MXene based Heterostructures for electrode materials of Batteries: A Review

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Abstract
Growing concerns about fossil fuel's environmental impact, along with the recent breakthrough of electric vehicles, have turned research focus to energy storage solutions. Despite the fact that batteries were invented about 200 years ago, modern technologies are required to store energy in a larger grid with a high density. The electrode materials used in energy storage devices such as batteries and supercapacitors play a major role in their overall performance. A lot of materials have been explored but due to appealing electrical and electrochemical properties, MXene has received a lot of interest for energy storage devices. Because of their layered structure and high conductivity, MXenes are promising candidates for energy storage applications. Two-dimensional heterostructured materials are more advantageous than individual building blocks for batteries and supercapacitors. In this review work, we looked at different MXene based heterostructures and their electrochemical performance as electrode materials of batteries. A particular application of MXene in Lithium-ion batteries has been studied. Synthesis and characteristics of MXenes are briefly discussed here. Finally, future prospects and challenges are highlighted.

Keywords: MXene; Lithium-ion Batteries; Supercapacitors; Heterostructures.

1. Introduction
As the world's population grows, energy is becoming one of the most major challenges. Scientists are deeply concerned about the production, use, and storage of fuel because it is restricted beneath the earth's surface. If we can store renewable energy on a larger scale, it may be a viable option [1]. Batteries, capacitors, and supercapacitors are being used extensively as electrical energy storage devices [2-4]. Lithium-ion batteries and sodium-ion batteries are becoming very popular day by day because of their extensive uses in electric vehicles, laptops, digital cameras [5-8]. Electrode materials are very important for these devices because outcome depends on materials of electrodes. Ru, Pt, Ir are common materials for electrodes that have been used for energy storage. But these materials have limitations such as availability; cost [9–15]. Graphene has been employed as anode material, although its specific capacity is restricted to 372 mAh/g [16]. MXenes, a new group of 2D materials consisting of transition metal nitrides, carbides, and carbonitrides, have received huge attention due to their outstanding properties. It is unique in the way that it has excellent electrical conductivity, easily accessible structure, fast diffusion, good
thermal stability, large interlayer spacing, large surface area, and thickness controllability. Restacking in MXene sheets is one of the major challenges to design electrodes for energy storage devices but that can be avoided by introducing different spacers between two MXene layers [19]. In terms of energy storage technology, hybrid structures have a lot of potential. Researchers have recently introduced MXene/Graphene [73], Phosphorene/MXene [77], Bi2MoO6/MXene[76], MoS2-on-MXene[72]. These heterostructures provide novel properties, enhance electrochemical performance, and also provide opportunities to design and manipulate nanoelectronic devices [20-22]. Researchers discovered Mo2TiC2T6 MXene in 2015 in which atomic layer of Titanium is inserted between two layers of Molybdenum. This arrangement produces features that differ from those previously reported for Ti3C2T6 MXene [23]. They produced a MoS2/Mo2TiC2T6 heterostructure that exhibits metallic characteristics due to the presence of MXene and exhibits high Coulombic efficiencies, high specific capacities, and great cycling stability when utilized as the electrode material of LIBs [23].

**Figure 1.** Number of papers on Mxene published per year (Data has been taken from sciencedirect.com)

MXene hybrid structures are vastly under research, and it will take a lot of effort to meet the energy storage needs. So, for the development and advancement of electrode materials for batteries, supercapacitors, and other energy storage devices, a highly focused evaluation is necessary. In this review, we looked at the most recent MXene synthesis technique, as well as the properties of MXene and their possible applications. It has been discussed how MXene can be used in lithium-ion batteries. The electrochemical performance of many heterostructures is investigated in depth in this work.

