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Preparation and Electrochemical Performance of NCM622/Carbon-Fiber-Network Composite Cathodes via Electrospinning and Carbonization

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Abstract

LiNi_{0.6}Co_{0.2}Mn_{0.2}O₂ (NCM622) is a representative nickel-rich layered cathode material for lithium-ion batteries, but its practical performance can be limited by discontinuous electronic pathways and contact deterioration within composite electrodes. In this work, an NCM622/carbon-fiber-network composite (NCM622-C) was prepared by electrospinning followed by nitrogen-protected carbonization at 600 °C. NCM622 particles were incorporated into a polyvinylpyrrolidone (PVP)/ethanol spinning precursor and subsequently integrated with the PVP-derived carbon-fiber framework. Scanning electron microscopy (SEM) showed a particle/fiber interwoven morphology, while X-ray diffraction (XRD) confirmed preservation of the layered alpha-NaFeO₂-type NCM622 structure after carbonization. The estimated I(003)/I(104) ratio was approximately 1.80, indicating that the layered ordering was maintained. At 1 C, NCM622-C delivered initial charge and discharge capacities of about 147 and 146 mAh g⁻¹, respectively, with an initial Coulombic efficiency of 99.3%, compared with 143/140 mAh g⁻¹ and 97.9% for pristine NCM622. After 50 cycles at 1 C, NCM622-C retained about 141.6 mAh g⁻¹ (96.1% retention), exceeding the 129.8 mAh g⁻¹ (92.7%) of pristine NCM622. NCM622-C also showed higher capacities at low-to-medium rates and better capacity recovery after returning to 0.5 C. These results support electrospinning-derived carbon-fiber networking as a practical route to improve electronic connectivity and short-term capacity retention of NCM622 cathodes.

Keywords: NCM622; electrospinning; carbon fiber network; lithium-ion battery; cathode; electrochemical performance

1. Introduction

Lithium-ion batteries are central to portable electronics, electric vehicles, and stationary energy storage because of their high energy density, long service life, and mature manufacturing base [1-3]. Among the major cell components, the positive electrode strongly influences energy density, cost, safety, and cycle life, which has motivated sustained development of layered oxide cathodes with higher reversible capacity and improved structural durability [3,4].

Layered nickel-cobalt-manganese oxides, LiNi_xCo_yMn_zO₂ (NCM), are widely studied because they combine relatively high capacity with reduced cobalt usage and tunable structural properties [4,5]. LiNi_{0.6}Co_{0.2}Mn_{0.2}O₂ (NCM622) is particularly useful as a representative Ni-rich composition: increasing the Ni fraction raises accessible capacity, while Co and Mn contribute to electronic/structural stabilization [5,23]. However, repeated Li⁺ extraction and insertion can cause lattice-parameter changes, local stress accumulation, particle-contact deterioration, and surface reconstruction. Li/Ni cation mixing and interfacial reactions can additionally impede reversible Li⁺ transport and accelerate polarization [6-10,24].

A variety of strategies have therefore been explored for NCM622 and related Ni-rich cathodes, including elemental doping, surface coating, precursor/morphology control, and conductive-carbon modification [7,12-17,25,26]. Coatings and dopants can stabilize the near-surface or bulk structure, but they may introduce additional synthesis complexity. Carbon-based modification is attractive because a conductive carbon phase can complement the intrinsically limited electronic conductivity of layered oxides. Nevertheless, conventional addition of particulate conductive agents mainly forms point-to-point contacts and may not provide a continuous conductive framework when local particle contacts evolve during cycling [15-18].

The spatial architecture of the conductive phase is therefore important. Kang et al. demonstrated that interweaving bulk carbon fibers with highly loaded NCM622 can establish a three-dimensional electronic network and improve

rate capability [18]. This result highlights the value of fibrous conductive frameworks, while also indicating a remaining opportunity to integrate the active material and fiber precursor more intimately before electrode fabrication. Electrospinning provides such a route because continuous polymer fibers can be formed directly from a precursor containing active particles and then converted to carbon fibers by heat treatment [19-22].

