Nanoparticles of Complex Oxides \( \text{Li}_1 + x (\text{Ni}_y \text{Mn}_z \text{Co}_{1 - y - z})_{1 - x} \text{O}_{2 - \delta} \) 
\( (0 \leq x \leq 0.2, \ 0.2 \leq y \leq 0.6, \ 0.2 \leq z \leq 0.4) \) Obtained by Thermal Destruction of Metal-Containing Compounds in Oil

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Abstract—Cathode materials in the form of \( \text{Li}_1 + x (\text{Ni}_y \text{Mn}_z \text{Co}_{1 - y - z})_{1 - x} \text{O}_{2 - \delta} \) (0 \( \leq x \) \leq 0.2, 0.2 \( \leq y \) \leq 0.6, 0.2 \( \leq z \) \leq 0.4) core–shell nanoparticles coated with a thin carbon shell were synthesized by thermal destruction of metal-containing compounds in oil and studied. The results of element analysis, X-ray diffraction analysis, scanning electron microscopy, X-ray photoelectron spectroscopy, and electrochemical tests of cathodes based on the obtained complex oxides in model cells were presented. The complex oxide \( \text{Li}_2 \text{Ni}_2 \text{Mn}_4 \text{Co}_2 \text{O}_{19} \) was the most promising composition because the loss of capacity after 50 cycles was 4% at a current density C/2 and an operating potential of 3.0–4.4 V relative to \( E (\text{Li}/\text{Li}^+) \). When the current density in discharge increased sixfold (3 C), the loss of capacity was 14% relative to the value obtained at a discharge current C/2 at voltages 3.0 to 4.4 V.

**Keywords:** cathode materials, complex oxides, core–shell nanoparticles, thermal destruction of metal-containing compounds in oil, nanoreactor, lithium-ion battery

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INTRODUCTION

Complex oxides \( \text{Li}_1 + x (\text{Ni}_y \text{Mn}_z \text{Co}_{1 - y - z})_{1 - x} \text{O}_{2 - \delta} \) (0 \( \leq x \) \leq 0.2, 0.2 \( \leq y \) \leq 0.6, 0.2 \( \leq z \) \leq 0.4) (below NMC) are the most promising cathode materials (CMs) for lithium-ion batteries (LIBs), which are of particular interest because of their ability to meet the ever-growing demand for energy storage devices. These multi-component oxides have high specific capacity (more than 160 mA h/g at a charge of C/20), electric conductivity, and thermal stability, as well as high reliability in cycling and lower cost due to the synergistic effect of their nickel, manganese, and cobalt ions [1–6]. For these materials, however, cation disordering is a problem, i.e., cation mixing of lithium and divalent nickel ions due to the similarity of their ionic radii (0.069 and 0.076 nm, respectively), which leads to deterioration of the electrochemical parameters of CMs and a decrease in the specific capacity of the entire LIB [7].

On the other hand, in order to improve the basic electrochemical characteristics, many research groups synthesized nanostructured complex oxides by various methods [8–11]. These methods lead to an increase in the charge–discharge rate due to a sharp reduction in the diffusion length of lithium ions and the increase of electrode–electrolyte contact area. However, due to their nanosized nature, CMs more actively interact with the electrolyte, causing a large number of limiting stages, which eventually leads to serious safety problems not inherent in microsized materials, especially at high temperatures.

When reducing the size of primary crystallites, it is advisable to cover them with a stabilizing surface layer, which would prevent changes in the phase composition and crystallographic parameters. This coating should not lead to decreased diffusion rate of lithium ions, thus annihilating the advantages of the use of nanosized particles [12].

