

Multilayer MXene Heterostructures and Nanohybrids for Multifunctional Applications: A Review

Sharif Tasnim Mahmud, Md Mehdi Hasan, Sudipta Bain, Sheikh Tamjidur Rahman, Mukitir Rhaman, Md Milon Hossain,* and Mustafa Ordu



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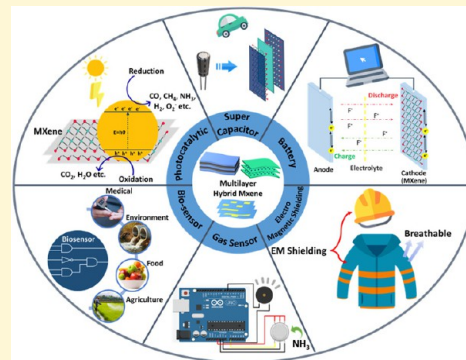
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ABSTRACT: MXenes (transition metal carbides and nitrides) have experienced exponential growth over the last two decades, thanks to their excellent physical, chemical, and mechanical properties. Intriguing properties like high conductivity, wear, and corrosion resistance while maintaining flexibility are the strong motivation behind the exploration of MXenes. Moreover, the large surface area and unique layered structure enhance the functionality of multilayer-MXene heterostructures and hybrids. This paper reviews the synthesis chemistry, structure properties of multilayer MXenes, and their multifunctional applications. MXene synthesis under different conditions, their hybrids and composites, intercalation, and structural geometries are discussed. The electrical, mechanical, optical, and magnetic properties of MXenes are briefly presented. Recent progress and development in MXene-based heterostructures and nanohybrids for supercapacitors, batteries, environmental and water treatment, antibacterial and tissue engineering, and electromagnetic absorption and shielding are systematically discussed. Finally, research challenges and a perspective in this specified area are addressed for potential developments.



The growth of nanotechnology drives the rapid development of high-performance technologies in the 21st century. Extraordinary computational and experimental progress in materials technology has paved the way to understanding matters better and allowed the modification of materials properties at molecular and atomic levels.^{1,2} The low dimensional nanomaterials are accelerating the fabrication and commercialization of nanodevices with low power consumption and superior performance.³ The nanomaterials range from zero-dimensional (0D) to three-dimensional (3D) structures, i.e., 0D quantum dots,⁴ 1D nanowires,⁵ 2D nanosheets,⁶ and 3D nanospheres. Since the successful exfoliation of 2D graphene in 2004,⁷ the 2D nanomaterials family has experienced tremendous progress, and different multifunctional applications of 2D nanomaterials are expanding. Notable 2D nanomaterials under investigation transition metal dichalcogenides (TMDs),⁸ hexagonal boron nitride (hBN),⁹ phosphorene,¹⁰ silicene,¹¹ 2D halide perovskite nanomaterials,¹² molybdenum disulfide (MoS₂),¹³ 2D metal–organic framework (MOF) nanosheets,¹⁴ and the transition metal carbides and/or nitrides (MXenes).¹⁵ 2D nanomaterials have already shown promise for diverse applications utilizing unique structural features and excellent physicochemical, optical, and magnetic properties. However, the

properties surprisingly differ due to the variation in crystal structure and compositions, i.e., dielectric, semiconductor, or semimetal.¹⁶ Ultrathin atomic thickness endows 2D nanomaterials with excellent mechanical flexibility and optical transparency. Combining large lateral size, ultrahigh surface areas, and superior thermal conductivity, 2D nanomaterials becomes ideal for myriad applications.^{17–19} Surface properties provide ample active sites to enhance the interaction with other nanomaterials and polymers to form hybrid structures.²⁰

MXenes have experienced growing research interest since their discovery at Drexel University in 2011 by the exfoliation of Ti₃AlC₂ at room temperature. Because the transition metal carbides and nitrides include a material family of more than 60 members, the report explored a theoretically unlimited number of MXenes.^{21–26} The presence of abundant surface termination

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MXenes (transition metal carbides and nitrides) have experienced exponential growth over the last two decades, thanks to their excellent physical, chemical, and mechanical properties. Intriguing properties like high conductivity, wear, and corrosion resistance while maintaining flexibility are the strong motivation behind the exploration of MXenes.

groups, i.e., oxygen (O), hydroxyl (–OH), and halogens (F, Cl), endows MXenes with superior physical and chemical properties along with ease of processability.^{27,28} MXenes consist of multilayer sandwich structures that usually contain an odd number of layers, where carbon or nitrogen (X) layers are sandwiched between metals (M).¹⁵ MXenes are represented in $M_{n+1}X_nT_x$ form, which includes transition metals (M elements), e.g., Sc, Y, Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Mn, carbon, and nitrogen (X elements) along with the functional surface termination groups (T_x elements).^{29,30} The unique structure-derived properties such as hydrophilicity, reactivity, and high negative ζ -potential induce stable colloidal solution combined with electrical, electrochemical, optoelectronic, and magnetic properties are driving the theoretical and experimental study of MXenes.^{31–33} The surface properties allow the formation of the MXene-based multilayer hybrid structure by combining MXenes with compatible nanomaterials using self-assembly,³⁴ polymerization,³⁵ and covalent functionalization.³⁶ The development of MXenes-based energy application,³⁰ sensors,³⁷ catalysts,³⁸ environmental remediations and water treatment,^{39,40} photonics and optoelectronics,⁴¹ electromagnetic interference,⁴² joule heating,² and membranes⁴³ have been reviewed and reported in recent years. However, the works on MXene-based heterostructures and hybrid structures for multifunctional applications except for supercapacitors⁴⁴ have not been reported yet, despite showing exciting properties and applications in most of the above-mentioned areas. A careful review of the multilayer MXene (ML-MXene)-based heterostructures and nanohybrid is essential to understand the trends in this research area and the areas that require attention in the future to develop structures with superior stability and functionality.

This review provides a comprehensive insight into the structure–property–application relationship of MXene-based hybrid structures and the introductory discussion on MXenes to clarify the basic concepts behind the development. The first section of the work provides a brief discussion on the synthesis of MXene and its hybrid structures. The subsequent sections highlight the structural attributes of MXenes, the geometrical morphology of the MXene hybrids, and the effects on the properties. Later, the properties of the MXenes, i.e., electrical, mechanical, optical, and magnetic, are introduced. Subsequently, the applications of MXene hybrid/composites in energy storage, i.e., capacitors and batteries, sensing, catalysis for the environment, biomedical, and electromagnetic shielding, have been presented. The review presents a critical discussion on each section to identify the research gaps. Finally, the paper provides pressing challenges and possible solutions for future directions of multilayer-MXene hybrid research.

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■ SYNTHESIS OF MXENE

MXenes are the class of transition metal carbide, nitride, and carbonitrides, synthesized by the selective etching of the “A” element in the MAX phase.⁴⁵ MAX particles are transformed into a loosely packaged accordion-like structure from the solid, dense state, resembling exfoliated graphite, and referred to as loosely formed, multilayer, or ML-MXenes flakes.^{45,46} Figure 1 shows a periodic table with highlighted elements that commonly occur in the formation of MXenes. The chemical composition of MXene is generally represented by $M_{(n+1)}X_nT_x$, where “M” is an early transition metal, “A” represents the materials from the group 13 or 14 of the periodic table, and “X” indicates the carbon or nitrogen, so basically, these materials are nitrides and carbides. The molar ratio number, “n”, usually means 1, 2, 3, or 5.⁴⁷ The surface chemical groups on MXene sheets are represented as “T”. When “A” is etched from the MAX phase, “T” preserves the charge neutrality in MXene, which depends on the nature of the etchants. The compositions of “T” are currently F, O, and OH because of an F-contained etchant. It is predicted that around 70 MXene varieties can be prepared from the MAX phase or non-MAX phase.^{48–50} The following section discusses the most significant MXene multilayer and hybrid multilayer formation methods.

Multilayer MXene. Two major routes explored for the synthesis of MXene are the top-down and the bottom-up approaches.⁵¹ The top-down approach in MXene production refers to the process of reducing the size of bulk material/powder or removing part of the bulk structure to leave micro to nanometer particles.^{48,49} On the one hand, selective etching and exfoliation are categorized as top-down processes. On the other hand, physical vapor deposition (PVD) and chemical vapor deposition (CVD) are categorized as the bottom-up processes.^{51,52} The top-down approach is usually preferred for the preparation of ML-MXenes because the first successful selective etching of aluminum from Ti_3AlC_2 for the production of multilayer Ti_3C_2 was achieved with a top-down method. MXene synthesis begins with the etching of the MAX phase to replace element “A” (e.g., aluminum) atomic layer with surface termination groups such as –OH, –O, and –F to form the $M_{(n+1)}X_nT_x$.⁵³ Hydrogen and van der Waals bonds resulting from the etching process hold the layers of the MXene together.⁵⁴ Wet chemical etching with hydrofluoric acid (HF) synthesizes multilayered MXene flakes of different compositions such as Ti_2CT_x , $Ti_3C_nT_x$, Nb_2CT_x , and V_2CT_x .^{46,55} Different structures of MXenes such as scrolls, nanotubes, and multilayer Ti_3C_2 were obtained following sonication in addition to nanosheets.^{49,56} A synthesis map of $Ti_3C_2T_x$ using HF and in situ HF at room temperature and delamination methods are presented in Figures 1 and 2.^{48,49,57}

Although HF is the primary etchant in the MXene fabrication processes, alternative etchants have been explored due to the highly volatile and hazardous characteristics of HF.⁵⁸ Molten salts,^{46,59} alkaline solutions,^{60,61} and hydrothermal treat-

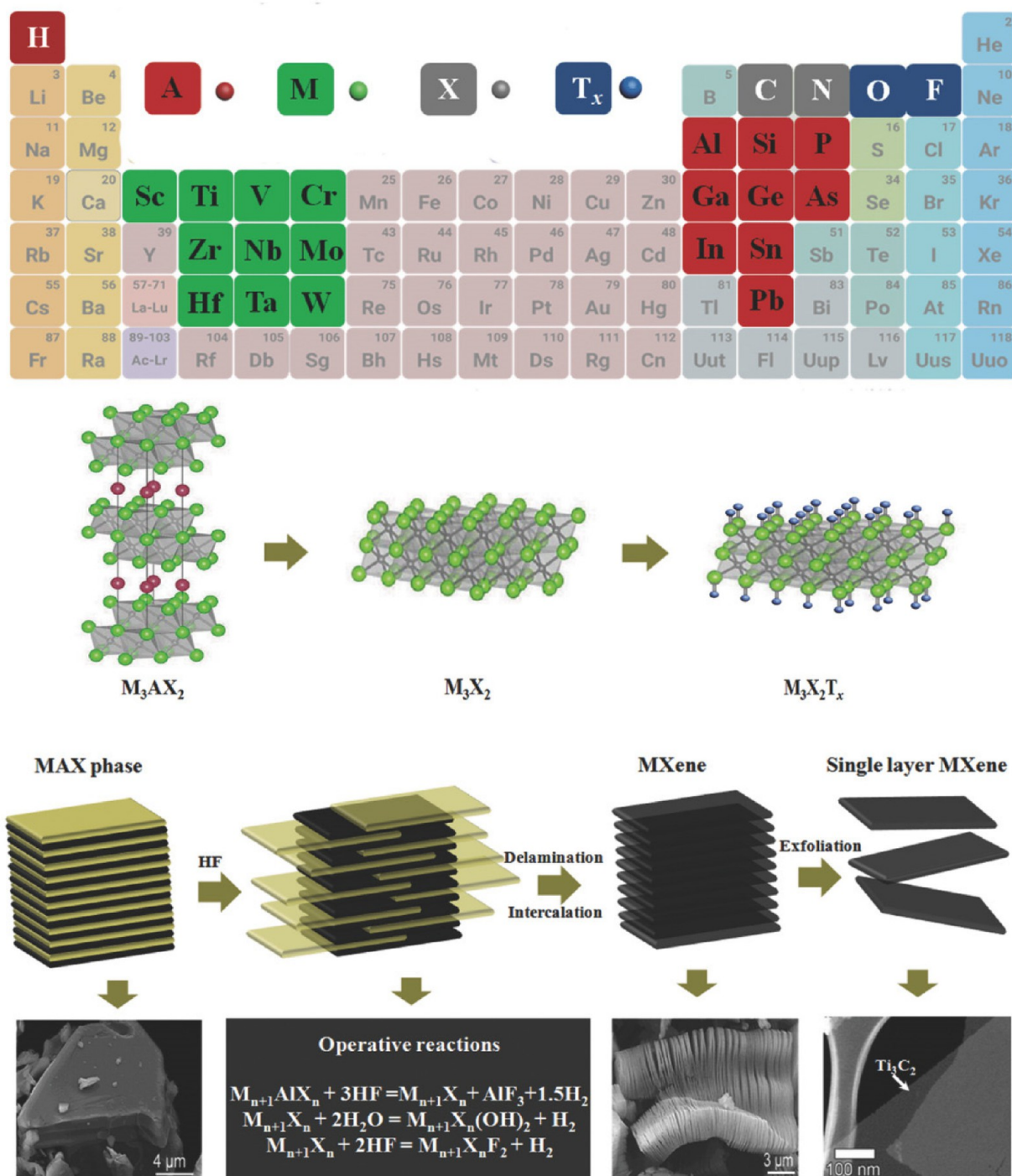


Figure 1. Periodic table with highlighted elements used in MAX processes and Schematic of MXene synthesis process using HF etchant. (Reproduced with permission from ref 56. Copyright 2018, WILEY-VCH Verlag GmbH & Co. KGaA.)

ment^{60,62,63} widened the synthesis routes and the resultant properties. Bifluorides of potassium, sodium, and ammonium are the reported alternative etchants for selective etching of Al from Ti₃AlC₂.^{58,64,65} However, HF-assisted synthesis of MXene causes intercalation of cations with fluoride or bifluoride salts and exhibits greater interlayer spacing. The interlayer spacing increases by the combination of HF and fluoride salts compared to the etching only with HF.^{58,64}

A lower HF concentration (5–10 wt %) containing acid mixtures is used for in situ HF synthesis. To synthesize Mo₂TiC₂ and Mo₂Ti₂C₃ from the Mo₂TiAlC₂ and Mo₂Ti₂AlC₃ MAX

phases, etchants based on HF/HCl are used.^{66,67} For the LiF/HCl etching route, the molar concentration of the two reactants defines the required amount/concentration of HF, LiCl production, and remnant HCl. In situ HF etchants utilizing LiF/HCl yielded processable Ti₃C₂ MXene when an intercalant (Li⁺) was introduced into the etchant solution. The concentration of H⁺ exchanged with Li⁺ is achieved by varying the concentration of exchanged cations which determines the intercalation mechanism of the LiF/HCl. However, the scalability of these methods is challenging due to the presence of unetched MXene during exfoliation.^{55,68} Alkaline etchants

Synthesis of $Ti_3C_2T_x$ MXene

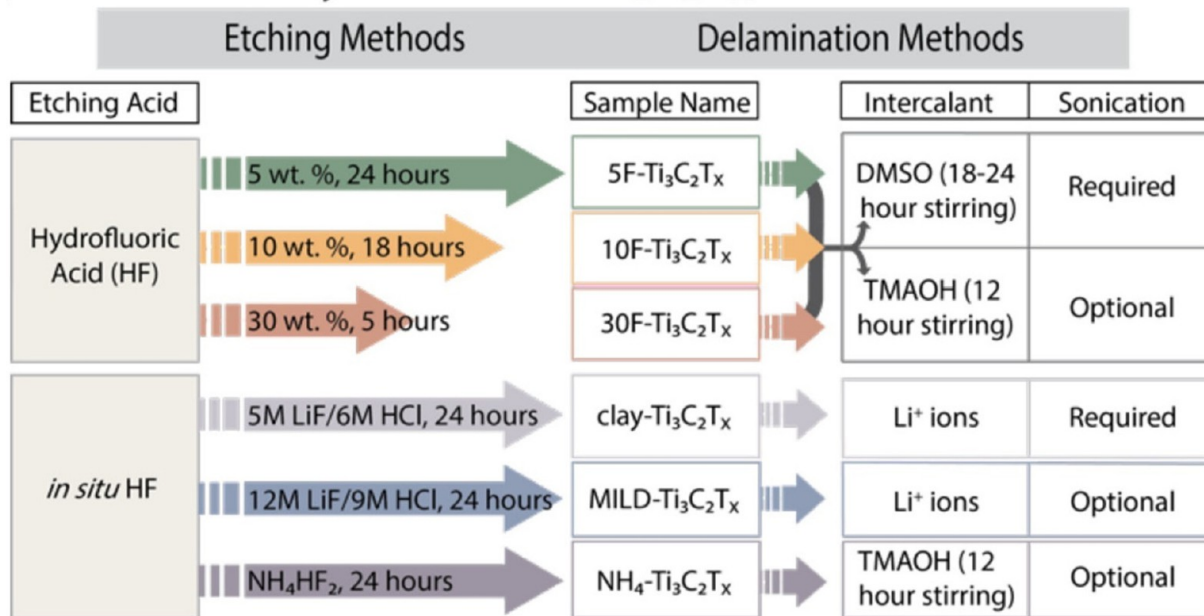


Figure 2. Etching with HF (top) and *in situ* HF (below). In the HF route, three different concentrations with different durations are used to synthesize $Ti_3C_2T_x$. (Reproduced with permission from ref 55. Copyright 2017, American Chemical Society.)

have the capability of etching Ti_3AlC_2 or other layered precursors utilizing suitable binding properties with Al. The removal of Al in alkaline conditions often involves hydrothermal treatment at higher temperatures or pressures.^{46,59,69} Conventional HF aqueous acidic solutions are ineffective in etching nitride-based MAX phases. Therefore, the nitride MXene, especially Ti_4N_3 , involves selective etching of Al from Ti_4AlN_3 by using molten salts such as potassium fluoride (KF), sodium fluoride (NaF), and lithium fluoride (LiF).^{46,66} A list of different types of MXenes synthesized from various precursors and etchants is presented in Table 1.

Multilayer MXene Heterostructures and Nanohybrids.