In contrast to ex situ addition of preformed carbon fibers, the present study incorporates NCM622 particles into a PVP/ethanol electrospinning precursor so that particle/fiber contact is established during fiber formation and retained after carbonization. The resulting NCM622-C composite is evaluated against a real pristine-NCM622 control prepared and tested under the same electrochemical conditions. The study focuses on three experimentally supported questions: whether the layered NCM622 structure is preserved after electrospinning/carbonization, whether the carbon-fiber network improves initial reversibility and 50-cycle capacity retention, and whether it improves the response to changing C-rates. The conclusions are intentionally limited to these measured structural and electrochemical outcomes rather than to unmeasured interfacial or diffusion mechanisms.

2. Materials and Methods

2.1. Materials

Commercial NCM622-A powder was supplied by Keluode (China). Polyvinylpyrrolidone (PVP) was obtained from Macklin, and anhydrous ethanol (analytical grade) and N-methyl-2-pyrrolidone (NMP) were obtained from Sinopharm Chemical Reagent Co., Ltd. Poly(vinylidene fluoride) (PVDF), a commercial battery-grade electrolyte, CR2016 coin-cell hardware, lithium metal, and separator materials were used as received. Super P conductive carbon was used as the conductive additive. The electrospinning-assisted design was based on the ability of electrospinning to generate continuous polymer-fiber networks that can be converted into carbonaceous frameworks after heat treatment [19-22].

2.2. Preparation of the NCM622-C Composite

The preparation route is shown in Fig. 1. For the spinning precursor, 0.3 g of NCM622-A, 1.0 g of PVP, and 13.2 g of anhydrous ethanol were used. PVP was slowly added to ethanol at room temperature and stirred at approximately 700 r min⁻¹ until a homogeneous polymer solution was obtained. NCM622-A powder was then added and the suspension was stirred for 12 h, followed by ultrasonic dispersion for 30 min. The total precursor volume was approximately 15 mL.

For electrospinning, the precursor was divided into three 5 mL syringes equipped with 27G needles and mounted on a YFSP-T high-voltage electrospinning system (Tianjin Yunfan Technology Co., Ltd., China). The feeding speed was set to approximately 0.001 mm s⁻¹, the swing amplitude to 40 cm, the positive voltage to 14 kV, and the collector-roller speed to 300 r min⁻¹. Aluminum foil was used on the collector. The obtained NCM622/PVP fibrous membrane was removed from the collector and vacuum-dried at 150 °C for 12 h.

The dried fibrous membrane was cut into approximately 10 cm × 3 cm strips and heat-treated in a tube furnace (F219793U, Hefei Kejing Materials Technology Co., Ltd., China). The sample was first heated in air to 250 °C and held for 3 h for stabilization. It was then heated to 600 °C under N₂ protection and held for 2 h. After the 600 °C hold, the sample was cooled to 200 °C at 5 °C min⁻¹ under N₂, after which the gas supply was stopped and the sample was allowed to cool naturally to room temperature. The carbonized product was ground to obtain the NCM622-C composite powder.

