

Article

Mn²⁺ Pre-Embedded V₂CT_x MXene as a Negative Electrode for Lithium-Ion Batteries

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Abstract

V₂CT_x MXene is a promising anode material for lithium-ion batteries due to its high electrical conductivity and abundant active sites. However, the spatial environment within its layers restricts the function of its energy storage electrode. Herein, V₂CT_x MXene was synthesized via an NH₄F–HCl-assisted hydrothermal etching method, followed by electrochemical pre-intercalation of Mn²⁺ using a three-electrode system. Structural characterizations confirm that Mn²⁺ pre-intercalation effectively modulates the interlayer environment, reduces surface F terminations, and maintains a stable layered structure. Electrochemical measurements demonstrate that the Mn²⁺-intercalated V₂CT_x MXene delivers an enhanced reversible capacity of 313.6 mAh·g^{−1} after 200 cycles, outperforming pristine V₂CT_x MXene. The improved rate capability and reduced charge transfer resistance indicate accelerated ion/electron transport kinetics. This study provides an effective interlayer engineering strategy for improving MXene-based lithium-ion storage performance.

Keywords: MXene; pre-intercalation; interlayer spacing; electrode materials

1. Introduction

With the rapid development of new energy technologies and energy storage devices, lithium-ion batteries are widely used in portable electronic devices, electric vehicles, and large-scale energy storage systems due to their high energy density, long cycle life, and environmental friendliness [1–3]. As the core component of lithium-ion batteries, the structural characteristics and electrochemical performance of electrode materials directly determine the specific capacity, rate performance, and cycling stability of the battery [4–6]. Therefore, the development of advanced electrode materials that combine high specific capacity, excellent electron/ion transport capability, and structural stability is currently a key direction in lithium-ion battery research.

MXene is a type of two-dimensional transition metal carbon/nitride material obtained by selective etching of element A from MAX phase [7,8]. Since its first report in 2011, it has shown great potential in the field of electrochemical energy storage due to its metallic conductivity characteristics, controllable interlayer structure, and abundant surface functional groups (–F, –O, –OH). Among them, the most mature Ti₃C₂T_x MXene exhibits a reversible capacity of about 320 mAh·g^{−1} in lithium-ion batteries, but its thick layered structure and limited active sites still limit its further performance improvement [9,10].



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Compared to Ti-based MXene, V_2CT_x MXene, has a smaller single-layer thickness and larger specific surface area due to its composition of only two layers of V atoms and one layer of C atoms, which can expose more electrochemical active sites [11–13]. Theoretical calculations show that the theoretical storage capacity of V_2CT_x MXene for Li^+ can be significantly higher than that of M_3X_2 and M_4X_3 MXene, and both its surface and interlayer provide sufficient reaction sites for Li^+ insertion/extraction. In addition, the accordion-shaped structure formed by the stacking of a large number of V-C-V monolayers constructs ordered nanochannels between layers, which is conducive to rapid electron transfer and ion diffusion, making it a significant advantage in high-power output electrode materials [14–16].

However, the intralayer spatial environment of V_2CT_x MXene is closely related to its electrochemical reaction mechanism. The key lies in the manner of optimizing its intralayer spatial environment for enhancing its ion storage performance [17–19]. Previous studies have shown that V_2CT_x MXene electrodes without interlayer structure regulation only maintain a specific capacity of about $270\text{ mAh}\cdot\text{g}^{-1}$ after hundreds of cycles in lithium-ion batteries. The ion transport resistance at the electrode/electrolyte interface is high, which limits their rate performance and cycling stability. Therefore, improving the ion transport kinetics and structural stability of V_2CT_x MXene through rational structural design and interlayer engineering regulation is a keyway to enhance its electrochemical performance.

In recent years, ion pre-embedding has received widespread attention as an effective interlayer regulation strategy for MXene. By introducing metal ions between MXene layers, the disordered stacking of nanosheets can be effectively suppressed, the layered structure can be stabilized, and the transport behavior of electrons and ions can be improved [20,21]. Transition metal ions, specifically, do not only have high conductivity but can also interact with functional groups on the surface of MXene, thereby regulating interlayer distance and surface chemical environment. Ion pre-embedding achieved by electrochemical methods has the advantages of controllable process, mild conditions, and uniform embedding, and it has been proven to be an efficient interlayer engineering method.

