THERMAL ANALYSIS AND SYNTHESIS OF NEW MATERIALS IN FLNP JINR

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Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) are developing along with neutron methods for condensed matter research in FLNP JINR. Netzsch TGA 209 F1 Libra gravimeter gives the opportunity to determine the content of volatile components in the samples, to determine the limit of thermal stability of compounds, and to precise the composition of the samples. For example, TGA allowed to confirm the presence of amorphous manganese oxide MnO_2 in the adsorbent based on iron oxide Fe_3O_4 [1].
Differential thermal analysis is useful addition to the study of phase transformations in materials. Netzsch 204 F1 Phoenix differential scanning calorimeter allows to do the research not only during heating, but also at low temperatures (to 93K). We carry out thermal analysis both in an inert atmosphere (argon, nitrogen) and in an air, which allows to investigate oxidation processes.
Along with the study of the physicochemical properties of materials, sometimes tasks of the synthesis of new materials arises. The synthesis of the studied materials directly in FLNP can significantly expand the area of our research. And we have this possibility to synthesize materials. Our equipment (furnaces and autoclaves) allows to conduct annealing in an inert or oxidative atmosphere, as well as to carry out hydrothermal (solvothermal) synthesis in solutions at elevated pressure. The main objects of synthesis are inorganic materials, including cathode materials for sodium-ion batteries (SIBs) and metal-organic frameworks (MOFs). SIBs are interesting as an alternative to lithium-ion batteries due to higher sodium content in nature compared with lithium. The synthesis of cathode materials with high capacity and good stability is an important task for wide use of SIBs.
MOFs are crystalline materials consisting of an infinite network of metal-ions, or metal-ion clusters, bridged by organic ligands through coordination bonds into porous two- or three- dimensional extended structures. They are attracting increasing interest due to their unique adsorption properties. In the last time, the problem of water purification from dangerous substances is becoming more and more acute. A large number of studies are devoted to the extraction of heavy metals from aqueous solutions [2]. The creation of solid adsorbents with high capacity and stability in the aquatic medium is a very important task. MOFs are one of the promising types of adsorbents. But very few MOFs are used as adsorbents from aqueous solutions because most MOFs are unstable in aqueous medium [2]. We synthesized Ni-MOF $\{[Ni(L\text{-trp})(bpe)(H_2O)]\cdot H_2O\cdot NO_3\}_n$ (*L*-trp = *L*-tryptophan, bpe = 1,2-bis(4-pyridyl)ethylene) and Zr-MOF MIP-202(Zr), based on *L*-aspartic acid. They are stable in an aquatic environment. With UV-Vis spectrometry, we investigated the properties of Ni-MOF and Zr-MOF in the process of ruthenium sorption from aqueous solutions of ruthenium chloride.
Another attribute of MOFs is magnetism. Most magnetic frameworks are those containing paramagnetic metal centers. Research of the magnetic properties of MOFs with Ni and Co synthesized in FLNP was carried out on the spectrometer of polarized neutrons REMUR (IBR-2). It was shown that samples are capable to magnetization. The biggest effect was observed on the compound bis(*L*-histidinato)nickel(II) monohydrate. Magnetic domains formation is possible at low magnetic field. We plan to continue this research.

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[2] N. Manousi, D.A. Giannakoudakis, E. Rosenberg and G.A. Zachariadis (2019). Extraction of Metal Ions with Metal–Organic Frameworks. Molecules. 24, 4605.