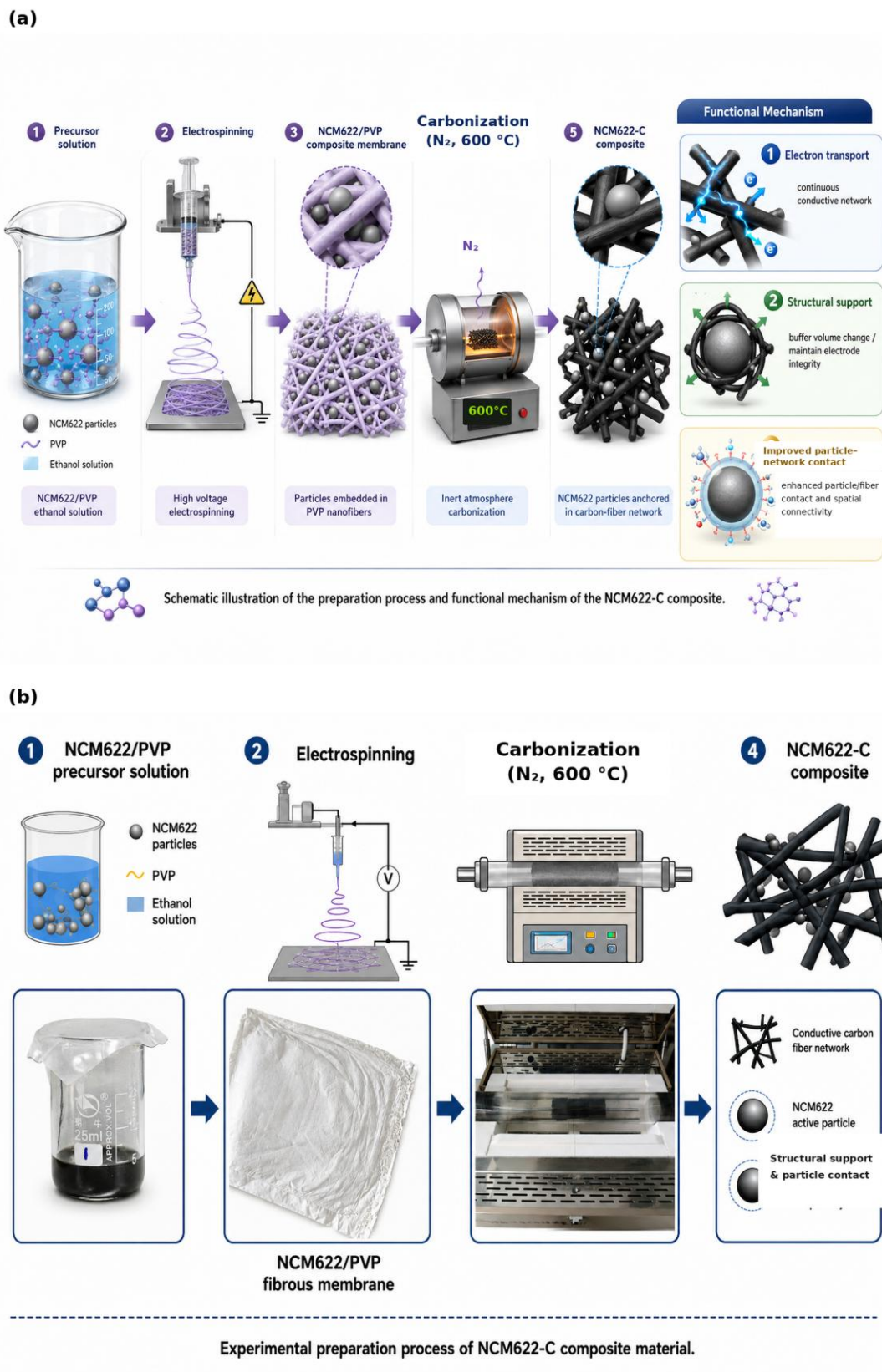


Fig. 1 (a) Schematic illustration of the electrospinning-carbonization route and the proposed roles of the carbon-fiber network; (b) experimental preparation process using the actual precursor solution, fibrous membrane, and heat-treatment product

2.3. Electrode Preparation and Coin-Cell Assembly

NCM622-C active material, Super P, and PVDF were mixed at a mass ratio of 8:1:1. For a representative batch, 0.16 g of active material, 0.02 g of PVDF, and 0.02 g of Super P were mixed, followed by addition of 600 μL of

NMP. The slurry was stirred for 5 h and coated onto aluminum foil with a 100 μm doctor blade. The coated electrodes were vacuum-dried at 100 $^{\circ}\text{C}$ for 12 h and punched into 13 mm disks. Pristine NCM622 control electrodes were prepared using the same active-material/Super-P/PVDF ratio, slurry preparation, coating, and drying procedure.

CR2016 half-cells were assembled in an Ar-filled glovebox (MKSS10045, Mikrouna, China) with H_2O and O_2 concentrations maintained below 0.01 ppm. Lithium metal was used as the counter/reference electrode. A commercial battery-grade electrolyte was used without further modification. During assembly, 20 μL of electrolyte was first added to wet the cathode, followed by placement of the separator and an additional 40 μL of electrolyte. The cells were sealed using an MRX-SF120 crimper and rested for 6 h at room temperature before testing.