Based on the above research background, this work uses NH_4F -HCl mixed solution as the etching system and successfully prepares high-purity V_2CT_x MXene through hydrothermal assisted etching method. On this basis, using a three-electrode system, Mn^{2+} was pre-embedded into the interlayer of V_2CT_x MXene through cyclic voltammetry. The structural characterization results indicate that the material still maintains a typical layered structure after Mn^{2+} pre-embedding, and its (002) interplanar spacing is adjusted from 0.9605 nm to 0.9501 nm, indicating a stable interaction between Mn^{2+} and MXene layers. The electrochemical test results showed that the V_2CT_x MXene electrode pre-embedded with Mn^{2+} increased its specific capacity to $313.6\text{ mAh}\cdot\text{g}^{-1}$ after 200 cycles in lithium-ion batteries, significantly higher than $271.9\text{ mAh}\cdot\text{g}^{-1}$ of the non-pre-embedded sample, while exhibiting lower charge transfer resistance and better rate performance. This work studies the effective regulation of the interlayer structure and surface chemistry of V_2CT_x MXene through electrochemical ion pre embedding, providing new ideas and experimental basis for improving the lithium-ion storage performance of MXene-based negative electrode materials. It has important reference value for the design of high-performance lithium-ion battery electrode materials.

2. Results and Discussion

2.1. Microstructure and Morphology

Figure 1a shows the XRD pattern of V_2CT_x MXene obtained after etching with NH_4F and HCl. From the figure, the (103) diffraction peak of V_2AlC phase significantly weakens after etching, and the (002) diffraction peak shifts towards a smaller angle, which is caused

by the increase in interlayer spacing of MXene after etching. XRD successfully demonstrated the acquisition of V_2CT_x MXene phase. After etching, there are still small amounts of vanadium, aluminum oxides, and other impurities present. Figures 1b,c and S1 show the scanning electron microscopy images of V_2AlC before etching and V_2CT_x MXene obtained after etching. In Figure 1b, the V_2AlC before etching exhibits a block-like structure with a certain texture on the side of the material and a relatively smooth surface. In Figure 1c, the V_2CT_x MXene obtained after etching exhibits a typical layered structure, further proving that the Al atomic layer of V_2AlC is etched away after etching.

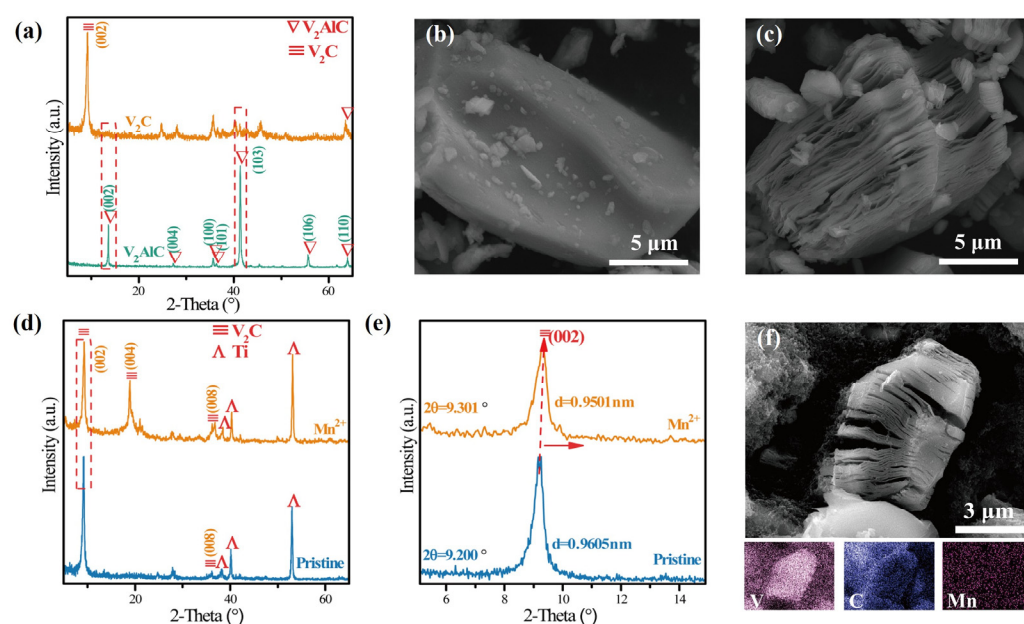


Figure 1. (a) XRD patterns of V_2AlC and V_2CT_x MXene; (b,c) SEM image of V_2AlC and V_2CT_x MXene; (d) XRD patterns of Mn^{2+} before and after pre-embedding in V_2CT_x MXene; (e) the enlarged XRD spectra of the (002) peak of Mn^{2+} before and after pre-embedding in V_2CT_x MXene; (f) SEM and EDS image of Mn^{2+} after pre-embedding V_2CT_x MXene.