Our previous studies [13, 14] showed that dropwise introduction of a solution of metal-containing compounds in a melt solution of polyethylene formed nanoreactors, from which the volatile components that formed as a result of the thermal decomposition of the introduced salts evaporate together with the solvent. This forms nanoparticles whose composition is given by the molar ratio of salts in the initial solution. The use of this approach allowed us to perform the
thermal decomposition of the introduced acetate solution in a polyethylene matrix and to obtain isolated nanoparticles consisting of a mixture of oxides of the same composition that was specified for the initial solution of the salts. The thermal treatment of the obtained precursors gave the core–shell nanoparticles with an average size of 90 nm, whose shell was the nanometer layer of amorphous carbon. During the electric tests of CMs in model cells it was found that in the case of intercalation—deintercalation of lithium ions into/from the structure of CMs coated with an amorphous layer of carbon, the lithium ions diffuse into the shell; i.e., it can serve as an intermediate reservoir for storing the lithium ions and easily damp the inevitable “swelling” of the CM core [15].

The goal of this study was to prepare complex oxides Li$_{1+x}$(Ni$_x$Mn$_{y}$Co$_{1-z}$)$_{2-δ}$O$_2$ (0 ≤ x ≤ 0.2, 0.2 ≤ y ≤ 0.6, 0.2 ≤ z ≤ 0.4) under different synthesis conditions and to perform comparative studies of the obtained oxides by modern physicochemical and electrochemical methods.

**EXPERIMENTAL**

The starting materials used were lithium CH$_2$COOLi · H$_2$O (99%, Aldrich), cobalt Co(CH$_3$COO)$_2$ · 4H$_2$O (99%, Aldrich), manganese Mn(CH$_3$COO)$_2$ · 4H$_2$O (99%, Aldrich), and nickel Ni(CH$_3$COO)$_2$ · 4H$_2$O (99%, Aldrich) acetates, acetic acid (99.9%), high-pressure polyethylene (HPPE) (Aldrich), and vacuum oil (VO) (State Standard GOST 38.01402-86).

The synthesis of complex oxides Li$_{1+x}$(Ni$_x$Mn$_{y}$Co$_{1-z}$)$_{2-δ}$O$_2$ (0 ≤ x ≤ 0.2, 0.2 ≤ y ≤ 0.6, 0.2 ≤ z ≤ 0.4) in the form of core–shell nanoparticles with a carbon coating consists of two stages: (1) preparation of nanoparticles of a mixture of metal oxides stabilized in the bulk of the polymer matrix and (2) high-temperature treatment.

At the first stage, the samples were synthesized by thermal decomposition of a solution of metal-containing compounds into a polyethylene–hydrocarbon oil (TMCC) solution melt [13, 14, 16] at 230–250°C. In a typical experiment, a solution of lithium, nickel, manganese, and cobalt acetates in a suitable molar ratio in acetic acid was introduced dropwise, while vigorously stirring, in the HPPE melt solution. In addition, an inert gas was fed to the reactor to ensure rapid and complete removal of volatile reaction products and to prevent the oxidation of metals. The amount of HPPE was calculated such that the concentration of the resulting nanoparticles relative to the matrix was 15–60 wt % [16]. The termination of gas formation indicated the end of reaction. After cooling, the liquid portion was separated from the precipitate. The second stage was the thermal treatment of the precipitate at 450–900°C for 8–24 h in an inert gas and air.

**Methods of Investigation**

The element analysis was performed by inductively coupled plasma atomic emission spectroscopy on an iCAP 6300 unit (Thermo Electron, United States). The phase composition was determined on a Bruker Advance 8 (Germany) X-ray powder diffractometer. The operating mode: 40 kV, 40 mA, Cu radiation, nickel filter, 2θ = 10°–80°, scan step 0.01°. To interpret the diffraction patterns, the International Center for Diffraction Data database was used. The morphology and microstructure of the samples were studied with a Carl Zeiss Supra 40–30–87 scanning electron microscope (SEM) (Germany). The accelerating voltage for obtaining images in secondary and backscattered electrons was 1–10 kV. The chemical and electronic states of atoms were determined by X-ray photoelectron spectroscopy (XPS) on a PHI 5500 ESCA X-ray photoelectron spectrometer (Physical Electronic, United States).