2. Synthesis & Preparation of MXenes

2.1 Synthesis

MXenes are synthesized from a specific etching of ‘A’ from M_{n+1}AX_{n} (MAX) Phase. Here ‘M’ can be any initial transition metals, for example, Sc (21), Ti (22), V (23), Cr (24) etc. ‘A’ is a metal member of group 12-16 like Cd (48), Al (13), Si (14), P (15) etc. & ‘X’ is carbon (C-6), nitrogen (N-7) [24-28]. ‘A’ is removed from MAX antecedent utilizing HF or NH4HF2 provided in equation (1) & (4). The tough metallic bond between M and A is changed by weaker H-bond like fluorine (F), Hydroxyl (OH), Oxygen (O). Metallic hydrophilicity is present, and it reacts with various groups such as O, OH, and F, as shown in the equations. (2), (3) & (5) [17,26,27,29].
2.1 Preparation of Ti$_3$C$_2$

At room temperature aqueous HF is used to separate weakly bonded Al layers from Ti$_3$AlC$_2$ phase in order to preparing Ti$_3$C$_2$. Following are the reactions,

\[ 3\text{HF} + \text{Ti}_3\text{AlC}_2 \rightarrow \text{Ti}_3\text{C}_2 + \text{AlF}_3 + 3/2\text{H}_2 \]  
(6)

\[ \text{Ti}_3\text{C}_2 + 2\text{H}_2\text{O} \rightarrow \text{Ti}_3\text{C}_2 (\text{OH})_2 + \text{H}_2 \]  
(7)

\[ 2\text{HF} + \text{Ti}_3\text{C}_2 \rightarrow \text{Ti}_3\text{C}_2\text{F}_2 + \text{H}_2 \]  
(8)

Equations (7) and (8) show OH and F termination, respectively. To mitigate solids, centrifugation is utilized, followed by washing with deionized water. These MXenes are made up of several layers. sonications are used to obtain a single structure [17, 46].

As an etchant, Halim [31] employed NH$_4$F instead of the toxic HF. Because the nature of NH$_4$HF$_2$ is not as strong as that of HF, and because of the cation's simultaneous intercalation, NH$_4$HF$_2$ is more suited and safe for making MXenes. The reactions are as follows:

\[ 3\text{NH}_4\text{HF}_2 + \text{Ti}_3\text{AlC}_2 \rightarrow \text{Ti}_3\text{C}_2 + \text{(NH}_4)_3\text{AlF}_6 + 3/2\text{H}_2 \]  
(9)

\[ \text{aNH}_4\text{HF}_2 + \text{Ti}_3\text{C}_2 + \text{bH}_2\text{O} \rightarrow (\text{NH}_3)c (\text{NH}_4)d\text{M}_{n+1}\text{X}_n(\text{OH})_x \text{F}_y \]  
(10)

2.2 Properties

3.1 Structural Properties

After extracting A from MAX phase MXenes are formed with various surface groups like F, O, and OH. MXenes with O/OH surface group are most stable. The surface group F can be switched with OH by rinsing or reserving in water [32]. High-temperature treatment or metal absorption process can be used to transform surface group OH into O. Bare MXenes can be found by contacting O terminated MXene with metals like Mg, Ca, Al [33]. MXene’s Structure modeling is very important to understand the structure. Previously it was assumed that the surface groups O, F, OH were located above the void among nearby three C atoms [34]. But later developments indicate position and arrangement of O, F, and OH groups are more complex and are not uniformly positioned. More studies are required to understand the coexistence and absorption of surface groups within MXenes including the factors interlayer interaction, Van der Waals force etc.

3.2 Stability

Bigger the negative worth of lattice energy, the stability of crystal is higher. By calculating structure of energy band using First principle, MXene’s lattice energy is found negative so MXene can exist stably [18]. It is found that stability of Ti$_{n+1}$C$_n$ (MXene with Carbon) is more than that Ti$_{n+1}$N$_n$ of (MXene with nitrogen). It is also found that the stability and resistance of electron beam emission of Ti$_3$C$_2$ film layer is more than that of graphene [29].