2.4. Characterization and Electrochemical Measurements

The morphology of the NCM622-C composite was examined using a Zeiss Supra55 scanning electron microscope (SEM). X-ray diffraction (XRD) was performed using a Rigaku Ultima IV diffractometer. Diffraction data were collected over $2\theta = 5\text{-}80^{\circ}$ at a scan rate of $10^{\circ} \text{ min}^{-1}$ and compared with reference peak positions for layered NCM622. For layered NCM materials, the I(003)/I(104) intensity ratio is commonly used as a qualitative indicator of layered ordering and Li/Ni cation mixing [9-11]. In this work, the ratio is used only as an auxiliary structural indicator and not as a quantitative determination of cation disorder.

Galvanostatic tests were performed using a LAND CT3001A battery testing system (Wuhan LAND Electronic Co., Ltd., China) at room temperature over a 3.0-4.2 V window versus Li/Li^+ . Initial charge/discharge profiles and the 50-cycle cycling test were measured at 1 C. The rate sequence was 0.5 C, 1 C, 3 C, 5 C, and then 0.5 C again, with ten cycles at each rate. The current settings used in the original test program were 0.117, 0.234, 0.702, and 1.170 mA for 0.5 C, 1 C, 3 C, and 5 C, respectively. A nominal reference capacity of 172 mAh g^{-1} was used only to calculate and set the C-rate currents in the original test program; it was not treated as a strict theoretical upper limit for the experimentally measured specific capacity.

Specific capacity (Q_s), Coulombic efficiency (CE), and capacity retention after the nth cycle (R_n) were calculated as follows:

$$Q_s = Q / m \quad (1)$$

$$CE = Q_{\text{dis}} / Q_1 \times 100\% \quad (2)$$

$$R_n = Q_n / Q_1 \times 100\% \quad (3)$$

where Q is the measured capacity, m is the active-material mass used for normalization, Q_{dis} and Q_{chg} are the discharge and charge capacities, respectively, and Q_1 and Q_n are the first-cycle and nth-cycle discharge capacities. All NCM622-C and pristine-NCM622 electrochemical data reported in this manuscript are experimental control data obtained under the stated test conditions.

Human and animal rights: This study did not involve human participants or animals.

Use of AI-assisted language tools: AI-assisted tools were used during manuscript preparation for English-language editing and structural organization. All scientific content, experimental data, interpretations, and the final manuscript were reviewed and approved by the authors, who take full responsibility for the work.

3. Results

3.1. Morphology of the NCM622-C Composite

Figure 2 shows SEM images of the carbonized NCM622-C composite at different magnifications. The material exhibits a particle/fiber interwoven morphology rather than a simple packing of isolated particles. At higher magnification, NCM622 particles are observed on fiber surfaces and around fiber-junction regions. The characteristic carbon-fiber diameter is approximately 1 μm , while the observed NCM622 particle size is about 0.4 μm .

Local particle agglomeration is also present. This morphology is consistent with two likely contributions during processing: the relatively high surface energy of inorganic NCM622 particles promotes local aggregation in the

precursor, while shrinkage and carbonization of the PVP fibers can reduce particle-particle spacing during heat treatment. Despite these local agglomerates, a continuous fibrous framework remains visible after carbonization, confirming that the electrospinning-derived network survives the thermal treatment.

