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Synthesis and Characterization of Metal-Organic Polymer based on Fe(III)

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Abstract—In this work, a coordination polymer based on iron trinuclear acetate was synthesized. The coordination polymer was obtained in two stages. At the first stage $[Fe_3O(CH_3CO_2)_6(H_2O)_3]\cdot 2H_2O$ – tricyclic iron acetate was synthesized. As the dicarboxylate ligand, we chose muconic acid, which is not toxic to the body. Double bonds in its structure create additional nodes for interaction with drugs.

The optimal conditions for the synthesis of a coordination polymer based on tricyclic iron acetate and muconic acid were selected: solvothermal synthesis at 78 °C, autogenous pressure and using ethanol as a solvent. The resulting coordination polymer is a nanosized mesoporous framework with a narrow pore distribution, an average radius of~1.18 nm, and a developed specific surface area of 512.1 m2/g. The composition and crystal structure of tricyclic iron acetate and a coordination polymer based on it have been confirmed by the methods of elemental and X-ray phase analysis.

Keywords—Polymers, ferrous oxide, X-ray diffraction, composites, muconic acid.

I. Introduction

ORGANOMETALLIC coordination polymers are a promising class of ordered porous compounds built from metal-containing units and organic linkers. Due to their specific properties and structure, such polymers are one of the most important research subjects among new scientific directions in chemistry and materials science [1]-[15]. But this class of connections also has a number of problems. Any change in the conditions for obtaining the material can lead to different directions of the synthesis, as a result of which it is possible to obtain different final reaction products. In this regard, it is possible to obtain chemically and thermally unstable compounds [16]-[24].

One of the modern achievements of nanotechnology is the creation of nano- and micro-sized composite materials and technologies for their industrial production. Among these materials, powders of magnetically controlled sorbents play important role in medicine. The name itself defines the most important property of magnetically controlled sorbents - the ability to remotely control them using an external magnetic field. The combination of this property with the possibility of drug loading and those biochemical properties that can be endowed with particles with the help of a suitable coating make it possible to create fundamentally new methods of treating various diseases. Therefore, the issues related to the use of magnetically controlled sorbents in general medical practice are relevant. In particular, one of these issues is the development of a methodology for the practical calculation of the parameters of the extracorporeal detoxification system, designing the appropriate composite materials.

The term “nanocomposites” appeared relatively recently, but natural nanocomposites have been known for a long time [25]-[35]. Clay mineral nanoparticles are widely used to control the viscosity properties of polymer solutions and to stabilize gels. The nature of the effect of nanoparticles on the properties of composite nanomaterials and the directions of their use largely depend on the matrix (the medium where the nanoparticles are dispersed) [36]-[42].

The development of technologies for creating nanocomposite systems is currently mainly moving towards the development of filled composites with the introduction of functionally active compounds into an inorganic or polymer matrix, which makes it possible to create fundamentally new

materials [43]-[76].

The main purpose of the polymer binder is to bind the filler together, to ensure the joint operation of all monofilaments (or particles, if a dispersed filler is used), to ensure the solidity of the material and the transfer (distribution) of stresses. The properties of the binder almost completely depend on: heat and heat resistance, resistance to the action of various working media (water, steam, fuel, oils, etc.), impact strength, impact strength, resistance to prolonged exposure to alternating loads, creep, stress relaxation.

Coatings on nano and micro-sized particles can serve for many purposes. First of all, modification of the surface with coatings makes it possible to make the particles compatible with various matrixes [14]-[20]. For medical purposes, the biocompatibility with the environments of a living organism is of crucial importance. It is equally important that coatings can significantly enhance or decrease the sorption properties of magnetically controlled sorbents. This provides prerequisites for the creation of magnetically controlled particles with specific sorption properties. It is also known that the coatings prevent the core from leaching out. The presence of a coating also often facilitates the stabilization of particles in an environment with an alkaline pH or significant salt concentration. For example, the isoelectric point of SiO_2 is reached at pH 2-3. Therefore, the particles coated with silica are negatively charged at the pH of the blood, which causes electrostatic repulsion, which avoids the formation of clumps.

The unique physicochemical properties of magnetically controlled sorbents, noted above and many others, attract great interest of researchers of various specialties around the world. This is evidenced by the growth in the number of scientific publications, patents and innovative works related to both the study of the fundamental properties of magnetically controlled particles and the solution of applied problems associated with the development of methods for their targeted use in medicine and biology.

