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Investigation of Physical Properties of Fe(III) Containing Metal-Organic Polymers

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Abstract—In this work, we studied the properties of a specially synthesized organometallic coordination polymer - a porous coordination polymer with biocompatible structural elements based on oxoclusters of iron muconate (III). The samples were investigated by scanning electron microscopy, thermogravimetric analysis combined with differential scanning calorimetry, and the study of low-temperature nitrogen adsorption of a sample obtained by a modified solvothermal technique.

It is shown that most of the pores of the sample have an average radius of 18,8 Å ~ 1,88 nm. Also, as a result of the study, it is necessary to conclude that the synthesized material has a developed surface area - it is 512,1 m^2/g and the pore volume is ~ 0,48 cm^3/g.

It should be concluded that such materials are promising as components for a new generation of various kinds of functional materials with improved or unique characteristics. It is obvious that further research in this area is important from both fundamental and applied points of view.

Keywords—Polymers, nanoparticles, adsorption, mechanical properties.

I. Introduction

ORGANOMETALLIC coordination polymers are a new developing class of compounds built on the basis of inorganic building blocks consisting of metal ions or their clusters connected to each other through organic linkers in such a way that one-, two- or three-dimensional structures are formed. Organic units, in this case, are mono- or polydentate ligands. Some of these materials can find their application for preparation of magnetically controlled sorbents.

One of the modern achievements of nanotechnology is the creation of nano- and micro-sized composite materials and technologies for their industrial production [1]-[13]. 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.

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.

Taking into account the works on substantiating the method of practical calculation of the parameters of the extracorporeal detoxification system, it seems appropriate to determine the dependence of the concentration of the adsorbed component in the solution on the characteristics of the type of magnetically controlled sorbents used (mass, sorption capacity). Obtaining quantitative estimates will make it possible to reasonably set the required dosage of magnetically controlled sorbents for a specific detoxification procedure. Important, that temperature conditions can strongly affect the material and its behavior, so, a range of works was devoted to modelling of temperature conditions of materials [28]-[38].

In order to derive the equation of the adsorption isotherm, a number of simplifications are introduced. All the places where the adsorbed particles are fixed are the same, and adsorption on one of them does not affect the state of the other. The interaction between the adsorbed particles is negligible. The adsorption layer is monomolecular, i.e. it consists of one layer of molecules. In this case, the bond of the adsorbate with the adsorbent is sufficiently strong, which excludes the movement of the adsorption complex along the surface of the adsorbent (localized adsorption).

It is rather difficult to predict with accuracy the structure, chemical (reaction ability) and physical (density, conductivity etc.) 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]-[80]. 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.

II. Study of the Properties of Organometallic Coordination Polymers

The adsorption capacity of any adsorbent is determined primarily by its specific surface area $s_0 = s/m$, where s_0 is the surface area of the adsorbent; $m = Vd$ - its mass; d (kg/m^3) - the density of the adsorbent, V(m^3) - the volume. From this we obtain $s_0 = s/(Vd) = (1/d)\delta$, where $\delta = s/V$ is the degree of dispersion (fragmentation) of the adsorbent.

Specific surface area of the adsorbent s_0, and, consequently, its adsorption capacity will be the greater, the greater its degree of dispersion δ, or the smaller the linear dimensions of the particles into which the adsorbent is crushed.

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.

As you know, the surface of solids, like liquids, has an excess free Gibbs energy. However, solids, unlike liquids, cannot change the surface area by spontaneous change in shape. Therefore, the tendency to a decrease in the excess surface Gibbs energy in systems where the solid phase (the surface of magnetically controlled sorbents) is in contact with a liquid solution manifests itself mainly in the ability to retain solute molecules on the surface. The latter is possible if the molecules of the solute interact with the surface more strongly

than with the molecules of the solvent - the liquid to be purified (adsorption).

The dependence of the adsorbed amount of toxin Γ on its concentration in solution at a constant temperature is called the adsorption isotherm, a typical form of which is shown below in Fig.1.

The Langmuir adsorption isotherm in coordinates $\Gamma = \Gamma(C_a)$ is a hyperbola and is shown in Fig. 1.

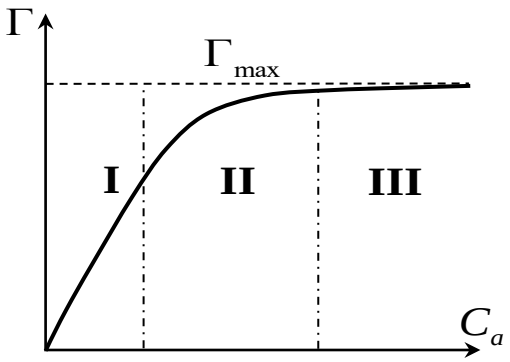

Fig. 1 Adsorption isotherm and its characteristic regions.