The electric measurements were carried out in model cells according to two- and three-electrode schemes in galvanostatic and/or potentiostatic modes. The positive electrode was prepared by the following procedure. A homogenized mixture (80% cathode material, 10% acetylene black (Timcall), 10% polyvinylidene fluoride (PVdF) dissolved in N-methylpyrrolidone) was applied to aluminum foil and dried at 120°C for 12 h in a vacuum drying cabinet. The electrolyte was a 1 M solution of LiPF$_6$ in ethylene carbonate—dimethyl carbonate (1 : 1). Metallic lithium was a counterelectrode and reference electrode.

**RESULTS AND DISCUSSION**

In our previous studies [16, 17], we prepared complex oxides Li$_{1+x}$(Ni$_x$Mn$_{y}$Co$_{1-z}$)$_{2-δ}$O$_2$ (0 ≤ x ≤ 0.2, 0.2 ≤ y ≤ 0.6, 0.2 ≤ z ≤ 0.4) and found that after the first stage of the process, nanoparticles of a mixture of metal oxides were obtained; they consisted of transition metal and lithium oxides uniformly distributed over the entire volume of the polymer matrix and had an ellipsoidal shape with an average size of 2.3 ± 0.4 nm.

Li$_{1.2}$Ni$_{0.2}$Mn$_{0.4}$Co$_{0.2}$O$_{1.9}$ (below LNMC) was used as an example to study the effect of the nanoparticle concentration in the polymer matrix at the first stage and the temperature mode of processing at the second stage on the basic physicochemical and electrochemical properties of the synthesized complex oxides.

During the heat treatment of the precursors obtained at the first stage of the process, the diffraction pattern showed reflections characteristic of a layered crystal structure of α-NaFeO$_2$ type already after annealing at 300°C due to their nanosized nature (space group $R_3m$, no. 166); further heat treatment (above 600°C) led to more intense reflections, which indicated the agglomeration of the primary crystallites (Fig. 1). According to [18], single-phase NMC forms at 500°C, but termination of crystallographic ordering
requires temperatures above 750°C. The diffraction patterns contained signals of impurity phases: the reflections in the 2θ range 22°–23° corresponded to the cation orientation present in the Li₂MnO₃ layers; the reflections in the 2θ range 30°–33° were characteristic for lithium carbonate formed during the thermal decomposition of metal-containing compounds. It is important to note that the Li₂MnO₃ phase formed due to the excess mole content of lithium (a > 1) and manganese (c > b and d) in LiₓNi₄Mn₁₀Co₉O₂ and had dual properties: (1) at a charge of >4.4 V, there was an irreversible increase in the charge capacity (of more 40%); this process is associated with the removal of O²⁻ and Li⁺ anions from the crystal structure of Li₂MnO₃ aimed at maintaining electroneutrality in the structure, and the removal of oxygen seems thermodynamically more advantageous than the oxidation of Mn above 4⁺; (2) if the cycling was limited to a voltage below 4.4 V, Li₂MnO₃ worked as a frame and reservoir for lithium, preventing deterioration of the capacity during electric cycling [19, 20].

After further high-temperature treatment to 800°C, the initial stoichiometric composition of the metals remained unchanged. Thus, due to the dropwise addition of the initial solution of salts to the rector, this method made it possible to achieve constant element composition during the agglomeration of crystallites (Table 1). During treatment to 900°C, this ratio of metals in the composition changed because of the partial evaporation of Li₂O. In addition, heat treatment from 600 to 900°C leads to a decrease in the carbon content from 4.4 to 2.3 wt %, respectively.