MXenes are thermodynamically metastable with great surface energy and a high percentage of exposed metal atoms present on the surface. These properties are negatively influenced by oxygen regardless of oxygen phases, even under ambient conditions.^{90,91} MXene in the ambient atmosphere imparts new functionalities combined with different nanoparticles to produce hybrid MXenes.^{92,93} Among different 2D materials, MXenes have exceptional surface tunability and adjustable physical and chemical properties, making them a promising material for hybridization. In particular, exfoliated 2D MXene hybrids with nanoparticles and polymers are utilizable as the base for constructing new functional hybrid materials.⁵⁶ The high surface-to-volume ratio, surface control, and outstanding electrochemical properties of MXene are beneficial for the applications of MXene hybrids.⁶⁹

Combining the compatible components in a heterostructure material at the molecular level often leads to superior physical, mechanical, chemical, and electrochemical properties suitable for high-performance applications. These properties can be further improved by tuning the elemental compositions, morphologies, shapes, and molecular-level interactions/interfaces.^{94,95} The broader spectrum of organic molecules and the existence of more than 20 compositions of MXenes with distinct properties exhibit the enormous potential of MXene hetero-

structures and hybrids. MXene hybrids and heterostructures with tunable shapes, properties, and morphologies can be synthesized by rationally choosing the MXene and components within a hybrid. ML-MXene hybrid synthesis can be categorized into organic molecules and polymer self-assembly,^{96–98} oxidant-free monomer polymerization into polymers,^{99–101} and covalent functionalization.^{102–104} The complex molecular interaction such as π - π stacking interlinkage, hydrogen bonds, and van der Waals interconnection between the organic functional group and the 2D solid-state materials dictates the self-assembled hybrid structure.^{105,106} Polymerization of chemical oxidants requires the deposition of the conducting polymer in the existence of a host material, and the fabrication of conducting polymer hybrids requires monomer addition to the acidic solution of an oxidant.^{66,68,107} *In situ* polymerization improves the dispersion of MXene in polymers, interfacial adhesion between MXene and the polymer matrix, and improvement in mechanical, electrical, and thermal properties. To initiate the polymerization process, an oxidant such as ammonium persulfate (APS) is used in the oxidative chemical deposition of polyaniline (PANI) on a substrate and the surface of various materials.¹⁰⁷ Several other methods including electrophoretic deposition (EPD),¹⁰⁸ ultrasonication treatment,¹⁰⁹ facile self-assembly process,¹¹⁰ stirrings followed by vacuum filtration,¹¹¹ primitive calcination,¹¹² incubation,⁸⁷ and mechanical high-speed agitation¹¹³ have also been reported. The mechanical agitation method involves a simple physical mixture and affects the dispersion and the interface. Table 2 summarizes the synthesis conditions and potential applications of the ML-MXene heterostructures and hybrids.

The stability of the MXene in a hybrid structure is critical for various applications and the long-term stability is determined by preventing oxidation. The degradation mechanisms differ depending on the type of MXene; for example, in the presence of water, Ti_3C_2 MXene degrades to a cloudy-white solution of

Table 1. Synthesis of ML-MXene with Different Etchants and Conditions

ML-MXene	precursors	etchants (time, temperature)	purpose/application	ref
Ti ₃ C ₂	Ti ₃ AlC ₂	5 wt % HF (24 h, RT) 10 wt % HF (24 h, RT) 30 wt % HF (4 h, RT)	optimization of synthesis parameters	55
Ti ₂ C	Ti ₂ AlC	13.5 M LiF in 6 M (15 h, 40 °C)	hybrid Li-ion battery (LIB)	70
Ti ₃ C ₂	Ti ₃ AlC ₂	34.5 M LiF in 10 M HCl (24 h, 35 °C)		
Ti ₂ C	Ti ₂ AlC	1.6 M FeF ₃ in 6 M HCl (25 h, 50 °C)	performance of new etchants on MAX phases	71
Ti ₃ C ₂	Ti ₃ AlC ₂	2.3 M FeF ₃ in 6 M HCl (24 h, 30 °C)		
Ti ₂ C	Ti ₂ AlC	21.5 M KF in 12 M HCl (24 h, 30 °C) 21.5 M NaF in 12 M HCl (24 h, 30 °C) 21.5 M NH ₄ F in 12 M HCl (24 h, 30 °C)	storage of natural gas	72
Ti ₃ C ₂	Ti ₃ AlC ₂	31 M KF in 12 M HCl (24 h, 30 °C) 31 M NaF in 12 M HCl (24 h, 30 °C) 31 M NH ₄ F in 12 M HCl (24 h, RT)		
Ti ₃ C ₂	Ti ₃ AlC ₂	40 wt % HF (24 h, RT)	fabrication of Sn ₄ ⁺ ion decorated nanocomposites for Lithium ion battery (LIB)	73
Ti ₃ C ₂	Ti ₃ AlC ₂	50 wt % HF (2 h, 50 °C) 50 wt % HF (10 h, RT)	effect of HF solution on Ti ₃ AlC ₂ powders	53
Ti ₃ C ₂	Ti ₃ AlC ₂	50 wt % HF (22 h, RT)	interaction of layered MXene	74
Ti ₃ C ₂	Ti ₃ AlC ₂	50 wt % HF (18 h, RT)	MXene for high volumetric capacitance	75
Ti ₃ C ₂	Ti ₃ AlC ₂	5 M LiCl in 6 M HF (24 h, 25 °C)	effects of the presence of LiCl during the chemical etching	76
Ti ₃ C ₂	Ti ₃ AlC ₂	1 M NH ₄ HF ₂ (12 h, 60 °C) 1 M NaHF ₂ (12 h, 60 °C) 1 M KHF ₂ (12 h, 60 °C)	etching with bifluoride (NaHF ₂ , KHF ₂ , NH ₄ HF ₂) in single-stage process	64
Ti ₃ C ₂	Ti ₃ AlC ₂	27.5 M NaOH (12 h, 270 °C)	fluorine-free method, e.g., alkali-etching strategy at high temperature	61
Ti ₃ C ₂	Ti ₃ AlC ₂	NH ₄ F, hydrothermal (24 h, 150 °C)	preparation by hydrothermal method and analysis of electrochemical properties	63
Nb ₂ C	Nb ₂ AlC	50 wt % HF (90 h, RT)	anode material for LIB	77
V ₂ C	V ₂ AlC	50 wt % HF (90 h, RT)		
V ₂ C	V ₂ AlC	3.35 M NaF in 12 M HCl (48 h, 90 °C)	preparation with NaF at high temperature as anode for LIB	78
Zr ₃ C ₂	Zr ₃ Al ₃ C ₅	1 M NaHF ₂ (12 h, RT) 1 M KHF ₂ (12 h, RT)	comparison between structural stability of Zr ₃ C ₂ T _z and Ti ₃ C ₂ T _z MXenes	79
Mo ₂ TiC ₂	Mo ₂ TiAlC ₂	50 wt % HF (48 h, 55 °C)	synthesis and analysis of Mo ₂ TiC ₂ T _z , Mo ₂ Ti ₂ C ₃ T _z , and Cr ₂ TiC ₂ T _x	80
Mo ₂ Ti ₂ C ₂	Mo ₂ Ti ₂ AlC ₂	50 wt % HF (96 h, 55 °C)		
Cr ₂ TiC ₂	Cr ₂ TiAlC ₂	5 M LiF in 6 M HCl (42 h, 55 °C)		
Nb ₄ C ₃	Nb ₄ AlC ₃	50 wt % HF (96 h, RT)	synthesis of phase-pure Nb ₄ C ₃ with formula M ₄ X ₃	81
(Nb,Ti) ₄ C ₃	(Nb,Ti) ₄ AlC ₃	50 wt % HF (90 h, 50 °C) 10 M LiF in 12 M HCl (180 h, 50 °C)	Nb ₄ C ₃ T _x multilayer MXene for energy storage applications	82
Ti ₃ C ₂	Ti ₃ SiC ₂	30 wt % HF + (oxidant), e.g., (HNO ₃ , KMnO ₄ , (NH ₄) ₂ S ₂ O ₈ , or FeCl ₃) (47 h, 40 °C)	oxidant-assisted selective etching of Si from Ti ₃ SiC ₂	83
Ti ₃ C ₂ T _z	Ti ₃ AlC ₂	10 wt % HF (24 h, 25 °C) + LiCl, LiBr, LiI 48 wt % HF + HCl, HBr, HI, H ₃ PO ₄ , or H ₂ SO ₄	intercalation and deintercalation mechanism	84
Ti ₃ C ₂	Ti ₃ AlC ₂	40 wt % HF (48 h, 60 °C) + Ti ₃ C ₂ calcinated high temp., 200–1200 °C	vacuum calcination for better electrochemical and thermal properties for LIB	85
Ti ₃ C ₂ T _x	Ti ₃ AlC ₂	5, 10, and 30 wt % HF (24, 18, 4 h, RT) 5 wt % HF + H ₂ SO ₄ (96 wt %, HCl (37 wt %))	thermal gravimetry and mass spectrometry up to 1500 °C under a He atmosphere	86
Nb ₂ CT _x	Nb ₂ AlC	50 wt % HF (48–120 h, 50 °C)		
Mo ₂ CT _x	Mo ₂ Ga ₂ C	50 wt % HF (48–120 h, 50 °C)		
Ti ₃ C ₂ T _x	Ti ₃ AlC ₂	40% wt. HF + 30 min sonication and stirred (168 h, RT)	biomedical application with their biocompatibility, hydrophilic property	87
Ti ₃ C ₂ T _x	Ti ₃ AlC ₂	6 M HCl + LiF (40 h, 45 °C)	humidity sensor	88
Ti ₃ C ₂ T _x	Ti ₃ AlC ₂	9 M HCl + LiF (ice bath) (24 h, 35 °C)	exfoliation via water freezing method	89

titanium dioxide (TiO₂) anatase. Hybridization has the potential to improve the long-term stability of MXene.

The conducting polymer/monomers on the MXene surface are polymerized by oxidants such as ammonium persulfate and other oxidants. MXenes decompose into TMO-carbon hybrids with a high oxide load on the carbon sheets. The majority of

MXenes oxidation research focuses on Ti-based compounds.¹¹⁴ Wang et al.¹¹⁵ reported hydrothermal oxidation of Ti₃C₂T_x and applied the oxidized composite for photocatalysis. The electron microscopy image showed that heterogeneous TiO₂ nanoparticles nucleated to form a heterojoint structure on the surface of Ti₃C₂.^{115,116} The sonication of dimethylformamide (DMF)-

Table 2. Synthesis Conditions for Different ML-MXene Heterostructure and Hybrid with Their Potential Application

ML-MXene	precursors	etchants (time, temperature)	hybrid synthesis method	purpose/application	ref
Ti ₃ C ₂ T _x -sodium alginate (SA)	Ti ₃ AlC ₂	LiF in 6 M HCl (24 h, 35 °C)	self-assembly	electromagnetic interference (EMI) shielding	66
2D niobium carbide (Nb ₂ C)-PVP (polyvinylpyrrolidone)	Nb ₂ AlC	60 mL of a 50% HF (48 h, RT)	mixing	phototherapy of cancer	113
Ti ₃ C ₂ T _x /poly-EDOT (3,4-ethylenedioxythiophene) hybrids	Ti ₃ AlC ₂	LiF (2 g) in HCl (9 M, 20 mL), 24 h, 35 °C	mixing	EDOT induced polymerization mechanism of charge-transfer	120
Ti ₃ C ₂ -DOX (doxorubicin hydrochloride)	Ti ₃ AlC ₂	10 mL of 9 M HCl containing 1 g of LiF (72 h, RT)	mixing	biocompatible, tumor-specific effective cancer cell killing and tumor tissue destruction	121
Mo _{0.133} C MXene- PEDOT:PSS (poly(3,4-ethylenedioxythiophene) polystyrenesulfonate)	(Mo _{2/3} Sc _{1/3}) ₂ AlC	20 mL of 48% HF (24 h, RT)	mixing	ultrathin flexible solid-state supercapacitor	122
Ti ₃ C ₂ -PPy (polypyrrole)	Ti ₃ AlC ₂	10 mL of 48% HF (8 h, 60 °C)	self-assembly	improve both PPy-based electrode capacity and cycle stability	123
Ti ₃ C ₂ T _x -SO ₃ H	Ti ₃ AlC ₂	20 mL of 49% HF (72 h, 25 °C)	mixing	efficient proton transfer pathways for enhanced conduction	124
Ti ₃ C ₂ -LLDPE	Ti ₃ AlC ₂	49% HF (24 h, 60 °C)	mixing	MXenes enhance the crystallization and kinetics of thermal degradation of LLDPE	125
Ti ₃ C ₂ T _x /PVA (poly vinyl alcohol) composites	Ti ₃ AlC ₂	10 mL of 50 wt % HF solution (18 h, RT)	mixing	Raman spectroscopy	126
Ti ₃ C ₂ /UHMWPE (ultrahigh molecular weight polyethylene)	Ti ₃ AlC ₂	100 mL of 49% HF solution for (24 h, RT)	mixing	morphology, structure, and properties of the Ti ₃ C ₂ /UHMWPE nanocomposites	127
Ti ₃ C ₂ T _x -PDAC (poly diallyldimethylammonium chloride)	Ti ₃ AlC ₂	30 mL of 6 M HCl (45 h, 40 °C)	layer-by-layer (lbl) assembly	conductive coatings for wearable electronics and biometric sensors	98
Ti ₃ C ₂ T _x /PPy hybrids	Ti ₃ AlC ₂	15 mL of 40 wt % HF solution (24 h, RT)	oxidative polymerization	high-performance electromagnetic wave absorption	128
polyaniline (PANI)-Ti ₃ C ₂	Ti ₃ AlC ₂	20 mL of 1 M HCl (6 h, 2 °C)	mixing	supercapacitor electrode material with high specific capacitance and excellent cycling stability	129
PANI@TiO ₂ /Ti ₃ C ₂ T _x	Ti ₃ AlC ₂	3 M HCl (24 h, 180 °C)	self-assembly	hierarchical structure composite for next-generation electrochemical capacitors electrode	130
Ti ₃ C ₂ T _x /glycine hybrids	Ti ₃ AlC ₂	LiF (2 g) stirred in 20 mL of 9 M HCl (24 h, 35 °C)	mixing	interaction mechanism of MXene, Ti ₃ C ₂ T _x , and amino acid-glycine on charge storage	103
pyrrole (C ₄ H ₄ NH) and Ti ₃ C ₂ T _x hybrids	Ti ₃ AlC ₂	20 mL of 9 M HCl (was added to 7.5 M (2 g) of LiF (2 h, 35 °C)	mixing	pyrrole intercalation, orientation, and metal-free polymerization on Ti ₃ C ₂ T _x MXene	100
CNF (cellulose nanofiber) and Ti ₃ C ₂ T _x hybrids	Ti ₃ AlC ₂	2 g of LiF, 40 mL of 9 M HCl (24 h, 35 °C)	mixing	high-performance EMI films	131
MXene/MPFs nanosheets	Ti ₃ AlC ₂	LiF (1.5 g), 30 mL of 9 M HCl (72 h, 60 °C)	mixing	flexible supercapacitor	132
Silver nanowire (AgNW) and MXene nanosheets	Ti ₃ AlC ₂	LiF (3.2 g), 40 mL of 9 M HCl (24 h, 35 °C)	mixing	lightweight EMI shielding materials	133
MCF(MXene/C hybrid foam) and MCF/epoxy	Ti ₃ AlC ₂	3.6 g LiF, 40 mL of 9 M HCl (24 h, 35 °C)	mixing	EMI shielding	134
PDMAEMA (poly dimethylamino ethyl methacrylate)-Ti ₃ CT _x hybrid	Ti ₃ AlC ₂	10 mL of concentrated HF (40%) (24 h, 80 °C)	mixing	temperature sensing	135
Cu-Ti ₃ C ₂ T _x	Ti ₃ AlC ₂	40% 50 mL HF (24 h, 60 °C)	mixing	all-solid-state super-capacitor	136
Ti ₃ C ₂ T _x -rGO (graphene oxide) hybrids	Ti ₃ AlC ₂	1.0 g of LiF in 20 mL of 12 mol/L HCl (24 h, 35 °C)	self-assembly	high temperature and fire-resistant thermoplastic polyurethane elastomer (TPU)	137
Ti ₃ C ₂ T _x and Ni nanochain hybrid	Ti ₃ AlC ₂	2 g of LiF, 40 mL of 9 M HCl (24 h, 35 °C)	mixing	EM wave absorption and shielding	138
SnS ₂ /Sn ₃ S ₄ -Ti ₃ C ₂ hybrid	Ti ₃ AlC ₂	40 mL of 45% HF (72 h, 25 °C)	self-assembly	facile solvothermal and calcination process	139
MoS ₂ @Ti ₃ C ₂ T _x MXene hybrid	Ti ₃ AlC ₂	48% 50 mL of HF (24 h, RT)	mixing	electrochemical apt sensor (MEA) for sensitive and rapid quantification of thyroxine (T4)	116

intercalated MXene and other materials (e.g., graphene oxide [GO]) in deionized (DI) water followed by centrifugation results in the MXene-hybrid dispersion.^{117,118} Yan et al.¹¹⁹ reported the hybrid electrode films via electrostatic self-assembly of the MXene/rGO (reduced graphene oxide). The diallyl dimethylammonium chloride (G-PDDA) suspension was injected into the MXene solution under stirring and later exposed to a sonic probe.¹¹⁹

Structure and Intercalation of Multilayer MXene.

