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Metal-Ion-Intercalated MXene for Enhanced Capacitance in Supercapacitors

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Abstract

MXenes are high-performance pseudocapacitive materials known for their excellent conductivity, large surface area and fast redox reactions occurring at the surface. Despite these advantages, their practical application is hindered by the tendency of MXene nanosheets to aggregate and restack, which significantly compromises cycling stability. In this work, post-delamination metal-ion intercalation was employed to successfully expand the interlayer spacing of Ti_3C_2 while simultaneously optimizing its surface functional groups. Benefiting from the enlarged interlayer spacing and improved surface chemistry, the Mn-intercalated MXene (Mn-MXene) delivers a high specific capacitance of 285 F g^{-1} at a scan rate of 10 mV s^{-1} in $1 \text{ M H}_2\text{SO}_4$ electrolyte, which represents a 26% enhancement compared with pristine Ti_3C_2 . Notably, Mn-MXene exhibits nearly 100% capacitance retention after 3000 cycles.

Keywords: supercapacitor; MXene; intercalation

1. Introduction

Supercapacitors (SCs) occupy a unique position in energy storage technology, offering a combination rarely found in other devices: they deliver both the high power density of capacitors and a significantly enhanced energy density compared to conventional dielectric capacitors [1,2]. This capability is complemented by safe, high-power operation ($>10 \text{ kW kg}^{-1}$), rapid charge–discharge rates, and exceptional long-term cycling stability [3,4]. Owing to these advantages, supercapacitors exhibit great potential for applications in transportation systems, electronic devices, and implantable biomedical devices [5,6]. However, compared with rechargeable batteries, the relatively low energy density of supercapacitors remains a critical limitation. Therefore, improving the energy density of supercapacitors has become an urgent challenge and a key research focus [7].

MXenes are a class of two-dimensional transition metal carbides, nitrides, or carbonitrides with a general formula of $\text{M}_{n+1}\text{X}_n\text{T}_x$, where M represents an early transition metal from groups III to VII (e.g., Ti, Nb, Mo, V, Cr, and Ta), X denotes carbon, nitrogen, or their combinations, and T_x refers to various surface functional groups (such as $-\text{OH}$, $-\text{Cl}$, and $-\text{F}$) [8,9]. Owing to their unique conductivity and ultrahigh volumetric capacitance, MXenes have attracted increasing attention for supercapacitors applications [10,11]. Among the various MXene compositions, $\text{Ti}_3\text{C}_2\text{T}_x$ exhibits several distinct advantages [12]. First, its MAX-phases, such as Ti_3AlC_2 and Ti_3SiC_2 , are widely used in industrial applications and are readily available [13]. Second, $\text{Ti}_3\text{C}_2\text{T}_x$ generally demonstrates superior structural stability compared with nitrogen-containing MXenes, enabling improved cycling stability [14].



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Third, the conductivity of $\text{Ti}_3\text{C}_2\text{T}_x$ is typically higher than that of many other MXenes, including TiNbCT_x , Ti_2CT_x , $\text{Ta}_4\text{C}_3\text{T}_x$, and $\text{Ti}_3\text{CN}_x\text{T}_y$ [15,16]. As a result, $\text{Ti}_3\text{C}_2\text{T}_x$ -based electrodes exhibit longer cycle life and favorable rate capability.

Nevertheless, MXene nanosheets are prone to restacking and aggregation, which leads to inefficient out-of-plane electron and ion transport [17,18]. When MXenes are directly employed as supercapacitor electrodes, their electrochemical performance is often constrained by film thickness, leading to low areal capacitance. Intercalation is an effective strategy for tuning the properties of two-dimensional materials, and can modify both physical properties and chemical reactivity [19]. By increasing the interlayer spacing, it not only enhances ion diffusion but also exposes a greater number of electrochemically active sites [20,21]. In previous studies, ion intercalation has commonly been employed in MAX phase etching and multilayer MXene delamination. For example, a one-step etching method used a mixture of hydrochloric acid (HCl) and lithium fluoride (LiF) as the etchant. In this process, Li^+ intercalation facilitates MXene delamination and results in an enlarged interlayer spacing [22]. Li et al. demonstrated that by immersing multilayer MXene powders in a CsOH solution, Cs^+ ions were successfully introduced into the MXene structure. This intercalation expanded the interlayer spacing, leading to a significant enhancement in the gravimetric specific capacitance, which increased by approximately 725% [23]. These methods typically initiate intercalation from multilayer MXenes, which limits the extent of intercalation due to the already constrained interlayer spacing in the bulk structure. As a result, the degree of ion insertion becomes difficult to control, leading to challenges in precisely tuning the intercalation process and achieving uniform modification across the material. There have also been reports on the intercalation of single-layer MXenes. Sun et al. applied the electrochemistry-driven cation intercalation (ECI) method to insert metal cations into Ti_3C_2 interlayers followed by calcination. The resulting T-Mn-C film delivered an ultrahigh volumetric specific capacitance of 1655.5 F cm^{-3} at a scan rate of 1 mV s^{-1} [24]. Yang et al. reported the fast gelation of MXene in an aqueous solution initiated by divalent metal ions. The fast gelation effectively suppressed the restacking of MXene NSs and led to excellent rate performance of the hydrogel [25]. However, these products typically consist of a mixture of intercalated and non-intercalated MXene nanosheets. Therefore, the intrinsic influence of fully intercalated MXene on energy storage performance remains to be systematically clarified.