A three-electrode system was prepared in a 0.2 M $Mn(NO_3)_2$ aqueous electrolyte using titanium foil as the counter electrode, Ag/AgCl (3 M KCl) electrode as the reference electrode, and ML MXene electrode as the working electrode. Using an electrochemical workstation, 10 cycles of cyclic voltammetry (CV) testing were performed to achieve Mn^{2+} insertion. Negative potential between -0.3 and -1 V at a scanning rate of 1 mV/s was applied to drive Mn^{2+} insertion/extraction into V_2CT_x MXene. Figure 1d,e shows the XRD patterns of the prepared ML V_2CT_x MXene and Mn^{2+} pre-embedded V_2CT_x MXene electrodes. The (002) diffraction peaks of the V_2CT_x MXene electrode before and after Mn^{2+} pre-embedding appeared at 9.200° and 9.301° , respectively, indicating that the layered structure was still maintained after Mn^{2+} pre-embedding in ML V_2CT_x MXene. Compared with the (002) diffraction peak of ML V_2CT_x MXene, the (002) diffraction peak of Mn^{2+} pre-embedding in ML V_2CT_x MXene shifted towards higher angles, indicating that the interlayer spacing decreased after Mn^{2+} embedding. The interlayer spacing calculation results of the samples are shown in Table 1. The interlayer spacing of ML V_2CT_x MXene is 0.9605 nm, and the interlayer spacing of Mn^{2+} pre-embedded V_2CT_x MXene is 0.9501 nm. The reduction in interlayer spacing may be due to the interaction between the pre-embedded Mn^{2+} and V_2CT_x MXene layers, which leads to the contraction of interlayer spacing. In addition, FWHM of the (002) diffraction peak decreases after Mn^{2+} pre-embedding into ML V_2CT_x MXene, indicating that ion pre-embedding can cause rearrangement of ML V_2CT_x MXene nanosheets and increase long-range orientation. Figure 1f shows the SEM and elemental

mapping images of Mn^{2+} pre-embedded V_2CT_x MXene. From the figure, it can be seen that after Mn^{2+} pre-embedding, V_2CT_x MXene still maintains its typical accordion-like structure. In addition to the mapping of C and V elements contained in V_2CT_x MXene shown in Figure 1f, it can also be seen that the Mn element is uniformly distributed in the sample.

Table 1. The (002) peak position, interplanar spacing, and FWHM before and after Mn^{2+} pre-embedding in V_2CT_x MXene.

	2θ [°]	<i>d</i> -Spacing [nm]	FWHM [°]
V_2CT_x	9.200	0.9605	0.415
$\text{V}_2\text{CT}_x\text{-Mn}^{2+}$	9.301	0.9501	0.423

XPS was used to characterize the content and chemical state of surface elements in the sample. From Figures 2 and S2, it can be seen that the V, C, O, and F elements are present in V_2CT_x MXene before and after Mn^{2+} pre-embedding. The Mn element in V_2CT_x MXene is present after Mn^{2+} pre-embedding. From the percentage content of the F element, it was found that the F content in the sample before pre-embedding was 15.15%, and after embedding Mn^{2+} , the F content decreased to 6.08% (Figure 2a,b). The embedded Mn^{2+} provides a strong thermodynamic driving force (manifested as a significant difference in formation energy), promoting the transformation of the electrochemically inert -F terminal on the MXene surface to the active -O terminal, thereby indirectly reducing the F content. In Figure 2c, it can be seen that, compared with V_2CT_x MXene, a new peak at 685.22 eV appears in the XPS spectrum of F 1s after Mn^{2+} pre-embedding into V_2CT_x MXene, which can be attributed to the Mn-F bond. From the XPS spectrum of C 1s in Figure 2d, it can be seen that after embedding Mn^{2+} , the O-C=O bond is enhanced, indicating that a large amount of Mn^{2+} embedding breaks the previous V-C-F bond, and some -OH is reduced to hydrogen gas and forms O-C=O bond. Figure 2e shows the V 2p XPS spectra of V_2CT_x and Mn^{2+} - V_2CT_x , where the peaks at 525.0 eV and 517.5 eV belong to V^{4+} $2p_{1/2}$ and V^{4+} $2p_{3/2}$, respectively, and the peaks at 531.8 eV and 530.4 eV belong to oxygen-containing functional groups, the mixture of oxides, and H_2O , respectively. In the XPS spectrum of Mn 2p in Figure 2f, peaks observed at 653.8 eV and 642.3 eV belong to Mn $2p_{1/2}$ and Mn $2p_{3/2}$, respectively, indicating the successful embedding of Mn^{2+} in V_2CT_x MXene.