Besides the surface termination group, intercalation significantly affects the properties of the MXene. Water, organic molecules, and metal ions are commonly intercalated into MXene and play a remarkable role in tailoring the functionality of MXene. On the

one hand, organic molecules improve the electrochemical capacitance, while water intercalation surges the interlayer spacing, and surface absorption and also influences the gas transport behavior.^{51,140–146} Simulation shows the effect of different termination groups on altering the bandgap and the Fermi level of MXene.¹⁴⁷ On the other hand, exposure of the “M” metal to the air decreases the dimension of the structure.⁴⁹ Modification of the surface porosity and microstructure using the heteroatoms leads to higher performance.¹⁴⁸ A higher surface area is advantageous for the fabrication of energy storage and advanced electrode materials.¹⁴⁹

To achieve a single atomic layer in 2D, material delamination is done. Different procedures can be followed to delaminate the

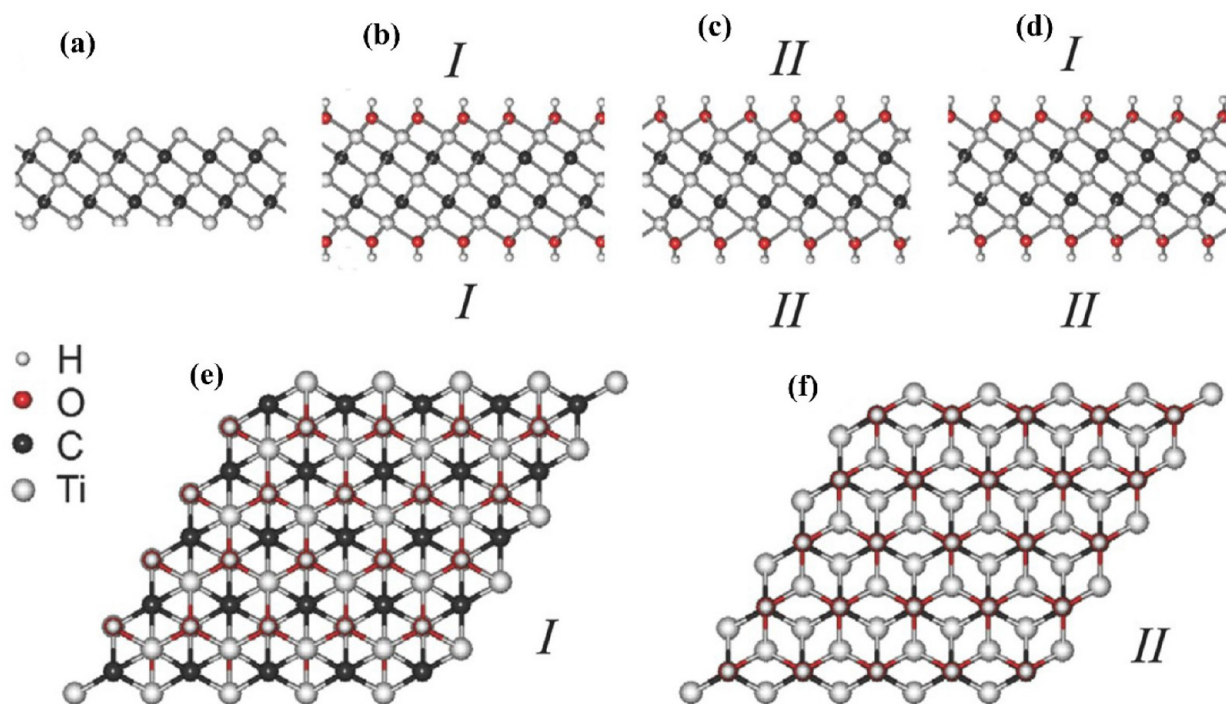


Figure 3. Surface atom arrangements of functionalized MXenes in various configurations: (a) pure Ti_3C_2 , (b) I- $\text{Ti}_3\text{C}_2(\text{OH})_2$, (c) II- $\text{Ti}_2\text{C}(\text{OH})_2$, and (d) III- $\text{Ti}_3\text{C}_2(\text{OH})_2$ (combination of b and c). Top views of (e) I- $\text{Ti}_3\text{C}_2(\text{OH})_2$ and (f) II- $\text{Ti}_3\text{C}_2(\text{OH})_2$. (Reproduced with permission from ref 29. Copyright 2013, WILEY-VCH Verlag GmbH & Co. KGaA.)

multilayer structure, i.e., mechanical cleavage, chemical intercalation and exfoliation, and liquid exfoliation.^{150,151} The delaminated $\text{Ti}_3\text{C}_2\text{T}_x$ shows uneven edges, tiny dark particles on the surface, and the size decreases incrementally. The particle may be partially oxidized, and the vacancies progressively become holes on the surface that lead to strength loss.^{152,153} The defects in MXene batteries react irreversibly with the cations and lower the initial Coulombic efficiency. In turn, this affects the conductivity of MXene.^{154,155} In contrast, atomic defects like vacancies and antisites in 2D materials can alter the electronic behavior, leading to changes in the electronic structure as well as optical and magnetic properties. Defects in ML-MXenes become more challenging to examine due to the increased number of atoms over the material.¹⁵⁶

Intercalation and surface group modification enhance the performance and capacitance of the MXene sheets as an electrode material. Cation intercalation improves the gravimetric capacitance in $\text{Ti}_3\text{C}_2\text{T}_x$, whereas controllable surface modification exploits the energy storage capacity. Termination in the presence of low terminal group concentration results in a higher redox reaction on the surface of $\text{Ti}_3\text{C}_2\text{T}_x$ and increases the capacitance.^{157,158}

CHARACTERIZATION OF STRUCTURAL GEOMETRY OF MULTILAYER MXENE

The first structure of ML-MXenes was proposed as a stacked Ti_3C_2 layer at the OH end based on density functional theory (DFT) simulations.^{74,159} Using the X-ray diffraction (XRD) pattern, researchers determined that the empirical C parameter value of the geometrically optimized structure of the fully hydroxylated MXene corresponds to the experimental finding. Therefore, the presence of hydroxyl and fluorine in combination cannot be ruled out. The accordion-like expanded structure normally observed in scanning electron microscopy (SEM)

should not be taken as the only indication of effective etching of Al from MAX phases, and thus selective etching should be verified by both XRD and energy dispersive X-ray analysis (EDX) analyses.^{29,84} Morphology of MXene MAX phase powder represents MXene multilayer powder produced with a lower HF concentration. In this particular case, selective removal of Al can be confirmed by the absence of the MAX phase (e.g., Ti_3AlC_2) peak in the X-ray diffraction pattern.⁵⁵

Mixed surface termination groups, such as $-\text{F}$, $-\text{OH}$, and $-\text{O}$, affect the stability of MXene, which is exacerbated by the possibility of the presence of water molecules in the interlayer space between the layers. DFT investigations have revealed the existence of two orientations that are energetically advantageous for T in $\text{Ti}_3\text{C}_2\text{T}_2$. $\text{Ti}_3\text{C}_2\text{T}_2$ undergoes two different configurations (Figure 3). A hybrid arrangement (configuration III, Figure 3d) of configuration I and configuration II, as opposed to each other, is frequently explored in situations when one or both of the configurations are incompatible.^{159,160} To determine the structural stability of distinct $\text{Ti}_3\text{C}_2\text{F}_2$ and $\text{Ti}_3\text{C}_3(\text{OH})_2$ configurations, relative DFT simulations are used to compare the results as total energy diminishes in the order of $\text{I} > \text{III} > \text{II}$. Therefore, both the $-\text{F}$ and $-\text{OH}$ groups are more likely to use configuration I than the other groups. The steric repulsion between T groups and C atoms causes the lowest structural instability in configuration II, which is the most stable configuration. During geometry optimization, another possible configuration in which T terminations are bonded just above the Ti(1) atoms of Ti_3C_2 is unstable and regularly switches from configuration I to configuration III. The stacking of the layers, as well as the intermediate materials between them, has a considerable impact on the framework's energy efficiency.^{29,159}

The molecular level combination of compatible materials into a hybrid enhances physical, mechanical, chemical, and electrochemical properties.¹⁶¹ Additionally, molecular-level interac-

tions/interfaces, morphologies, and shapes can be changed by tuning the chemical compositions. The intercalation of polymers/organic molecules/metal oxide/2D materials (e.g., graphene) between multilayer MXene is end-group dependent as demonstrated by XRD patterns of different hybrids.⁶² Furthermore, self-assembly of the hybrid MXenes is possible due to the electrostatic interaction between the positively charged side chain functional groups of the polymer/organic compound and the negatively charged layered MXene sheets.^{55,62}

Electrical Properties. MXenes have good electronic properties, thanks to the availability of narrow band gaps and demonstrate excellent conductivity resulting in fast charge transfer.^{90,162,163} Generally, MXene has a conductivity ranging from 1 to a few thousand S cm^{-1} .¹⁶⁴ For instance, the electrical conductivity of $\text{Ti}_3\text{C}_2\text{T}_x$ can exceed 2000 S cm^{-1} .¹⁶⁵ The multilayered $\text{Ti}_3\text{C}_2\text{T}_x$ increases the contact between electrode and electrolyte and increases the electron transfer rate due to Ti–C bare layers that enhance the interfacial interaction between the electrolyte and electrode and result in better electrochemical energy storage.¹³⁹ In lithium-ion batteries (LIBs), the multilayered $\text{Nb}_4\text{C}_3\text{T}_x$ showed a better capacity and cycling performance resulting from large interlayer spacing after etching.¹⁶⁶ Large interlayer spacing and specific surface area can be obtained by exfoliating multilayered MXene to a few layers or a single-layer structure.²⁹

Moreover, MXenes showed metallic properties with a high density of state when the surface was not terminated. The outer surface of the transition metal plays an essential role in the electronic behavior of MXenes than the inner surface.¹⁶⁷ Replacing the two exterior transition metals of $\text{Ti}_3\text{C}_2\text{T}_x$ with $\text{Mo}(\text{Mo}_2\text{Ti})\text{C}_2\text{T}_x$, electronic properties changed from metallic to a semiconductor.¹⁶⁸ Resistance of the $\text{Mo}_x\text{V}_{4-x}\text{C}_3$ increased with the decrease in the Mo content; however, the resistivity showed a significant change with the rise in temperature.¹⁶⁹ Electrochemical impedance spectroscopy of pristine Ti_3C_2 , alkaliized Ti_3C_2 , and sulfur decorated Ti_3C_2 MXenes showed that the consumption of the sulfur group had a better electronic contact upon sodiation/desodiation. The merged resistance of sulfur decorated MXene was simulated and the lowest value of 202.4Ω was reported.¹⁷⁰

Mechanical Properties. MXenes have high mechanical strength, excellent flexibility, dimensional stability, and flame retardancy.^{134,164,171,172} As in the electrical properties, surface termination processes also alter the mechanical properties of MXenes.¹⁶⁷ The matrix interface bonding is improved by the functional groups, which results in improved properties of bonding and electrical conductivity in polymer composites.¹⁷² The integration of MXene to polymer matrix or epoxy-based composites improves the mechanical properties.^{164,173} For MCF (MXene coated cotton fabric), the addition of $\text{Ti}_3\text{C}_2\text{T}_x$ increased the cell density and improved the mechanical properties like Young's modulus and hardness of fiber due to the cross-linking. This also enhanced the EMI shielding performance of MCF.¹³⁴ It is reported that some mechanical properties like toughness, flexural strength, and hardness are increased by 300%, 150%, and 300% respectively in the 2 wt % composite of $\text{Ti}_3\text{C}_2\text{T}_x/\text{Al}_2\text{O}_3$.¹⁷⁴ For a 5 vol % composite of $\text{Ti}_3\text{C}_2\text{T}_x/\text{Cu}$, the tensile strength increases by 40%.¹⁷² PVC has a tensile strength of 20.8 MPa, but in PVC/MXene composites it is found that it increases by 21.73%, 51.53%, 156.73%, 173.55%, and 174.08% with the addition of 2, 4, 8, 10, 15 wt % of MXene, respectively. The same thing happens for Young's Modulus,

which increases by 1.64, 18.3, 173.62, 177.47, and 177.19%.¹⁷⁵ The TPU (thermoplastic polyurethane)/MXene shows improvement in tensile strength and elongation at break with a 0.5 wt % composition of MXene. The elongation at break increases by 15.4% and tensile strength by 41.2%.¹⁷⁶ Another study shows that an MXene/CNF composite indicates a nacre-like structure, which shows a variation in the mechanical properties with a change in the content of d- $\text{Ti}_3\text{C}_2\text{T}_x$. However, for an optimal value of 50 wt %, $135.4 \pm 6.9 \text{ MPa}$, $16.7 \pm 0.7\%$, $14.8 \pm 0.4 \text{ MJ/m}^3$, and $3.8 \pm 0.3 \text{ GPa}$ are found as tensile strength, fracture strain, toughness, and Young's Modulus, respectively. The composite of $40 \times 15 \text{ mm}$ dimensions can withstand a weight of $\sim 500 \text{ g}$ without breaking.¹⁷⁷ The MXene composite with PSZ (polysilazane) and ABN (aggregated boron nitride) shows another improvement in tensile strength. Without the presence of MXene in the composite MXene-PSZ, the tensile strength is 4.22 MPa while with MXene that number increases to 6.20 MPa, an increase of 147%. MXene-PSZ-ABN/PVA shows a 283% increase to 11.96 MPa.¹⁷⁸ However, filler material content higher than the threshold value may degrade the mechanical properties and cause processing issues.

Optical Properties. MXenes show a plasmonic resonance suitable for laser science.¹⁷⁹ The capability of demonstrating second harmonic generation, Kerr effect, optical rectification, and saturable absorption are essential for compact, high-power, and ultrafast femtosecond pulsed lasers.¹⁸⁰ The formation of a hybrid surface oxide layer with $\text{Ti}_4\text{N}_3\text{T}_x$ MXene showed visible light absorption at energies greater than 2.0 eV.¹⁸¹ The $\text{Ti}_3\text{C}_2\text{T}_x$ demonstrated 77% of transmittance with visible light at 550 nm and increased to 90% when intercalated with NH_4HF_2 . In contrast, the transmittance value for the thin film of the Ti_3AlC_2 MAX phase is 30%. The absorbance showed a linear relationship with the thickness of the intercalated film of $\text{Ti}_3\text{C}_2\text{T}_x$. The imaginary part of the dielectric function tensor or function of the photon wavelength is used to evaluate absorption, reflection, and transmittance. The plasmonic energy is estimated to be 10, 11.63, 10.81 and 11.38 eV for Ti_2C , Ti_2N , Ti_3C_2 and Ti_3N_2 , respectively, using the reflectivity and energy loss curve.¹⁸² 2D $\text{Ti}_3\text{C}_2\text{T}_x$ shows a superior electromagnetic wave absorption capacity, making it ideal for light to heat conversion devices. The localized surface plasmon resonance effect renders ultrathin $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets with higher adsorption and conversion efficiency of near-infrared laser irradiation.¹⁴⁹

Magnetic Properties. Spin-polarized density functional calculations showed the nonmagnetic behavior of MXene at the ground state. This phenomenon originated from the strong covalent bond between the metal, the "X" element, and the attached group. The previous transition metals in MXenes have a lower number of valence electrons that resulted in limited unpaired electron spins and are the key reason for the nonmagnetic properties of such MXene.¹⁸³ External strain alters the covalency of the bonds, which showed a release in the "d-orbital" electron. As a result, magnetism can appear even in the absence of the magnetic system.¹⁸²

The magnetic behavior of MXenes depends on the d-orbital of the transition metal atom. There are bonding σ and antibonding σ^* states in the M–X, and M–T bonds. Under oxidation, the antibonding conditions remain unfilled. So, the only electron in the nonbonding d-orbitals remains responsible for the magnetic behavior.¹⁸⁴ The pristine monolayer Ti_2X (C, N) (like Ti_2C , Ti_2N) showed a magnetic behavior due to the third electrons present on the surface of the Ti atom.^{185–187} Cr_2C is the first in the MXene family to have half-metallic

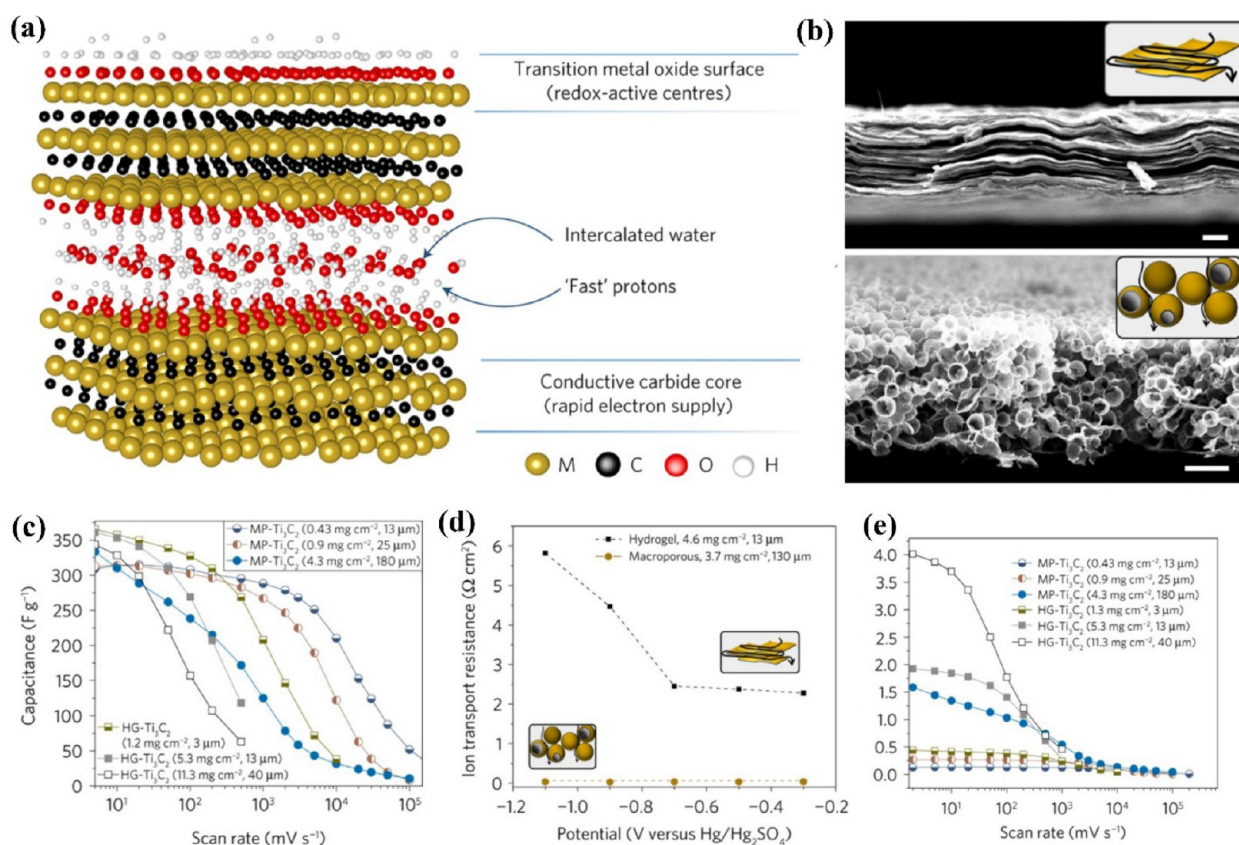


Figure 4. (a) Illustration of MXene structure. (b) SEM image of a cross-section of Ti₃C₂T_x ML-MXene and microporous templated Ti₃C₂T_x electrode (scale 5 μm). (c, d, and e) Rate performance of different MXene films and mass loadings represented in gravimetric and areal capacitance between the hydrogel and microporous MXene. (Reproduced with permission from ref 203. Copyright 2017, Springer Nature.)