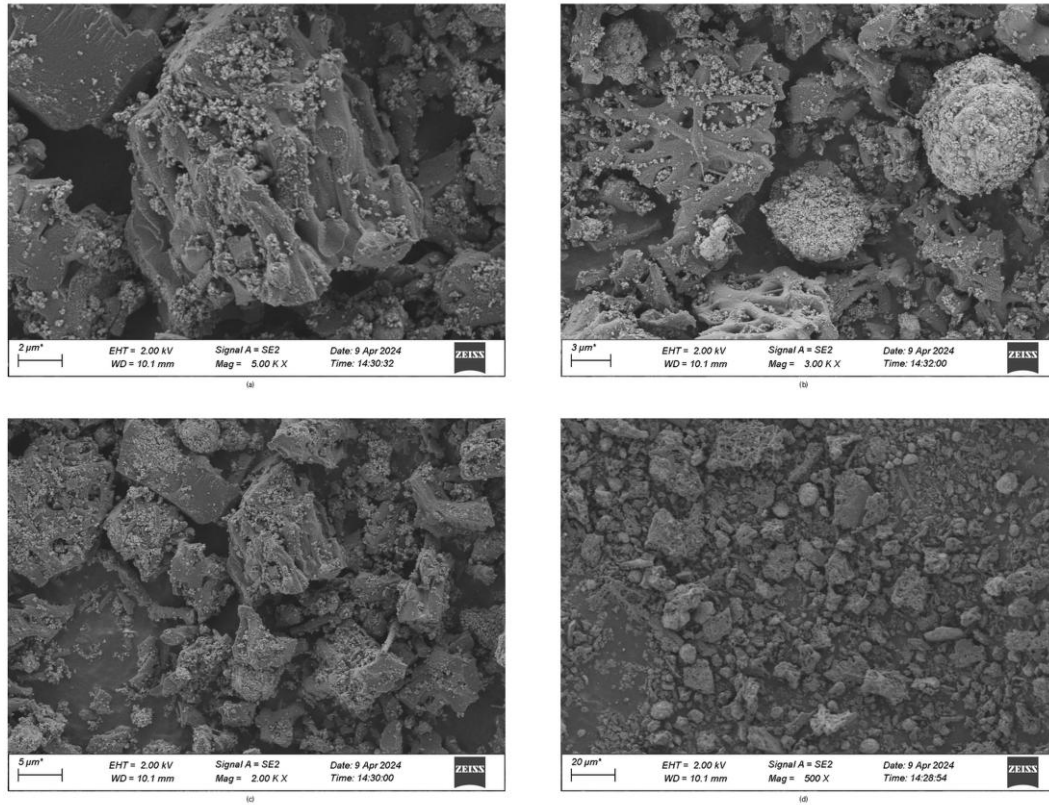


Fig. 2 SEM images of the NCM622-C composite at different magnifications

3.2. Crystal Structure

Figure 3 compares the XRD pattern of NCM622-C with schematic reference peak positions for layered NCM622. The NCM622-C sample exhibits characteristic reflections at approximately 18° , 36° , 44° , 52° , 58° , and 75° , assignable to the (003), (006)/(012), (104), (105), (108)/(110), and (113) planes of the α - NaFeO_2 -type layered structure. The positions of the principal reflections show that the host layered NCM622 structure is retained after electrospinning and carbonization.

A broad feature around 25° is consistent with amorphous or low-graphitization carbon formed from the PVP-derived phase. No strong additional crystalline impurity peaks are evident in the measured pattern. The estimated $I(003)/I(104)$ ratio is approximately 1.80. Because this value is obtained from peak intensities without Rietveld refinement, it is interpreted only as a qualitative indication that the sample retains a relatively ordered layered structure; quantitative Li/Ni site-occupancy analysis would require more rigorous diffraction or microscopy methods [9-11].

