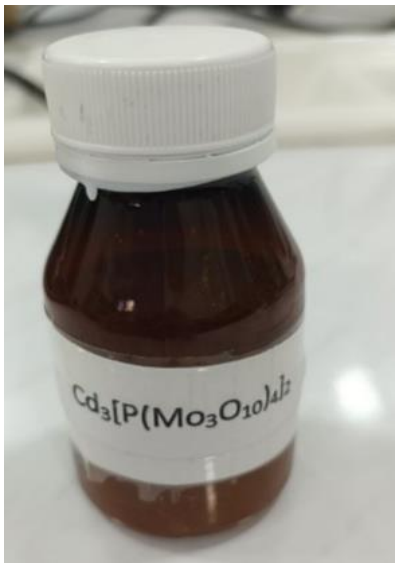

Figure 1. Cadmium phosphomolybdate associate

Method of preparation: 1. 0.6 ml of dibutyl phthalate and 0.315 g of polyvinyl chloride were added to 3 ml of tetrahydrofuran solution; 2. The resulting mixture was thoroughly stirred on a magnetic stirrer in a closed state for 1 hour.

This process produces a membrane material with a uniform structure, which is subsequently used as the basis for a cadmium-selective electrode.



Figure 2. Membrane preparation process

Method of preparation: 1. Add 0.05 g of nickel molybdophosphate to the mixture and continue stirring for another 1 hour; 2. Pour the resulting solution into specially prepared containers of different sizes and leave to dry for 24 hours.

This process allows obtaining a stable membrane with the necessary physical and chemical properties for further use in electrochemical sensors.

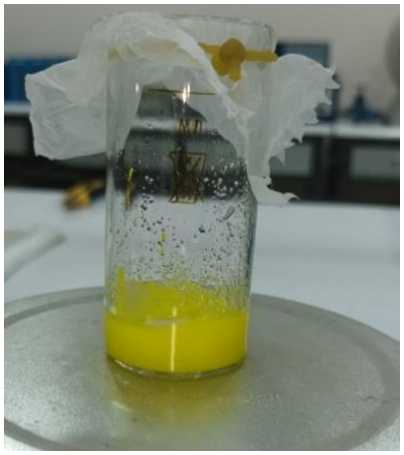

Figure 3. Electrode preparation and drying: The resulting membrane is used to prepare the electrode, which is then dried for another 3 days

Experimental equipment and procedure: Morphology analysis: The morphology of the ISE membrane samples obtained on the surface of the printed electrode was studied using a Supra 50VP (LEO) scanning electron microscope.

Spectroscopy: Combined scattered spectra of ionic salts were recorded using a Renishash Raman Microscope. Lasers with wavelengths of 514 and 633 nm were used as excitation sources. Recording was carried out at room temperature. The spectral resolution of the spectrometer was 1 cm-1.

Raman vibration spectra: Vibration spectra of coordinate complexes contain high-frequency vibrations (4000-600 cm-1) of ligands and low-frequency vibrations (below 600 cm-1) of metal-ligand bonds. The former provide information on the influence of the ligand's electronic structure on coordination, the latter on the structure of the coordination region and the nature of the metal-ligand bond.

Infrared Spectrometer Analysis: All data were obtained using the IQ-Fure IRAffinity-1S infrared spectrometer from Shimadzu. This spectrometer enables the synthesis and study of organic

compounds, determination of the structure of organic substances, synthesis and study of complex compounds, study of substances in inorganic chemistry, study of the properties of nanomaterials in materials science, and the development of optical sensors with high sensitivity and selectivity for various gases in analytics.