3.3 Metallic, Electronic and Electrical

Bare MXenes variants Ti$_{n+1}$X$_n$ have metallic characteristic. Increasing of value of n decreases the metallic behavior because of additional M-X bond. Ti$_{n+1}$N$_n$ has more metallic characteristics than Ti$_{n+1}$C$_n$ as nitrogen atom has additional electron than carbon [37-38]. The density of Ti$_{n+1}$C$_n$ and Ti$_{n+1}$N$_n$ near Fermi energy states is 2.5-4.5 times than their antecedent MAX phase, according to the first principle. 3D state of Ti in the broken bond of Ti-Al transforms into the metal bond state of Ti-Ti, causing MXene to have metallic characteristics, which is compatible with the obtained results of [29]. MXene exhibits semiconductor properties depending on the type and order of surface groups (O, OH, Fetc.) [39]. Using the first principle, M. khazaei [40] discovered M$_2$C (M = Ti, Zr, SC, V, Cr, Nb, HF, Ta) and M$_2$N (M = Ti,
Zr, Cr, Ta, Nb, HF). They demonstrated that Sc$_2$CF$_2$, Hf$_2$CO$_2$, Sc$_2$CO$_2$, Zr$_2$CO$_2$, Ti$_2$CO$_2$, Sc$_2$(OH)$_2$ are semiconductor with a band gaps of 0.25 to 2.0 eV[40]. Electric conductivities of MXenes pressed discs are alike to multi-layered graphene and greater than graphene oxide materials and carbon nanotubes [43-44]. Resistance increases with the number of layers and the different types of functional surface groups (OH, O, F) [29,46-47]. Because of variations in (i) delamination yield, (ii) d-spacing between MXenes flakes (iii) surface functional groups, (iv) lateral diameters (v) defect concentration generated by each etching technique, electrical conductivities of Ti$_3$C$_2$Tx ranged from 850 to 9880 Scm-1[57-60].

3.4 Magnetic

Various magnetic properties are found in several kinds of MXenes. For example, Ti$_3$CNTx and Ti$_3$C$_2$Tx are non-magnetic with the surface groups O, OH, F etc. [39,50,52], while at room temperature Cr$_2$CTx and Cr$_2$NTx are ferromagnetic with OH and F surface groups [18,40,54] and Mn$_2$NTx is ferromagnetic independent of the surface groups [17]. These are calculated predictions and more experimental observations are needed.

3.5 Optical

For optoelectronic, photovoltaic, photocatalytic, and transparent conductive electrode technologies, UV and visible light absorption are important optical activities. Experiments on the Ti$_3$C$_2$Tx (T: surface group F, OH, or O etc.) thin film demonstrate that it transmits 77 percent of visible light with a wavelength of 550nm [55]. The transmittance of a thin film of the Ti$_3$C$_2$Tx (MAX) phase is 30%, which is not as transparent as MXene [31]. Intercalated Ti$_3$C$_2$T$_x$ with NH$_4$HF$_2$ has a transmittance of 90%. The absorbance of Ti$_3$C$_2$Tx (T: surface group F, OH, or O, etc.) and intercalated films are not dependent on their thickness [31]. The optical characteristics of pure and functionalized Ti$_3$C and Ti$_3$C$_2$ with O, OH, F are presented by Wang[56]. Between infrared and ultraviolet, as well as visible light, MXenes containing F & OH functional groups have lower in-plane absorption coefficients than bare MXenes. OH and F surfaces clustered Ti$_3$C and Ti$_3$C$_2$ should emit white color, according to refractivity analysis [56].

4. Application of MXenes in Lithium-ion Batteries

Lithium-ion batteries were first commercialized in 1990, and research has progressed at a rapid rate since then. Improved energy density and lightweight make it advantageous to conventional batteries in mobile phones, computers, and digital cameras. One of the most recent advancements (electric vehicles) needs a large amount of power at a high current[61]. Rapid charge/discharge rate degrades the electrochemical performance of lithium-ion batteries. As a result, new materials and technologies are necessary to enhance the performance of LIBs[62]. Graphite has been emerged as a promising alternative as an anode material for LIBs due to its good electronic conductivity, cost-effectiveness, and stability. However, when graphite is employed, a difficulty arises since it limits the specific capacity to 372 mAh/g[63]. It is demonstrated from the theoretical calculations that MXenes, which have a wide range of structural and chemical properties, are one of the most attractive candidates for various energy storage applications[64].