In this work, we intercalated metal ions (Li^+ , Mn^{2+} , and Al^{3+}) into single-layer Ti_3C_2 dispersions using a simple physical mixing method. Additionally, we ensured complete intercalation of MXene with multiple centrifugation steps. This approach successfully increased the interlayer spacing of Ti_3C_2 while optimizing its surface functional groups. The intercalation process not only prevented the restacking of Ti_3C_2 but also facilitated the expansion of electron and ion transport pathways, enhancing the material's overall performance. As a result, both the specific capacitance and rate capability were significantly improved. The XRD results show that Mn-MXene increases the interlayer spacing to 1.550 nm, compared with 1.471 nm for pristine Ti_3C_2 . Mn-MXene delivers a high specific capacitance of 285 F g^{-1} at a scan rate of 10 mV s^{-1} in a 1 M H_2SO_4 electrolyte, which represents a 26% improvement compared with pristine Ti_3C_2 . After 3000 charge-discharge cycles in a Swagelok cell, the capacitance remains close to 100%, indicating the long-term cycling stability. These results demonstrate that metal-ion intercalation is an effective modification strategy for enhanced capacitance.

2. Materials and Methods

2.1. Preparation of Metal-Ion Intercalated MXene

Figure 1A illustrates the synthesis procedure of metal-ion intercalated MXene (M-MXene). First, 4 mL of a single-layer Ti_3C_2 dispersion (99%, 5 mg mL^{-1} , FoShan XinXi Technology Co., Ltd.) was stirred uniformly. Then, Ti_3C_2 dispersion was slowly added dropwise into 20 mL of 0.06 mol L^{-1} MSO_4 solution ($\text{M} = \text{Li}^+$, Mn^{2+} , or Al^{3+}). The mixture was stirred for 90 min under Ar atmosphere. After stirring, the suspension was centrifuged at 12,500 rpm to remove excess ions. Notably, metal cations were found to interact with functional groups on Ti_3C_2 . This interaction destroyed the electrostatic repulsive forces between the nanosheets [26]. As a result, M-MXene formed a precipitate after centrifugation. After five centrifugation cycles, the black precipitate was collected and freeze-dried to obtain M-MXene powder. To prepare M-MXene films, an appropriate amount of M-MXene powder was dispersed in deionized water and stirred thoroughly. The above suspension was vacuum filtrated through a membrane (Celgard 3501, $0.22 \mu\text{m}$ pore size, Celgard LLC, USA). The mass loading of the film was 1.04 mg cm^{-2} .