behavior for its metallic spin-up channel and an insulating spin-down channel in the structure.¹⁸⁵ Other research indicated that functional groups inhibit the ferromagnetic properties of MXene.¹⁸⁸ Wang et al. stated that if the tensile strain is increased regularly, the magnetic moment increases, and the transition in ferromagnetism from nonmagnetism is observed.¹⁸⁹ Yue et al. found that the magnetic moments of Fe₂C are a total of (3.95 μ_B). This moment comes from the Fe site, which is 1.96 μ_B, and the adjacent C atoms are polarized antiferromagnetically with a moment of -0.10 μ_B.¹⁹⁰ For the Mn centered double transition MXene, it was found that the moment value is up to 4 μ_B per unit cell and the Curie temperature is 1133 K for single-metal MXene.^{191,192} As mentioned earlier, surface termination also dictates the magnetic behavior. Lv et al. have predicted that unterminated Cr₂C is a ferromagnetic half-metal, and both properties are lost upon termination.¹⁹³ This same behavior was observed for Cr₇FeC₄. Before termination, Cr₇FeC₄ is metallic and after termination with O, the resultant Cr₇FeC₄X₈ becomes half metal. On the other hand, doping with Fe, the bandgap of Cr₈C₄F₈ decreased but remained as a bipolar antiferromagnetic semiconductor.¹⁹⁴ In other cases, it was found that a transition metal having more valence electrons can increase the magnetic moment. Other research also found that Fe₂C shows more robust ferromagnetic ordering than Ti₂C, where Fe has more valence electrons than Ti.¹⁹⁰ Bae et al. performed PBE+U, SCAN, and HSE06, and they found V₂CO₂, V₂CF₂, and Mo₂CF₂ to be magnetic.¹⁹⁵ Research on Zr₂N showed a max of 1626 μeV per cell magnetic anisotropy energy if the strain value is 2%. The value is 203 μeV for Zr

atoms, which is greater than Co(65 μeV) in bulk. This indicates that the Zr₂N has a greater magnetic behavior.¹⁹⁶

Supercapacitor. Electrochemical energy storage devices, such as the supercapacitor (SC), have gained attraction with the global energy demand for hybrid vehicles and flexible electronics. SCs store energy on the electrode surface through electrosorption of charge carrier ions.^{197,198} 2D materials have an atomic thickness, ample active surface sites, and excellent mechanical properties ideal for the electrode material of SCs.^{157,197} MXenes have shown superior super capacitance, thanks to the outstanding conductivity, large specific surface area, and available redox sites.¹⁵⁷ MXene multilayer and hybrid multilayer capacitance have been explored using the electroactive redox pseudocapacitance characteristics, embedding ability, and conductivity characteristics to improve energy storage capacity.¹⁹⁹ Because of the fast redox (pseudocapacitive) surface storage process, devices are capable of storing more energy than electrical double-layer capacitors (EDLCs). Furthermore, the high capacitance of ML-MXene electrode materials leads to an increase in the energy density and cyclic ability of SCs. Because of its high electrical and thermal conductivity, Ti₃C₂T_x is predicted to be a suitable choice for solid-state SCs according to theoretical projections.^{152,199,200} The thin-film electrodes used in conventional SCs are typically sandwich structures, and the in-plane architecture will open the door to further downsizing.²⁰¹ The in-plane configuration of the electrode array offers significant advantages over traditional sandwich structures and fiber-based structures. Accessibility to the edges of the electrodes to the electrolyte results in ultrahigh power density superior to that of batteries and traditional

Table 3. Progress in Multilayer MXene and Their Heterostructures-Based SC with Related Parameters

ML-MXene and ML-hybrid MXene	electrode type	electrolyte	rate	capacity retention	cycle number	volumetric capacitance	ref
Ti ₃ C ₂ T _x	macroporous electrode	3 M H ₂ SO ₄	10 V s ⁻¹	210 F g ⁻¹	10000	1500 F cm ⁻³	203
V ₂ C	sheet-like free-standing electrode	1 M Na ₂ SO ₄	2 mV s ⁻¹	164 F g ⁻¹	10000	—	205
V ₂ CT _x	film electrode	seawater	2 A g ⁻¹	181.1 F g ⁻¹	5000	317.8 F cm ⁻³	204
Ti ₃ C ₂ T _x -G	hybrid films-like electrode	3 M H ₂ SO ₄	2 mV s ⁻¹	—	20000	1040 F cm ⁻³	119
Ti ₃ C ₂ T _x -MPFs	hybrid films-like electrode	3 M H ₂ SO ₄	1 mA cm ⁻³	—	30000	694.2 F cm ⁻³	132
TiO ₂ /C-Ti ₃ C ₂ T _x -MXene/ NiO	free-standing electrode	1 M KOH	1 A g ⁻¹	92.0 mA h cm ⁻³	5000/58.6%	—	93
Ti ₃ C ₂ /CNTs	hybrid films-like electrode	6 M KOH	1 A g ⁻¹	134 F g ⁻¹	10000	—	108
Ti ₃ C ₂ T _x -MgO (expanded MXene)	sheet-like free-standing electrode	1 M KOH	1 A g ⁻¹	180 F g ⁻¹	8000/87%	203 F g ⁻¹	208
Co ₃ O ₄ -MXene/rGO	hybrid films-like electrode	6 M KOH	1 A g ⁻¹	345 F g ⁻¹	10000/90%	—	209
d-Ti ₃ C ₂ T _x	binder-free film electrodes	1 M KOH	1.5 A g ⁻¹	140 F g ⁻¹	10000	—	210
Nb ₂ CT _x /CNT	hybrid films-like electrode	1 M H ₂ SO ₄	5 A g ⁻¹	—	5000/80.3%	—	211
Ti ₃ C ₂ T _x /BC composites	porous electrodes	1 M H ₂ SO ₄	30 mA cm ⁻²	416 F g ⁻¹	10000/ 96.5%	2084 mF cm ⁻²	212
Ti ₄ C ₃	free-standing electrode	0.1 M H ₂ SO ₄	1 V s ⁻¹	—	2000/89%	520 F cm ⁻³	213
Ti ₃ C ₂	three-electrode system	1 M H ₂ SO ₄	1 A g ⁻¹	517 Fg ⁻¹	10000/99%	550 F g ⁻¹	157
Ti ₂ CT _x	porous electrodes	30 wt % KOH	1 A g ⁻¹	51 F g ⁻¹	6000/86%	—	92
Ti ₃ C ₂ T _x	three-electrode system	3 M H ₂ SO ₄	0.5 A g ⁻¹	351 F g ⁻¹	10000/ 90.5%	1142 F cm ⁻³	152
PPy/Ti ₃ C ₂	polymer-based free-standing flexible electrodes	0.5 M H ₂ SO ₄	10 mA cm ⁻²	126 F g ⁻¹	20000/70%	406 F cm ⁻³	123
p-C ₃ N ₄ / Ti ₃ CT _x	hybrid films-like electrode	6 M KOH	1 A g ⁻¹	327 F g ⁻¹	5000/96.2%	—	214
Ti ₂ CT _x -GO	hybrid films-like electrode	1 M H ₂ SO ₄	1.5 A g ⁻¹	—	3000/90%	586.4 F cm ⁻³	117
TCBOC/Ti ₃ C ₂ T _x	three-electrode system	1 M H ₂ SO ₄	1 A g ⁻¹	—	5000/85%	396.5 F cm ⁻³	215
NiO/C-Ti ₃ C ₂ T _x	porous electrodes	1 M KOH	1 A g ⁻¹	51 F g ⁻¹	5000/72.1%	60 F cm ⁻³	93

SCs.^{201,202} Lukatskaya et al.²⁰³ reported that the proper design of electrodes could bring MXene capacitance closer to its theoretical limit. For example, macroporous electrode structures allow outstanding capacitance retention even at charge–discharge rates above 1 V s⁻¹, i.e., 210 F g⁻¹ at 10 V s⁻¹, better than hydrogel-based electrodes (Figure 4).²⁰³

Apart from electrode design, different etching processes also affect the specific capacity by forming different functional groups on the MXene surface. Superior electrochemical performance of V₂CT_x (etching Al layers from the V₂AlC MAX phase using NaF +HCl at 90 °C for 72 h) has made this preferable as an SC electrode to other MXenes and used as an SC electrode in adequate seawater electrolytes. V₂CT_x MXene is thinner than Ti₃C₂T_x, resulting in higher ion diffusion speeds.²⁰⁴ For example, an etching process with a combination of lithium fluoride (LF) and HCl acid was used to successfully synthesize high-purity V₂C.²⁰⁵ The uniform multilayer structure of V₂C showed a higher capacity after 10 000 cycles, good cycle stability, and a 90% specific capacity retention rate. However, V₂C's capacitance gradually decreased with increasing scan rates because of incomplete penetration of the electrolyte within a short time. The combination of the 2D conductive carbide layer and hydrophilic hydroxyl-terminated surface facilitated excess capacitance of 300 F cm⁻³, higher than that of porous carbon. This higher capacitance of MXene is further enhanced by the intercalation of various cations like Na⁺, K⁺, NH₄⁺, Mg²⁺, and Al³⁺.⁷⁵

However, pure multilayer MXene is easy to collapse and pile up, affecting the performance and stability of electrode material for SCs. Intercalation, doping, or composite of MXene improve the electrochemical performance of SCs.^{93,206} Two methods have been discussed to avoid the restacking of Ti₃C₂T_x-MXene layers: a straightforward hard templating technique and a pore-

forming approach. Furthermore, to overcome the limitations of the SC, different modifications and hybridizations have been performed along with the design as listed in Table 3.

A multilayer MXene-based hybrid electrode for SC was prepared by the electrophoretic deposition (EPD) method. The EPD method produces a strong binder-free Ti₃C₂/CNTs composite film, preventing the restacking of MXene nanosheets and improving electrochemical performance.¹⁰⁸ For SCs, negatively charged MXene nanosheets and charged reduced graphene oxide (rGO) nanosheets were used for electrostatic self-assembly to fabricate a highly conductive film SC.¹¹⁹ The hybrid structure as self-restacking of MXene multilayers increased interlayer spacing and facilitated the rapid diffusion and transport of electrolyte ions. The fabricated SC demonstrated superior performance with an ultrahigh volumetric energy density of 32.6 Wh L⁻¹. The symmetric SC also exhibited a maximum volumetric power density of up to 74.4 kW L⁻¹.^{119,207} As flexible free-standing electrodes, hydrogen-bonded MXene, Ti₃C₂T_x, and 2D metal-porphyrin frameworks (MPFs) hybrid films were proposed to overcome the low conductivity and low structural stability of 2D MPFs. The synergistic effect of MXene (Ti₃C₂T_x) and MPF resulted in larger interlayer spacing, “MPF to MXene to MPF” interconnected conductive network, and complete utilization of active sites. This facilitated the rapid migration of ions and electrons and reduced the electrolyte migration path of ions (Figure 5a–c).¹³²

Metal oxide-based Ti₃C₂T_x MXene showed higher conductivity and a larger specific surface area, which are of great importance and considered the main factors in improving SC efficiency. By a simple and efficient hydrothermal process, NiO nanosheets were effectively formed on the surface of Ti₃C₂T_x MXene in a highly hierarchical porous honeycomb-like structure

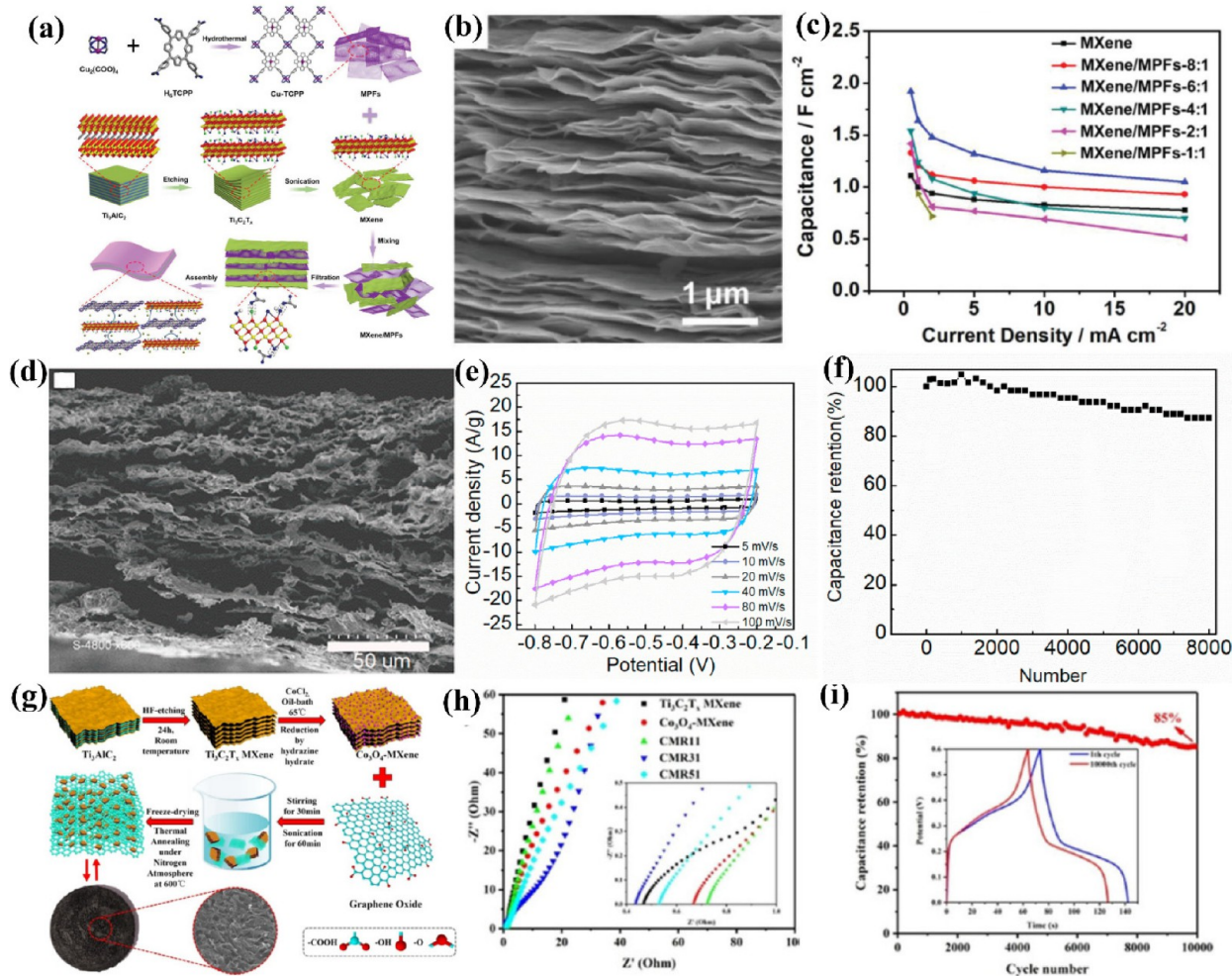


Figure 5. (a) Synthesis of interlayer hydrogen-bonded MXene/MPFs films is illustrated in this diagram. (b) SEM images (cross-section) of MXene/MPFs film. (c) MXene and MXene/MPFs areal capacitances in various ratios. (Reproduced with permission from ref 132. Copyright 2019, WILEY-VCH Verlag GmbH & Co. KGaA.) (d) SEM image shows crumpled layer-like morphology MgO-expanded MXene. (e) CV curves for different scan rates. (f) Cyclic capacity for 8000 charge–discharge cycles at current rate 5 A g^{-1} . (Reproduced with permission from ref 208. Copyright 2020, Elsevier Ltd.) (g) Step-by-step preparation of the Co_3O_4 -MXene/rGO hybrid porous material is depicted in this diagram. (h) Nyquist plots of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and Co_3O_4 -MXene and Co_3O_4 -MXene: rGO (CMR) with different percentage ratio electrodes on the EIS. High-frequency range is shown in the inset. (i) CMR31 electrode's cycling stability was tested over 10 000 cycles at 3 A g^{-1} . The CV curves recorded at the 1st and 10 000th cycles are shown in the inset. (Reproduced with permission from ref 209. Copyright 2019, Wiley-VCH Verlag GmbH & Co. KGaA.)

by Xia et al.⁹³ The Ni-dMXNC ($\text{TiO}_2/\text{C}-\text{Ti}_3\text{C}_2\text{T}_x$ -MXene/ NiO) electrode showed a great specific capacity of 60.7 mA h g^{-1} at a high current rate of 1 A g^{-1} , corresponding to 92.0 mAh cm^{-3} volumetric specific capacity and a reasonable rate of retention capacity.⁹³ The open morphology of the expandable MXene multilayer, which was created utilizing metal oxide (MgO) nanoparticles as rigid templates, was based on crumpled layers (Figure 5d–f). As the volume of active expanded electrode materials was considered, the volumetric energy density reached 10 W h L^{-1} . The device capacitance gradually increased to more than 110% at 25 F g^{-1} during the first 5000 charge–discharge cycles.²⁰⁸

A significant amount of MXene research has been reported developing alternative or inexpensive electrode materials with strong capacitances. Co_3O_4 is considered one of the best alternative materials due to its low cost, high theoretical capacitance, and environmental sustainability. A hybrid multilayer porous MXene aerogel was prepared by Liu et al.²⁰⁹ as Co_3O_4 -MXene/rGO as an SC electrode. The higher specific

capacitance of 345 F g^{-1} was achieved at 1 A g^{-1} , which is notably higher than that of the electrodes $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, rGO, and MXene/rGO (Figure 5g–i).²⁰⁹ Figure 5 also shows how metal oxide, MPF, Co_3O_4 , and graphene-based hybrid composite significantly improve the capacitance. MXenes are predominantly studied in aqueous electrolytes and are outperformed by the capacitance of most carbon-based and pseudocapacitive materials. However, X-ray diffraction/adsorption, electrochemical quartz crystal microbalance in gravimetric and dissipative modes, and nuclear magnetic resonance were used to examine the charge storage behavior of multilayer MXene electrodes in aqueous electrolytes. They are often combined with molecular dynamics (MD) and DFT modeling to validate the obtained results. However, further research is required for a better understanding of the charge storage mechanism for SCs. Table 3 lists the capacitance and related parameters for multilayer MXene and their heterostructures.

