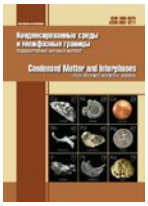

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# Effect of the phase composition of cathode material based on vanadium oxide nanofibers on the electrochemical characteristics of a lithium-ion battery

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**Abstract**

*Objectives:* Progress in improving the performance characteristics of lithium-ion batteries is largely determined by the cathode material. Various modifications of vanadium oxides attract interest as cathode materials. For example, a vanadium ion or a vacancy at a vanadium pentoxide lattice site can accommodate two positively charged lithium ions, resulting in a high theoretical specific capacity (~294 mAh·g$^{-1}$). However, during operation, the material rapidly degrades and loses its initial specific capacity. A variety of strategies has been proposed to mitigate capacity degradation, including structural modification, phase composition control, and heteroatom doping with foreign metal ions. The main aim of this work was to evaluate the effect of the phase composition of cathode material based on vanadium oxide nanofibers on the performance characteristics of a lithium-ion battery.

*Experimental:* The cathode material was fabricated from vanadium oxide nanofibers synthesized via electrospinning. The phase composition and surface morphology of the nanofibers were studied by X-ray diffraction and scanning electron microscopy. The cathode material was evaluated using standard electrochemical techniques.

*Conclusions:* All samples were mixtures of vanadium oxides ($V_2O_5$, $VO_2$, $V_4O_9$, $V_6O_{13}$) in various ratios. With increasing molar fraction of oxalic acid during annealing, the percentage of vanadium pentoxide decreases while that of vanadium dioxide increases. Electrochemical studies of the material showed that replacement of vanadium pentoxide nanofibers in the cathode material with nanofibers consisting of a mixture of vanadium oxides led to a decrease in initial specific capacity and did not increase cycling stability.

**Keywords:** Cathode material, Lithium-ion battery, Vanadium oxides, Electrospinning, nanofibers

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# Влияние фазового состава катодного материала на основе нанонитей оксидов ванадия на электрохимические характеристики литий-ионного аккумулятора

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**Аннотация**

*Цель статьи:* Прогресс в улучшении эксплуатационных характеристик литий-ионных аккумуляторов в значительной степени определяется материалом катода. В качестве катодного материала интерес исследователей привлекают различные модификации оксидов ванадия. Например, ион ванадия или вакансия в узле кристаллической решетки пентаоксида ванадия могут удерживать два положительно заряженных иона лития, что приводит к большой теоретической удельной ёмкости (~294 мАч·г$^{-1}$). Однако в процессе работы материал быстро деградирует и теряет свою изначальную удельную ёмкость. Для сохранения емкости исследователи предлагают ряд методов модификации материала: изменение структуры, фазового состава, добавка ионов других металлов и др. Главная цель представленной работы – оценка влияния фазового состава катодного материала на основе нанонитей оксидов ванадия на эксплуатационные характеристики литий-ионного аккумулятора.

*Экспериментальная часть:* Катодный материал изготавливался на основе нанонитей оксидов ванадия, полученных методом электроспиннинга. Фазовый состав и морфология поверхности нанонитей исследовались методами рентгеновской дифракции и сканирующей электронной микроскопии. Материал катода исследовался электрохимическими методами.

*Выводы:* Все образцы представляют собой смесь оксидов ванадия ($V_2O_5$, $VO_2$, $V_4O_9$, $V_6O_{13}$) в различных соотношениях. По мере увеличения молярной доли щавелевой кислоты при отжиге процентное содержание пентаоксида ванадия уменьшается, а диоксида возрастает. Электрохимические исследования материала показали, что замена в катодном материале нанонитей пентаоксида ванадия на нанонити из смеси оксидов ванадия приводит к уменьшению начальной удельной емкости и не увеличивает стабильность при циклировании.