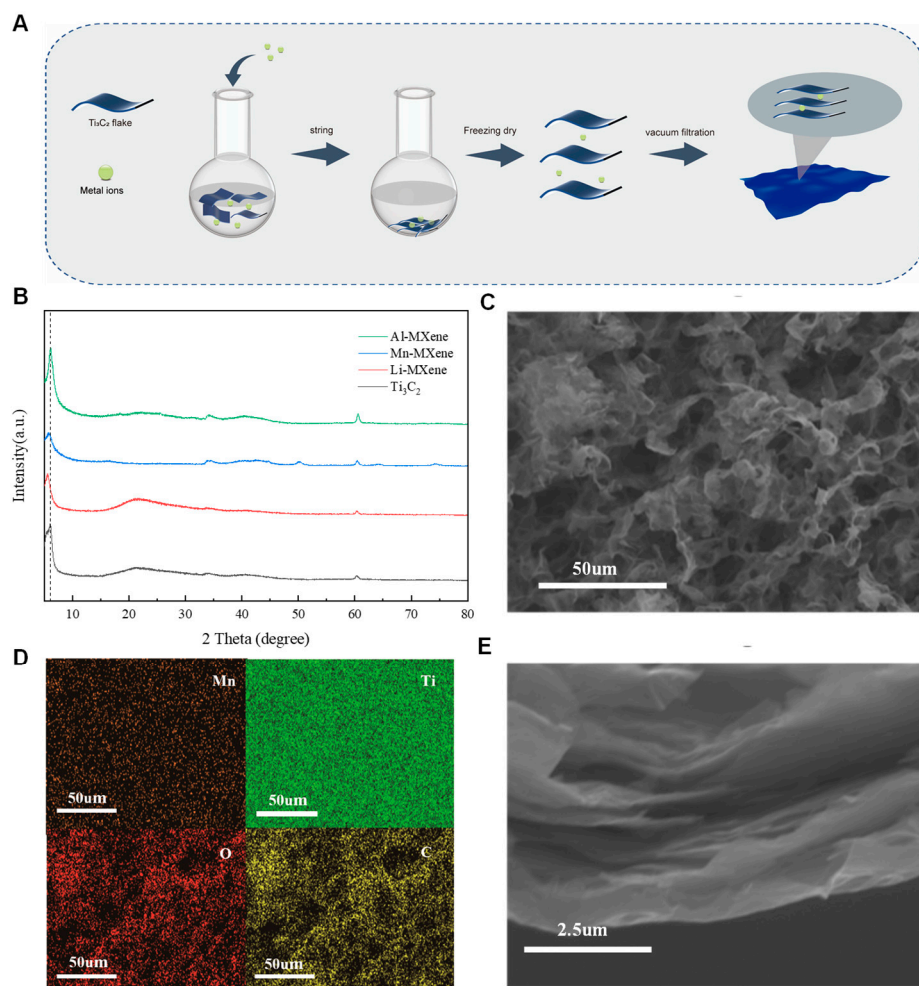


Figure 1. (A) Schematic illustration of the synthesis process of M-MXene. (B) X-ray diffraction (XRD) patterns of M-MXene powders. (C,D) Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) mapping of Mn-MXene. (E) Cross-sectional SEM image of Mn-MXene film.

2.2. Electrochemical Measurements

The synthesized materials were used as working electrodes for electrochemical measurements. A total of 5 mg Ti_3C_2 and 20 μL Nafion-117 solution (used as binder) were added to 5 mL deionized water and stirred for 1 h. The mixture was then ultrasonicated for

5 min in an ice bath. The ink was coated onto a glassy carbon current collector (0.785 cm^2) as the working electrode. The mass of the active material on the working electrode was calculated based on the volume and concentration of the ink. The counter electrode was a graphite rod and the reference electrode was a Hg/Hg₂SO₄ electrode saturated with K₂SO₄. For measurements in the Swagelok cell, self-supporting films (0.79 cm^2) were directly used as the working electrodes, with activated carbon as the counter electrode and a Hg/Hg₂SO₄ reference electrode. The activated carbon electrode was prepared by uniformly coating a slurry onto carbon paper (a mass loading of 4 mg cm^{-2}). The slurry contained activated carbon powder, carbon black (XC-72R), and Poly (vinylidene fluoride) (PVDF) powder dissolved in N-methyl-2-pyrrolidone (NMP). The composition of the activated carbon electrode was 80 wt.% activated carbon, 10 wt.% carbon black, and 10 wt.% PVDF [27]. A polypropylene (PP) membrane (Celgard LLC, USA) was used as the separator. All electrochemical measurements were conducted at room temperature using an electrochemical workstation (CHI 660E, Shanghai CH Instruments). Electrochemical impedance spectroscopy (EIS), galvanostatic charge–discharge (GCD), and cyclic voltammetry (CV) were performed for electrochemical characterization. The potential range was from -1.1 V to -0.3 V (versus Hg/Hg₂SO₄) and $1\text{ M H}_2\text{SO}_4$ aqueous solution was used as the electrolyte. The EIS was obtained from 10 mHz to 100 kHz [28].

Equations (1) and (2) were used to calculate the specific capacitance from the discharge part of CV and GCD, respectively.

$$C_{s-CV} = \frac{\int_{V_1}^{V_2} i dV}{\nu (V_2 - V_1)} \quad (1)$$

$$C_{s-GCD} = \frac{2i \int V dt}{V^2} \quad (2)$$

where C_{s-CV} represents the specific capacitance calculated from CV curves (F g^{-1}). V_1 and V_2 denote the upper and lower limits of the voltage window (V), i is the current density (mA/g), and ν is the scan rate (V s^{-1}). C_{s-GCD} represents the specific capacitance calculated from GCD curves; t is time (s) [29,30].

2.3. Material Characterization

X-ray diffraction (XRD) patterns were collected using a Shimadzu X-ray diffractometer (XRD-6100, SHIMADZU, Japan) with Cu K α radiation ($\lambda = 1.54178\text{ \AA}$). Data were acquired over a 2θ range of $5\text{--}80^\circ$. The interlayer spacing d was calculated using Bragg's law:

$$d = \frac{\lambda}{2\sin\theta} \quad (3)$$

where λ is the X-ray wavelength and θ is the Bragg's angle in radians [31].