During adsorption, the surface of the adsorbent is gradually filled with molecules of the adsorbed substance: first, the most active areas are filled, and then the entire surface. After filling it, molecules can form a second, third, etc. layers. Therefore, a distinction is made between monomolecular and polymolecular adsorption. Given the monomolecularity of the adsorption layer, its thickness is obviously limited. In this case, the amount of toxin absorbed by the sorbent cannot exceed the limiting value $\Gamma = \Gamma_{max}$.

III. Results and Discussion

One of the main problems in the field of coordination polymers is their thermal stability. Therefore, to study these properties, the resulting compound was subjected to thermal analysis. As can be seen from Figure 2, the thermal decomposition of the sample obtained by the solvothermal method up to 250 ˚C occurs monotonically with a slight endo-effect on DSC. Then the DSC curve shows an exo-effect at 288.3 ˚C. It can be concluded that when the sample is heated to 300 ˚C, water molecules and solvent residues are removed, accompanied by polymerization. The weight loss corresponds to this when calculated from the found molecular weight of the polymer. Table 1 presents data on the change in the mass of the obtained coordination polymer.

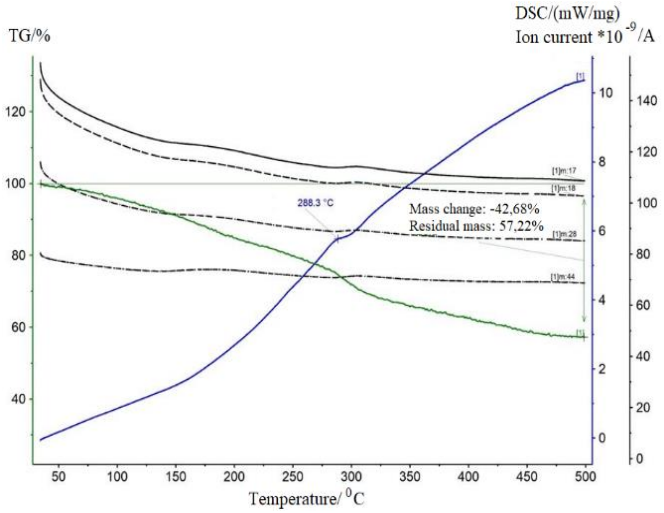

Fig. 2 Thermogravimetric analysis of the obtained coordination polymer combined with differential scanning calorimetry.

Table 1. Results of thermogravimetric analysis of the obtained coordination polymer combined with differential scanning calorimetry.

Sample	TGA data				DSC data	Residue at 500 °C % wt.
	$T_{5\%}$[°C]	$T_{10\%}$[°C]	$T_{20\%}$[°C]	$T_{30\%}$[°C]	Peak [°C]	
$[Fe_3O(C_6H_6O_4)_2 \cdot (H_2O)_3]H_2O$	110	158	248	309	288.3(max)	57.22
$T_{5\%}$, $T_{10\%}$, $T_{20\%}$, $T_{30\%}$ — Temperatures of 5%, 10%, 20%, 30% mass lost						

The data from Table 1 allows to calculate the weight of the residual mass of oxoclusters of iron muconate. Scanning electron microscopy was used to confirm the scaffold morphology. According to the results obtained, it can be seen that the topological structure of trinuclear iron acetate (Figure 3) is represented by particles of regular shape, characteristic of crystalline substances. Morphology of the iron-containing complex is retained for the resulting coordination polymer (Figure 4).

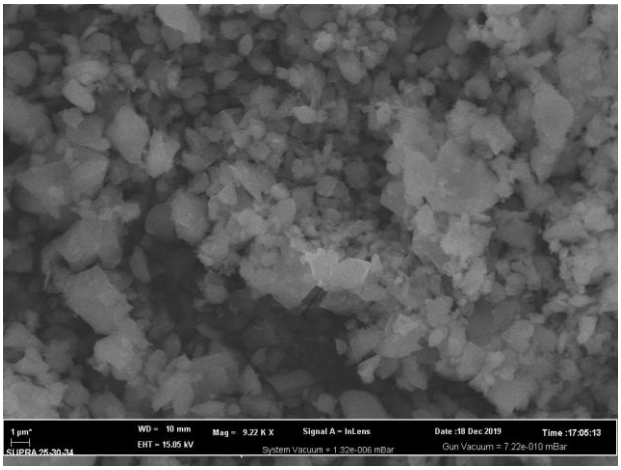

Fig. 3 sem photomicrographs of tricyclic iron acetate.