As is known, in the structure of NMC, the MeO₆ octahedra (Me = Ni, Mn, Co) share edges in pairs and form negatively charged MeO₂ layers stabilized and screened by layers of octahedrally coordinated lithium ions. In this case, the lithium ions can move freely in the plane of the layer and be reversibly intercalated to and deintercalated from this structure, with a simultaneous change in the formal oxidation state of the transition metals [21]. As is known from the literature [22], the intensity ratio of the (003) and (104) peaks is affected by the degree of ion disordering in the 3a (lithium layers) and in the 3b (transition metal layers) positions of the crystal lattice. The undesirable cation disordering will be present at an intensity ratio of (003) to (104) peaks of less than 1.2 and also of (101) to (006) and (012) of over 1.6 [23]. The nickel Ni²⁺ ions replace the lithium ions in their positions, which hinders Li⁺ ion diffusion, i.e., the intercalation of lithium ions during cycling; therefore, it leads to a significant drop in the capacity of the CM from cycle to cycle [24].

All the diffractograms (Fig. 2) contained reflections characteristic of the R₃m layered structure and the Li₂MnO₃ phase (space group C2/m). LNMC were obtained at temperatures from 600 to 900°C (hereinafter LNMC600–LNMC900, respectively). The samples had low cation disordering of lithium ions and transition metals in the crystal lattice, as evidenced by high integrated intensities of the (003)/(104) peaks, which were 1.35, 1.72, 1.84, and 1.66, and (101)/(006) + (012) 1.23, 1.31, 1.36, and 1.42 for LNMC600, LNMC700, LNMC800, and LNMC900, respectively (Table 2). A more pronounced splitting of the pairs of

![Diffraction patterns](image1.png)

**Fig. 1.** Diffraction patterns of Li₁₂Ni₀.²Mn₀.⁴Co₀.²O₁.⁹: (a) after dropwise addition of the salt solution in the rector; (b) after annealing at 300°C; and (c) after annealing at 600°C; (d) Li₂MnO₃ and (2) Li₂CO₃.

![Diffraction patterns](image2.png)

**Fig. 2.** Diffraction patterns of Li₁₂Ni₀.²Mn₀.⁴Co₀.²O₁.⁹ after annealing at (a) 600°C, (b) 700°C, (c) 800°C, and (d) 900°C. *Li₂MnO₃.

| Table 1. Molar ratio of metals in Li₁₂Ni₀.²Mn₀.⁴Co₀.²O₁.⁹ after annealing at temperatures from 600 to 900°C |
|---|---|---|---|---|
| Sample | Li | Ni | Mn | Co |
| LNMC600 | 1.20(3) | 0.20(1) | 0.40(5) | 0.20(6) |
| LNMC700 | 1.20(4) | 0.20(1) | 0.39(8) | 0.20(6) |
| LNMC800 | 1.20(4) | 0.20(6) | 0.40(7) | 0.19(7) |
| LNMC900 | 1.18(1) | 0.20(8) | 0.40(4) | 0.20(4) |
reflections (006)/(102) and (108)/(110) was observed for LNMC800, probably due to larger primary crystallites and high degree of cation ordering. The ratio of the calculated lattice parameters $c/a$ was within 4.989–4.993, which also confirmed the low degree of cation mixing between lithium ions and divalent nickel. For the LNMC900 sample, the degree of cation disordering increased, as evidenced by an increase in the ratio of the peak intensities (101) to (006) and (012) relative to the values for other samples, and this is probably due to the start of the thermal decomposition or phase transformation of LNMC.

A decrease in the width and increase in the intensity of reflections (Fig. 2) at elevated temperature suggested growth of primary crystallites, the mean values of which were 90, 140, 230, and 360 nm for LNMC600, LNMC700, LNMC800, and LNMC900, respectively, which was confirmed by SEM micrographs (Fig. 3). The carbon shell significantly sup-

<table>
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<td>1.4224</td>
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<td>$c/a$</td>
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<td>4.991</td>
<td>4.993</td>
<td>4.989</td>
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<tr>
<td>$I(\text{(101)}/I(\text{006}+(012))]$</td>
<td>1.23</td>
<td>1.31</td>
<td>1.36</td>
<td>1.42</td>
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<tr>
<td>$I(\text{003)}/I(\text{104)}$</td>
<td>1.35</td>
<td>1.72</td>
<td>1.84</td>
<td>1.66</td>
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Fig. 3. SEM micrographs of Li$_{1.2}$Ni$_{0.2}$Mn$_{0.4}$Co$_{0.2}$O$_{1.9}$ after annealing at (a) 600°C, (b) 700°C, (c) 800°C, and (d) 900°C.
pressed agglomeration of primary crystallites at the temperature increased to 900 °C [25].