Scanning electron microscopy (SEM) images and energy dispersive X-ray (EDX) elemental mapping were obtained using a Thermo Fisher Scientific Apero 2S. X-ray photoelectron spectroscopy (XPS) measurements were conducted using a Kratos Axis Supra XPS spectrometer with an Al K α radiation source ($h\nu = 1486.6\text{ eV}$). The binding energies in the XPS spectra were calibrated by referencing the C 1s peak to 284.8 eV .

3. Results

The synthesis of M-MXenes is illustrated in Figure 1A. In the intercalation process, the negative MXene surface attracts positive cations, which adhere to the MXene via electrostatic interactions, leading to the formation of the corresponding M-MXene precipitate [25]. In this study, high-purity single-layer Ti₃C₂ nanosheets were utilized. The X-ray diffraction

(XRD) pattern (Figure 1B) displays a characteristic (002) peak at 6.0° , indicating the successful etching and intercalation. The Tyndall effect of the Ti_3C_2 dispersion is illustrated in Figure S1, demonstrating its good dispersibility and delamination.

Since the intercalation behavior of MXenes is closely related to the ionic radius of metal ions [24], We chose Li^+ , Mn^{2+} , and Al^{3+} as intercalation metal in this work. The crystal structure of M-MXenes was first characterized by XRD. As shown in Figure 1B, all materials exhibit a distinct (002) peak, indicating that metal ion intercalation does not disrupt the layered structure of MXenes. The (002) peak positions of Li-MXene and Mn-MXene are lower than those of Ti_3C_2 , appearing at 5.6° and 5.7° , respectively. The calculated interlayer spacings of Li-MXene and Mn-MXene are 1.577 nm and 1.550 nm, respectively, in contrast to the interlayer spacing of 1.471 nm for Ti_3C_2 , demonstrating a successful expansion of the interlayer distance. Notably, the largest interlayer spacing was observed in Li-MXene, which is likely attributed to the higher degree of hydration of Li^+ ions and their weaker electrostatic interactions with the MXene surface [32].

The morphologies of Ti_3C_2 and Mn-MXene were further compared. Figure 1C shows the SEM image of freeze-dried powders of Mn-MXene. Compared with Ti_3C_2 (Figure S1), Mn-MXene exhibits a more porous and fluffy surface, which can be attributed to the disruption of the dense, layered structure of MXene during the ion intercalation process [14]. This porous structure is considered favorable for ion transport between MXene layers. Figure 1D shows the EDX mapping of the powder, revealing a uniform elemental distribution and confirming that Mn remains intercalated within the MXene layers even after multiple centrifugation steps, contributing to structural support [33]. Figure S2 shows the percentage composition of the EDX mapping of elements. The atomic fraction of Mn is only 0.87%, indicating that only few metal ions remain within the MXene after multiple washing steps. Figure 1E presents a cross-sectional SEM image of the Mn-MXene film, in which the multilayered structure can be directly observed. Meanwhile, the Mn-MXene film formed after vacuum filtration could be peeled off intact from the filter paper, demonstrating the structural stability after metal intercalation.

To investigate the electrochemical performance of M-MXenes, electrochemical measurements were first conducted in a conventional three-electrode system. Prior to collecting CV data, all electrodes were pre-cycled for 50 cycles at a scan rate of 50 mV s^{-1} to ensure complete wetting. Figure 2A presents the CV curves of M-MXenes and Ti_3C_2 recorded at a scan rate of 20 mV s^{-1} . All M-MXenes exhibit pronounced redox peaks, indicating the typical pseudocapacitive redox reactions of MXene. During the charging process, the reduction peak at approximately -0.9 V corresponds to the surface intercalation of H^+ , while the oxidation peak at around -0.8 V during discharge is associated with the deintercalation of H^+ [34]. With increasing scan rate, only slight shifts in the anodic and cathodic peak positions are observed, indicating the reversible nature of the surface redox reactions. Figure 2B shows the GCD curves measured at a current density of 1 A g^{-1} , where distinct voltage plateaus are observed, further confirming the pseudocapacitive characteristics of M-MXenes. The specific capacitances at different scan rates are summarized in Figures 2C and S3. At a scan rate of 10 mV s^{-1} , Mn-MXene exhibits the highest specific capacitance of 285 F g^{-1} . Meanwhile, the specific capacitances of Li-MXene and Al-MXene reach 265 and 261 F g^{-1} , respectively, all of which are higher than that of pristine Ti_3C_2 (227 F g^{-1}). These results demonstrate that metal ion intercalation can effectively enhance the electrochemical performance of Ti_3C_2 . The CV and GCD curves of Mn-MXene are shown in Figures 2D and 2E, respectively. Mn-MXene exhibits excellent pseudocapacitive behavior at low scan rates and maintains a high capacitance retention of 83% (238 F g^{-1}) even at a high scan rate of 200 mV s^{-1} , indicating favorable rate capability. At a current density of 1 A g^{-1} , the specific capacitance of 288 F g^{-1} and a Coulombic efficiency of

~100% were achieved, indicating good electrochemical reversibility. Figure 2F presents the EIS plots of M-MXenes and Ti_3C_2 . Mn-MXene exhibits the smallest charge-transfer resistance, demonstrating the superior ion transport kinetics.