**Ключевые слова:** катодный материал, литий-ионный аккумулятор, оксиды ванадия, электроспиннинг, нанонити

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## 1. Introduction

Now, lithium-ion batteries are one of the most common types of batteries. They are used in mobile devices (smartphones, tablets, laptops, cameras), medical devices (pacemakers), and electric vehicles. Modern batteries can withstand up to 2,000 charge–discharge cycles but have insufficient specific capacity, which limits the expansion of their application areas.

The characteristics of batteries are largely determined by the cathode material. Various modifications of vanadium oxides attract the attention of researchers as cathode materials. For example, each lattice site of vanadium pentoxide can accommodate two positively charged lithium ions, resulting in a high theoretical specific capacity (~294 mAh·g$^{-1}$) [1]. However, during operation, the material rapidly degrades and loses its initial specific capacity. One possible way to improve the electrochemical characteristics is to increase the concentration of $V^{4+}$ ions [2].

This work aimed to evaluate the effect of the phase composition of cathode material based on vanadium oxide nanofibers on the performance characteristics of a lithium-ion battery.

## 2. Experimental

Vanadium pentoxide nanofibers were synthesized by electrospinning. The precursor was synthesized by dissolving vanadium pentoxide powder in hydrogen peroxide; high-molecular-weight polyvinylpyrrolidone (PVP) was added to the resulting gel [3]. The solution feed rate was 0.3–0.35 mL/h, and the voltage between the collector and the needle was 17 kV.

Then, the vanadium pentoxide nanofibers were annealed: first in air, then in argon in the presence of oxalic acid powder [4]. At the first stage, PVP was removed from the fibers. The second stage led to partial reduction of vanadium pentoxide. Three variants of molar ratios between vanadium pentoxide nanofibers and oxalic acid were used: (2:1), (1:2), and (1:3). As a result, different ratios of $V^{4+}/V^{5+}$ ions in the fibers were obtained.

Photographs of the samples were obtained using a Melytec SM 32 scanning electron microscope with an EDAX Octane Elite Super detector (accelerating voltage – 20 kV); elemental analysis was also performed (data processing was carried out using EDAX APEX Advanced software without the use of standards).

X-ray phase analysis was performed using an automatic powder diffractometer D2 Phaser (Bruker) with Cu$K\alpha_1\alpha_2$ X-ray tube radiation, wavelengths $\lambda_{CuK\alpha1}$ = 1.54059 Å and $\lambda_{CuK\alpha2}$ = 1.54443 Å. Samples were prepared by dry pressing of the studied substance into a low-background monocrystalline silicon cuvette. Phase identification and quantitative Rietveld analysis were carried out using PDXL2 (Rigaku) and TOPAS software, the Powder Diffraction File (PDF-2, 2020) powder diffraction database, as well as structural data from the Inorganic Crystal Structure Database (ICSD, 2023).

The cathode material was prepared by grinding in a mortar to a homogeneous state the studied material (active component), conductive carbon black (SuperP), and a binder (polyvinylidene fluoride, PVDF, battery-grade) in a ratio of 60:30:10. Then, N-methylpyrrolidone was added. The resulting mixture was applied to graphitized aluminum foil using an applicator (coating thickness – 150 μm). The samples were dried in an oven at 100 °C for 24 h. Then, the samples were rolled on a roller press to improve contact between the coating and the aluminum substrate. Subsequently, the electrodes were punched out, and the average areal mass loading of the finished electrodes was 1.01 mg/cm$^2$.