Zhou reported 2D multilayered vanadium carbide (V$_4$C$_3$) MXene as an electrode material of LIBs. They synthesized this specific MXene by etching Al from V$_4$AlC$_3$ in HF solution (Figure-2). To get larger interlayer spacing and the specific surface they used Ball-milling treatment on V$_4$AlC [65]. As anode material, it shows outstanding performance with a specific capacity of 225 mAh/g after 300 cycles [65]. Rao studied the interaction of lithium sulfide on Ti-based MXene and found that Ti-S bond dominates the interaction between MXene and Lithium sulfide which prevents the loss of sulfur from cathode of lithium-sulfur batteries [66]. Recent advancements of MXene based heterostructures for Lithium-ion batteries will be reviewed in the latter part of this paper.
5. MXene based Heterostructures for batteries

MXene-based heterostructures have paved the way for the development of high-capacity rechargeable batteries. Researchers have been paying close attention to it since it outperforms individual building blocks. They're working on new hybrid architectures that use a variety of catalysts to overcome the limitations of battery and other energy storage device design. [72-76].

Xu[67] fabricated an ultrafast kinetics electrode for sodium-ion batteries by synthesizing MoSe₂/MXene hybrid architecture. DFT calculation shows that diffusion barrier between Na⁺/MXene effectively reduced which enables high ion and electrical conductivity. This heterostructure shows high reversible capacities at high current (490 mAh/g at 1 Ag⁻¹, 1250 mAh/g at 10 Ag⁻¹), 99.8% coulombic efficiency indicates outstanding electrochemical performance[67]. Researchers design and fabricated a Co₃S₄/MXene hybrid architecture as an effective sulfur host for Mg–S batteries. DFT calculation and XPS result showed that polysulfide could be absorbed and it provides fast Mg ion transportation. A specific capacity of 1220 mAhg⁻¹, and after 100 cycles 528 mAhg⁻¹ capacity has been obtained in their work[68].

A representation of the 2D transition-metal dichalcogenide MoS₂ appears to be a strong contender for lithium-ion storage[69]. However, there are significant challenges, such as MoS₂'s weak cycle stability and rate capabilities[70]. MXenes give an enhancement in electrochemical performance of MoS₂ based electrodes. MXene’s layered structure and excellent conductivity make them ideal for energy storage, especially lithium-ion storage[71]. MoS₂ on Mo₂TiC₃Tx MXene heterostructures with a few layers optimize the potential of MXene as a support for MoS₂[72].

Obtained a-LPs from MoS₂ and Mo₂TiC₃Tx/MoS₂ heterostructures are 3.16 and 3.06 e, accordingly. In the heterostructure, MoS2 is such a semiconductor that has a bandgap of 1.7 eV and metallic characteristics. Higher conductivity is required for high-rate charging and discharging. It is concluded that the combination of MoS₂ and Mo₂TiC₃Oₓ considerably improves the conductivity of MoS₂[72]. Initial charge, discharge capacities of pure Mo₂TiC₃Tx are 134 and 268 mAhg⁻¹. From Figure 3, we see MoS₂/Mo₂TiC₃Tx - 500 gives a 4 times improvement in charge, discharge capacities (554 and 646 mAhg⁻¹ at 0.1 A⁻¹). Charge capacity of the Mo₂TiC₃Tx/MoS₂ heterostructure is likewise significantly higher than that of multilayer Mo₂TiC₃Tx previously reported [72].

MXene/graphene heterostructure functions exceptionally well as Li-ion battery’s electrodes. The presence of graphene prevents MXene layers from restacking effects and improves electric conductivity, Li
adsorption strength, and mechanical stiffness [73]. Charge changes from graphene to MXene have been discovered in large quantities. Weakly coupled Ti$_2$C$_x$/MoS$_2$ heterostructures have already shown similar high charge transfer [74]. Single layers of Ti$_2$C$_2$O$_2$ are semiconducting, whereas, Ti$_2$CF$_2$ and Ti$_2$C(OH)$_2$ are known to be metallic[75]. The electrical conductivity of graphene states is higher than that of isolated MXenes, resulting in metallicity, as seen in Figures 2a-c of [73]. MXene/Graphene heterostructures outperform Ti$_2$C$_2$O$_2$ electrodes in terms of electrical conductivity and electrolyte infiltration, resulting in improved electrochemical performance [73].