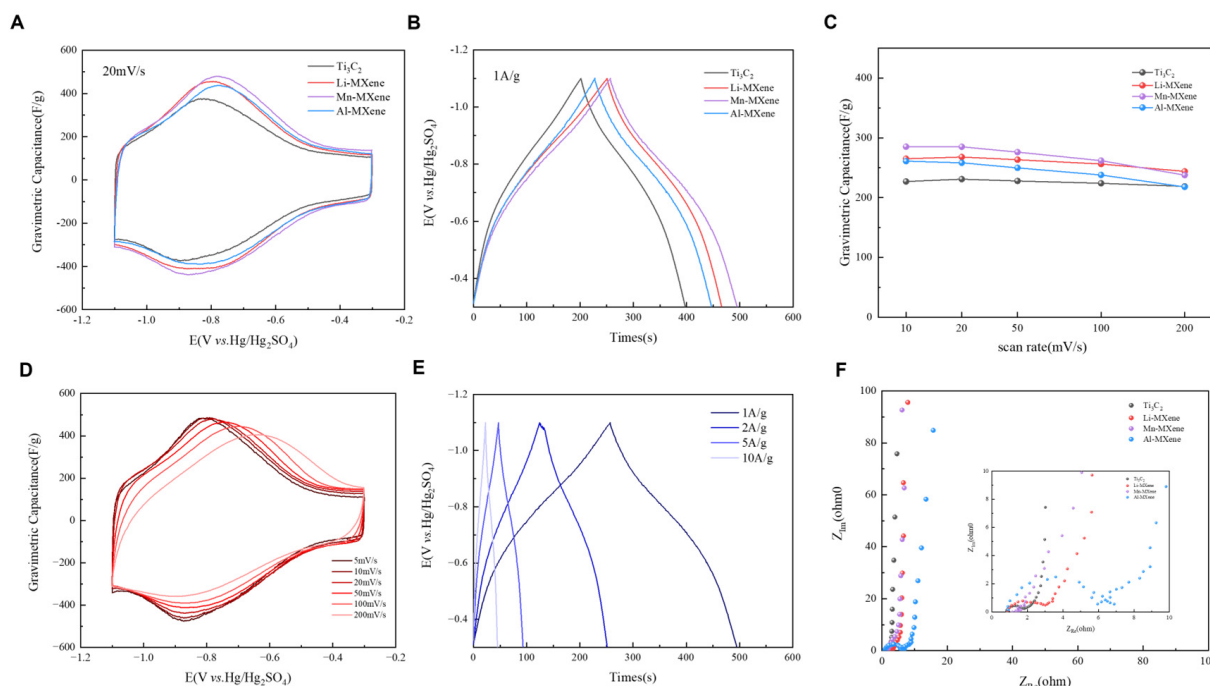


Figure 2. Electrochemical performance of M-MXene. (A) CV curves of M-MXene and Ti_3C_2 . (B) GCD curves of M-MXene and Ti_3C_2 . (C) Specific capacitance as a function of scan rate for M-MXene and Ti_3C_2 . (D) CV curves of Mn-MXene. (E) GCD curves of Mn-MXene. (F) Electrochemical impedance spectroscopy (EIS) plots of M-MXene and Ti_3C_2 .

The superior specific capacitances exhibited by Mn-MXene and Li-MXene are consistent with the XRD results, which can be attributed to their enlarged interlayer spacings. Although the interlayer spacing of Mn-MXene is smaller than that of Li-MXene, its higher specific capacitance is likely attributed to the modification of surface functional groups, which can be confirmed with the XPS survey. The XPS characterization results are presented in Figures 3 and S4. Figure 3A presents the survey spectra of M-MXenes and Ti_3C_2 . No obvious characteristic peaks corresponding to the intercalated metal ions are observed, corresponding to the trace presence of Mn detected in the EDS results (Figure S2). Figure 3B presents the relative ratios of oxygen and fluorine to titanium, determined by area integration of the respective XPS peaks. The oxygen content of Al-MXene and Mn-MXene is significantly higher than that of Ti_3C_2 and Li-MXene, which is attributed to their higher oxidation states [25]. Concurrently, XPS analysis reveals a marked decrease in the -F content on the surface of Mn-MXene. This loss of -F terminations, typically replaced by -OH groups, is believed to enhance the electrochemical performance of the material.