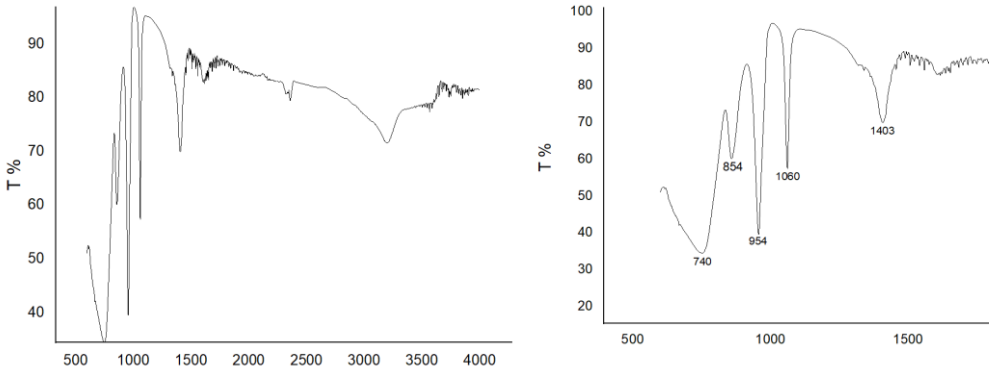

Figure 4. Infrared spectrometry results**.**

From the infrared spectrometry results of the cadmium-phosphomolybdate ion complex, only the characteristic regions were extracted and the corresponding frequencies of their peaks were determined. The obtained peaks and their corresponding characteristic groups are shown in the following table.

From the infrared spectrometry results of the cadmium-phosphomolybdate ion complex, only the characteristic regions were extracted and the corresponding frequencies of their peaks were determined. The obtained peaks and their corresponding characteristic groups are shown in the following table.

Explanations: 4000-3000 cm^{-1}: This range indicates O - H or N - H bonds present in the molecule; 1600-1400 cm^{-1}: C = O and C = C bonds may be visible in this range ; 1200-1000 cm^{-1}: Phosphoryl and molybdenyl oxides, as well as Mo = O bonds, are observed in this region ; 700-600 cm^{-1}: This range indicates possible molybdenum oxide or sulfide bonds (Mo - O or Mo - S). This analysis allows a more accurate understanding of the structure of the cadmium phosphomolybdate ionic complex, since each peak and its corresponding group reflect the chemical structure of the material.

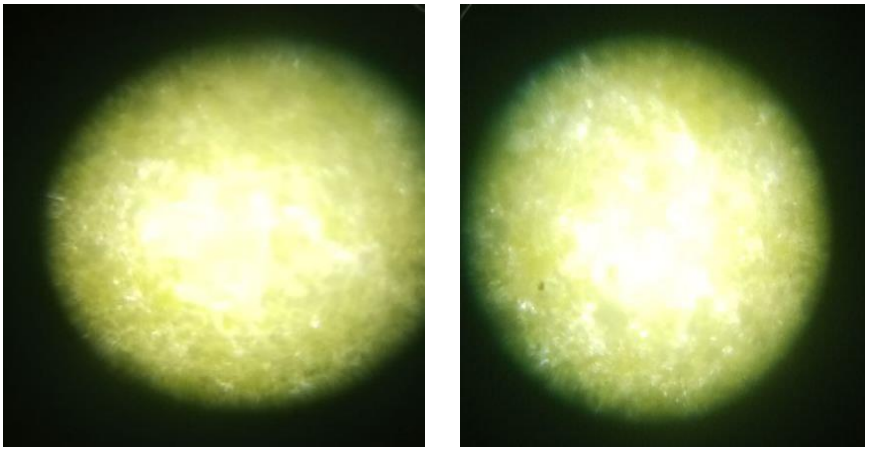

Figure 5. Optical microscope images of membrane and ionic fortus and their discussion.

Image of the membrane of a cadmium selective electrode taken with an OPTIKA optical microscope B -150. In this case, the microscope objective size is 40/0.65-160/0.17, which means the membrane is magnified by 40-160 times. The image shows that the internal structure of the cadmium phosphomolybdate film is presented in an amorphous greenish-yellow state.

Conclusion. This section presents the results of the analysis and discussion of the ion fortues used in the membranes, as well as the results obtained from their study using modern analytical methods. It should be noted that the results obtained using the IQ - Fure spectrometer IRAffinity -1 S Shimadzu, equipment for elemental analysis and optical microscope OPTIKA B -150, were analyzed and explained in detail. Using Shimadzu X-ray diffractometry Maxima X XRD 7000 was used for elemental analysis, which allowed to accurately determine the composition of the components in percentage. At the end of the chapter, images of the membrane and ion fortify under a microscope are given.

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