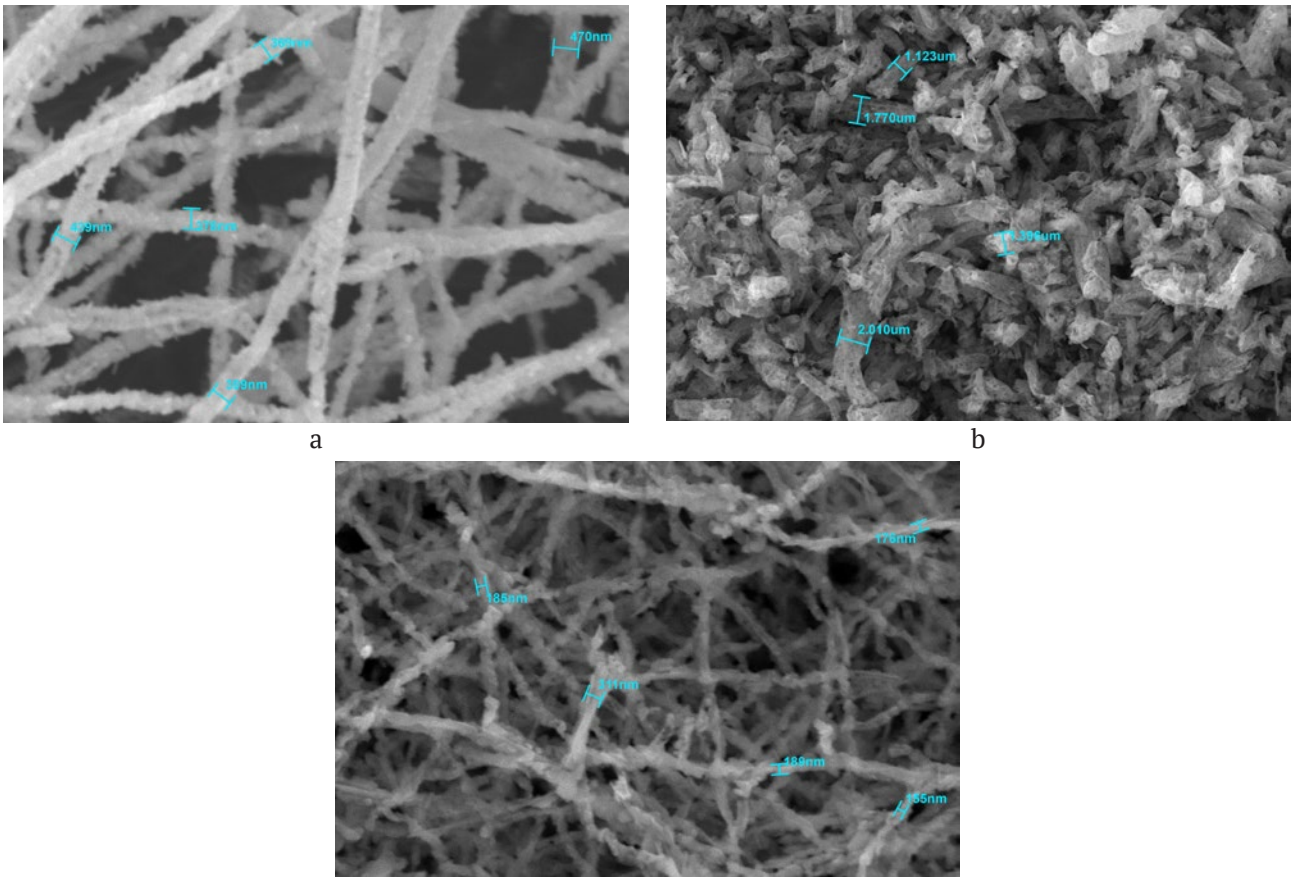
To study the electrochemical characteristics of the material, CR2032 disc lithium-ion batteries (coin-type cells) were assembled. The assembly was performed in an argon atmosphere in a glove box. Lithium foil was used as the counter electrode. The electrodes were separated by a Celgard separator; 1 M $LiPF_6$ in an EC:DMC:DEC (1:1:1) mixture was used as the electrolyte.

Cyclic voltammetry was performed using a Biologic VMP-3 potentiostat–galvanostat in the potential range of 2.0–4.0 V at a scan rate of 0.1 mV/s. Charge–discharge cycling at constant and variable current densities was carried out using a Neware BTS4000 battery cycler in the same potential range at a current density of 0.2 A/g.