This conclusion is further substantiated by the analysis of the Ti 2p and O 1s spectra (Figure 3C,D). Specifically, the Ti 2p spectrum can be deconvoluted into four peaks corresponding to Ti-C, Ti^{2+} , Ti^{3+} , and Ti^{4+} (TiO_2) states [35]. The enhanced intensity of the Ti^{3+} and Ti^{4+} peaks in Mn-MXene indicates an increase in the oxidation state of titanium. Additionally, the O 1s spectrum confirms a rise in oxygen-containing functional groups in Mn-MXene.

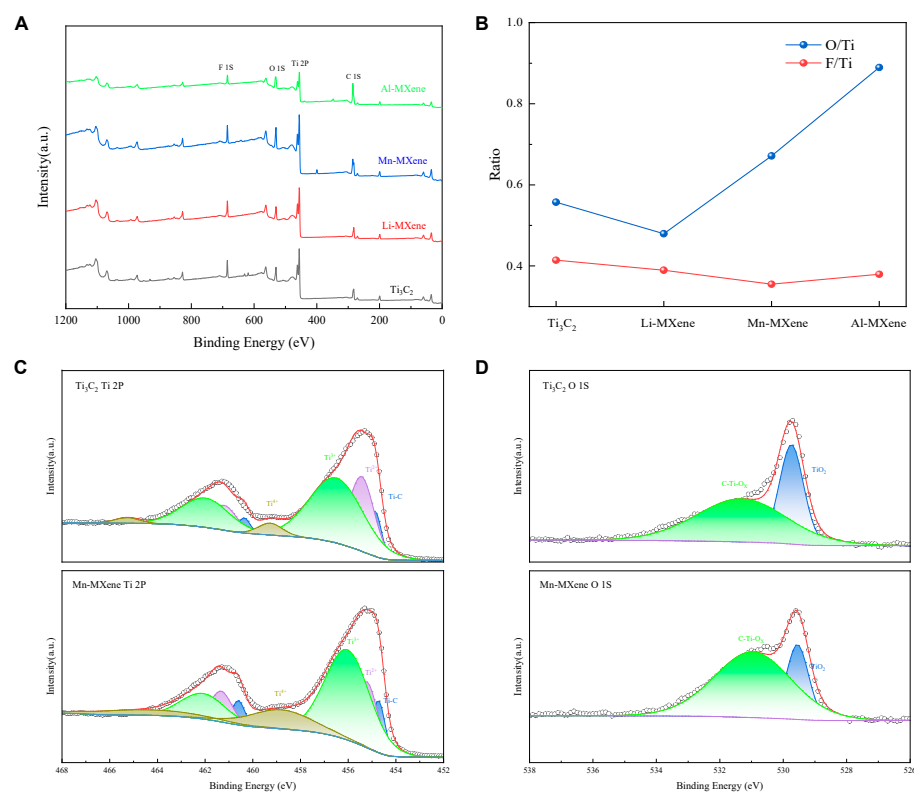


Figure 3. (A) X-ray photoelectron spectroscopy (XPS) survey spectra of Ti_3C_2 and M-MXene. (B) O/Ti and F/Ti calculated by integrating the areas of their corresponding XPS peaks. (C,D) High-resolution Ti 2p and O 1s XPS spectra comparing Mn-MXene with pristine Ti_3C_2 , the red lines show the fitted results.

To further analyze the electrochemical kinetics of Mn-MXene, electrochemical measurements were conducted in a Swagelok cell due to its low resistance (Figure 4A). The electrolyte and reference electrode were the same as in the previous tests, while the working electrode was replaced with MXene-based self-supporting films as the working electrodes.