To obtain LNMC800 with a narrower particle size distribution, it was suggested that the amount of HPPE be chosen such that the concentration of the formed nanoparticles relative to the polymer matrix was 15–60 wt % (hereinafter LNMC800-15, LNMC800-30, and LNMC800-60). It was found, however, that the average size of agglomerates (D50) was 9.4, 7.6, and 6.3 μm, and the scatter of primary crystallites was 80–450, 140–310, and 130–630 nm for LNMC800-15, LNMC800-30, and LNMC800-60, respectively. A more pronounced splitting of the (006)/(102) and (108)/(110) pairs of reflections and high degree of cation ordering were observed in the LNMC800-30 sample; the integrated intensity ratio was 1.66, 1.84, 1.93 for the (003)/(104) peaks and 1.34, 1.36, and 1.45 for the (101)/(006) + (012) peaks for LNMC800-15, LNMC800-30, and LNMC800-60, respectively (Fig. 4).

The electronic state of transition metal ions in LNMC800 corresponded to Ni2+, Co3+, and Mn4+ (Fig. 5). According to quantum-chemical calculations [26], the following pairs are involved in the redox process during the cycling of Li1+x(Ni0.2Mn0.4Co0.2)1–xO2–δ (0.2 ≤ y ≤ 0.6, 0.2 ≤ z ≤ 0.4): the Ni2+/Ni3+ pair at x in the range 0 < x < 1/3; Ni3+/Ni4+ in the range 1/3 < x < 2/3; and Co3+/Co4+ in the range 2/3 < x < 1 (>4.3 V). The Mn4+ ions remain electrochemically neutral. In this connection, the Li1+x(Ni0.2Mn0.4Co0.2)1–xO2–δ solid solutions with a high nickel content (y > 0.3) are of great interest from the viewpoint of increasing capacity [27, 28].

In addition to the fundamental Ni2p3/2 and Ni2p1/2 lines, the Ni2p spectrum contains intense lines with an energy ~6–7 eV higher than that of the fundamental lines. According to the literature, the presence of intense peaks is characteristic of paramagnetic compounds containing Ni2+ ions [29, 30]. At the same time, an increase in the nickel content in the samples leads to a substantial shift of the maximum of the Ni2p3/2 line toward higher binding energies. The observed shift is generally associated with the oxidation of nickel and formation of Ni3+ ions [31].

Today, nickel-enriched complex oxides NMC are of particular interest as the most promising cathode materials in lithium-ion batteries. However, the main problems in the synthesis of these materials are: sharp increase in the degree of cation disordering at increased nickel contents, which drastically deteriorates the electrochemical parameters from cycle to cycle; reduced thermal stability of the electrode on its basis; and increased irreversibility of the capacity of the first cycles.
All the samples had low degree of cation disordering, and the (003)/(104) and (101)/(006) + (012) peak intensities were in the admissible range [23] (Fig. 6). The cation mixing increased with the nickel content in NMC; the (101)/(006) + (012) peak intensity ratio was 1.36, 1.33, 1.37, and 1.51; the (003)/(104) ratio changed insignificantly and was 1.84, 1.85, 1.96, and 1.87 for LNMC, LiNi0.33Mn0.33Co0.33O2 (NMC111), LiNi0.5Mn0.3Co0.2O2 (NMC532), and LiNi0.6Mn0.2Co0.2O2 (NMC622), respectively. The calculations also showed that the lattice parameters and unit cell volume increased with the nickel content in NMC [25, 32].

Due to the larger electrode–electrolyte contact area leading to higher charge–discharge rates and small diffusion distances for ion and electron transport, nanostructured CMs can be charged within a shorter period of time [33].