## 3. Results and discussion

### *3.1. Scanning electron microscopy*

Photographs of samples with different molar ratios between vanadium pentoxide nanofibers and oxalic acid during annealing are shown in Fig. 1. The average nanofiber diameter and mass percentage ratios between vanadium and oxygen




a b c

**Fig. 1.** Photographs of samples with different molar ratios between vanadium pentoxide nanofibers and oxalic acid obtained by a scanning electron microscope: a – 2:1; b – 1:2; c – 1:3

according to the elemental analysis results are presented in Table 1. All samples were multiphase.

During annealing in an argon atmosphere, vanadium pentoxide is reduced by carbon monoxide (oxalic acid decomposes with the release of carbon monoxide). The elemental analysis results show that as the molar fraction of oxalic acid during annealing increased, the percentage of vanadium increased while that of oxygen decreased.

### *3.2. X-ray phase analysis*

X-ray diffraction patterns of samples with different molar ratios between vanadium pentoxide nanofibers and oxalic acid during annealing are shown in Fig. 2. According to the X-ray phase analysis results, the samples were mixtures of vanadium oxides ($V_2O_5$, $VO_2$, $V_4O_9$, $V_6O_{13}$) in various ratios. For samples with molar ratios of 2:1 and 1:2, the quantitative phase ratios were determined and are presented in Table 2. For the sample with a molar ratio of 1:3, quantitative determination was not possible since structural data for some of the vanadium oxide reference cards are unavailable. From the diffraction pattern of the third sample, it can be stated that the predominant phases were $V_6O_{13}$ and $VO_2$. As the molar fraction of oxalic acid during annealing increased, the percentage of vanadium pentoxide decreased while that of vanadium dioxide increased.

**Table 1.** Elemental analysis results of samples with different molar ratios between vanadium pentoxide nanofibers and oxalic acid

| Molar ratio | Average particle size, nm | Oxygen, % | Vanadium, % |
|---|---|---|---|
| 2:1 | 408 | 51.5 | 39.2 |
| 1:2 | 1666 | 30.0 | 67.1 |
| 1:3 | 281 | 8.8 | 87.2 |