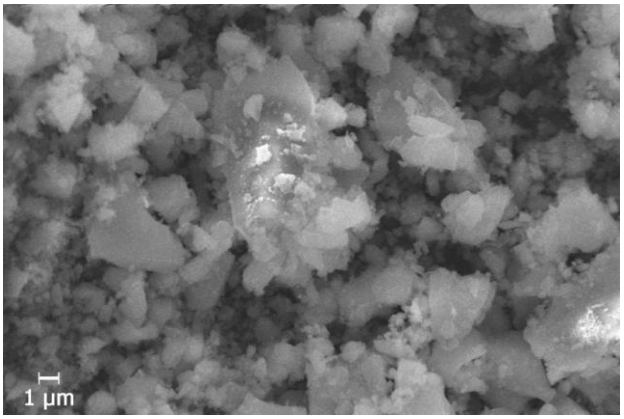

Fig. 4 sem micrographs of the coordination polymer.

Since coordination polymers are characterized by a large surface area, it is important to study the textural characteristics of the synthesized compound. It is necessary in order to characterize the pore size and their distribution, hence, the adsorption capacity of the composite. Figure 5 shows the results of low-temperature nitrogen adsorption of the sample obtained by the modified solvothermal technique. It can be seen that the obtained coordination polymer has an isotherm with a steep rise in the region of low P/P_0 and an almost horizontal section in the saturation region, which indicates polymolecular adsorption on the mesoporous adsorbent.

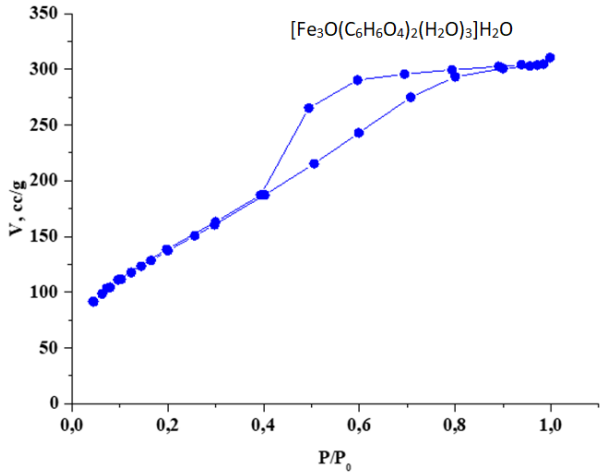

Fig. 5 Adsorption - desorption isotherms of nitrogen for the obtained coordination polymer.

The data obtained as a result of low-temperature (77K) nitrogen adsorption confirm that it was possible to reticulate (replace) the acetate linker with the muconic one. Muconic acid, unlike acetate, is capable of binding several inorganic blocks, due to which an extended structure is created. This is confirmed by the surface area obtained as a result of the analysis - it is 512,1 m^2/g and the pore volume is~ 0,48 cm^3/g. As a result, after activation of the sample by thermal vacuum treatment, a mesoporous framework was obtained.

Figure 6 shows the distribution of pore volume along the radii.

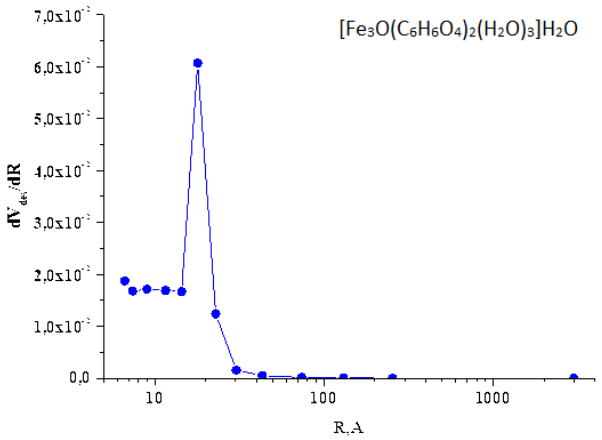

Fig. 6 Differential distribution curve of pore volume along the radii *r*.

IV. Conclusion

From the data obtained, it can be concluded that most of the pores have an average radius of 18,8 Å ~ 1,88 nm. Also, as a result of the study, it is necessary to conclude that the synthesized material has a developed surface area according to the BET theory (Brunauer, Emmett, Teller) - it is 512,1 m^2/g and the pore volume is ~ 0,48 cm^3/g. It should be concluded that such materials are promising as components for a new generation of various kinds of functional materials with improved or unique characteristics. It is obvious that further research in this area is important from both fundamental and practical points of view, since it opens the prospective for tailored fabrication of functional nanocomposites for subsequent application as sorbents and construction materials.

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