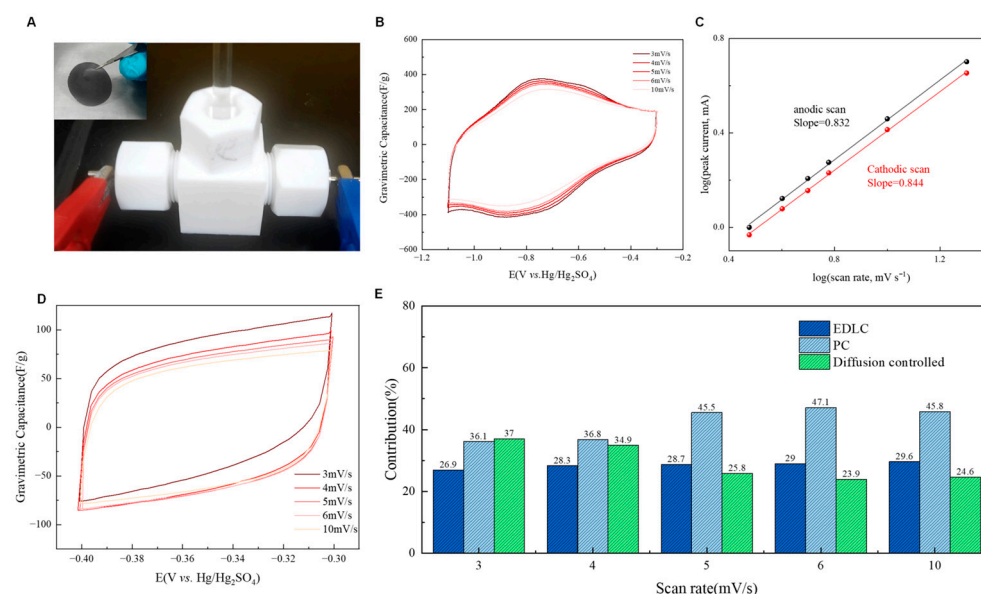


Figure 4. (A) Digital photograph of the Swagelok cell; inset shows the (B) CV curves of Mn-MXene film, (C) *b*-value analysis, (D) CV curves of EDLC survey, (E) capacitive contribution analysis of Mn-MXene.

Kinetic analysis of Mn-MXene was evaluated using CV at different scan rates (Figure 4B). For an electrochemical process, the total stored charge can be divided into two main contributions: (i) a capacitive-controlled part arising from fast Faradaic charge-transfer processes at surface atoms, together with a non-Faradaic contribution from electric double-layer adsorption; and (ii) a diffusion-controlled Faradaic intercalation process. The current in the CV curves follows Equation (4) with scan rate (v).

$$i = ab^v \quad (4)$$

where a is a constant depending on system-specific parameters, v is scan rate, and b characterizes the nature of the process. For a capacitive process, b is closer to 1, and for a diffusion-controlled process, b is closer to 0.5 [36].

Thus, the b -values can effectively differentiate between non-diffusion-limited capacitive effects and diffusion-controlled intercalation processes. To further assess the charge storage mechanism, logarithmic plots of peak current ($\log i$) versus scan rate $\log v$ for both the anodic and cathodic peaks within the range of 3–20 mV s^{-1} are presented in Figure 4C. The b -values for the anodic and cathodic peaks are 0.832 and 0.844, respectively, indicating that the charge storage process is predominantly non-diffusion-controlled, which accounts for the excellent rate capability of Mn-MXene. In comparison, the cathodic peak b -value of Ti_3C_2 is 0.753 (Figure S5), demonstrating the improved reaction kinetics of Mn-MXene.

To further distinguish and quantify the capacitive contributions to the overall current response, it is assumed that the current at a fixed potential arises from a combination of two independent currents: surface capacitive current and diffusion-controlled current.

$$i(V) = k_1v + k_2v^{1/2} \quad (5)$$

where v is the scan rate (mV s^{-1}), k_1v represents the current contribution from surface capacitive effects, and $k_2v^{1/2}$ corresponds to the current arising from diffusion-controlled Faradaic processes. So, k_1 and k_2 can be derived from the linear plot of $i(V)/v^{1/2}$ versus $v^{1/2}$ with different scan rates.

Capacitive-controlled behavior can be categorized into two components: EDLC (associated with the intercalation of hydrated ions between MXene layers) and PC (linked to redox reactions involving surface absorption and functional groups). As shown in Figures 4D and S5, the EDLC was measured by cycling the electrode within a non-faradaic potential region, where no charge-transfer reactions occur and only adsorption/desorption processes take place [24]. In this work, the selected potential window was -0.4 to -0.3 V versus $\text{Hg}/\text{Hg}_2\text{SO}_4$. At the center of the potential range (-0.35 V vs. $\text{Hg}/\text{Hg}_2\text{SO}_4$), the difference in current densities between the anodic (j_a) and cathodic (j_b) current densities was calculated for each scan rate (v). The value of C_{dl} can be determined by the following equation:

$$C_{dl} = \frac{j_a - j_b}{2v} \quad (6)$$

Figure 4E shows the capacitive contribution with scan rate of Mn-MXene. Mn-MXene, owing to its improved structural features, achieves a superior capacitive contribution of 63–75% over the range of 3–10 mV/s , reflecting enhanced kinetics even at high scan rates. Furthermore, the capacitive behavior of Mn-MXene is mainly dominated by PC. As the scan rate increases, the proportion of pseudocapacitive contribution increases progressively, whereas the EDLC contribution remains essentially constant. In contrast, Ti_3C_2 exhibits a capacitive contribution of 47.7% at 3 mV s^{-1} , which increases to 55.1% at 6 mV s^{-1} (Figure S5). The PC contribution of Ti_3C_2 is only 1.9% at 3 mV s^{-1} and increases to merely

12.6% at 10 mV s^{-1} . These results indicate that the enhanced specific capacitance of Mn-MXene is primarily attributed to the increased pseudocapacitive behavior.