This work is a continuation of previous works, which presents the results of comparative studies of the structure and sorption capacity of nano- and micro-sized particles to low-, medium- and high-molecular objects [21]-[27].

In addition to being used in devices for the standardized extraction of reagents in biofluid, magnetically controlled sorbents can find no less effective use for introducing useful substances (vitamins, microelements, or other additives) into biofluid. Indeed, the release of the active reagent from the shallow pores of the specified sorbent (a thin layer of the carbon shell) is facilitated, does not require much time and is easily controlled. All this makes the specified process well predictable. Accordingly, the rate of establishment of the equilibrium concentration of the active reagent introduced into the biofluid is high, and the current value of the reagent concentration in the solution is quite accurately determined by the surface density of the reagent and the thermodynamic characteristics of the system, taking into account the Langmuir isotherm, and can be purposefully changed depending on the solution temperature.

It is rather difficult to predict with accuracy the structure, chemical and physical properties of the future polymer due to the huge variety of organic and inorganic building units [1]-[12]. It should be noted that organic components are capable, depending on the solvent and synthesis conditions, in different ways to bind metal ions and clusters [13]-[29]. But choosing the initial blocks and the method of connecting them, relying on the already known methods of synthesis, it is possible to assume and select the necessary structure of the material and its properties [30]-[42]. The coordination sphere of the metal and the directionality of the bond of donor atoms, as well as the geometry of the bridging ligand, to a large extent affect the structure and properties of the polymer [43]-[58]. Therefore, a particularly important task in the synthesis of MOCP is the control of the interaction of components in the solvent, as well as the correct selection of organic and inorganic components.

Active (well absorbing) adsorbents have a very large specific surface area. For example, the absorbers most often used for scientific purposes and industrial practice - activated carbon, silica gel, zeolites – have s_0 up to several hundred and even thousands of square meters per 1 g.

The ability of the adsorbent to absorb the adsorbate is characterized by the amount of adsorption. The amount of adsorption is the excess mass of the adsorbate in the boundary layer over its mass in an equal volume of the environment, referred to the unit surface of the adsorbent.

Sometimes the adsorption value is expressed in moles of adsorbate per 1 m^2 (or 1 cm^2) of the adsorbent surface. Since quite often the surface of the adsorbent is unknown, the value of adsorption is expressed in moles of adsorbate per 1 g of adsorbent (mol/g). It is customary to evaluate the process of toxin sorption by the adsorbing surface using the curves of Langmuir sorption isotherms.

Currently, there is a constant search for optimal synthesis methods and reaction conditions for obtaining coordination polymers with the required properties and structure [25]-[33]. The coordination polymers obtained as a result of various methods should be further studied using physicochemical methods to determine their properties. X-ray diffraction analysis is usually used to identify the structure of coordination polymers, since these materials are highly crystalline and porous. The structure is recognized by comparing the obtained diffractogram of the synthesized coordination polymer with the already presented known one. This method is considered the most effective, but it is not always possible to use it, since it is not always possible to obtain single crystals. Due to this method, along with structure recognition, it becomes possible to determine various polymorphic forms, as well as to estimate the percentage of crystallinity of the material. After the crystal structure has been identified, the crystallographic parameters of the polymer can be determined: unit cell size, crystallite size, and lattice parameters. They can be calculated using various methods, including mathematical correction [34]-[42].

In this work, the goal is to create a porous coordination polymer with biocompatible structural elements based on oxoclusters of iron (III) muconate. Iron (III) was chosen as a metal-forming center, since it is considered biocompatible, and as an organic component, muconic acid, which is also not toxic to the body, was chosen. It is of interest to develop an optimal route for obtaining such a nanoscale framework and to study the composition and physicochemical properties of the resulting compound.

II. Synthesis and Study of the Properties of the Coordination Polymer

The coordination polymer was obtained in two stages. At the first stage $[Fe_3O(CH_3CO_2)_6(H_2O)_3]\cdot 2H_2O$ – tricyclic iron acetate was synthesized. A trinuclear inorganic block of iron (III) acetate was chosen as the inorganic component of the coordination polymer. Iron is considered biocompatible, since this trace element takes part in a large number of metabolic processes in the human body and is important in the erythrocyte part of the blood system [40]. At the second stage, to obtain a mesoporous coordination polymer based on tricyclic iron acetate and muconic acid.

