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Hydrothermal Synthesis and Electrochemical Properties of Metal Oxide Based Nanocomposites

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Abstract: This article analyzes approaches to the preparation and evaluation of metal oxide composite electrodes based on studies by researchers from Uzbekistan and Russia. Particular attention is paid to the intermediate phase formed during hydrothermal synthesis, subsequent heat treatment, and contact with the carbon substrate. Studies of NiO-based materials are considered alongside publications on carbon–$NiCo_2O_4$ composites, reduced graphene oxide, and gel polymer electrolytes. The findings are distinguished according to the synthesis method and testing configuration. This study is a targeted literature review rather than a new laboratory investigation. Based on the analysis, an experimental plan is proposed that focuses on control samples, independent synthesis replicates, and electrode–electrolyte compatibility testing.

Keywords: Hydrothermal synthesis, nickel oxide, nanocomposite, carbon substrate, supercapacitor, specific capacitance, gel polymer electrolyte.

Introduction: The properties of an electrode combining a metal oxide and a carbon component cannot be explained solely by its chemical composition. The distribution of the active phase, its formation conditions, and its contact with the current collector are also important. Studies of nickel oxide by T. L. Simonenko and co-authors demonstrate the need to distinguish the hydrothermal stage from the formation of the final oxide [1, 2]. T. A. Larichev and co-authors investigated a composite incorporating nickel cobaltite into a carbon matrix [3]. A study led by S. P. Gubin examined the electrochemical reduction of graphene oxide and the dependence of the resulting electrode response on charge–discharge conditions [4]. These studies are useful for distinguishing the contributions of the oxide phase and the conductive substrate, although not all of them involve hydrothermal synthesis. Research by J. Shodmanov and A. Boymirzaev from Uzbekistan addresses the mechanical and structural properties of gel polymer electrolytes for flexible supercapacitors [5, 6]. These studies are relevant to this review because electrode material development also requires consideration of the electrolyte.

The aim of this study is to clarify the relationships among synthesis, structure, and electrochemical properties using the selected sources and to formulate a testable approach for subsequent experiments.

A nanocomposite consists of at least two constituent components and contains structural features on the nanometer scale. The interface is the region where these components meet. Electron transport to the current collector and electrolyte ion transport to the active surface are distinct processes. Improving one does not automatically accelerate the other.

Specific capacitance is the electrical capacitance per unit mass. It should not be equated with surface area: geometric surface area, surface area determined by gas adsorption, and electrochemically active surface area are different quantities. Pores that are inaccessible to the electrolyte may not contribute fully to charge storage during measurement, even though they are present in the geometric structure.

METHODS

Source Selection

Six publications by authors working at scientific institutions in Russia and Uzbekistan were selected for this review. Bibliographic information was checked against publisher webpages and DOI records. Two studies on NiO synthesis [1, 2] were used as the main examples of preparation methods, while studies on carbon–$NiCo_2O_4$ and graphene electrodes [3, 4] provided comparative material. Two publications on gel polymer electrolytes [5, 6] were used as evidence concerning the electrolyte component of the device. This selection does not constitute a systematic review of all available publications. Sources were selected according to the authors' institutional affiliations and their relevance to the topic; publication language was not treated as an indicator of the authors' research affiliations. In the source manuscript, the titles of some papers published in English were identified as translations in the reference list, while their DOIs were retained unchanged.

Criteria for Data Comparison

For each publication, the synthesis method, resulting phase, use of heat treatment, electrode configuration, and measurement units were identified. Capacitance values obtained for different compositions were not combined into a single ranking. The chart uses only two values measured at different current densities for the same electrode type within a single study [2]. No intermediate values were estimated.

Provenance of Images and Results. The micrographs were taken from an openly licensed publication by Russian authors [2]. They are not laboratory images acquired by the authors of this review. The original source and license are indicated below each image. The chart was redrawn using numerical values reported in the publication and should not be interpreted as new experimental data.

A targeted literature review should explicitly state its selection criteria and the limits of its conclusions. An analysis based on six sources cannot provide a statistically representative account of an entire research field. Because the selected publications differ in material composition and experimental configuration, direct comparisons of their maximum capacitance values are limited.