The long-term cycling stability of Mn-MXene was also evaluated in a Swagelok cell, as shown in Figure 5A. Mn-MXene films were subjected to GCD tests at a current density of 10 A g^{-1} , and nearly 100% capacitance retention was maintained after 3000 cycles, demonstrating excellent cycling stability. Notably, the specific capacitance of Mn-MXene slightly increased during cycling, likely due to the expansion of interlayer spacing induced by electrochemical intercalation. Figure 5B presents the XRD patterns of Mn-MXene before and after cycling. The (002) peak shifts from 6.2° to 5.9° after 1000 cycles, further confirming both the structural integrity and the expansion of the interlayer spacing. These findings suggest that the intercalation of Mn^{2+} successfully expanded the interlayer spacing of Ti_3C_2 and modified its surface functional groups, providing Mn-MXene with enhanced ion transport channels and significantly improving electrochemical performance.

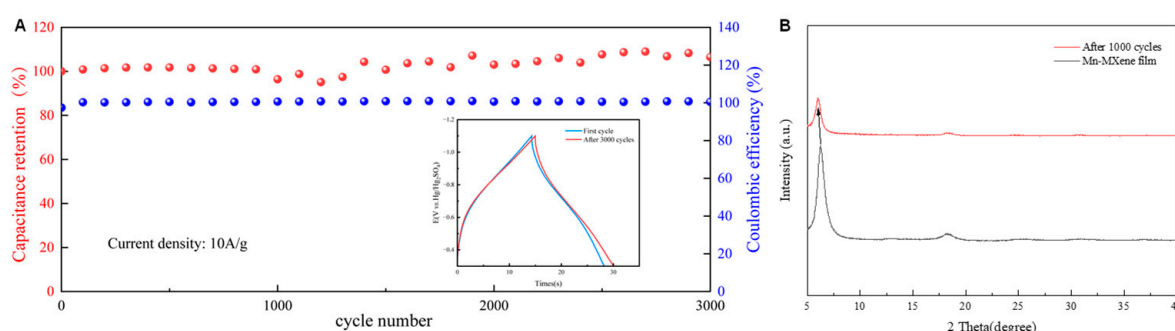


Figure 5. (A) Cycling stability of Mn-MXene. (B) The X-ray diffraction (XRD) patterns of Mn-MXene before and after GCD testing, the black arrow shows the shift of the peak.

4. Conclusions

In this study, we investigated a post-delamination metal-ion intercalation strategy to address the restacking issue of Ti_3C_2 MXene. Various metal ions with different valences and ionic radii were explored to assess their impact on the material's structure and performance. Specifically, the interlayer spacing of Mn-MXene was expanded to 1.55 nm, along with an increase in surface oxygen content, leading to a specific capacitance of 285 F g^{-1} at a scan rate of 10 mV s^{-1} in 1 M H_2SO_4 , which represents a 26% enhancement over Ti_3C_2 . Furthermore, Mn-MXene retains nearly 100% of its capacitance after 3000 charge–discharge cycles. Kinetic analysis confirms that Mn-MXene significantly enhances the capacitive contribution and improves the intercalation kinetics of electrolyte ions. These findings demonstrate that metal cation intercalation is a promising approach to optimizing the performance of two-dimensional materials beyond MXenes.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/nano16050331/s1>, Figure S1. (A) Tyndall effect of Ti_3C_2 dispersion. (B) Scanning electron microscopy of Ti_3C_2 powder. Figure S2. Percentage composition of the EDX mapping of elements. Figure S3. Cyclic voltammetry (CV) curves of Ti_3C_2 , Li-MXene, Al-MXene. Figure S4. (A,B) High-resolution Ti 2p and O 1s X-ray photoelectron spectroscopy (XPS) spectra of Li-MXene. (C,D) High-resolution Ti 2p and O 1s XPS spectra of Al-MXene. Figure S5. (A) CV curves of Ti_3C_2 film in the Swagelok cell. (B) b -value analysis of Ti_3C_2 . (C) CV curves of EDLC survey. (D) Capacitive contribution analysis of Ti_3C_2 . Table S1. Comparative study of MXene for specific capacitances. References [37–40] are cited in the supplementary materials.

Author Contributions: Conceptualization, Y.Z.; methodology, Q.J.; investigation, Y.Z.; writing—original draft preparation, Y.Z.; writing—review and editing, Q.J.; supervision, Q.J. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The original contributions presented in this study are included in the article/Supplementary Materials. Further inquiries can be directed to the corresponding author.

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