One of the most informative methods for studying the structure of coordination polymers is IR spectroscopy. According to the IR spectra, it can be seen that the coordination polymer (Figure 1) has strong and broad absorption bands in the range of 2800 - 3500 cm^{-1}, corresponding to the stretching vibrations of the OH group of both coordinated and water molecules, as well as the stretching vibrations of the CH group of muconic acid.

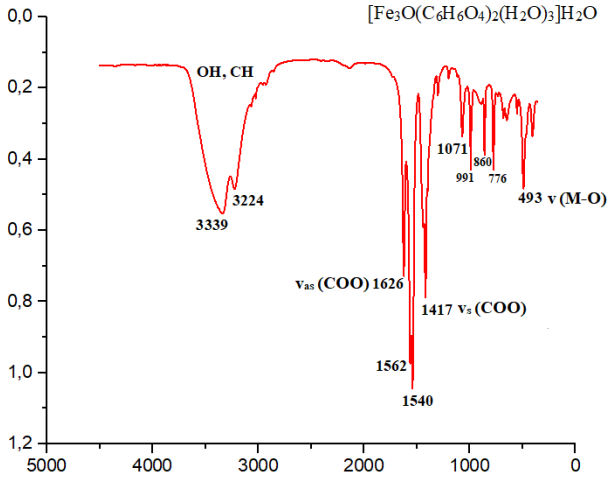

Fig. 1 ir - spectrum of the coordination polymer.

For comparison, Figure 2 shows the IR spectrum of the organic ligand, muconic acid.

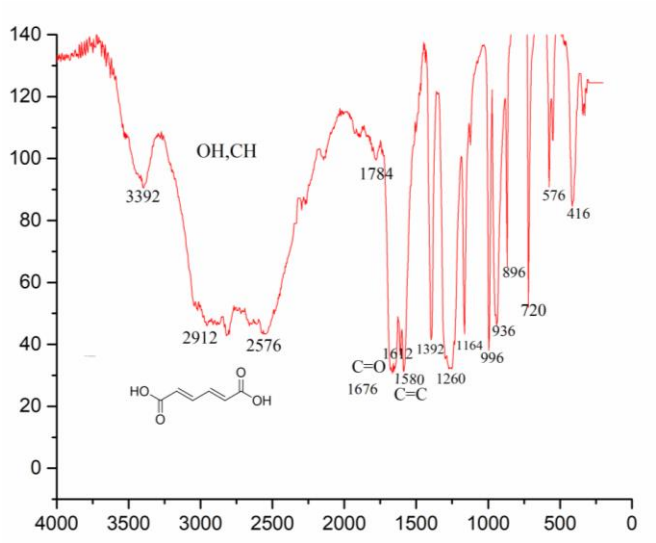

Fig. 2 ir - spectrum of muconic acid.

As it can be seen from the Figure 2, stretching vibrations of the carbonyl group C = O at 1676 cm^{-1} of the starting muconic acid (according to the table values) are significantly shifted by 1626 cm^{-1} in the structure of the coordination polymer. The difference between asymmetric stretching vibrations vas (COO) and symmetric stretching vibrations vs (COO) (1417 cm^{-1}) provides useful information on the nature of carboxylate coordination. The ligand is coordinated with the metal cation in the monodentate coordination mode ($\Delta\nu \geq 200$ cm^{-1}). Metal-oxygen bond is evidenced by a band at 493 cm^{-1}.

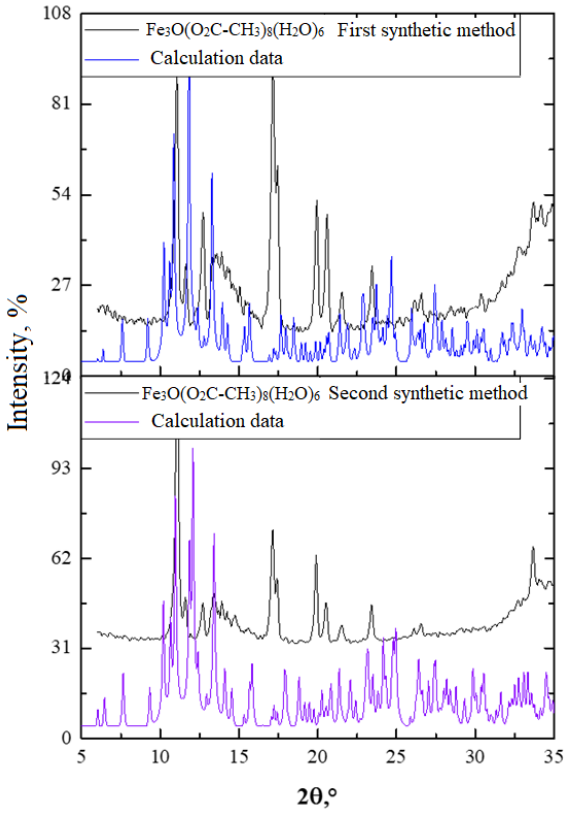

Fig. 3 Radiographs of tricyclic iron acetates in comparison with calculated data.