Batteries. It is particularly favorable to use 2D MXenes and their heterostructures in rechargeable batteries because of their

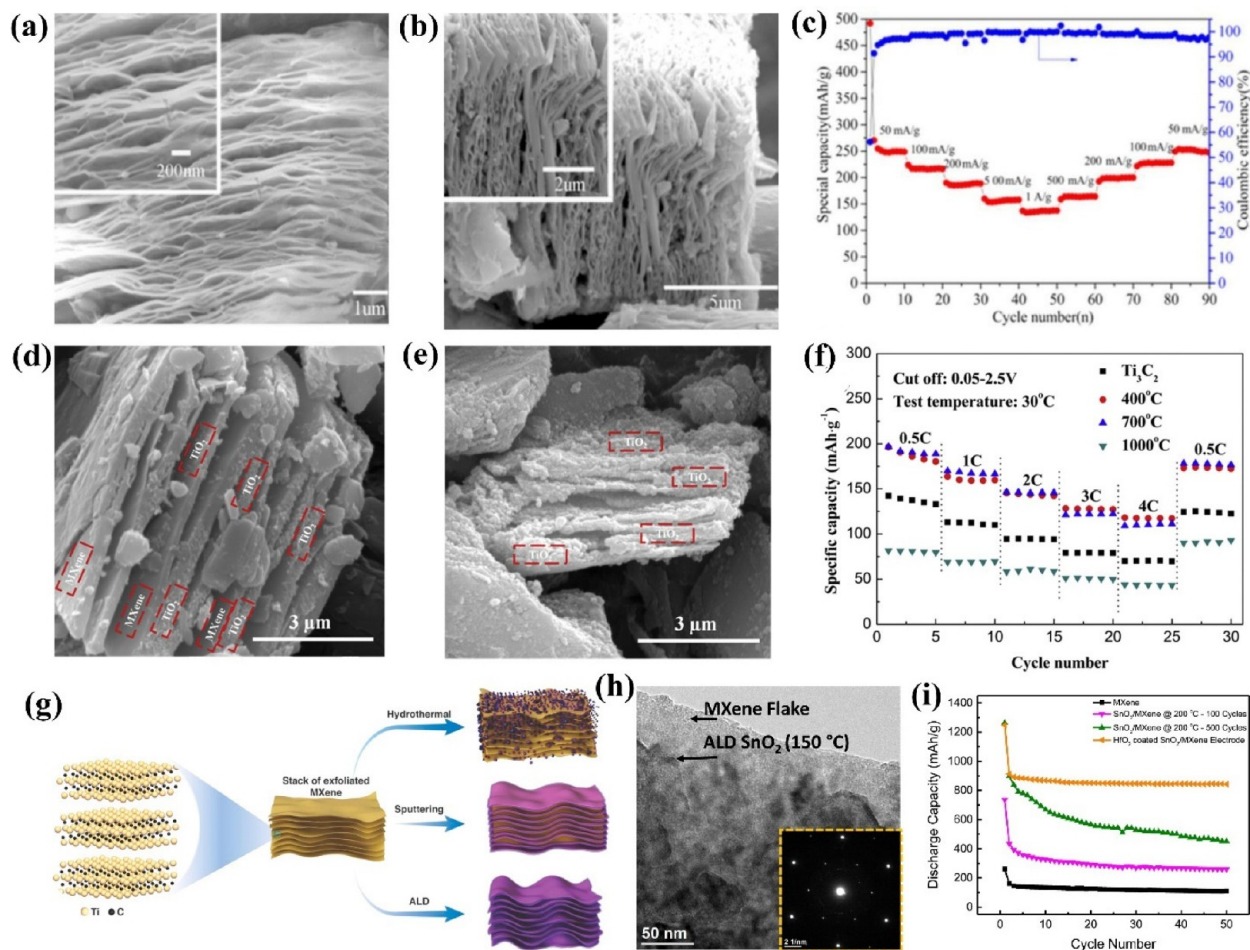


Figure 6. (a and b) SEM images of exfoliated V_2AlC . (c) Specific capacity of V_2C . (Reproduced with permission from ref 78. Copyright 2017, The Electrochemical Society.) (d and e) Ti_3C_2 surface morphologies before and after vacuum calcination when heated in air at different temperatures. (f) Rate capacity at various current rates. (Reproduced with permission from ref 85. Copyright 2018, Elsevier Ltd.) (g) Different methods for depositing SnO_2 on Ti_3C_2 MXene sheets. Exfoliated MXene sheets are shown on the left, and MXene hybrid electrodes after SnO_2 deposition is on the right. An exfoliated MXene sheet stack is presented in the center. (h) TEM image of MXene sheets coated with a 50 nm-thick layer of SnO_2 . (i) Discharge capacity over 50 cycles at 500 mA g^{-1} HfO_2 coated $SnO_2/MXene$ electrodes. (Reproduced with permission from ref 227. Copyright 2017, Elsevier Ltd.)

high electrical conductivity and high hydrophilicity, which are both beneficial for improved efficiency. The high capacity for various cations in the interlayer of MXene contributes to the overall efficiency of the battery system. Reversible energy storage in rechargeable batteries is essentially dependent on the insertion and extraction of alkali metal ions, such as Li^+ , K^+ , Na^+ , and Mg^{2+} , in and out of the battery.^{216,217} The most frequently used LIB uses graphite anodes as the commercial energy storage. However, the limited capacity restricts the graphite anodes and may fail to meet the ever-growing need for energy storage in the future. However, metallic or narrow bandgap semiconductor characteristics of ML-MXene and their derivatives are highly advantageous for LIB applications.^{144,218,219} Naguib et al.⁴⁹ evaluated the viability of Ti_3C_2 MXene for LIBs as anode materials and later widely investigated both experimentally and computationally as LIB anodes and cathodes.^{220,221} The adsorption and migration of Li ions on the exposed F- and OH-terminated Ti_3C_2 MXene surface were validated. Furthermore, Ti_3C_2 MXene is a potential candidate for LIB anodes where low operating voltage and diffusion barriers are required.²¹⁷ The electrochemical behaviors, Li^+ uptake potential of Ti_3C_2 , and intercalation with dimethyl

sulfoxide (DMSO) showed the charge and discharge capacities of 264.5 and 123.6 mA h g^{-1} , respectively, with a Coulombic efficiency of 47%.²²⁰ The superior initial irreversible capacity of Ti_3C_2 contributes to the formation of the electrode surface SEI (solid electrolyte interphase) film and the irreversible reduction of electrochemically active surface groups. DMSO intercalation expands the d -spacing of MXene, and increases the storage capacity of Li ions.²²⁰ A high degree of conversion was achieved (>90 wt %) for V_2C MXene prepared using the quasi-2D structure V_2AlC powders with NaF and HCl, etching at 90°C . This can be attributed to prolonged cycling, especially at high charge/discharge rates, which expose increased electrochemically active sites (Figure 6a–c).⁷⁸ Li diffusion is expected to occur in ML-MXenes through the [1000] and [0100] crystalline directions. Using DFT simulations, the Li storage potential of functionalized MXenes like Sc_2C ,²²² Ti_2C ,²²³ Ti_3C_2 ,²²⁰ V_2C ,⁷⁸ Cr_2C ,²²⁴ and Nb_2C ²²⁵ were examined.²²⁶ Among those MXenes, the reversible capacity is the largest for V_2CO_2 , and the highest diffusion barrier for MXene, indicating that it may be ideal for lightweight and high-capacity batteries.¹⁶⁸

While laminated MXenes have high electronic conductivity, MXene electrodes as a hybrid composite with carbon

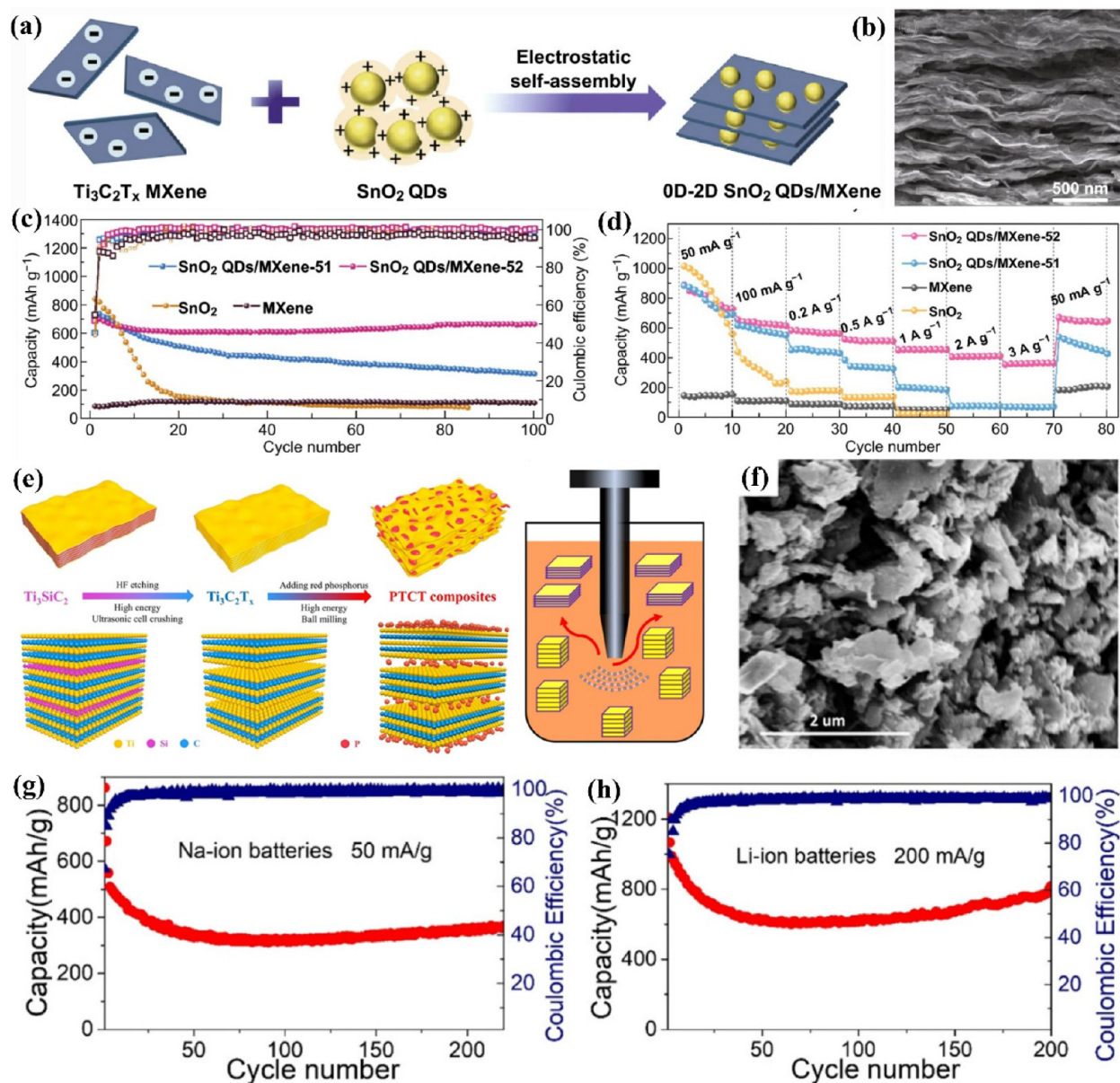


Figure 7. (a) Schematic illustration and (b) morphology for the prepared 0D-2D QDs SnO_2 /MXene hybrids. (c) Cycle stability and (d) rate performance of LIB electrodes of all specimens. (Reproduced with permission from ref 162. Copyright 2019, The Authors.) (e) Fabrication of PTCT composite materials. (f) SEM image of PECT-MXene hybrid composite. (g) PTCT composite hybrid cycling performance of sodium-ion battery at current rate 50 mA g^{-1} . (h) 200 mA g^{-1} cycle performance with greater red P concentration for LIBs. (Reprinted (adapted) with permission from ref 230. Copyright 2019, American Chemical Society.)

additives,^{69,118,228} metal oxide,^{227,229} or polymer derivatives²³⁰ demonstrated enhanced conductivity. Researchers have proposed many methods for producing highly conductive MXenes for high-performance applications, including oxidation, doping with metal oxides, and doping with heteroatoms. In the case of LIB anodes, the oxidation of $\text{Ti}_3\text{C}_2\text{T}_x$ in an oxygen-poor environment can result in the formation of TiO_2 (anatase) disordered composite material with excellent electrochemical performance that can be used as a negative electrode material.^{115,231} As an example, a hierarchical composite of 2D niobium carbide MXene (Nb_2CT_x) in CO_2 synthesized via one-step controllable partial oxidation enhanced electrochemical performance. The oxidized Nb_2CT_x charge storage extends 198 and 152 C g^{-1} at 5 and 20 mV s^{-1} , respectively.⁶⁹ A systematic study on thermal stability in different atmospheres showed that

vacuum calcination improves the electrochemical properties of MXene Ti_3C_2 multilayer for Li-ion batteries.⁸⁵ Ti_3C_2 powders, after vacuum calcination, showed much higher capacitance than initial Ti_3C_2 as LIBs anode. After heating at 400 and 700 $^\circ\text{C}$ for 100 cycles at a current rate of 1 C, the primary capacitance of 126.4 and 147.4 mA h g^{-1} rose by 45 and 69%, respectively. It exhibited outstanding rate capability due to changes in surface chemistry of TiO_2 (Figure 6d–f).⁸⁵

The utilization of metal oxide in conjunction with ML-MXene hybridization can considerably boost the cycle capacity of the LIB, as previously stated. As an excellent chemical modifier, metal oxides such as tin dioxide (SnO_2) improve the cycling stability of MXene by acting as a catalyst. As a result of its high theoretical potential, SnO_2 is regarded a suitable choice as the anode material in LIBs.²³² It substantially increases the

Table 4. ML-MXene-Based Structures as Electrode Material for Batteries

structures	battery type	electrode	initial capacity (mAh g ⁻¹)	current density	capacity retention (mAh g ⁻¹)	number of cycles	coulombic efficiency	ref
Ti ₃ C ₂ T _x and nonaqueous Na ⁺ electrolyte	Na-ion	anode	270	20 mA g ⁻¹	70	100	95%	240
2D Ti ₃ C ₂	Li-ion	anode	264.5	1 C	118.7	75	47%	220
V ₂ C MXene	Li-ion	anode	467	1 C	291	20	98.6%	78
Ti ₃ C ₂ T _x	Li-ion	anode	426	1 C	180	50	87%	221
f-Ti ₃ C ₂	Li-ion	anode	410	1 C	200	50	—	226
V ₂ CO ₂	Li-ion	anode	335	1 C	276	50	—	218
2D (Ti ₂ CT _x) nanosheets	Li-ion	anode	1015	100 mA g ⁻¹	389	50	—	246
Nb ₂ C	Li-ion	anode	780	0.5 C	420	100	near 100%	247
Ti ₂ CT _x	Na-ion	anode	360	1 C	175	100	65%	248
V ₂ O _x @V ₂ CT _x	Zn-ion	cathode	304	2 mA g ⁻¹	84	200	81.6%	244
SnO ₂ -Ti ₃ C ₂	Li-ion	anode	1030.1	100 mA g ⁻¹	360	200	34.4%	229
Ti ₃ C ₂ X ₂	Na-ion	anode	379	1 A g ⁻¹	80	120	56%	236
PDDA-BP/Ti ₃ C ₂ nanosheet	Na-ion	anode	1112	0.1 A g ⁻¹	658	2000	68.7%	249
red phosphorus Ti ₃ C ₂ T _x (PTCT)	Li-ion	anode	906	50 mA g ⁻¹	505	90	97.9%	230
SnS-Ti ₃ C ₂ T _x	Na-ion	anode	412.8	100 mA g ⁻¹	255.9	50	70.4%	237
CoNiO ₂	Na-ion	anode	463	100 mA g ⁻¹	188.4	140	98.7%	238
sulfur-Ti ₃ C ₂	Na-ion	anode	413	2 A g ⁻¹	135	1000	near 100%	170
Ti ₃ C ₂ T _x @Sb-0.5	Na-ion	anode	353	2 mA g ⁻¹	200	500	98%	239
Ti ₃ C ₂ T _x	Na-ion	anode	110	30 mA g ⁻¹	73	70	100%	241
Ti ₃ C ₂ T _x @C	Mg-ion	cathode	198.7	10 mA g ⁻¹	123.3	400	85%	242
V ₂ C	Li-ion	anode	492	0.2 C	290	50	99.4%	250
MoS ₂ /MXene	K-ion	anode	453.5	50 mA g ⁻¹	145.5	50	66.5%	245
V ₂ CT _x	Al-ion	cathode	335	10 mA g ⁻¹	112	20	90%	243
SnO ₂ @MXene	Li-ion	anode	736	50 mA g ⁻¹	258	50	92%	227
MoS ₂ @Ti ₃ C ₂ T _x	Li-ion	anode	1140	0.05 C	501	500	91.4%	251
SnS ₂ /Sn ₃ S ₄ @Ti ₃ C ₂ MXene	Li-ion	anode	708	100 mA g ⁻¹	426.3	100	37.3%	139

electrochemical efficiency of the composite SnO₂-MXene.^{227,229}

For SnO₂-Ti₃C₂ nanocomposites synthesized by the hydrothermal deposition of SnO₂ nanoparticles on Ti₃C₂, MXene retained almost 50% of the initial capacitance even after 200 cycles, i.e., showed capacitance of 1030 mAh g⁻¹ at 100 mA g⁻¹ and 360 mAh g⁻¹ at 200 cycles.²²⁹ A low-temperature atomic layer deposition (ALD) of metal oxides on MXene provides advantages such as prevention of oxidation and structure over the hydrothermal process.²²⁷ To increase the stability even further, ALD is followed by the deposition of a conformal thin passivation layer of inactive HfO₂ in conjunction with SnO₂-MXene. At 500 mA g⁻¹, the HfO₂ coated SnO₂-MXene electrodes demonstrated a specific capacity of 843 mA h g⁻¹ after 50 cycles of operation (Figure 6g–i).²²⁷

To further improve the capacity retention and cyclic ability of SnO₂-based MXene hybrid, SnO₂ quantum dots (QDs) were self-assembled on Ti₃C₂T_x MXene sheets electrostatically to produce SnO₂/MXene hybrids and have a distinctive 0D-2D structure. Dimensionless SnO₂ QDs (about 4.7 nm) are distributed uniformly over 2D Ti₃C₂T_x ML-MXene sheets with a controlled loading percentage. The hybrid of SnO₂ and QDs/MXene exhibited excellent high-density lithium storage of 887.4 mAh g⁻¹ at 50 mA g⁻¹. Besides, the lithium storage capacity after 100 cycles was 659.8 mAh g⁻¹ at 100 mA g⁻¹ with 90% capacitance retention. The special 0D-2D geometry provides large electrochemically active sites with high specific capacitance to augment the electrochemical efficiency of electrode materials (Figure 7a–d).¹⁶²