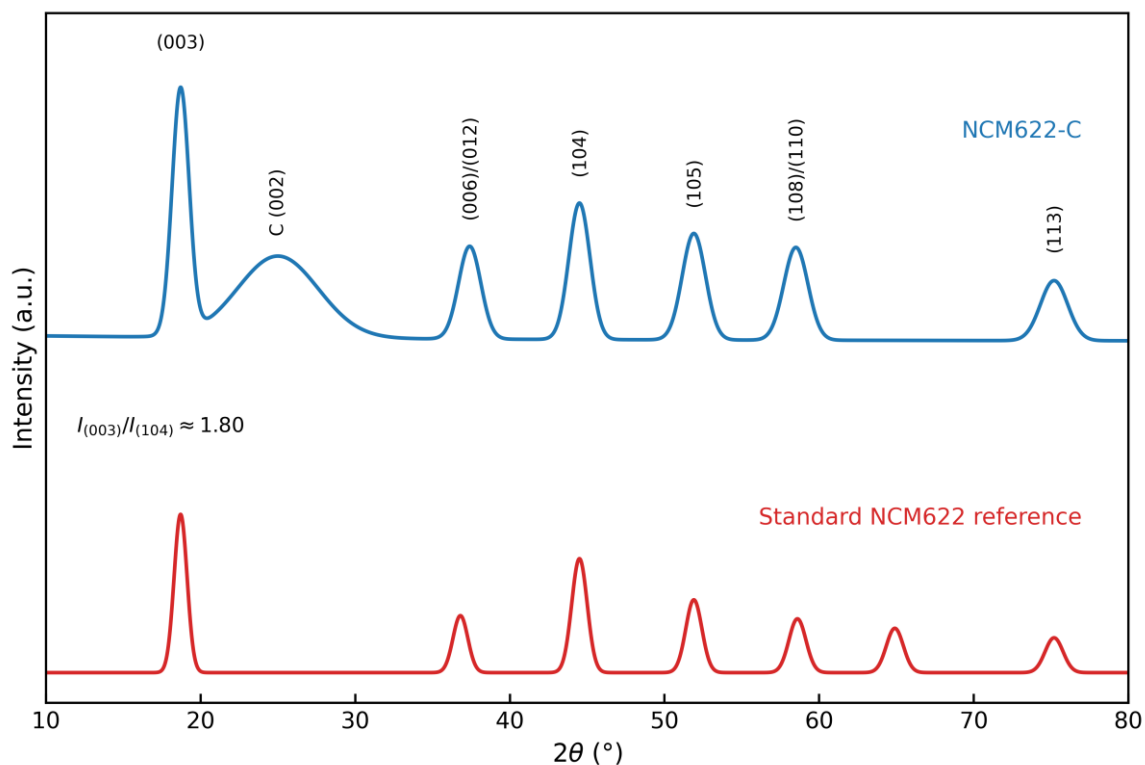


Fig. 3 XRD pattern of the NCM622-C composite and schematic reference peak positions of standard layered NCM622

3.3. Electrochemical Performance

3.3.1. Initial charge/discharge behavior

Figure 4a compares the first charge/discharge profiles of pristine NCM622 and NCM622-C at 1 C. Both electrodes show the continuous voltage variation expected for layered NCM cathodes over the 3.0-4.2 V window. NCM622-C delivers an initial charge capacity of approximately 147 mAh g⁻¹ and an initial discharge capacity of approximately 146 mAh g⁻¹, corresponding to an initial Coulombic efficiency of approximately 99.3%. The pristine NCM622 control delivers approximately 143 mAh g⁻¹ on charge and 140 mAh g⁻¹ on discharge, with an initial Coulombic efficiency of approximately 97.9%. Thus, NCM622-C exhibits a smaller first-cycle irreversible capacity difference under the same test conditions.

3.3.2. Cycling performance

Figure 4b shows the cycling behavior at 1 C. The discharge capacity of NCM622-C decreases from approximately 147.3 mAh g⁻¹ in the first cycle to approximately 141.6 mAh g⁻¹ after 50 cycles, giving a capacity retention of approximately 96.1%. In comparison, pristine NCM622 decreases from approximately 140.1 to 129.8 mAh g⁻¹, corresponding to a retention of approximately 92.7%. The Coulombic efficiency of NCM622-C remains near 99-100% through most of the test. These data demonstrate better 50-cycle capacity retention for the carbon-fiber-network composite under the conditions used here.

3.3.3. Rate performance

The rate response is shown in Fig. 4c. The average discharge capacities of NCM622-C at 0.5 C, 1 C, 3 C, 5 C, and the recovered 0.5 C step are approximately 199.9, 125.0, 63.1, 20.0, and 145.9 mAh g⁻¹, respectively. The corresponding values for pristine NCM622 are approximately 173.5, 116.2, 54.8, 18.3, and 124.5 mAh g⁻¹. NCM622-C therefore shows the clearest advantage at low-to-medium rates and during recovery to 0.5 C, whereas the difference becomes small at 5 C.