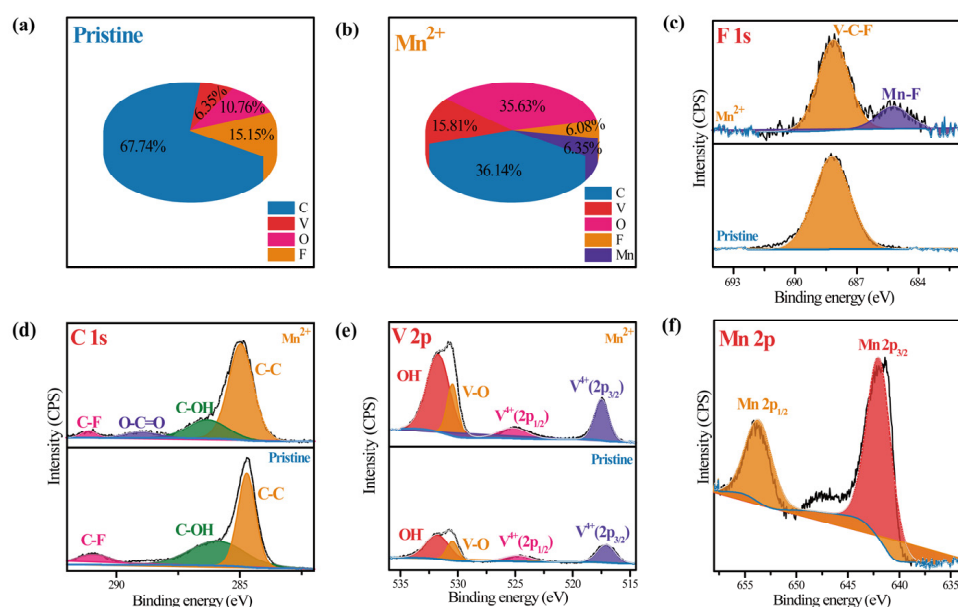


Figure 2. XPS spectra of Mn^{2+} before and after pre-embedding V_2CT_x MXene; (a,b) the presence of element; (c) F 1s; (d) C 1s; (e) V 2p; (f) Mn 2p.

Figure 3a shows the FT-IR spectra of pre-embedded Mn^{2+} and original V_2CT_x MXene. The peak at 3450 cm^{-1} in Figure 3a is caused by the stretching of water molecules, O-H, adsorbed on the surface of MXene. The peaks at 1630 cm^{-1} and 1384 cm^{-1} are attributed to the stretching vibrations of the C=O bond and O-H bond, respectively. The peaks at 1060 cm^{-1} and 650 cm^{-1} belong to the C-F bond and V-F bond. The strength of the C-F bond and V-F bond is weakened after Mn^{2+} pre-embedding, which further proves that the previous V-C-F bond is destroyed after Mn^{2+} pre-embedding in V_2CT_x MXene. In order to determine the molecular structure of the material surface, Raman testing was conducted, and Figure 3b shows the Raman spectra of V_2CT_x MXene before and after Mn^{2+} pre-embedding. The vibration at 298 cm^{-1} (E_{1g}) corresponds to the in-plane vibration of the V atom, the vibration at 417 cm^{-1} (A_{1g}) corresponds to the out-of-plane vibration of the V atom, and the Raman vibration at 496 cm^{-1} is attributed to the E_g mode in $\text{V}_2\text{C}(\text{OH})_2$, corresponding to the in-plane vibration of the V atom, indicating the presence of both out-of-plane tensile and in-plane biaxial compressive stresses. The other two peaks located at 1344 cm^{-1} and 1603 cm^{-1} correspond to the D peak of sp^3 vibration of disordered structured carbon and the G peak of sp^2 vibration of fully graphitized structured carbon in V_2CT_x MXene electrodes, respectively. Comparing the Raman peaks of MXene before and after Mn^{2+} embedding, it was found that with the pre-embedding of Mn^{2+} , the I_D/I_G values significantly decreased, indicating an increase in the orderliness of MXene electrodes during the pre-embedding of Mn^{2+} . This may be attributed to the conductive carbon black introduced during the electrode preparation process, along with the embedded Mn^{2+} , which also intercalates into the carbon material.

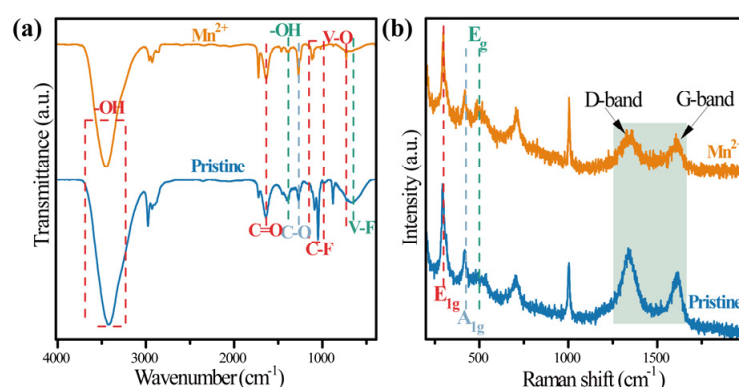


Figure 3. (a) FT-IR spectra and (b) Raman spectra of Mn^{2+} pre-embedded V_2CT_x MXene.