One of the most prevalent elements on the planet is silicon dioxide (SiO₂), which is a cost-effective and environmentally friendly option as an energy source. Although SiO₂ remains inactive against Li, it can be engineered to react directly via a

simple process.²³³ Mu et al.²³⁴ successfully prepared a microspherical SiO₂/MXene hybrid material composed of a layered MXene. The Stober and spray drying methods were combined for the first time, and the SiO₂ nanoparticles were securely anchored by bonding. Bonding between the SiO₂ nanoparticles and the MXene matrix enhanced structural stability throughout long-term cycling. In addition, the laminated MXene matrix has greater elasticity to reduce the volume change of SiO₂ nanoparticles and facilitate the transmission of electrons and (Li⁺) lithium ions. Consequently, with a high reversible power of 838 mA h g⁻¹ at 100 mA g⁻¹, the SiO₂/MXene anode offers superior electrochemical efficiency with excellent cycle stability with a capacity retention of 97% over 100 cycles.²³⁴

Fe₂O₃ nanoparticles are suitable anode materials for batteries because of their high cyclic rate performance. A series of Fe₃O₄@Ti₃C₂ hybrids were synthesized by the ultrasonic mixture of Ti₃C₂ MXene and Fe₃O₄ nanoparticles.¹⁰⁹ Ti₃C₂ ML-MXene can hold a certain amount of Fe₃O₄ nanoparticles as a carrier and the Fe₃O₄@Ti₃C₂ hybrids showed superior electrochemical efficiency than pure Fe₃O₄ or Ti₃C₂. The Fe₃O₄@Ti₃C₂ hybrid prepared as anode material with a weight ratio of 2:5 indicates a significant reversible power of 747.4 mAh g⁻¹ at a current rate of 1 C after 1000 cycles. The Fe₃O₄@Ti₃C₂ electrode showed a much lower charge transfer resistance and a better cyclic ability for LIBs.¹⁰⁹ Furthermore, a lightweight, freestanding and binder-free silicon/MXene composite paper has been directly used as an anode for LIBs.¹¹¹ This unique architecture showed adaptability to large volume expansion, enhanced the conductivity of composite materials, prevented MXene sheets from restacking, provided additional active sites, and promoted effective ion migration. The device exhibited

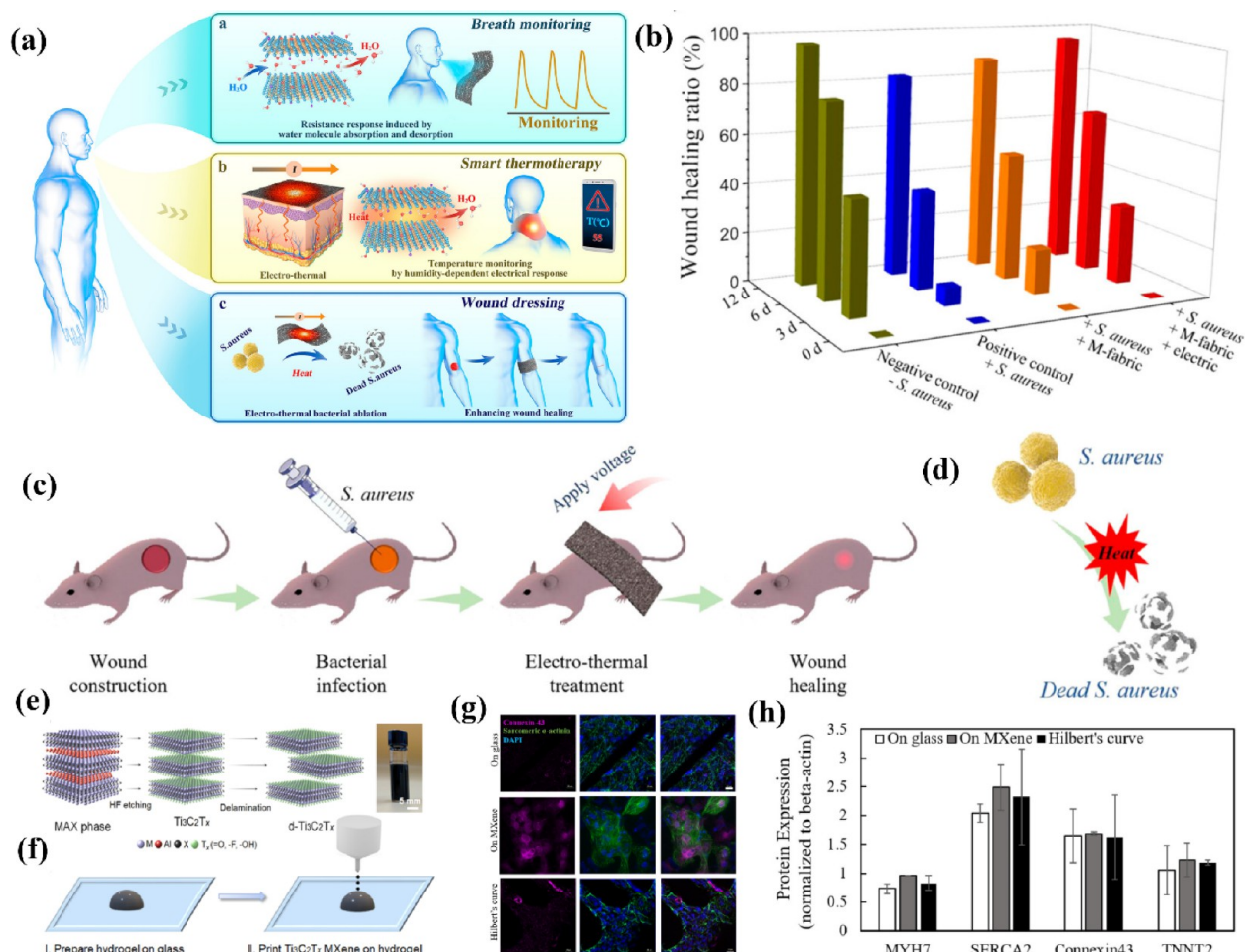


Figure 8. (a) Schematic diagram showing potential applications of MXenes in mobile healthcare and medical therapy fields. (b) Wound healing/tissue regeneration percentage in various recovery timeframes with different methods of treatment. (c) Representation for the development of a bacteria-infected model together with Joule therapy with the use of MXene. (d) Bacterial killing by resulting heat is depicted schematically. (Reproduced with permission from ref 266. Copyright 2020, American Chemical Society.) (e) Schematic diagram of selective HF etching of layers of Ti_3AlC_2 MAX phases to prepare $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. (f) Schematic representation of printing $\text{Ti}_3\text{C}_2\text{T}_x$ MXene-hydrogel composite. (g) On day 7, immunostaining and qRT-PCR examination of iCMs were performed. (h) Western blotting quantitative analysis. (Reproduced with permission from ref 267. Copyright 2020, Elsevier Ltd.)

excellent performance after 100 cycles at a high capacity of 2118 mAh g^{-1} with a 200 mA g^{-1} current density.¹¹¹

A successful method for investigating more efficient anode materials for LIBs is combining transitional metal oxides with MXene. The high specific surface area of the nanosized $\text{SnS}_2/\text{Sn}_3\text{S}_4$ hybrid also improves the interaction between electrolytes and electrodes. The hybrid confines in the multilayer stacks serve as a spacer to reduce the restacking layer propensity and enhance the limited capacitance of Ti_3C_2 . After 100 cycles, the $\text{SnS}_2/\text{Sn}_3\text{S}_4$ -MXene hybrid shows strong cycling stability of $462.3 \text{ mA h g}^{-1}$ at 100 mA g^{-1} and a superior rate efficiency of $216.5 \text{ mA h g}^{-1}$ at 5000 mA g^{-1} .¹³⁹ Apart from LIB, lithium-sulfur (Li-S) and lithium-oxygen (Li-O₂) batteries have also drawn great attention due to their energy intensity. 3D metal carbide MXene/rGO hybrid layered nanosheets were examined as the cathode host material for Li-S batteries.¹¹⁸ This composite cathode achieved a good initial capacity of $1144.2 \text{ mAh g}^{-1}$ and a high capacity retention level of $878.4 \text{ mA h g}^{-1}$ after 300 cycles. The unique MXene and rGO nanosheets 3D layer formation with functional 2D surfaces are more effective at trapping sulfur and lithium polysulfides.¹¹⁸ The Ni- $\text{Ti}_3\text{C}_2\text{T}_x$ hybrid produced via a simple hydrothermal and high-temper-

ature calcination method was recently introduced as a cathode for the Li-O₂ battery. Because of their unique catalytic activity, Ni nanoparticles with a diameter of 20 nm can accelerate the decomposition of Li_2O_2 . Utilizing the advantages of the two materials, the Ni- $\text{Ti}_3\text{C}_2\text{T}_x$ hybrid battery is used as the positive electrode of the Li-O₂ battery, with a maximum specific capacity of $20264 \text{ mA h g}^{-1}$ at 100 mA g^{-1} and $10699 \text{ mA h g}^{-1}$ at 500 mA g^{-1} for the first cycle.²³⁵

Electricity grid storage solutions on a large scale are necessary to store and use renewable resources efficiently. Because the relative abundance of Li in the Earth's crust is only 20 ppm, it is time to reconsider the feasibility of Li for large-scale electrochemical energy storage.²³⁶ In recent years, Na,^{170,230,237–241} Mg,²⁴² Al,²⁴³ Zn,²⁴⁴ and P²⁴⁵ have demonstrated many advantages over LIBs, such as abundant raw materials, less costly, and less standard potential. With different components, conductivity, hydrophilicity, and adjustable surface functional groups, ML-MXene is a two-dimensional layered substance that has significant potential to improve the quality of Li-, K-, Ca-, Na-, Al-, and Mg-ion batteries and different types of energy storage systems (Table 4). Excellent electrochemical efficiency of ML-MXene materials for all nonlithium energy storage

Table 5. Antibacterial Activity and Tissue Engineering Application ML-MXene and Composites

multilayer hybrid MXene	result	application	ref
Ti ₃ C ₂ T _x /cellulose composite hydrogel with doxorubicin hydrochloride drug	100% tumor cell killing efficiency achieved at 808 nm light illumination.	photothermal treatment	275
Au/MXene and Au/Fe ₃ O ₄ /MXene composites	No teratogenic effect was found for these composites. In addition, the ability to destroy tumor cells increased with an increase in laser power density at 1.5 W cm ⁻² .	photothermal treatment	276
surface modification of Ti ₃ C ₄ /MnO _x composite with soybean phospholipid	A high rate of photothermal conversion and tumor cell ablation activity was achieved.	photothermal treatment	277
Ti ₃ C ₄ MXene and chitosan	The range limit of detection was between 18 nM and 7.8 μM for sarcosine.	biosensor	278
TiO ₂ /Ti ₃ C ₂ T _x /Cu ₂ O	The sensor can detect the glucose level in the blood serum with a range between 100 nM and 10 μM.	biosensor	279
Ti ₃ C ₂ /CuS	Presence of cholesterol in a range of 10–100 μM was detected.	biosensor	280
Ti ₃ C ₂ /Prussian blue (PB)	The sensitivity of MXene/PB was higher in comparison with carbon nanotubes/PB and graphene/PB where the value was 52.3, 40.5, and 29.74 μA mM ⁻¹ , respectively.	biosensor	281
Ti ₃ C ₂ T _x /graphene hybrid film	The open structure of the hybrid film allows glucose oxidase to enter the pores, which helps to control the stability of the film. Therefore, the film exhibits excellent electrochemical activity toward glucose biosensing.	biosensor to detect glucose	282
Aβ ₄₂ imprinted MIP/delaminated Ti ₃ C ₂ T _x /MWCNTs/GCE composite	The sensitivity range was achieved between 1.0 and 100.0 fg mL ⁻¹ and the detection limit (LOD) was 0.3 fg mL ⁻¹ .	biosensor for alzheimer's disease	283
molecularly imprinted polymer (MIP) multiwall carbon nanotube (MWCNT) glassy carbon electrode (GCE)			
by using a hydrothermal technique, a Ti ₃ C ₂ and nickel–cobalt layered double hydroxide (NiCo-LDH) composite was created, which was then coated on a GCE	The sensitivity of this sensor was achieved at 64.75 μA m M ⁻¹ cm ⁻² at a low response time of 3 s with a linear range of 0.002–4.096 mM and a low limit of detection of 0.53 μM.	glucose biosensor	284

systems has been demonstrated. Recently, a new strategy for preparing multilayer Ti₃C₂T_x nanosheets used abundant Ti₃SiC₂ MAX phase by high-energy ultrasonic cell breaking and extraction and a low-concentration etchant to break the Ti–Si bond of the MAX phase. To form a nanodots/Ti₃C₂T_x (PTCT) composite material, a unique P–O–Ti bonded red phosphorus was combined with Ti₃C₂T_x ML-MXenes. When used as an electrode for a LIB or sodium-ion battery (SIB), the PTCT electrode showed excellent cycle stability in 1000 cycles. By increasing the P content, the capacitance of the LIB increases to 818.2 mA h g⁻¹ after 200 cycles. For the SIB, it showed an initial capacity of 863.8 mA h g⁻¹ at a current density of 50 mA g⁻¹. It retains the capacity of 370.2 mAh g⁻¹ after 200 cycles, indicating good sodium storage in MXene-based materials (Figure 7e–h).²³⁰ There are many significant advances for implementing multilayer MXene-based hybrid materials in the field of energy storage. Further studies into the practical applications of MXene-based materials for electrochemical energy storage are still desirable.

Biomedical Applications. The nanostructure and the controllable chemical structure of ML-MXene provide unique properties for biomedical applications.^{252–257} This section discusses the applications of the multilayer MXene in antibacterial activity, tissue engineering, photothermal therapy, bioprinting, bioimaging, and biosensors.

Antibacterial Activity. Skin is the largest organ in the human body. When any disruption occurs in the skin, it is known as a wound; the antibacterial activity plays a crucial role during the healing process of the wound.^{258,259} Recently, hybrid ML-MXene was used to fabricate wound dressing because of its unique properties. The glutaraldehyde cross-linked Ti₃C₂T_x/chitosan electrospun composite mat showed excellent antibacterial activity against *Escherichia coli* as a wound dressing.²⁶⁰ Direct adsorption of chitosan onto the bacterial cell surface blocks the membrane transport channel and causes cell deaths. The MXene-based composite can absorb water quickly to form a hydrogel.²⁶¹ A composite structure was prepared by incorporating multilayered Ti₃C₂T_x into chitosan/sodium hyaluronate through the freeze-drying process. This 2D MXene was

stabilized by L-ascorbic acid, which helps to prevent surface oxidation of MXene. The freeze-dried composite structure formed a hydrogel in Biocorp and showed excellent antibacterial activity. However, the higher amount of MXene reduced the porosity of the composite system. The addition of 5 wt % MXene into the chitosan/sodium hyaluronate exhibited 99% removal of *Staphylococcus aureus* bacteria with controlled porosity of the composite structure.²⁶² Although hydrogels play a significant role in the healing, these might cause problems with inflammatory responses.²⁶³ Therefore, the electrical stimulation method of wound healing was adopted to utilize the skin's electrical conductivity of 2.6 to 1 × 10⁻⁴ mS.²⁶⁴ Regenerated bacterial cellulose with a 2 wt % Ti₃C₂T_x MXene hydrogel fabricated by a freeze-drying method showed higher electrical conductivity of 7.04 × 10⁻⁴ S cm⁻¹ and thermal stability was achieved. This composite hydrogel wound dressing was treated with a skin defect rat model with and without electrical stimulation. The regenerated bacterial cellulose/MXene wound dressing with electrical stimulation effectively decreased the infected area within 14 days.²⁶⁵ Apart from the electrical stimulation, the Joule heating effect is also effective for the antibacterial activity of wound dressing. Dip-coated nonwoven cellulosic fabric in Ti₃C₂ MXene generates 100 °C heat at 6 V to kill *S. aureus* bacteria with 100% repaired wounds without damaging any cells at the wound site (Figure 8a–d).²⁶⁶

Tissue Engineering. Increasing global demand for organ transplantation is one of the most severe concerns nowadays. This huge demand for organs can be met by fabricating artificial organs through tissue engineering.^{268,269} Three key factors, i.e., cell seed, scaffold, and stimulating factors, significantly affect tissue engineering.²⁷⁰ Along with different stimulating factors, electrical stimulation helps cell alignment, migration, proliferation, and differentiation.²⁷¹ Therefore, electroconductive materials like polyaniline, polypyrrole, poly(3,4-ethylene dioxythiophene), polythiophene, polyacetylene, and poly(p-phenylene), poly(p-phenylenevinylene), poly p-phenylene-sulfide, silicon, and melanin are popular for tissue engineering.²⁷² In addition, MXenes have become an attractive material for tissue engineering due to their antibacterial, anti-

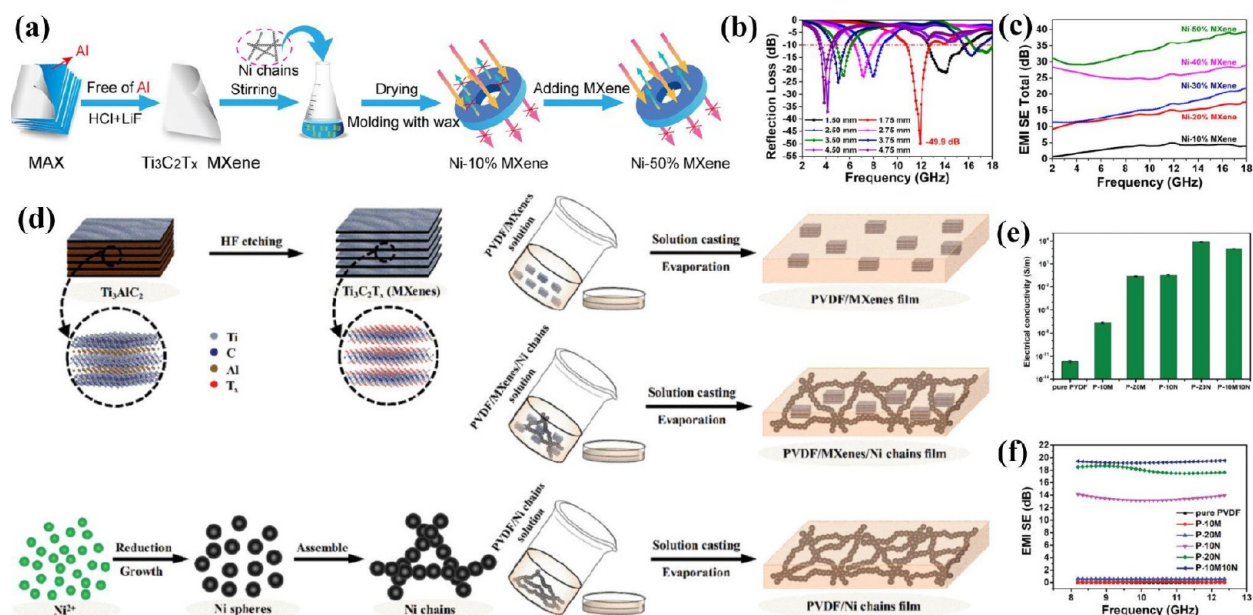


Figure 9. (a) Schematic representation of the fabricated MXene/Ni hybrids. (b) Electromagnetic absorption performance of Ni-10% MXene at various thicknesses with minimal reflection loss curves. (c) MXene/Ni hybrids shielding performance. (Reprinted with permission from ref 138. Copyright 2019, American Chemical Society.) (d) Diagram depicting the PVDF/MXene/Ni chain composite film production process. (e) Various composite films' electrical conductivities. (f) EMI shielding efficiency (SE) of 0.10 mm composite films. (Reproduced with permission from ref 293. Copyright 2019, WILEY-VCH Verlag GmbH & Co. KGaA.)

inflammatory, immunomodulatory properties, and cytocompatibility.⁵⁰ A composite hydrogel scaffold fabricated by incorporating ML-MXene and honey into chitosan showed excellent biocompatibility with no cytotoxicity to the stem cells. Honey and chitosan both have therapeutic properties, and the addition of MXene helped enhance the mechanical properties of the scaffold and swelling ability.²⁷³ Ti₃C₂T_x MXene was printed on polyethylene glycol hydrogel by aerosol jet printing with different patterns such as straight line, bowtie, and Hilbert's curve. Induced pluripotent stem cell obtained Cardiomyocyte (iCM) cells were seeded onto these patterns to improve the alignment. In addition, the number of living cells experiences a rise from 85% to 93% 7 days after seeding.²⁶⁷ Figure 8e–h shows different patterns for iCM cells on a MXene-polyethylene glycol composite hydrogel. To accomplish linear alignment of iCMs, the straight line was adopted. Hilbert's curve patterns, which can track the propagation of Ca²⁺ signal, were picked for their unique characteristics in addition to their iCM alignment capabilities. The Ti₃C₂T_x MXene-PEG hydrogels had a better alignment of the iCMs for all patterns.