It is useful to distinguish three levels of evidence: results measured by the original authors, quantities calculated from those results, and hypotheses proposed for future experiments. This distinction improves the verifiability of scientific conclusions. A calculated ratio is not presented as a new laboratory measurement.

RESULTS

The Hydrothermal Stage and Oxide Formation. Simonenko and co-authors investigated the preparation of nickel oxide powder in the presence of triethanolamine. Hydrothermal treatment was performed at 140 °C for 1 h. Subsequent heat treatment of the intermediate α-$Ni(OH)_2$ product at 350 °C for 1 h yielded NiO with an average coherent diffraction domain size of approximately 6 nm [1]. This quantity must not be equated with the diameter of an entire agglomerate. A different procedure was used to prepare the NiO/CP electrode on carbon paper: hydrothermal treatment at 200 °C for 1 h followed by heating at 300 °C for 2 h [2]. The synthesis parameters for the powder and the coating therefore cannot be assumed to be identical.

In hydrothermal synthesis, a precursor solution is heated in a sealed reactor. A precursor is a starting chemical compound from which the final product is formed. Nucleation refers to the formation of nuclei of a new phase, whereas crystal growth is the enlargement of the nuclei that have formed. These processes depend on the solution composition, temperature, and treatment duration.

A complexing agent changes the chemical speciation of metal ions in solution. The role of triethanolamine therefore cannot be limited to creating an alkaline medium. However, specific chemical evidence is required to establish a particular mechanism. During heat treatment, the conversion of the hydroxide into the oxide may be accompanied by particle coalescence; the resulting morphology need not be identical to that of the initial precipitate.

Morphological and Microstructural Analysis

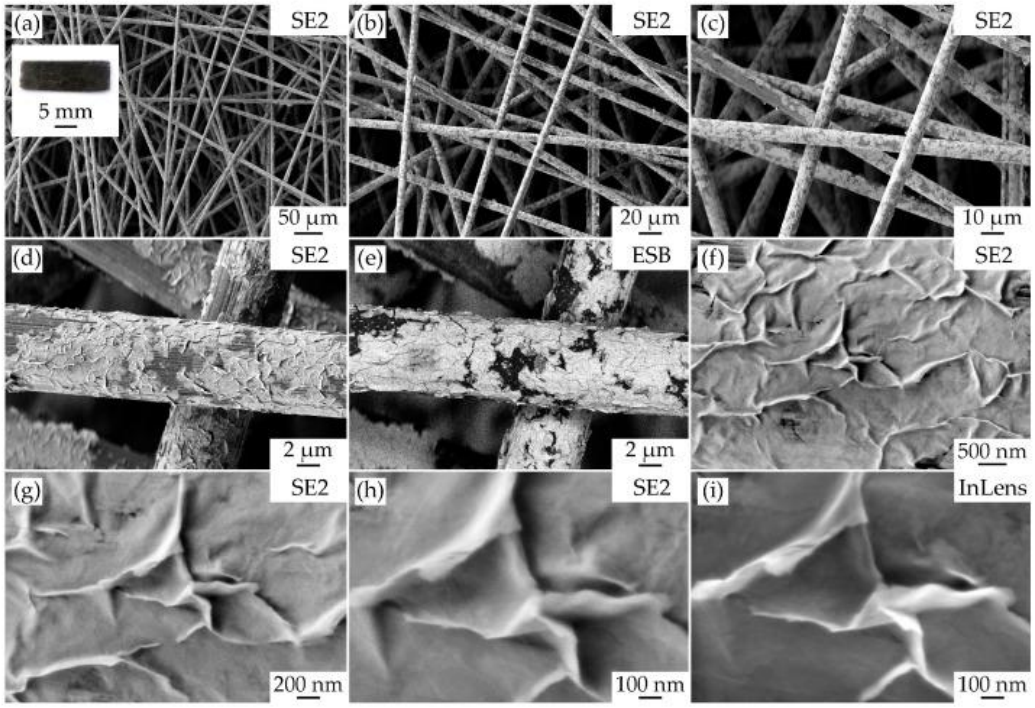


Figure 1. SEM micrographs of the NiO/CP electrode; the inset in panel (a) shows the macroscopic appearance of the sample.