2.2. Electrochemical Properties

Using a half-cell approach based on two electrodes, electrochemical performance tests were conducted on the electrodes before and after pre-embedding. In order to elucidate the redox reaction behavior and the reversibility of lithium-ion insertion and extraction, the CV curves of V_2CT_x MXene before and after Mn^{2+} pre-insertion were tested at different scan rates and cycles, as shown in Figure 4. It is worth noting that during the first lithiation process of V_2CT_x MXene, there is an irreversible reduction peak located at 0.31 V (see Figure 4b), which subsequently disappears during the insertion of lithium ions. The irreversible peak and corresponding capacity loss exhibited can be attributed to the irreversible electrochemical reduction reaction caused by the SEI film generated on the electrode and the functional groups on the surface of the electrode material. Starting from the second cycle, the shape of the CV curve of V_2CT_x MXene electrode before and after Mn^{2+} pre-embedding remained basically unchanged, and the curves almost overlapped, indicating that Li ions are reversible during the insertion and extraction process of the material, demonstrating good reversibility. From the CV curves of V_2CT_x MXene electrodes

before and after Mn^{2+} pre-embedding at different scanning rates, it can be observed that the capacity gradually increases with the increase in scanning speed during the cycling process, possibly due to the polarization phenomenon caused by the continuous insertion and extraction of Li^+ during the cycling reaction.

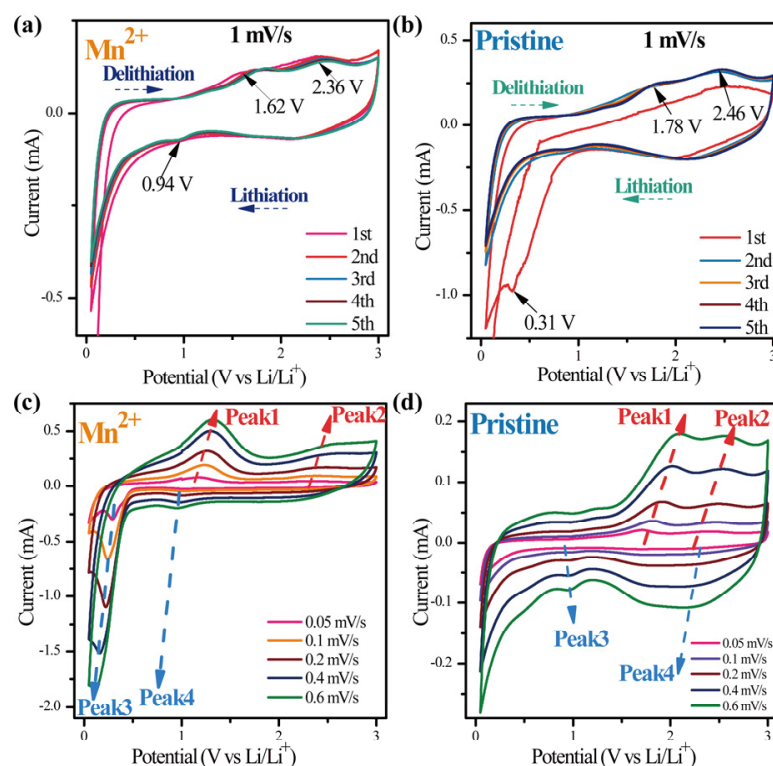


Figure 4. (a,b) CV curves of V_2CT_x MXene after and before Mn^{2+} pre-embedding; (c,d) CV curves at different scanning rates.

At 0.1 mV/s, the CV curves of V_2CT_x MXene before and after Mn^{2+} pre-embedding were compared, with a voltage range of 0.05–3.0 V, as shown in Figure 5a. In Figure 5a, it can be seen that the CV curve area of V_2CT_x MXene after Mn^{2+} embedding is larger than that of V_2CT_x , indicating that V_2CT_x MXene has higher capacity. To further demonstrate the superiority of Mn^{2+} pre-embedded V_2CT_x MXene, its cycling performance in lithium-ion batteries was tested, as shown in Figure 5b. The fluctuations shown in the figure may be caused by the instability of the battery during the testing process. The capacity of the original V_2CT_x MXene electrode after 200 cycles is 271.9 mAh/g. The specific capacity of Mn^{2+} pre-embedded V_2CT_x MXene first decreases and then gradually increases with the number of cycles, which is common in two-dimensional materials. After 200 cycles, the capacity of Mn^{2+} pre-embedded V_2CT_x MXene electrode reached 313.6 mAh/g. This may be because Mn^{2+} pre-embedded MXene layers serve as support points between layers, providing more stable and multiple reaction channels. During repeated Li^+ insertion/extraction, electrolyte penetration continuously increases, resulting in high capacity during the cycling process. Figure 5c shows the performance rate of V_2CT_x MXene and Mn^{2+} pre-embedded V_2CT_x MXene. After pre-embedding Mn^{2+} into V_2CT_x MXene, its charge and discharge capacities were higher than the original V_2CT_x MXene at current densities of 0.2, 0.5, and 1 C. When the current density is restored to 1 C, the capacity of Mn^{2+} pre-embedded V_2CT_x MXene is basically restored, indicating its good reversible performance. Figure 5d shows the GCD curve at 1 C (10th cycle). The V_2CT_x MXene electrode pre-embedded with Mn^{2+} has higher capacity, with capacities of 254.5 and 258.6 mAh/g, respectively. The capacity of the original V_2CT_x MXene is 228.7 and 238.3 mAh/g, respectively.