The presence of functional groups on the MXene provides a particular environment for cell attachment, cell proliferation, and distribution. The fiber diameter of the MXene/poly-L-lactic acid (PLLA)/polyhydroxyalkanoates (PHA) electrospun composite was larger than that of the PLLA-PHA nanosheets. However, the water contact angle of the MXene composite scaffold was found to be 112.76°, which was lower than for PLLA-PHA sheets. Functional groups in MXene improved the uniform cell distribution and decreased the contact angle.²⁷⁴ MXenes have also demonstrated efficient application in cancer treatment, biosensing, bioimaging, and 3D printing of organs, as listed in Table 5.

Electromagnetic Absorption and Shielding. Electronic devices have become inseparable parts of daily life in the modern era, and the world has started the transition toward the 5G

telecommunication technology.^{285,286} These electronic devices generate electromagnetic waves that have a significant detrimental effect on the human body. The electromagnetic interference (EMI) shielding devices restrict the waves within a particular area to lower the adverse impact on living organisms.²⁸⁷

The efficiency of EMI shielding devices depends on the electrical conductivity of the material which is at least 1 S/m. However, the shielding efficiency is not only dependent on the inherent properties of materials such as conductivity, permittivity, and permeability but also on the structure. Multilayered MXenes have been used for improving the EMI shielding efficiency and EM wave absorption by altering each layer's depth and dielectric characteristics.^{288–290} The multilayer structure and the large specific area of Ti₃C₂T_x help to provide additional electron transit routes that improve the EM wave absorption.²⁹¹ Although the absorption band of Ti₃C₂T_x hybrid absorbers is outstanding, issues such as a high matching impedance and a restricted effective absorption bandwidth range constrained their uses.²⁹²

Ti₃C₂T_x MXene/Ni chain hybrids with Ni-10% MXene, fabricated by a hydrothermal process, demonstrated an effective electromagnetic wave absorbing property at a thickness of 1.75 mm with the reflective loss of -49.9 dB at 2.1 GHz. At this point, high dielectric and magnetic losses were experienced. The total EMI shielding effectiveness experienced a rise with the increase of MXene from 10% to 50%, as a higher content of MXene helped to increase the electrical conductivity. A similar trend was also observed in the case of sample thickness. The highest EMI shielding effectiveness of 66.4 dB was achieved for Ni-50% MXene with a thickness of 2.8 mm and 33.8 dB for the thickness of 1.3 mm (Figure 9a–c).¹³⁸

A polyvinylidene fluoride (PVDF)/MXene/Ni chain composite film with 20 wt % MXene, produced by a solution casting method, failed to meet the practical requirement of electrical

Table 6. Electromagnetic Wave Absorption Properties of Hybrid MXene

hybrid material	manufacturing method	sample thickness (mm)	reflection loss (dB)	effective absorption bandwidth (GHz)	sample thickness (mm)	frequency (GHz)	ref
Ti ₂ CT _x /PVA	freezing-dry	3.9	-18.7	—	—	8.2	295
TiO ₂ /Ti ₃ C ₂ T _x /RGO	hydrothermal method	2.5	-65.3	4.3	2	—	296
MXene Ti ₃ C ₂ T _x @RGO aerogel	hydrothermal method with freeze-drying	2.05	-31.2	5.4	2.05	8.2	297
Ti ₃ C ₂ T _x MXene/Metal nanoparticles	cosolvent method	—	-52.6	3.7	3	8.4	298
rGO/Nb ₂ CT _x /Fe ₃ O ₄	hydrothermal and an electrostatic self-assembly method	—	-59.17	6.8	2.5	11.8	299
Ti ₃ C ₂ /Fe ₃ O ₄ /PANI	—	—	-40.3	5.2	1.9	15.3	300
RGO/Ti ₃ C ₂ T _x /Fe ₃ O ₄	ultrasonic spray	—	-51.2	6.5	2.9	14.4	301
Ti ₃ C ₂ T _x MXene/gelatin nanocomposite aerogel	freeze casting method	2	-59.5	6.24	2	14.04	302
CoFe ₂ O ₄ /Ti ₃ C ₂	solvothermal process	1.5	-30.9	8.5	1.5	8.3–16.8	303
FeCo-Ti ₃ C ₂ MXene	hydrothermal method	1.6	-17.86	8.8	1.6	9.2–8.0	304
Ti ₃ C ₂ T _x /TiO ₂ /PANI	hydrothermal and in situ polymerization	2.18	-65.61	5.92	2.10	13.92	305

Table 7. EMI Shielding Properties of Hybrid MXene

hybrid material	method	filler content	conductivity	shielding effectiveness (dB)	sample thickness	frequency (G Hz)	ref
Ti ₃ C ₂ T _x /PVDF	solvent assisted mixing and compression molding	22.55 vol %	0.988 S m ⁻¹	48.47 ± 3.5	2 mm	12.5	306
Ti ₃ C ₂ T _x /Epoxy	solution casting	15 wt %	105 S m ⁻¹	41	2 mm	8.2–12.4	307
Ti ₃ C ₂ T _x /PEDOT:PSS	vacuum-assisted filtration	7:1	340.5 S m ⁻¹	42.10	11.1 μm	8.2	308
aramid nanofiber/Ti ₃ C ₂ T _x	vacuum-assisted filtration	80 wt %	879.0 S cm ⁻¹	40.6	3.2 μm	8.2	309
d-Ti ₃ C ₂ T _x /electrospun regenerated cellulosic nanofibers	vacuum-assisted filtration	—	46.3 S cm ⁻¹	42.7	15 μm	2–18	310
Ti ₃ C ₂ T _x /cellulose nanofiber/silver	vacuum-assisted filtration	17.67 wt %	588.2 S m ⁻¹	50.7	46 μm	12.4	311
Ti ₃ C ₂ T _x /delignified wood cellulose	—	—	6333 S m ⁻¹	39.3	2 mm	12.4	312
PDMS coated M-filter (Ti ₃ C ₂ T _x /cellulose filter paper)	dip coating	1.89 vol %	2756 S m ⁻¹	43	0.2 mm	12.4–18 GHz	313
Ti ₃ C ₂ T _x /PVA	multilayer casting	19.5 wt %	716 S m ⁻¹	44.4	0.027 mm	8–12	314
Ti ₃ C ₂ T _x /Fe ₃ O ₄ -PANI	vacuum-assisted filtration	10:5	—	62	16.7 μm	8.2–12.4	315
d-Ti ₃ C ₂ T _x /aramid nanofiber	vacuum-assisted filtration	10:1	—	34.71	12 μm	8.2	316
chitosan/Ti ₃ C ₂ T _x	vacuum-assisted filtration	75 wt %	1402 ± 70 S m ⁻¹	34.7 ± 2	37 μm	8.2–12.4	317

conductivity for EMI shielding materials, i.e., 1 S m⁻¹. This is due to the low number of electron transport between the MXenes. On the other hand, the electrical conductivity was increased to 33.3 S m⁻¹ for 50 wt % of MXene. Interestingly, the superior electrical conductivity was achieved of 892 S m⁻¹ for PVDF with 10 wt % MXene and 10 wt % Ni chains composite. This composite exhibited the highest EMI shielding effectiveness value of 26 and 34.4 dB for a sample thickness of 0.24 and 0.36 mm, respectively (Figure 9d–f).²⁹³ A MXene/polyurethane composite was produced by spray coating of Ti₃C₂T_x MXene on single (SMPUF) and both sides (DMPUF) of an electrospun polyurethane fabric. The DMPUF experienced a rise in electrical conductivity of 11.5 S m⁻¹, which achieved the highest EMI shielding effectiveness of 21 dB above the commercial level compared to SMPUF.²⁹⁴ The electromagnetic wave absorption and the EMI shielding properties of hybrid MXene are listed in Tables 6 and 7.

Environment and Water Treatment. MXenes have a potential application for environmental issues^{318–321} such as water treatment, heavy metal removal by working as ion separation membranes,³²² capacitive deionization,³²³ antimicrobial coatings,³²⁴ and photocatalytic degradation.³²⁵ Moreover, the carbonyl functionalized Ti₃C₂T_x MXene (TCCH) showed superior performance in the removal of U(VI) and Eu(III).³²⁶

Wang et al.³²⁷ used 2D V₂CT_x for removing actinides from aqueous solutions, which was highly efficient for U(IV) sorbent (Figure 10a–c).³²⁷ Delaminated Ti₃C₂T_x nanosheets demonstrated a metal ions reduction capability, consisting of Cr (VI), Cu, and Ag.^{328–331} The test of U(VI)/Eu(III) contained radioactive wastewater showed a remarkable 80% contaminant removal in just 30 min by the TCCH adsorbent, which increased to 90% with the increase in treatment time up to 3 h. Carboxyl groups play a significant role in the efficiency of MXene and the aryl diazonium salt also enhanced the firmness/solidity of the material in water. The maximum adsorption capacity was reported as 344.8 and 97.1 mg g⁻¹ for U and Eu.³²⁶

ML-MXenes as a separation membrane could be used in the following three ways: (i) as a skeleton while fabricating, (ii) mixing of materials with MXene for the fabrication, and (iii) as a coating material.³³³ Alkalized Ti₃C₂T_x showed good performance for the Pb(II) removal and assumed that 1 kg of MXene is efficient for the treatment of 4500 kg of water (Table 5). MXenes showed great hydrophilic properties and strong interaction with polymers, providing a better fabrication faculty. A casting method for the fabrication of Ti₃C₂-SO₃H resulted in increased water uptake and proton conduction properties in the membrane.³²² MXene showed the capability of Cr ion removal,

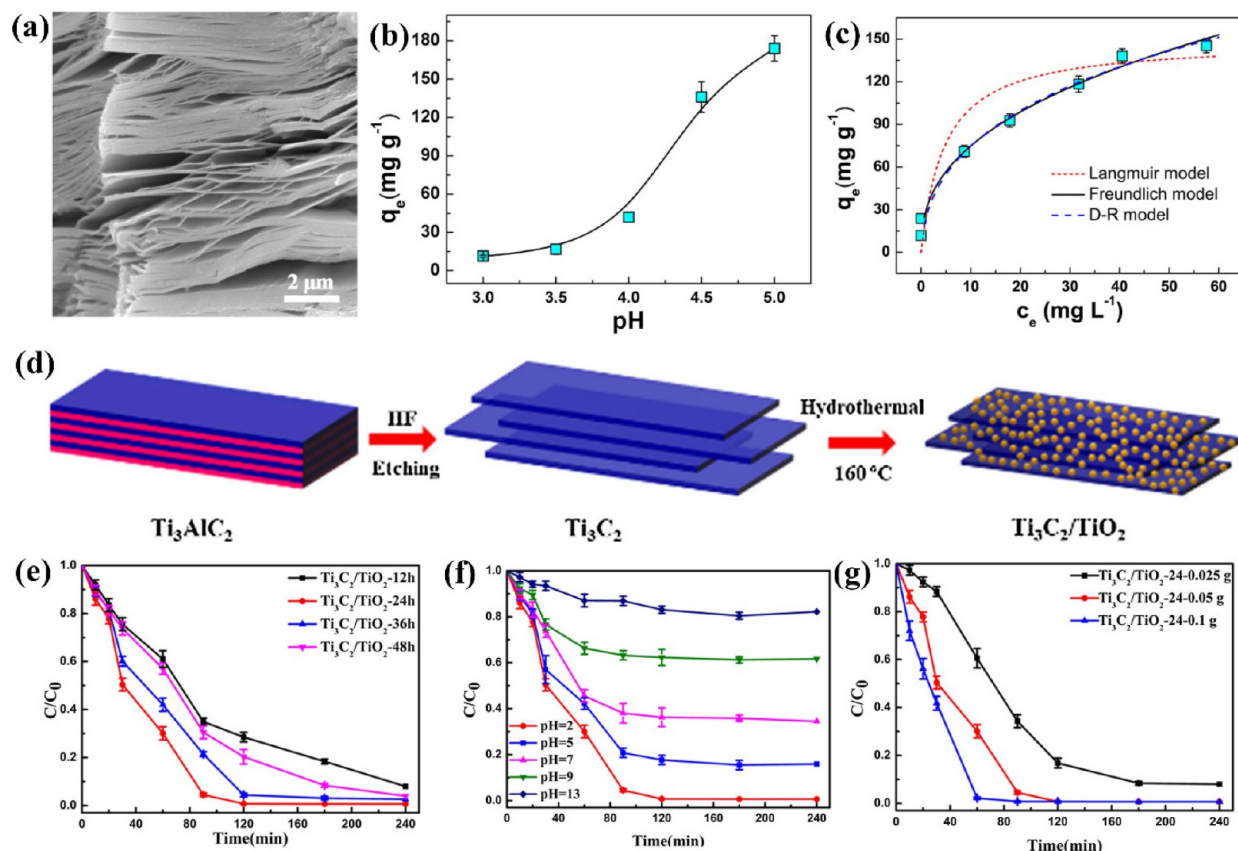


Figure 10. (a) At pH 4.5, SEM images of U(VI) absorbed. (b and c) Function of pH and earliest U(VI) concentration, U(VI) absorption from aqueous solution onto multilayered V_2CT_x . (Reproduced with permission from ref 327. Copyright 2016, American Chemical Society.) (d) Schematic diagram of the composition of Ti_3C_2/TiO_2 hybrid composite. (e) For different hydrothermal treatment times, the percentage extraction of Cr(VI) in 50 mL 50 mg L^{-1} solution (pH 2.0). (f) Cr(VI) extraction in 50 mL of a 50 mg L^{-1} solution Ti_3C_2/TiO_2 composite material concentration: 0.05 g L^{-1} (pH dependent). (g) Effectiveness of the Ti_3C_2/TiO_2 -24 composite material's dosage on Cr(VI) elimination in 50 mL of a 50 mg L^{-1} solution (pH 2.0). (Reproduced with permission from ref 332 Copyright 2019, Elsevier Ltd.)