Source: Simonenko et al. [2], Materials, 2023, 16, 5208. CC BY 4.0. Figure 2 in the original article.

A micrograph reveals morphology; on its own, it does not fully establish phase composition, electrical conductivity, or the charge storage mechanism. Such conclusions require joint consideration of structural and electrochemical analyses.

Electron microscopy images are interpreted at different magnifications. An overview shows the distribution of the coating along the fibers, while higher magnification helps distinguish local surface features. Images from multiple locations are required to assess whether a single field of view is representative of the entire electrode.

An agglomerate is an assembly of several particles and may be larger than an individual crystallite. Using the same term for a coherent diffraction domain measured by X-ray diffraction and a particle diameter observed in an SEM image misrepresents the meaning of the measurements.

Interfacial Contact and Elemental Distribution

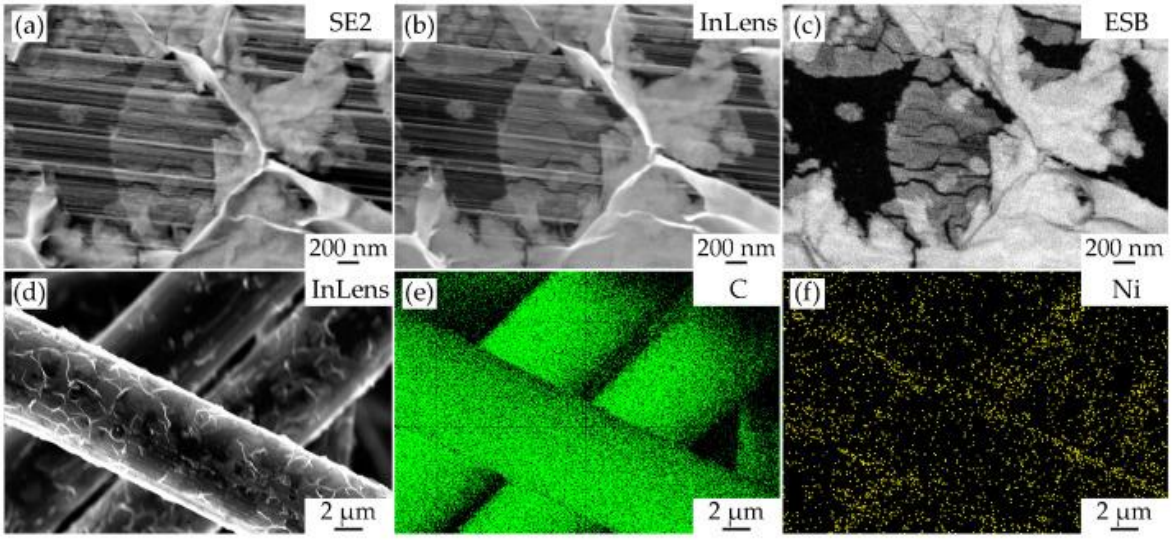


Figure 2. Contact between NiO nanosheets and a carbon fiber (a–c), the analysis region (d), and the distributions of carbon (e) and nickel (f).

Source: Simonenko et al. [2], Materials, 2023, 16, 5208. CC BY 4.0. Figure 3 in the original article.

An elemental map shows the spatial distribution of a particular element within a selected region. The co-location of the nickel signal and the carbon fiber helps establish the position of the coating, but does not, by itself, identify the type of chemical bonding. Appropriate spectroscopic evidence is required to draw conclusions about oxidation states.

Adhesion is the ability of a coating to adhere to its substrate. Coating delamination during bending may impair electrical contact. A flexible electrode should therefore be evaluated by comparing its electrical response before and after deformation, in addition to examining its appearance.

Calculation of Electrochemical Quantities

Capacitance derived from constant-current discharge can be normalized by mass or area using the following expressions:

$C_m = I\Delta t / (m\Delta V)$ (1) $C_a = I\Delta t / (A\Delta V)$ (2)

Here, I is the discharge current, Δt is the discharge time, m is the mass of active material included in the calculation, A is the electrode area, and ΔV is the potential window after excluding the ohmic drop. When interpreting the result, it is necessary to state whether the current collector or binder is included in the mass used for normalization. For a nonlinear discharge curve, these expressions yield the average capacitance over the selected interval.