MXene nanocomposite in Plate-to-Layer Bi$_2$MoO$_6$/MXene-Heterostructure performs as a very conductive substrate for loading and anchoring Bi$_2$MoO$_6$nanoplates, resulting in a great improvement in electronic conductivity, as well as structural stability, is obtained [76]. Detailed electrochemical performance of (i)Bi$_2$MoO$_6$/MXene-50%;(ii) Bi$_2$MoO$_6$/MXene-30%; (iii)Bi$_2$MoO$_6$/MXene-10% and pristine Bi$_2$MoO$_6$ has been analyzed in the figure 5 of [76] to find out the best possible Bi$_2$MoO$_6$/MXeneheterostructure[76].

Figure 3. In LIBs, the electrochemical performance of MoS$_2$/Mo$_2$TiC$_x$Tx heterostructures[72].

The surface area of MXene increases as the MXene content increases, resulting in a more irreversible interaction between functional groups on the MXene surface and Li$^+$ [76]. From figure 5 of [76] we see that MXene effectively enhances a composite electrode’s rate performance. Figure 6 of [76] demonstrates Li storage behavior of Bi$_2$MoO$_6$/MXene-30%. During the lithiation/delithiation, Bi$_2$MoO$_6$/MXene-30% exhibits higher diffusion coefficients and lower over potential compared to Bi$_2$MoO$_6$, which implies better reaction kinetics. Because of their high conductive nature, MXenenanoplates considerably increase charge transmission[76]. When MXene was used as a Bi$_2$MoO$_6$/MXene substrate during fabrication, the lithium storage capacity of Bi$_2$MoO$_6$ was considerably boosted. MXene improves charge transport and reduces Bi$_2$MoO$_6$ volume change, resulting in higher rate capability, long-term cycling stability, and high specific capacity.

Phosphorene/Ti$_2$C$_x$Tx MXene reduces structural expansion and allows electrons and sodium ions to migrate more easily, consequently improving in the hybrid structured anode’s cycling performance.
The increased radius of Na+ makes it difficult to construct traditional electrodes due to structural expansion [78-79]. Based on the alloying reaction, phosphorus (P) has the largest theoretical capacity (2600 mAh g⁻¹). Among all the prospective candidates for Na+ storage, phosphorus has the largest capacity (2600 mAh g⁻¹) [80-82]. Scientists have looked into red phosphorus as a potential anode material for SIBs intensively. However, when used in batteries, it reduces ionic diffusion rate, and weak intrinsic conductivity hinder rapid charge/discharge performance and cycle life [83-84]. Figure 3 of [77] shows the initial 3 cyclic voltammetry of the phosphorene/MXene heterostructure. The initial cathodic scan revealed a strong peak at 0V, indicating sodium intercalation, alloying processes, and SEI development. The anodic scan displays a peak around 0.55, 0.7, and 0.92V, indicating a stepwise desodiation process of phosphorene [77]. Second cycle reveals a very reversible chemical process, as evidenced by the overlapping CV profile. The phosphorene/MXene hybrid anode's subsequent discharge and charge curves are nearly unchanged, which is compatible with the CV results. At a current density of 0.1Ag⁻¹, cycling performances of (i) phosphorene/MXene, (ii) mixed BP/MXene, (iii) Ti₃C₂Tx MXene, and (iv) BP electrodes are presented in figure 3(c) of [77]. With a capacity of 467 mAh g⁻¹, the phosphorene/MXene heterostructure demonstrates good cycling stability after 100 cycles [77]. The volume expansion of the phosphorene/MXene heterostructured anode after 5 cycles is around 120 percent, demonstrating that the existence of MXene in the composite is well suited to supporting the phosphorene volume change. Phosphorus electrode has faster diffusion of Sodium ion validated by low polarization change in galvanostatic titration [85]. It is concluded that MXene's high electrical conductivity, together with higher Sodium diffusion kinetics and Sodium affinities in fluorine-rich phosphorene/MXene hybrids delivers high rate capability [77].