Table 8. MXenes Used for Different Toxic Metal Removal from an Aqueous Environment

MXene	adsorption capacity or reduction efficiency	condition of adsorption			toxic materials	ref
		temperature	time	pH		
Ti_3C_2	80 mg g^{-1}	298 K	14 h	—	Cr(VI)	336
$Ti_3C_2T_x$	250 mg g^{-1}	at RT	72 h	5.0	Cr(VI)	328
(analogous urchined rutile) TiO_2 -C (u-RTC)	$\sim 225\text{ mg g}^{-1}$	—	120 min	3.0–6.0	Cr(VI)	337
Ti_3C_2/TiO_2	99.35%	—	12 min	> acidic	Cr(VI)	332
nZVI (Nano zerovalent iron)-alk- Ti_3C_2	194.87 mg g^{-1}	—	~ 1500 min	2.0	Cr(VI)	338
$Ti_3C_2T_x$	78.45 mg g^{-1}	298 K	3 min	5.0	Cu(II)	339
$Ti_3C_2T_x$ -PDOPA (poly dihydroxyphenylalanine) (Amino acid modified)	18.36 mg g^{-1}	298 K	1 h	7.0	Cu(II)	340
$Ti_3C_2T_x$ /PmPD (poly(m-phenylenediamine))-S/1	540.47 mg g^{-1}	—	~ 700 min	2.0	Cr(VI)	341
MoS_2 /MXene (functionalized MXene)	1435.2 mg g^{-1}	—	2 min	2.0–11.0	Hg(II)	342
$Ti_3C_2T_x$	9.3 mg g^{-1}	298 K	2 h	7.0	Ba(II)	343
MXene/alginate	382.7 mg g^{-1}	~ 323 K	15 min	5.0–7.0	Pb(II)	344
$Ti_3C_2T_x$ -KH570 (silane coupling agent)	147.97 mg g^{-1}	303 K	2 h	1.0–6.0	Pb(II)	345
magnetic $Ti_3C_2T_x$ nanocomposite	1128.41 mg g^{-1}	298 K	24 h	6.0	Hg(II)	346
$Ti_3C_2(OH)_x F_{1-x}$	2400 mg g^{-1}	—	~ 250 min	2.5–6.0	PO_4^{3-}	347
$Ti_3C_2T_x$ core-shell spheres containing sodium alginate molybdenum	932.84 mg g^{-1}	298 K	24 h	4.5	Hg(II)	348
Ti_2CT_x nanosheets biosurfactant functionalized	232.9 mg g^{-1}	303 K	24 h	5.0	Pb(II)	349
$Ti_3C_2T_x$	36.6 mg g^{-1}	293 K	2 h	6.0	Pb(II)	350

and 99.35% of Cr(VI) was successfully removed using Ti_3C_2/TiO_2 nanosheets (Figure 10d–g).³³²

The functional groups like –O and –OH enhance the ability of MXene as a water-absorbent. The high surface area of MXene

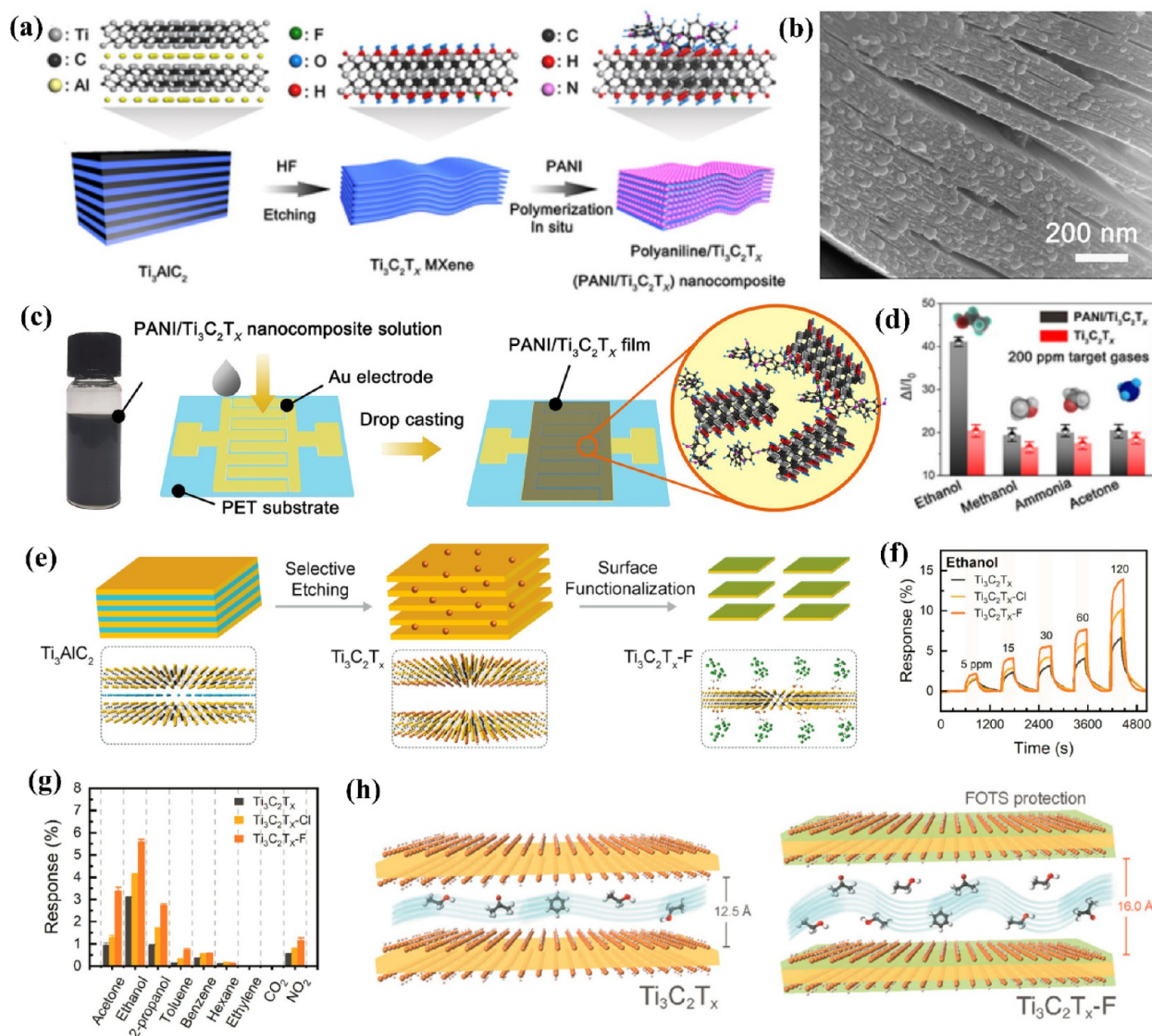


Figure 11. (a) Synthesis of PANI/Ti₃C₂T_x. (b) SEM image of PANI/Ti₃C₂T_x. (c) Illustration of the electrode before and after coating PANI/Ti₃C₂T_x nanocomposites and PANI particles size distribution. (d) Flexible Ti₃C₂T_x and PANI/Ti₃C₂T_x sensors for ethanol, methanol, ammonia, and acetone at room temperature. (Reproduced with permission from ref 360. Copyright 2019, The Authors.) (e) Surface-modified Ti₃C₂T_x nanosheets. (f) Pure MXene and modified MXene sensors dynamic response curves. (g) Maximum sensor response varies on the various gas exposure. (h) Ti₃C₂T_x and Ti₃C₂T_x-F schematics after exposure to VOCs (Reproduced with permission from ref 361. Copyright 2020, American Chemical Society.)

offers a direct ion exchange and reduction capability for various cations and organic molecules (Table 8).³³⁴ MXenes also possess outstanding catalytic activities to numerous pollutants, e.g., Ag nanoparticle-loaded MXenes prepared by intercalating dimethyl sulfoxide showed a superior catalytic behavior to 4-nitrophenol and 2-nitroaniline.³³⁵ Furthermore, Ti₃C₂T_x nanosheets demonstrated remarkable reduction ability by removing the Cr(VI) ions by concurrently reducing Cr(VI) to Cr(III) and absorbing the reduced Cr(III) ions.³²⁸

Organic materials, e.g., dyes and antibiotics, are some of the most dangerous pollutants of recent decades. Proper degradation of these materials is crucial for pollution management. ML-MXenes play a vital role as a cocatalyst in photocatalytic degradation, demonstrated using semiconductor materials like TiO₂, g-C₃N₄, Ag₃PO₄, and Bi₂WO₆.³⁵¹ The 2D MXene enhances charge carrier transfer from the photocatalysts to the MXene and forms a Schottky barrier on the photocatalyst/

MXene interface. The interface helps to inhibit the recombination of electron-hole.³⁵² Moreover, MXene's charge accumulation causes a negative shift and alignment of the Fermi level, improving the photocatalytic performance.³⁵³ For example, 2D Niobium Carbide Nb₂CT_x possesses a Fermi Level lower than the Ti₃C₂T_x, which can be used as a cocatalyst.³⁵⁴

The oil film layer of thickness greater than 1 μm on the water surface affects the diffusion of oxygen in the water, restricts the photosynthesis process of algae, and affects the regular growth of underwater organisms. Oils have a carcinogenic effect on aqueous life as well.³⁵⁵ Pure MXene membrane shows lower-level retention of dye; however, the addition of GO improves the wettability. A well-arranged layered structured membrane prepared by combining 2D GO and MXenes may balance the permeability of water and organic materials and the molecular repulsion of dye.³⁵⁵ This membrane showed excellent durability to washing, i.e., retained dimensional stability for 3 days when

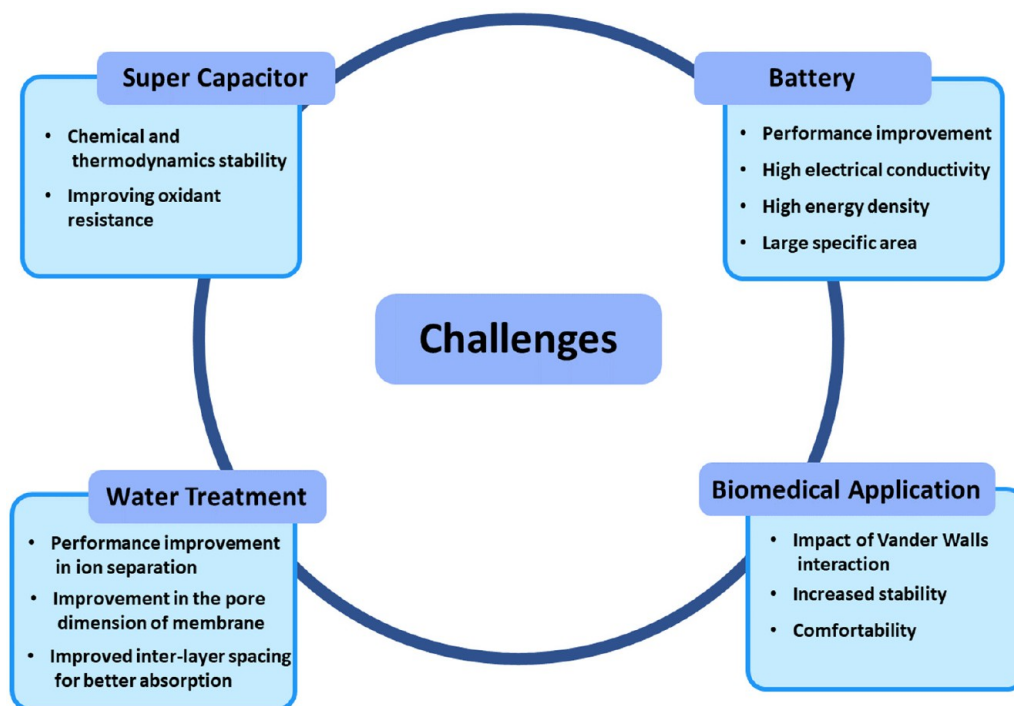


Figure 12. Schematic of insight on potential research and challenges scope in MXene-based hybrid nanocomposites.

washed in an ethanol solution. The presence of hydroxyl and fluorenyl groups on the surface of the MXenes forms a strong hydrogen bond with nylon and results in excellent stability.³⁵⁵

Anodic exposure of MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) to anodic potential in an aqueous solution oxidizes the nanomaterial generating TiO_2 layer, or TiO_2 domain. Then TiO_2 gets dissolved by F ions, and the electrochemical activity of the resulting nanomaterials gets lower than that of the original $\text{Ti}_3\text{C}_2\text{T}_x$.^{356,357} Therefore, $\text{Ti}_3\text{C}_2\text{T}_x$ demonstrates ultrasensitivity to H_2O_2 when used for the electrochemical reaction in the cathode potential window.³⁵⁸ 2D metal carbide MXenes, which offer good metallic conductivity for minimal noise and a functional surface for a high signal, overpower the sensitivity of traditional semiconductive channel materials. $\text{Ti}_3\text{C}_2\text{T}_x$ MXene-based gas sensors demonstrates low detection limits around 50 to 100 ppb for volatile organic compound (VOC) gases at room temperature.³⁵⁹ The very low noise also leads to 2 orders of magnitude greater signal-to-noise ratio than other 2D materials. The device surpasses the best-known sensors as the $\text{Ti}_3\text{C}_2\text{T}_x$ sensor shows elevated hydrogen-bonded gas selectivity. The practical LOD below 50 ppb and the conceptual LOD of VOC gas below 50 ppb are the lowest without pretreatment in any 2D material-based gas sensor working at room temperature.³⁵⁹

Nanocomposite like polyaniline/MXene (PANI/ $\text{Ti}_3\text{C}_2\text{T}_x$) mixtures have been reported by the integration of DFT simulation and bulk electrical sensitivity measurement to demonstrate the high electrocatalytic sensitivity.³⁶⁰ PANI nanoparticles are decorated on the surface of $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets by the low-temperature in situ polymerization. Integration of PANI induces significant sensitivity to detection, fast recovery/response, and good mechanical stability at room temperature. The compound sensing material showed high ethanol sensitivity of 200 ppm and 41.1% in ethanol utilizing the complementary properties of the composite material and the highly active $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. A fast response time of 0.4/0.5 s at room temperature, making it easy for real-time VOC gas

detected by next-generation sensing materials (Figure 11a–d).³⁶⁰

MXene's poor stability against oxidation and hydration in humid environments makes it unsuitable for long-term storage and application. It has been found that the surface treatment with fluoroalkyl silane (FOTS) containing $\text{Ti}_3\text{C}_2\text{T}_x$ compounds increases the mechanical and environmental stability of a superhydrophobic surface, as well as the sensing efficiency.³⁶¹ FOTS functionalization protects the sensor response when measuring the dynamic behavior of the $\text{Ti}_3\text{C}_2\text{T}_x$ -F sensor to 30 ppm ethanol in the range of 5–80% relative humidity. DFT simulation results showed that ethanol adsorption energy and the local structural deformation of $\text{Ti}_3\text{C}_2\text{T}_x$ -high F induced by ethanol adsorption help increase the sensitivity of the gas (Figure 11e–h).³⁶¹ As one of the most effective instruments for detecting noxious gases, gas sensors provide an essential means of monitoring gas concentrations and environmental data to ensure production safety. For these reasons, improving the sensitivity, selectivity, and stability of sensors have become prime factors of research.³⁶²

■ FUTURE PERSPECTIVE OF ML-MXENE AND ML-HYBRID MXENE

Recently, MXene, exfoliated from layered MAX stages, is experiencing exponential growth. The complexity in the synthesis process of MXenes has decreased over the years, and only etching and exfoliation are sufficient to make pure MXenes. The properties of the 2D materials depend on the synthesis process, so diversity in the material used for synthesis can escalate the applications. The developments include synthesizing simple MXenes to hybrid ML-MXenes and the exfoliation of novel MXenes, characterization of electronic and optical properties, and magnetic properties for diverse applications. MXenes can expand the application horizons by exploiting their numerous surface morphologies, surface function tunability, various magnetic orders, and significant spin–orbit coupling.

Furthermore, the addition of MXene can augment the conductivity of heterostructured and hybrid materials by several orders of magnitude, leading to future applications. The significant performance improvement of ML-MXene hybrid nanocomposites is attributed to the synergistic effects of high electrical conductivity, large specific area, and surface chemistry. Pristine MXene is susceptible to the environment and the environmental vulnerability of MXene could reduce by producing various heterostructures and hybrids. Investigation of the impact of van der Waals interactions on the stability and electronic properties of hybrid ML-MXenes will provide researchers with more flexibility to design new structures for various applications. The effect of multilayer and monolayer on the electronic property should also be studied thoroughly. The conventional electrodes show decay and make crystal-like structures by reacting with the Li-ions within the battery. A durable, eco-friendly, and efficient solution to the traditional battery is crucial and MXenes as an electrode material shows great potential. Comprehensive theoretical studies are required to understand the principles of the electrochemical factors that influence them and optimize the corresponding performances of 2D ML-MXenes as electrode materials in LIBs or SCs.

However, to fully utilize 2D heterostructures in energy storage applications, simple, rapid, and inexpensive synthesis procedures must be developed. Developing MXene synthesis methods that can improve accuracy in regulating the lateral size and number of layers of the delaminated MXene flakes is a bottleneck to the design and fabrication of high-performance ML-MXene-based electrodes. Molten Lewis salts have been used to make Ti- and V-based MXenes, but alternative MXenes with different "M" and surface terminations should be investigated. At this time, safer and more environmentally sustainable ways for manufacturing MXene have been proposed, and other solutions to this challenge are expected in the upcoming days.

The versatility of ML-MXene hybrid nanocomposites has demonstrated fascinating prospects and received widespread attention in water treatment. Although the new ML-hybrid MXene-based nanomaterials promise to develop high-performance membranes, there are still several challenges (Figure 12) to address for long-term stability. Also, eco-toxicological and life-cycle evaluations of ML-MXene-based nanomaterials require further exploration to widen the applicability of ML-hybrid MXene-based nanostructures. Designing and developing effective ML-MXenes and MXene-based hybrids requires identifying the catalytic mechanisms under reaction conditions using in situ characterization techniques and theoretical calculations. Analyzing the synergistic activity of reinforcing transition metals and other active components on ML-MXenes is required to fine-tune the behavior and selectivity. MXenes quickly oxidize in the air, and increasing the stability of the ML-MXenes hybrid is crucial for catalysis application. Furthermore, there is a lack of understanding of the relationship between surface termination and photocatalytic activity, particularly for electrocatalyst reactions. The composition relationship between catalytic efficiency and MXenes with unique surface terminations is critical to identifying the ever-increasing applications of ML-hybrid MXenes. However, strong chemical etching used in MXene production results in many flaws or vacancies on the surface of the generated MXene flakes. Defective sites are prone to oxidative degradation processes with water and/or oxygen, reducing MXenes' intrinsic characteristics and limiting their practical applicability. Because many MXene-derived materials and devices are typically manufactured using such solutions, the

longevity of MXene solutions is critical. MXenes distributed in aqueous solutions are not only oxidized but they also oxidize at a faster pace than freestanding MXenes. Furthermore, because of the numerous potential parallel processes involved, the oxidation mechanism of MXenes in an aqueous environment is highly difficult. As a result, the mechanism of MXene breakdown in an aqueous environment is yet unknown. Despite the numerous obstacles, more concentrated efforts should be made to investigate fluoride-free and ecologically friendly etchants as a viable alternative to traditional HF. By reducing the diameter of 2D MXene materials, the relative surface area and reactivity will be increased. This showed that the surface functional groups have a significant impact on MXene's electrochemical characteristics. Modifying morphology, on the other hand, is critical for increasing active sites. The customized porosity and curved geometry of 2D MXene flakes can create high surface area and adjusted pore size and volume, which could help versatile applications.

Comprehensive investigations into the dielectric relaxation and electromagnetic response of ML-MXene-based materials are also essential. ML-MXene and different heterostructures and hybrids of MXene can be specifically constructed and developed by hybridization and assembly based on advanced theoretical guidance for various applications with lightweight, high performance, good impermeability, thermal property, and excellent mechanical properties.

■ AUTHOR INFORMATION

Corresponding Author

Md Milon Hossain – Department of Mechanical and Aerospace Engineering, Cornell University, Ithaca, New York 14850, United States; Department of Textile Engineering, Chemistry and Science, North Carolina State University, Raleigh 27606, United States; orcid.org/0000-0001-8416-0631; Phone: +1919 986 5713; Email: mh2276@cornell.edu

Authors

Sharif Tasnim Mahmud – School of Textile Science and Engineering, Wuhan Textile University, Wuhan, Hubei 430079, China

Md Mehdi Hasan – UNAM - Institute of Materials Science and Nanotechnology, Bilkent University, Ankara 06800, Turkey

Sudipta Bain – Department of Fashion Design and Technology, Uttara University, Dhaka 1230, Bangladesh; orcid.org/0000-0002-9387-6145

Sheikh Tamjidur Rahman – Department of Textile Engineering, Khulna University of Engineering and Technology, Khulna 9203, Bangladesh

Mukitpur Rhaman – Department of Textile Engineering, Khulna University of Engineering and Technology, Khulna 9203, Bangladesh

Mustafa Ordu – UNAM - Institute of Materials Science and Nanotechnology, Bilkent University, Ankara 06800, Turkey

Complete contact information is available at:

<https://pubs.acs.org/10.1021/acsmaterialslett.2c00175>

Notes

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