Table 3 shows the results of testing the CMs of different stoichiometric compositions in model lithium cells. As expected, the stability decreased during the cycling as the nickel content in NMC increased. The CMs tested in milder modes (i.e., at a charge current density of 1C and discharge current density of 0.5C in the range of working potentials of 3.0–4.4 V with respect to the potential \( E (Li/Li^+) \)) showed more stable results during the cycling, the loss of capacity being 4–6% after 50 cycles. When the working potential increased to 4.6 V relative to \( E (Li/Li^+) \), the discharge capacity drastically increased by 20–30% relative to the previous modes, while the irreversible loss of the first cycle increased significantly and reached 24%. More aggressive cycling conditions accelerated the degradation of both the CM and the whole cell.

The samples with high nickel and lithium contents had the largest discharge capacity of 208, 183, and 201 mA h/g for LNMC, NMC532, and NMC622, respectively. The results of our studies showed that, the size of primary crystallites in the NMC532 and NMC622 samples decreased as the nickel content

![Fig. 6. Diffraction patterns of NMC after annealing at 800°C for (a) Li1.2Ni0.2Mn0.4Co0.2O1.9, (b) LiNi0.33Mn0.33Co0.33O2, (c) LiNi0.5Mn0.3Co0.2O2, and (d) LiNi0.6Mn0.2Co0.2O2. * Li2MnO3.](image-url)

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<td>208 [36]</td>
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<td>148</td>
<td>164</td>
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<td>Retained capacity, %</td>
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<td>Retained capacity, %</td>
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increased, i.e., the area of interaction between the electrode and electrolyte increased, as did the degree of cation disordering of the crystal structure and hence the hindrance to ion diffusion. At the same time, the surface carbon layer of primary crystallites partially prevented the destructurization of complex oxides during the cycling [25] but because of the high reactivity of these materials and aggressive conditions (>4.6 V), the shell did not completely protect the core from the formation of undesirable limiting stages, which led to a drop in the specific energy of the cell and to self-discharge and reduced number of cycles.

LNMC and NMC622 were tested, as materials with the highest capacity characteristics, by the Lithium-Ion Technologies Company at Ioffe Institute in two-electrode model cells at charge current densities of 1 С, discharge current densities of 0.5–3 С, and voltages of 3.0–4.4 V. It was found that at current densities increased to 3 С, the loss of capacity was 14 and 26% for LNMC and NMC622, respectively (Fig. 7).

The lower drop of capacity at a charge increased six-fold for LNMC may be explained by the presence of an additional phase, Li₂MnO₃, in the structure of LNMC, which acts as a framework and reservoir for lithium.

CONCLUSIONS

The thermal destruction of metal-containing compounds in oil followed by thermal treatment affords complex oxides Li₁₊ₓ(Ni₀.₅Mn₀.₅Co₃₋ₓ₋ₓ)₁₋ₓO₂₋₄ (0 ≤ x ≤ 0.2, 0.2 ≤ y ≤ 0.6, 0.2 ≤ z ≤ 0.4) with a narrow particle size distribution, low degree of cation mixing, and controlled molar ratio of metals. An increase in the concentration of the resulting nanoparticles relative to the polymer matrix above 30 wt % at the first stage and the processing temperature of the precursors at the second stage above 900°C significantly impair the basic physicochemical and electrochemical properties of the cathode materials. At the same time, increased nickel content (x > 0.3) leads to increased capacity and degree of cation disordering in the crystal structure of NMC and decreased life cycle of the cathode materials.

The Li₁.₂Ni₀.₄Mn₀.₄Co₀.₂O₁.₉ complex oxide is the most promising composition as the loss of capacity was 4 and 8% after 50 cycles at working potentials from 3.0 to 4.4 and 4.6 V relative to E (Li/Li⁺), respectively. Moreover, this material withstood a rate of 3 C at a discharge with 14% loss of capacity relative to the discharge capacity obtained at C/2 and at voltages of 3.0–4.4 V.

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