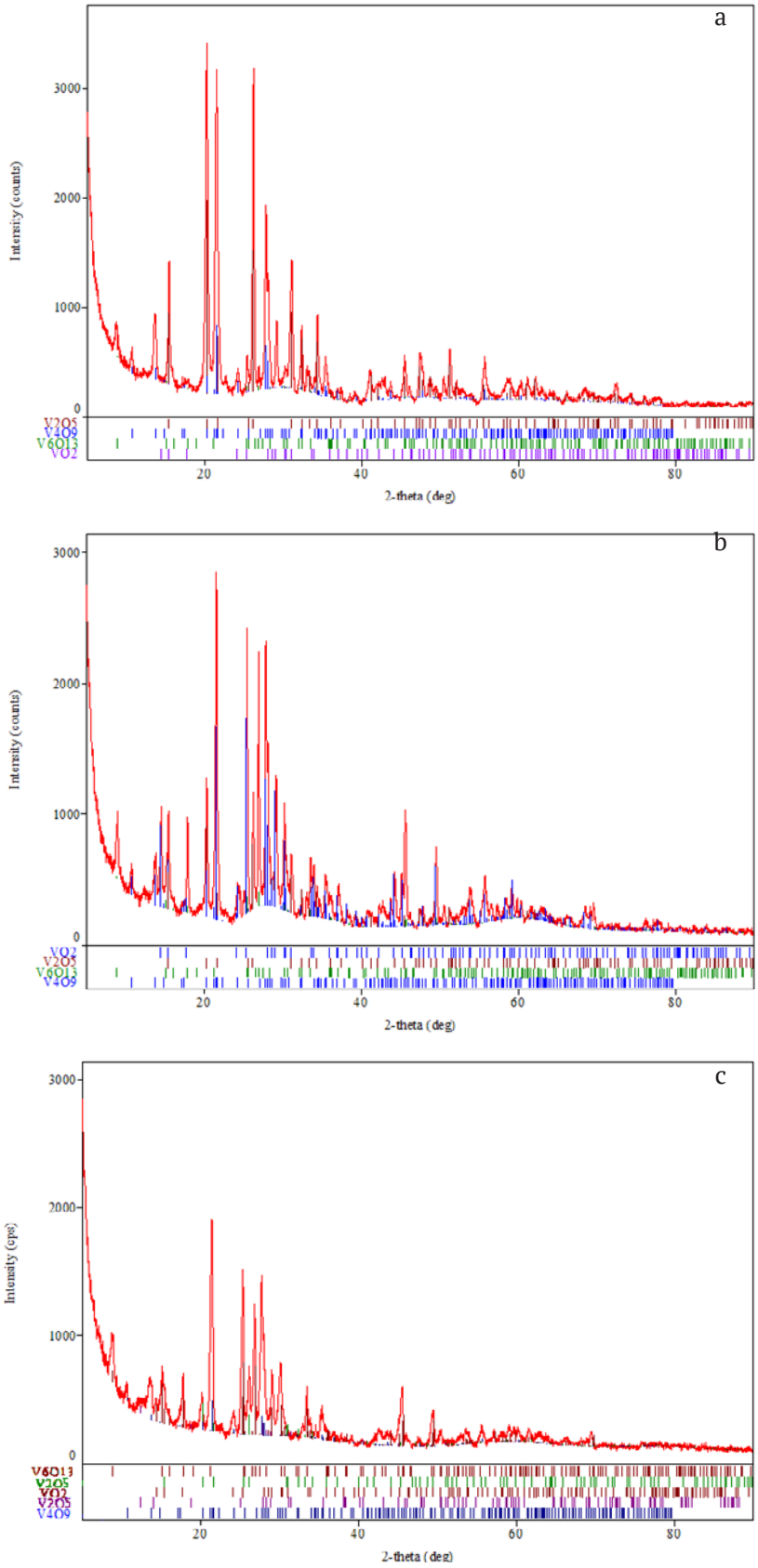



**Fig. 2.** X-ray images of samples with different molar ratios between vanadium pentoxide nanofibers and oxalic acid: a – 2:1; b – 1:2; c – 1:3

**Table 2.** Quantitative phase composition of samples with different molar ratios between vanadium pentoxide nanofibers and oxalic acid

| Molar ratio | $V_2O_5$, % ICDD 01-085-0601 | $VO_2$, % ICDD 01-084-7141 | $V_4O_9$, % ICDD 01-079-7317 | $V_6O_{13}$, % ICDD 01-072-1278 |
|---|---|---|---|---|
| 2:1 | 43.94 | 4.2 | 39.96 | 11.89 |

### *3.3. Electrochemical studies*

Cyclic voltammograms of samples with different molar ratios between vanadium pentoxide nanofibers and oxalic acid during annealing are shown in Fig. 3. The voltammograms were recorded in the potential range of 2.0–4.0 V vs. $Li^+$, which is a standard range for this material. As expected for multiphase materials, many peaks were observed in the graph. The peaks at ~2.67 V and ~2.54 V corresponded to $VO_2$; the peaks at ~3.44 V, ~3.38 V, ~3.24 V, ~3.18 V, ~2.25 V, and ~2.28 V corresponded to $V_2O_5$; the remaining peaks corresponded to other vanadium oxide modifications ($V_3O_4$, $V_6O_{13}$ [5–11]. With an increase in the oxalic acid fraction during annealing, the intensity of the peaks corresponding to vanadium pentoxide decreased, while the intensity of the peaks corresponding to $VO_2$ and $V_6O_{13}$ increased. In the sample with a molar ratio of 1:3 between vanadium pentoxide nanofibers and oxalic acid during annealing, the peaks of vanadium pentoxide were practically absent.