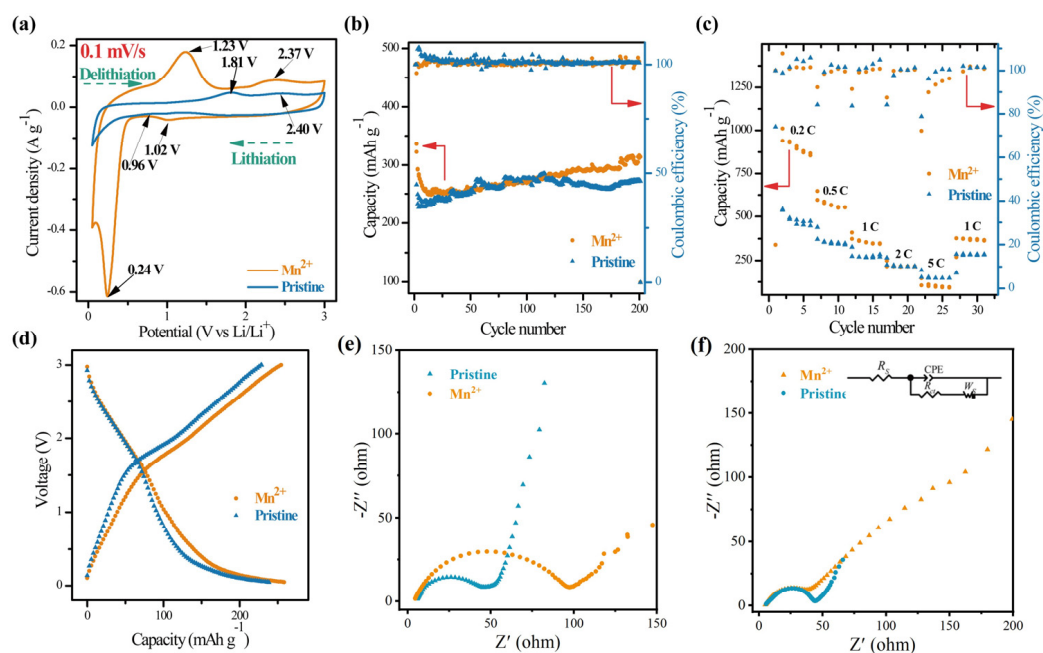


Figure 5. (a) CV curves; (b) cycle performance; (c) rate performance; (d) GCD curve; (e,f) the EIS spectra pre-cycle and (f) after 200 cycles of Mn^{2+} pre-embedded V_2CT_x MXene and the original V_2CT_x MXene.

In order to further investigate the electrochemical behavior of Mn^{2+} pre-embedded V_2CT_x MXene, the EIS spectra of V_2CT_x MXene and Mn^{2+} pre-embedded V_2CT_x MXene before and after cycling were measured, as shown in Figure 5e,f. It consists of two overlapping semicircles in the high-frequency and mid-frequency regions and a linear part in the low-frequency region. In the mid-high frequency region, the diameter of the semicircle of the sample after Mn^{2+} pre-embedding is smaller than that of the original V_2CT_x MXene, indicating that Mn^{2+} pre-embedding can reduce charge transfer resistance, which is beneficial for charge transfer and further confirms that Mn^{2+} can induce rapid charge transfer. This proves the reason why Mn^{2+} pre-embedded V_2CT_x MXene electrodes have more stable charge discharge cycling performance and higher specific capacity. Comparing the EIS spectra after 200 cycles, it can be seen that in the mid- to high-frequency region, the semi-circular diameter of the sample pre-embedded with Mn^{2+} is smaller than that of the original V_2CT_x MXene, indicating that the R_{ct} value of the sample pre-embedded with Mn^{2+} after cycling is lower than that before the original V_2CT_x MXene. This is because Mn^{2+} pre-embedding can induce larger interlayer distance, faster ion migration speed, and higher conductivity during the Li^+ insertion/extraction cycle.

In order to further investigate the embedding behavior of Mn^{2+} , structural characterization, morphology characterization, CV, and EIS testing were performed on the electrode after 200 cycles. Figure 6a shows the XRD patterns of ML V_2CT_x MXene electrode before and after Mn^{2+} insertion after 200 cycles. By calculation, it can be concluded that after 200 cycles, the (002) peak of both electrodes shifts towards a small angle compared to the XRD pattern of the sample before cycling (Figure 1e), confirming that the interlayer distance between the two electrodes further increases during Li^+ insertion and extraction cycling, thus explaining the capacity increase phenomenon of the ML V_2CT_x MXene electrode before and after Mn^{2+} insertion during cycling. For MXene, the significant decrease in the intensity of the (002) peak directly reflects an essential change in its layered structure, which may be due to the damage to the electrode structure caused by periodic charging and discharging. During the electrochemical cycling process, the in situ oxidation of Mn^{2+} to Mn^{4+} between layers and the formation of manganese oxide species may indeed lead to physical