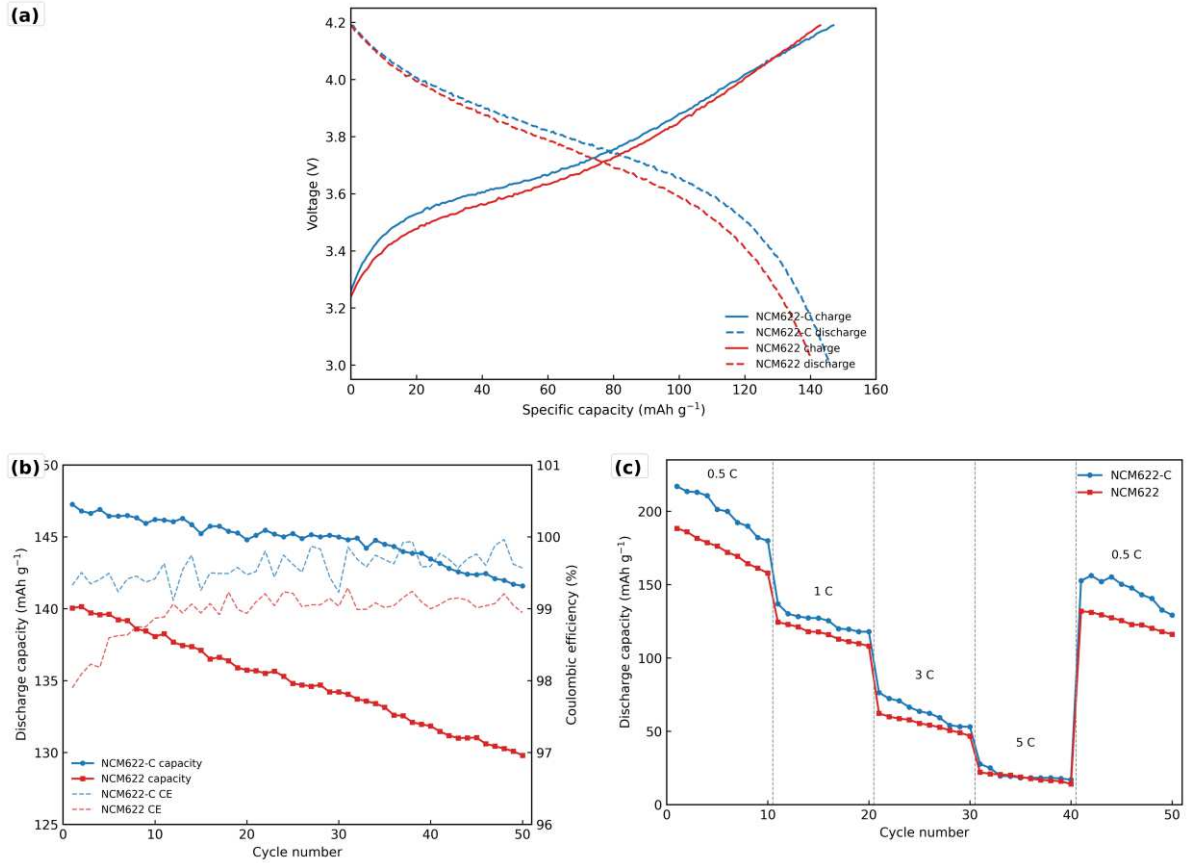


Fig. 4 Electrochemical performance of pristine NCM622 and NCM622-C: (a) initial charge/discharge profiles at 1 C; (b) cycling performance and Coulombic efficiency at 1 C; (c) rate performance at different C-rates

4. Discussion

The combined structural and electrochemical results support a structure-property relationship in which the electrospinning-derived carbon fibers mainly act as a spatially continuous electronic and mechanical connecting network. SEM directly shows that active particles are associated with a persistent fibrous framework, while XRD shows that the bulk layered NCM622 structure is retained after the 600 °C nitrogen-protected carbonization step. The higher initial Coulombic efficiency and improved 50-cycle capacity retention of NCM622-C are therefore consistent with better particle-to-particle electronic connectivity and more stable local electrode contact, rather than with a change in the fundamental NCM622 host structure.