The charge per unit mass is expressed as $q_m = I\Delta t/m$. When current is given in amperes, time in seconds, and mass in grams, q_m has units of C g^{-1}; conversion to mAh g^{-1} requires division by 3.6. Faradaic processes, capacitance, and energy density are not interchangeable concepts.

In a galvanostatic measurement, the current is held constant while the change in potential with time is recorded. The ohmic drop is a rapid potential change caused by electrical resistance. If the potential interval used in the calculation is not explicitly specified, capacitance values from different studies become difficult to compare.

In a three-electrode measurement, the potential of the working electrode is controlled relative to a reference electrode. In a two-electrode device, the measured voltage reflects the combined response of both electrodes. Consequently, the energy density of a complete device cannot be derived directly from the capacitance of an individual electrode. Active material mass, current collector mass, and total device mass represent different bases for normalization.

Rate Performance and Cycling Stability

The NiO/CP electrode was tested in a three-electrode configuration using 3 M KOH solution. As the current density increased from 0.5 to 20 A g^{-1}, the specific capacitance decreased from 207 to 160 F g^{-1}; 95% capacitance retention was reported after 2,000 cycles at 5 A g^{-1} [2]. These results pertain to an individual electrode.

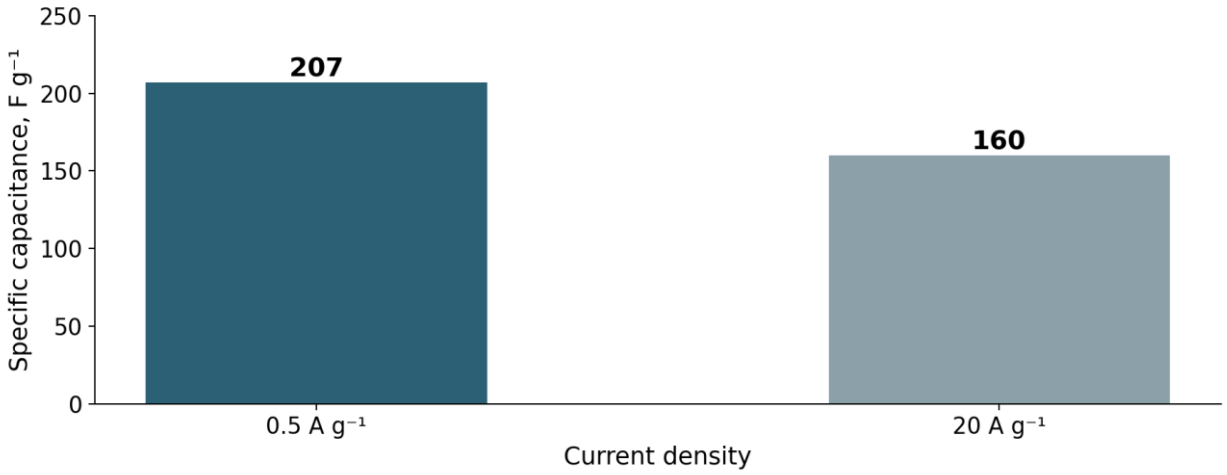


Figure 3. Capacitance of NiO/CP at two current densities.

The ratio 160/207 × 100 = 77.3% represents capacitance retention at the higher current density. It must not be confused with the 95% retention after 2,000 cycles: current density varies in the first comparison, whereas the number of cycles varies in the second.

Increasing the current density changes the fraction of active sites that can be accessed within a short time. A decrease in capacitance at high current should not be attributed solely to material degradation; limitations associated with ion transport and electrical contact must also be considered.

No general relationship or regression equation for all intermediate current densities was constructed from the two values shown in the chart. The absence of error bars does not imply zero measurement error. Without information on independent replicates, statistical

confidence intervals cannot be assigned arbitrarily.

DISCUSSION

Contributions of the Oxide and Carbon Phases

Larichev and co-authors prepared a carbon/$NiCo_2O_4$ composite by thermally decomposing mixed nickel–cobalt hydroxides on the surface of carbon nanofibers. Within the range of compositions investigated, they reported an increase in capacitance with increasing nickel cobaltite content [3]. Although this finding is relevant to oxide–carbon composites, the synthesis cannot be described as hydrothermal. Moreover, the observed relationship does not imply an unlimited increase across all compositions.