expansion between multiple MXene layers. After 200 cycles, the CV curve of the electrode (second cycle) is shown in Figure 6b. It can be observed that after Mn^{2+} pre-embedding, the oxidation peak of V_2CT_x MXene at 2.07 V shifts towards higher potential, indicating that Mn^{2+} pre-embedding expands the distribution range of Li^+ participating in electrode active sites. Meanwhile, compared with the original V_2CT_x MXene, Mn^{2+} embedding resulted in a larger integration area, explaining the phenomenon that the capacity of the ML V_2CT_x MXene electrode is higher than that of the ML V_2CT_x MXene electrode after Mn^{2+} embedding during the cycling process. Figure 6c shows the SEM and elemental mapping images after 200 cycles of charge and discharge. It can be seen that after Mn^{2+} pre-embedding, V_2CT_x MXene still maintains its original accordion-shaped morphology, with Mn element still uniformly distributed.

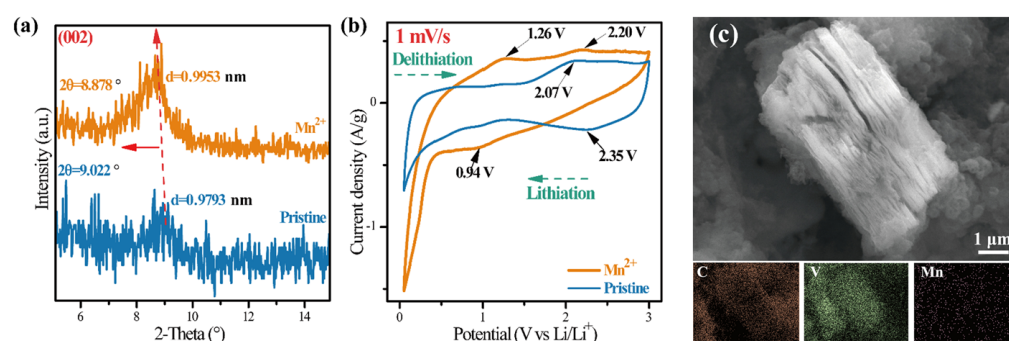


Figure 6. The XRD pattern of peak (a) (002) after 200 cycles; (b) CV curve (Period 2); (c) SEM and EDS images of Mn^{2+} embedded in V_2CT_x MXene after 200 cycles.

3. Experimental Section

3.1. Reagents and Materials

The materials and reagents required for the synthesis of samples are hydrochloric acid (HCl) with a concentration of 40%, V_2AlC MAX ceramic powder with a particle size of 400 mesh, 0.2 M manganese nitrate solution ($\text{Mn}(\text{NO}_3)_2$), ammonium fluoride (NH_4F), polyvinylidene fluoride (PVDF), carbon black, N-methyl pyrrolidone (NMP), 1 M lithium hexafluorophosphate solution (LiPF_6), 10×20 mm titanium sheet, glass fiber membrane (Whatman GF/D), Li sheet, ethylene carbonate (EC), diethyl carbonate (DEC), and methyl ethyl carbonate (EMC). V_2AlC MAX powders were purchased from Jilin 11 Technology Co., Ltd. (Jilin, China), and the rest were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). All the above chemicals used in this experiment are of analytical purity, and all the reagents were not further purified.

3.2. Preparation of V_2CT_x MXene Electrode

A total of 2 g of ammonium fluoride (NH_4F) was added to 20 mL of hydrochloric acid (HCl) (40%) solution and stirred for 30 min; then 2 g of V_2AlC MAX ceramic powder was added. The reaction mixture was sealed in a stainless steel autoclave, stored at 120°C for 24 h, and cooled naturally to room temperature. The black sediment was collected through centrifugation and washed with deionized water and ethanol three times. Finally, the precipitate was dried in a vacuum oven at 70°C for 12 h. The V_2CT_x MXene powder, carbon black, and PVDF were mixed uniformly according to the mass ratio of 8:1:1 with the addition of NMP. The obtained slurry was coated onto the titanium films (10 mm 20 mm, the covered area is 10 mm 10 mm) with a small brush. The mass density of the coated electrode was approximately $1 \text{ mg}/\text{cm}^2$. The prepared electrodes were dried under 120°C in a vacuum drying chamber.

3.3. Preparation of V_2CT_x MXene Pre-Intercalated with Mn^{2+}

Mn^{2+} pre-embedded V_2CT_x MXene electrode was prepared. Firstly, a three-electrode system was constructed with 0.2 M $Mn(NO_3)_2$ aqueous solution as an electrolyte, titanium foil as a counter electrode, Ag/AgCl (3 M KCl) electrode as a reference electrode, and MXene electrode as a working electrode. With the help of electrochemical workstation, the CV techniques were employed to drive the Mn^{2+} to intercalate into MXene via a negative potential of $-0.9 \sim -0.4$ V at 1 mV/s. The pre-embedded electrode needs flowing deionized water to clean the electrode surface. Using the above process, V_2CT_x MXene electrodes pre-intercalated with Mn^{2+} were prepared.