This interpretation is also consistent with earlier work on carbon-fiber-interwoven NCM622. Kang et al. reported that ex situ interweaving of bulk carbon fibers creates a three-dimensional conduction network in highly loaded NCM622 electrodes and improves rate performance [18]. The present route differs in that the active particles are introduced into the polymer spinning precursor before fiber formation, so particle/fiber contact is established during electrospinning and subsequently fixed by carbonization. This precursor-stage integration is the central processing distinction of the present study. The electrochemical data indicate that this architecture is beneficial under the tested 1 C cycling and low-to-medium rate conditions, while the small difference at 5 C suggests that strong polarization and Li⁺ transport limitations dominate at very high current.

The results do not directly prove suppressed electrolyte decomposition, a lower charge-transfer resistance, faster Li⁺ diffusion, or reduced microcrack formation. Electrochemical impedance spectroscopy, GITT, XPS, post-cycling SEM/TEM, and longer cycling would be required to establish those mechanisms quantitatively. Accordingly, the proposed mechanism is limited to improved electronic continuity, particle/network contact, and electrode structural integrity, all of which are consistent with the available SEM, XRD, and galvanostatic data.

Table 1 Electrochemical performance comparison of pristine NCM622 and NCM622-C cathodes

Sample	Initial charge/discharge (mAh g ⁻¹)	Initial CE (%)	Capacity after 50 cycles (mAh g ⁻¹)	Retention after 50 cycles (%)
NCM622-C	147 / 146	99.3	141.6	96.1
Pristine NCM622	143 / 140	97.9	129.8	92.7

5. Conclusions

An NCM622/carbon-fiber-network composite cathode was prepared by incorporating NCM622 particles into a PVP/ethanol electrospinning precursor and converting the fibrous polymer framework into carbon at 600 °C under N₂. SEM confirms a particle/fiber interwoven morphology with NCM622 particles located on fiber surfaces and at junction regions, while XRD confirms preservation of the layered alpha-NaFeO₂-type NCM622 host structure. The estimated I(003)/I(104) ratio of approximately 1.80 is consistent with retained layered ordering, although it is used only as a qualitative structural indicator.

Compared with the real pristine-NCM622 control tested under the same conditions, NCM622-C shows improved electrochemical reversibility and short-term capacity retention. At 1 C, its initial charge/discharge capacities are approximately 147/146 mAh g⁻¹ with 99.3% initial Coulombic efficiency, compared with 143/140 mAh g⁻¹ and 97.9% for pristine NCM622. After 50 cycles, NCM622-C retains approximately 141.6 mAh g⁻¹ (96.1%), whereas pristine NCM622 retains about 129.8 mAh g⁻¹ (92.7%). The composite also exhibits higher capacity at 0.5 C, 1 C, and 3 C and better recovery after returning to 0.5 C, while the improvement becomes limited at 5 C.

Within the evidence available in this study, the performance improvement is most reasonably associated with the continuous carbon-fiber framework improving electronic connectivity, particle/network contact, and electrode structural integrity. Further EIS, GITT, interfacial spectroscopy, post-cycling microscopy, and longer-term cycling are required to quantify changes in charge-transfer resistance, Li⁺ transport, interfacial chemistry, and structural evolution.

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Competing Interests: The authors declare no relevant financial or non-financial interests related to this work.

Author Contributions: Yichen Lei: Conceptualization, methodology, investigation, data curation, formal analysis, visualization, writing - original draft, and writing - review & editing. Weili Gu: Supervision, validation, and writing - review & editing. Zian Yang: Validation and writing - review & editing. YuChen Hu: Validation and writing - review & editing. All authors read and approved the final manuscript.

Data Availability: The source data underlying the charge/discharge, cycling, and rate-performance figures are provided as Supplementary Data files. Additional experimental data supporting the findings of this study are available from the corresponding author upon reasonable request.

Ethics Approval: Not applicable. This study did not involve human participants or animals.

Consent to Participate: Not applicable.

Consent for Publication: Not applicable.

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