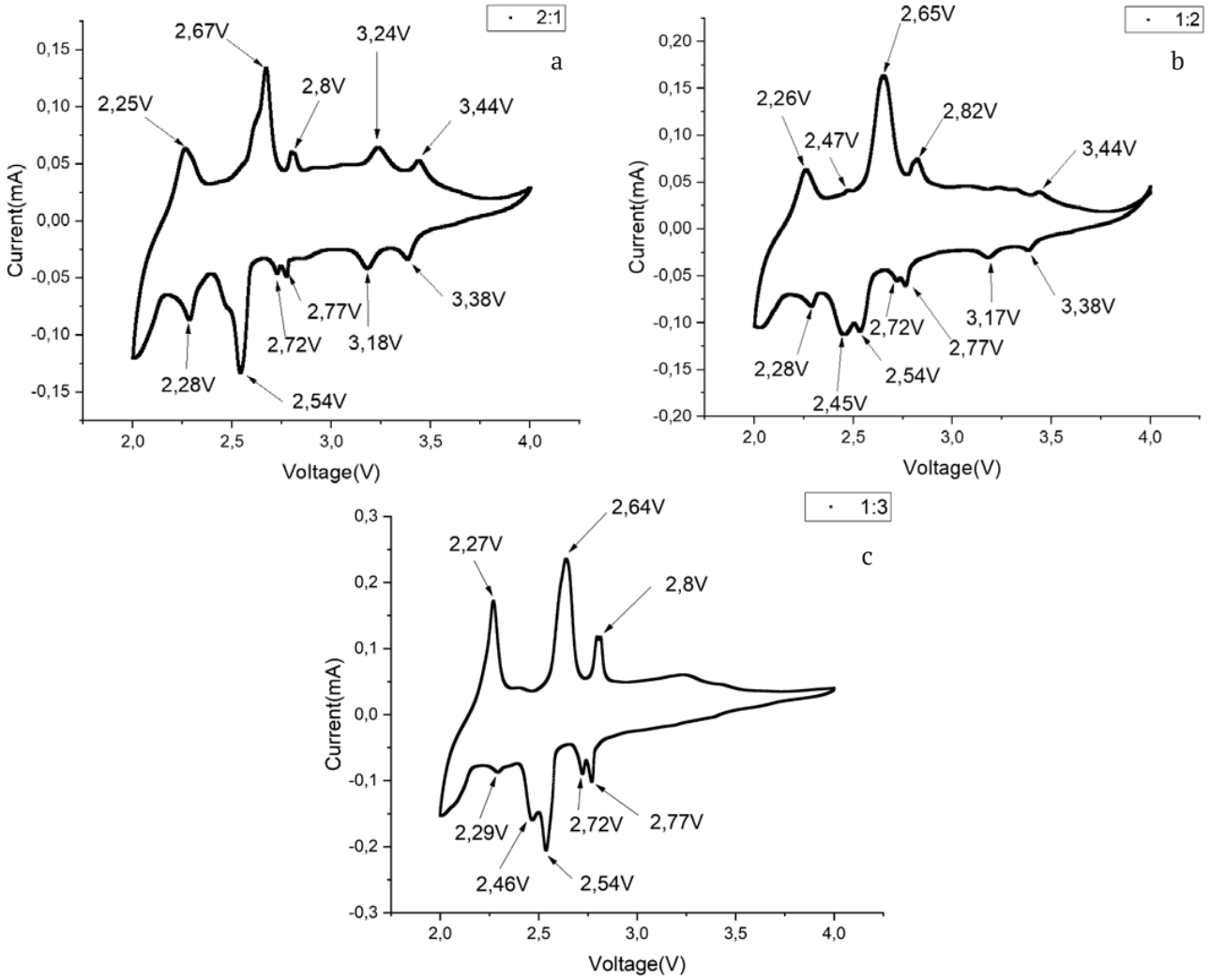



**Fig. 3.** Cyclic voltammograms of samples with different molar ratios between vanadium pentoxide nanofibers and oxalic acid: a – 2:1; b – 1:2; c – 1:3