Gubin and co-authors investigated the electrochemical reduction of graphene oxide electrodes in a symmetric capacitor. They demonstrated that specific capacitance depends on the charge–discharge rate and the operating voltage window [4]. This study provides a methodological example of how to monitor the response of the carbon component separately; it does not demonstrate the hydrothermal preparation of a metal oxide composite.

The carbon component can act as an electronically conductive network. Percolation refers to the formation of continuous conducting pathways. Increasing the oxide fraction may increase the number of active sites, but an excessively thick layer can hinder ion access to internal regions. The optimal composition must therefore be determined experimentally.

The term synergy should not be applied automatically whenever a composite exhibits high performance. To substantiate synergy, the individual components and the composite must be tested using the same electrolyte, loading, and potential window. Otherwise, the observed difference may arise from different experimental conditions rather than from interactions between the components.

Contributions of Researchers from Uzbekistan

J. Shodmanov and A. Boymirzaev investigated double-network gel polymer electrolytes based on gellan gum and polyacrylamide. Their study considered deformation tolerance and self-healing behavior alongside structural characterization of the material [5]. Such electrolytes are relevant to the development of flexible devices, but these results do not directly substantiate the capacitance of a NiO electrode.

A second study by the same authors examined the mechanical response of a compressible and bendable gel polymer electrolyte and its recovery under different conditions [6]. Because both publications are related studies from the same research group, they should not be regarded as corroboration by independent laboratories. A practical task arising from this research is to test the electrochemical compatibility of the gel with the selected oxide electrode separately.

In a gel polymer electrolyte, the polymer network acts as a framework that retains the liquid phase. Ionic conductivity characterizes the ability of ions to move through the medium; it differs from the electronic conductivity of the electrode. Increased mechanical strength does not necessarily imply improved electrochemical performance.

Wetting and the contact area between the electrode and the gel may affect device resistance. When evaluating compatibility, the gel composition, water retention, deformation state, and testing temperature should be recorded. If self-healing is observed in a mechanical test, complete recovery of the electrical properties must be verified separately.

Proposal for Subsequent Experiments

The testable hypothesis is that, at the same active material mass and under the same electrolyte conditions, the duration of hydrothermal treatment affects the distribution of the oxide on the carbon substrate and capacitance retention as the current increases. Initially, only one factor should be varied, and control samples comprising the bare substrate, pure oxide, and composite should be compared. At least three independently synthesized batches are proposed for each condition. Phase composition will be assessed by X-ray diffraction and morphology by electron microscopy. Electrochemical characterization will combine voltammetry, galvanostatic charge–discharge measurements, and impedance spectroscopy. The electrolyte, reference electrode, potential window, active material mass, and electrode area must be clearly specified in the report. This is a proposal for future work, not an experiment that has already been performed.

CONCLUSION

Studies of NiO by Russian researchers demonstrate the need to describe hydrothermal synthesis and subsequent heat treatment as distinct stages. When analyzing results for oxide–carbon composites, the synthesis route, active material loading, and testing configuration must be considered together. Studies of gel polymer electrolytes by authors from Uzbekistan also provide a basis for evaluating the mechanical properties of the electrolyte when developing a flexible device.

This review is limited to six selected publications; no new synthesis or independent electrochemical measurements were performed. Results from different sources were not statistically pooled. In future

research, control samples, independent replicates, and testing in a two-electrode device will be required to substantiate the advantages of the material.

The experimental design distinguishes an independent synthesis replicate from a repeated measurement of the same sample. The former evaluates the reproducibility of preparation, whereas the latter assesses measurement stability. Results should be reported as mean values together with a measure of dispersion.

X-ray diffraction for phase composition, SEM for morphology, and voltammetry and impedance spectroscopy for electrical response provide complementary information. Impedance describes the complex opposition to alternating current, and its frequency dependence helps distinguish different processes. An equivalent electrical circuit is not a unique physical interpretation; the choice of circuit and the quality of the fit must be justified.

The practical outcome of this review is the identification of factors that must be controlled in future experiments, rather than a new capacitance record.

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