Jiao fabricated TiO₂/Ti₃C₂Tx heterostructure for Lithium-Sulfur Batteries. TiO₂ is uniformly distributed on the MXene sheet and it works as capturing centers to polysulfides. Multifunctional catalyst TiO₂ improved the performance of Lithium-Sulfur batteries and it delivers 800 mAhg⁻¹ at 2C. Very low capacity decay (0.028%) is found even after 1000 cycles at 2C and 93% of capacity retention is maintained after 200 cycles [86]. The Co₃S₄@MXene electrode has a high initial capacity, but after 100 cycles, it loses half of its capacity. MoS₂/Mo₂TiC₂Tx ~500 has a smaller capacity than Co₃S₄@MXene but has a more stable performance. When current density rises, capacity decreases. To achieve large capacity and stable cycle performance, more analysis is needed.

**Table 1. Specific capacity for different heterostructures in various batteries**

| Structure Name | Battery Name | Electrode type | Capacity (mAhg⁻¹) |
|----------------|--------------|----------------|-------------------|
| Bi₂MoO₆/MXene-50% | LIBs | Anode | ~500 at 0.1 Ag⁻¹ |
| Bi₂MoO₆/MXene-30% | LIBs | Anode | ~600 at 0.1 Ag⁻¹ |
| Bi₂MoO₆/MXene-10% | LIBs | Anode | ~700 at 0.1 Ag⁻¹ |
| MXene/graphene | LIBs | Anode | 426(O/G), 392(OH/G) |
| Phosphorene/MXene | SIBs | Anode | 535 at 0.1 Ag⁻¹ |
| TiO₂/Ti₃C₂Tx | LSBS | Anode | 800 at 2C |
| Graphene/ReSe₂/Ti₃C₂ MXene | KIBs | Anode | 395.3 at 0.1 Ag⁻¹ |
| MoSe₂/MXene | SIBs | - | 490 at 1 A g⁻¹ |
| Co₃S₄@MXene | MG-SIBs | Cathode | 1220 at2C |
| Covalent triazine framework/Ti₃C₂MXenenanosheets | LSBS | Cathode | 1200 at 0.5C |
| V₂O₅/MXene | LIBs | Cathode | ~475 (discharge) at 0.5c |
| SnS₂/Sn₂S₄ on Ti₃C₂ | LIBs | Anode | ~1400(discharge)–800(charge) at 0.1 Ag⁻¹ |
6. Challenges and future prospects

It's necessary to make thin Mxene in order to enhance surface area by reducing thickness. The functional group of Mxene has a significant impact on the electrochemical performance of energy storage devices, although the relationship between functional group and Mxene features is still unknown. Researchers fabricated and analyzed electrochemical performance in great detail, but structural features of these heterostructures need to be studied further. Materials with a high dielectric strength are required to operate at high voltages. The dielectric strength of the above heterostructures is unknown. The resistance of a conductor changes as the temperature changes. So, the temperature Coefficient of Resistance needs to be calculated. The capacity of batteries to charge and discharge is determined by their electrode density. More physical and electrical features of electrode materials must be explored in order to arrive at a solution in battery design. Mxene will be a great material for future energy storage devices if the issues mentioned above can be resolved.

7. Conclusion

Energy storage is becoming a major concern due to recent advancement of electric vehicles. The high demand for electrochemical energy storage has led investigators to focus on the development of advanced electrode materials. MXene a new group of 2D materials is investigated in 2011. It has become an alternate material for electrodes to previously used materials due to its superior electrochemical characteristics. In this review paper, we discussed latest technology of MXene synthesis process and recent advancement of MXene heterostructures as electrode material of energy storage devices like lithium/sodium-ion batteries. MXene hybrid structures demonstrate superior performance compare to the individual building block that has been thoroughly discussed in this review work. We briefly discussed why MXene is promising in energy storage applications and many others as well due to its excellent properties. A specific application of MXene in lithium-ion batteries as electrode material has been reviewed. Because of several advantages like high electronic conductivity, improvement in ion transfer, good stability, eliminating one of the major problems in battery designing (restacking) MXene heterostructures opened a new door for research in the field of energy conversion and storage system. Hence it is reviewed here the advancement of MXene synthesis, specific application in Lithium-ion batteries, and heterostructures for electrode material in rechargeable batteries would be useful for further investigation of MXene in energy storage applications.

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