The results of long-term charge–discharge cycling of the materials are shown in Fig. 4. As expected, with an increase in the concentration of $V^{4+}$ ions in the nanofibers, the initial specific capacity decreased, which was consistent with literature data [6, 7, 12–14]. However, contrary to expectations, the material did not become more stable. In the sample with a molar ratio of 3:1 between vanadium pentoxide nanofibers and oxalic acid during annealing, the initial capacity increased again. This may be due to the greater stoichiometry of the material (vanadium dioxide and the very similar phase $V_6O_{13}$ predominated in the material). After the first cycle, all materials lost about 11–12 % of their initial specific capacity. This may be due to irreversible processes as well as initial structural rearrangement of the material. After 50 charge–discharge cycles, the materials lost about 15–20 % of their specific capacity relative to the capacity of the second cycle. The capacity losses after 100 charge–discharge cycles were: 31 % for vanadium pentoxide nanofibers; 43.2 % for the material with a molar ratio of 2:1 between vanadium pentoxide nanofibers and oxalic acid during annealing; 29.9 % for the material with a molar ratio of 1:2 between vanadium pentoxide nanofibers and oxalic acid during annealing; and 37.6 % for the material with a molar ratio of 1:3

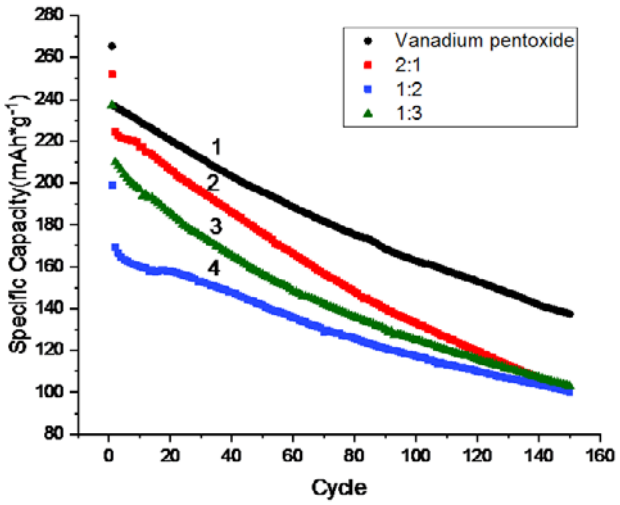



**Fig. 4.** Dependence of charge-discharge cycle number on the specific capacity of samples with different molar ratios between vanadium pentoxide nanofibers and oxalic acid: *1* – vanadium pentoxide; *2* – 2:1; *3* – 1:3; *4* – 1:2

between vanadium pentoxide nanofibers and oxalic acid during annealing.

## 4. Conclusions

Cathode materials consisting of a mixture of vanadium oxides with different phase ratios were synthesized. All obtained materials demonstrated a significant decrease in specific capacity during cycling. With an increase in the concentration of $V^{4+}$ ions in the nanofibers, the initial specific capacity of the material was lower compared to vanadium pentoxide nanofibers. An increase in the concentration of $V^{4+}$ ions did not lead to an improvement in stability during long-term cycling.

The results suggest that controlling the degree of $V_2O_5$ reduction alone is insufficient to ensure long-term cycling stability. Accordingly, future research should prioritize combined structural modification strategies, including the simultaneous implementation of reduction and cation doping, as such approaches may offer a viable pathway to materials with improved cycling performance.

## Authors' contribution

A. S. Sharlaev – conducted experimental studies, methodology development, writing of the original draft, conclusions. O. Ya. Berezina – supervision, methodology development, review and editing of the manuscript. F. S. Volkov – conducted experimental studies. S. N. Eliseeva – supervision, electrochemical methodology, review and editing of the manuscript.

## Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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