To confirm the crystal structure of the coordination polymer, X-ray phase analysis (XRF) data were used. In the results of the analysis of intermediate complexes of trinuclear iron acetate (Figure 3), narrow diffraction maxima are visible, indicating the presence of crystalline phases of the complex. They almost completely coincide with the calculated data.

The crystal structure is also preserved in the obtained organometallic coordination polymer (Figure 4). But it should be noted that its X-ray diffraction pattern contains an amorphous halo, the presence of which is possible due to the tendency of the ligand to stack-interaction (similar to the arrangement of coins in a stack), as a result of which the framework intergrowth, in other words, the formation of "intertwining" structures. It should be noted that under these conditions the yield of the organometallic coordination polymer is about 93% of the theoretical, which is very important for further practical use.

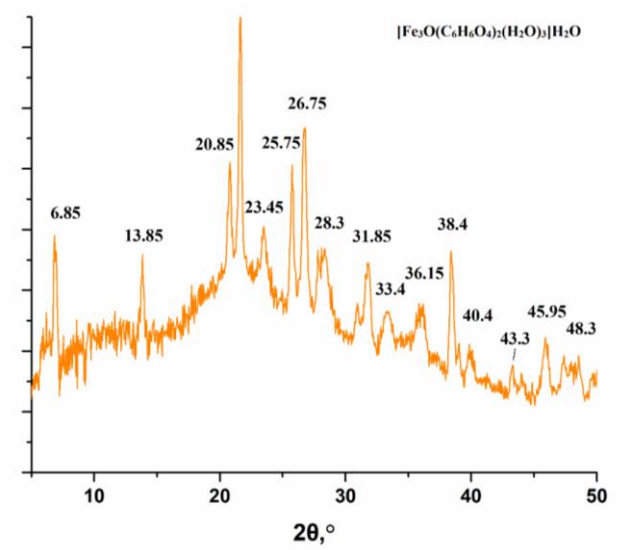

Fig. 4 x-ray diffraction pattern of one of the variants of the finished coordination polymer.

The resulting crystal structure corresponds to the topology of the known coordination polymers based on iron (III) oxoclusters (Figure 5).

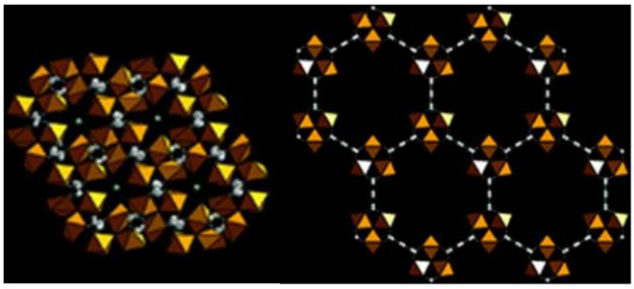

Fig. 5 Structure of the coordination polymer in dried form (left) and with open pores (right).

Elemental analysis was carried out to confirm the correctness of the obtained structure and its purity. The elemental analysis results confirm that an iron-based tricyclic acetate block was obtained in both synthesis methods. For the compound synthesized by the second method, the found elemental composition is closer in value to the calculated one, and it also has a high yield of the final product. Therefore, for further research, we chose tricyclic acetate obtained by the second synthesis method.

III. Conclusion

As a result of this study, the optimal conditions for the synthesis of a coordination polymer based on tricyclic iron acetate and muconic acid were selected: solvothermal synthesis at 78 °C, autogenous pressure and using ethanol as a solvent. The resulting coordination polymer is a nanosized mesoporous framework with a narrow pore distribution, an average radius of~1.18 nm, and a developed specific surface area of 512.1 m^2/g. The composition and crystal structure of tricyclic iron acetate and a coordination polymer based on it have been confirmed by the methods of elemental and X-ray phase analysis. The results allowed to develop an optimal route for obtaining such a nanoscale framework and to study the composition and physicochemical properties of the resulting compound.

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