3.4. Microstructural Characterization

X-ray diffraction (XRD) patterns of V_2CT_x MXene powders were obtained using a D/Max-2500 diffractometer (Rigaku, Tokyo, Japan). The test uses a $CuK\alpha$ ($\lambda = 0.15406$ nm) radiation source with a working voltage of 40 kV and a working current of 200 mA. The morphologies of V_2CT_x MXene powders were observed through scanning electron microscopy (SEM, JSM-7800F, JEOL, Tokyo, Japan). Nicolet iS50 Fourier transform infrared spectrometer (Thermo Fisher Scientific, MA, USA) was used to conduct FTIR tests on V_2CT_x MXene powders; the spectra in the range of $4000\text{--}500\text{ cm}^{-1}$ were collected with a wavenumber accuracy of 0.01 cm^{-1} . The chemical composition and elemental valence of the samples were characterized by X-ray photoelectron spectroscopy (XPS, Thermo Scientific escalab 250xi, MA, USA). Fourier transform infrared spectrometer (FT-IR, Thermo scientific Nicolet iS50, MA, USA) was used to collect infrared spectra. The Raman spectra of the samples were collected using confocal Raman microscope (Renishaw inVia, London, UK), and the excitation wavelength was 514 nm.

3.5. Electrochemical Measurements

Using V_2CT_x MXene pre-intercalated Mn^{2+} as anode (which contributes the charge storage between 0.05 and 3 V), lithium sheet as a counter electrode/reference electrode (to match the electrochemical reaction mechanism of lithium-ion intercalation/deintercalation, construct a stable lithium-ion transport cycle, and reduce interfacial side reactions), and commercial lithium-ion battery electrolyte (1 M LiPF₆ in ethylene carbonate/diethyl carbonate/ethyl methyl carbonate (EC/DEC/EMC), 1:1:1 in volume) as an electrolyte, the battery system was assembled with a CR2032 button electrode shell to explore the performance and mechanism of V_2CT_x MXene pre-intercalated Mn^{2+} electrode for lithium-ion storage at high temperatures. The cyclic voltammetry (CV) curves and electrode performance were collected using the electrochemical workstation (Metrohm, Autolab PGSTAT302N, Herisau, Switzerland) and the battery test system (NEWARE, CT-4008T, Shenzhen, China). During the testing process, the voltage window was set to 0.05–3 V, and the cyclic voltammetry curve of the lithium-ion battery was tested at a scanning speed of 1 mV/s. In addition, CV curves at different scan speeds (0.5, 1, 2, 5, and 10 mV/s) were tested and compared to further investigate the electrochemical performance of V_2CT_x MXene electrodes. The battery testing system was used to test the constant current charge discharge curve and cycling characteristics of the V_2CT_x MXene as the anode of lithium-ion batteries in the potential range of 0.05–3 V. At the same time, the rate characteristics of the V_2CT_x MXene as the anode of lithium-ion batteries were tested with current densities of 0.2, 0.5, 1, 2, and 5 C, and the changes in its cycling and rate performance were analyzed.

4. Conclusions

In this study, high-purity V_2CT_x MXene was successfully prepared through etching with $NH_4F\text{--}HCl$ mixed solution and combined with hydrothermal-assisted method. Based

on this, controllable pre-embedding of Mn^{2+} between V_2CT_x MXene layers was achieved through a three-electrode electrochemical system. The structural characterization results indicate that after Mn^{2+} pre-embedding, the material still maintains a typical layered accordion structure, with the (002) interplanar spacing adjusted from 0.9605 nm to 0.9501 nm. At the same time, the surface F functional group content is significantly reduced, and the material's orderliness and conductivity are improved. The electrochemical performance test results showed that Mn^{2+} pre-embedding effectively improved the lithium-ion storage performance of V_2CT_x MXene. Compared with the original V_2CT_x MXene, the specific capacity of the pre-embedded electrode increased from 271.9 mAh g^{-1} to 313.6 mAh g^{-1} after 200 cycles and exhibited better rate performance and lower charge transfer resistance. The above results indicate that electrochemical ion pre-embedding is an effective interlayer engineering control strategy for MXene, which can significantly improve its electrochemical reaction kinetics and structural stability, providing important references for the design of high-performance MXene-based lithium-ion battery electrode materials.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/inorganics14020065/s1>, Figure S1. SEM image of V_2AlC and V_2CT_x MXene. Figure S2. XPS spectra of Mn^{2+} before and after pre-embedding V_2CT_x MXene, (a) full spectra; (b) O 1s; (c) V 2p.

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